U.S. Department of Justice

Federal Bureau of Investigation

Washington, D. C. 20535-0001

February 28, 2007

Norman Gahn
Calumet County District Attorney's Office
206 Court Street
Chilton, WI 53014

RE: State of Wisconsin v. Steven Avery

Dear Mr. Gahn:

I am writing in response to a letter from Jerome F. Buting dated February 26, 2007, requesting discovery in the above-captioned matter. Each request relating to the analysis performed by the FBI Laboratory is addressed individually below.

1. "The protocol issue date is February 15, 2007..."

Enclosed is a copy of the relevant analysis protocols utilized by the FBI Laboratory in this case.

2. "Any data on tests the FBI has done or culled... "

There were no tests performed by the FBI that determined the amounts of EDTA found in ordinary household or automotive objects.

3. "Any and all lab sheets, work sheets, bench sheets..."

Enclosed is a copy of all of the file material generated by the FBI Laboratory relating to the analysis performed in this case. This material includes bench notes, computer printouts, chain of custody documents, and all other specific information regarding the case. A CD Rom is also included with this material containing raw data files. These files cannot be accessed unless the proprietary software which is available from Thermo Finnigan, is installed on your computer.

EXHIBIT 446
05 CF 381
DATE: 03-06-07 JB
Initials

Ex 446
(1)

4. "Any and all validation studies the FBI has done..."

Enclosed is a copy of the validation studies.

5. "Any and all "limitation of detection" studies..."

Information regarding studies referred to in the protocol in this case is found in the material provided for response 4.

6. "Any and all "selectivity" studies referred to in..."

Information regarding studies referred to in the protocol in this case is found in the material provided for response 4.

7. "Any and all "matrix effects" studies referred to..."

Information regarding studies referred to in the protocol in this case is found in the material provided for response 4.

8. "Citations to any and all "literature" referred to..."

Information regarding literature referred to in the protocol in this case is found in the material provided for response 4.

9. "A list of any and all cases where Marc LeBeau..."

Inasmuch as this request if not for documentation relating to the present case or for documentation regarding underlying scientific data, it is beyond the scope of discovery.

10. "A complete curriculum vitae for any individual..."

The analysis in this case was performed by Unit Chief Marc A. LeBeau. Chemist Jason Brewer was the Technician. Examiner Madeline Montgomery was the Technical Reviewer and Examiner Eileen Waninger was the Administrative Reviewer in this case. A copy of their curriculum vitae is enclosed.

11. "All data reflecting the rate of degradation or..."

Inasmuch as this request is not for documentation relating to the present case, it is beyond the scope of discovery. This information can be researched by any defense expert and is in the public domain.

Ex 446
(2)

12. "Any complaints or negative performance evaluations..."

Pursuant to FBI Policy, a review of the Examiner's file for complaints or negative performance evaluations may be requested in writing by the prosecutor to the appropriate Chief Division Counsel, who will coordinate a search with appropriate legal personnel at Headquarters. Laboratory personnel may not have access to certain personnel files containing such information.

13. "Any and all inside or outside proficiency tests..."

A copy of the completed proficiency test summaries for Unit Chief Marc A. LeBeau, Examiners Eileen Waninger and Madeline Montgomery and Chemist Jason Brewer is enclosed.

14. "Laboratory chain of custody records, including all..."

All information, regarding chain of custody of evidence for this case will be found in the case notes provided for response 3.

15. "Copies of traceability documentation for standards..."

All information regarding traceability for this case will be found in the case notes provided for response 3.

16. "Instrument run log with identification of all..."

All information, regarding instrument run logs for this case will be found in the case notes provided for response 3.

17. "Records of instrument maintenance status and..."

Enclosed is a copy of the maintenance records of the instruments used in the analysis of this case, for the time period surrounding the examination in this case.

18. "An error or contamination log covering any and all..."

There were no instances of contamination in this case. Had any instances occurred, the documentation, including actions taken, would be included in the material provided in response to request 3. Any request for documentation regarding error or

Ex 446
(3)

contamination that occurred in other cases is beyond the scope of the discovery.

19. "Raw data for the complete measurement sequence..."

All information regarding the raw data for this case, is found on the CD provided in response to request 3.

I hope this material will assist you in this effort.

Joseph DiZinno, D.D.S
Assistant Director
Laboratory Division

by

Robert Fram
Section Chief
Scientific Analysis Section

Enclosures

Ex 446
(4)

02/28/07
JDB

- EDTA DONOR TUBES ON NON-FTA TREATED CARDS (50 uL BLOOD/STAIN, 5/2004)
  - LABELED (A) - (I)
- CREATED EDTA (-) BLOOD SAMPLE (MAL 2/2/07, YELLOW TOP) ON NON-FTA TREATED CARDS JB, 50 uL/STAIN, CREATED 3 STAINS.
- BLANK CARD, NO BLOOD STAIN (NON-FTA TREATED CARD).

- CUT OUT FULL CIRCLE FOR EACH SAMPLE, PLACED IN LABELED CFD. ADDED 200 uL 500 ppm D/2-EDTA (aq) - SIT FOR 45 min, ROOM T.
- FILTRATES → VIALS

Ex 446

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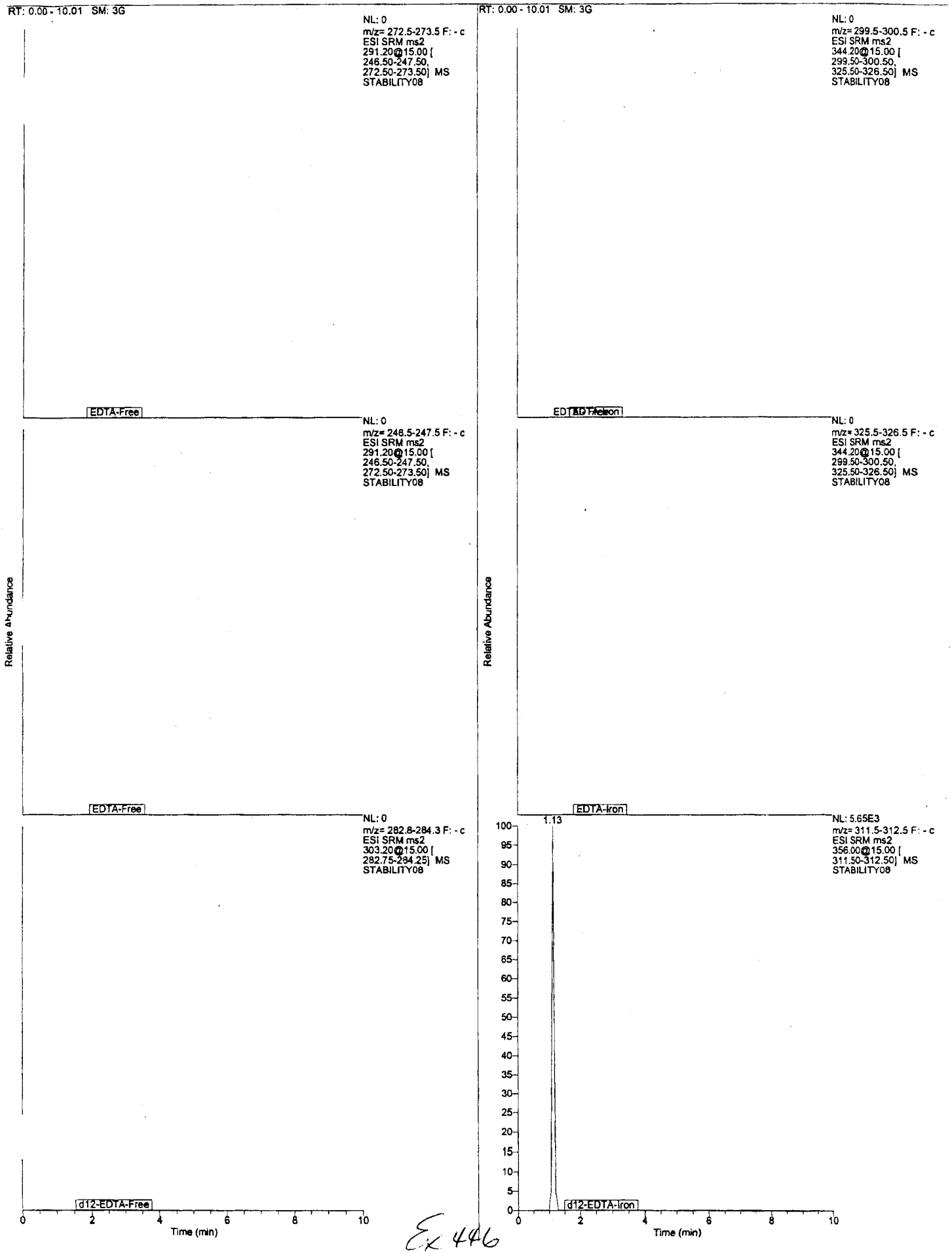
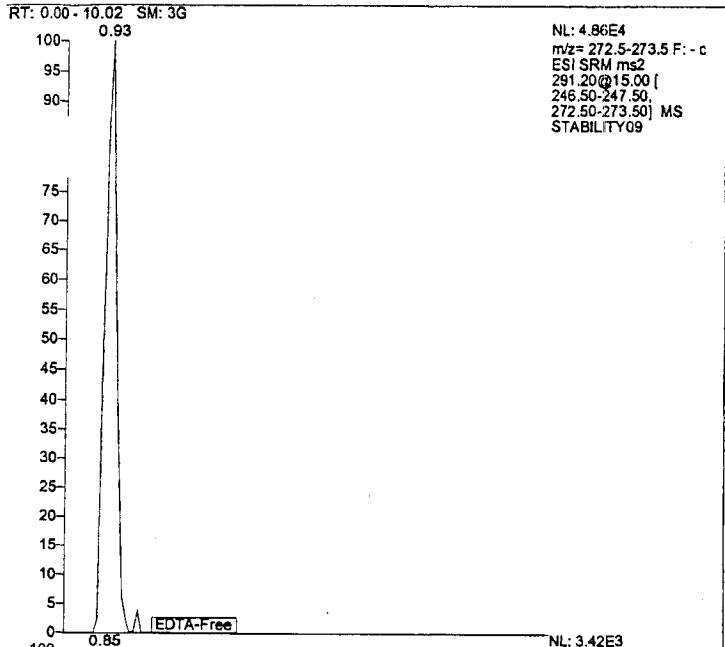
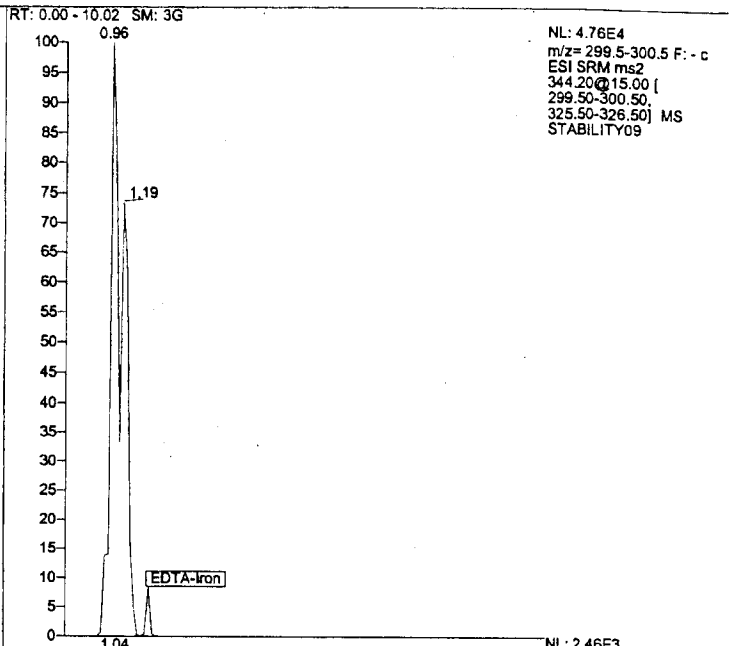
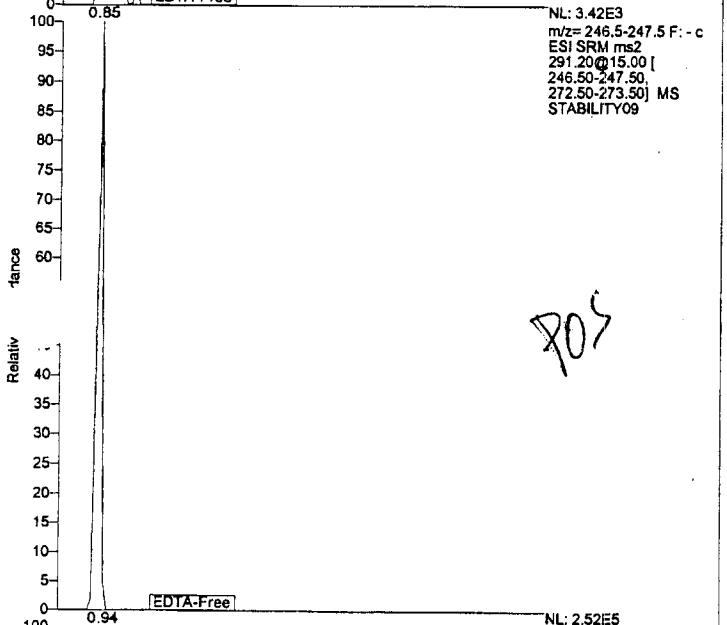


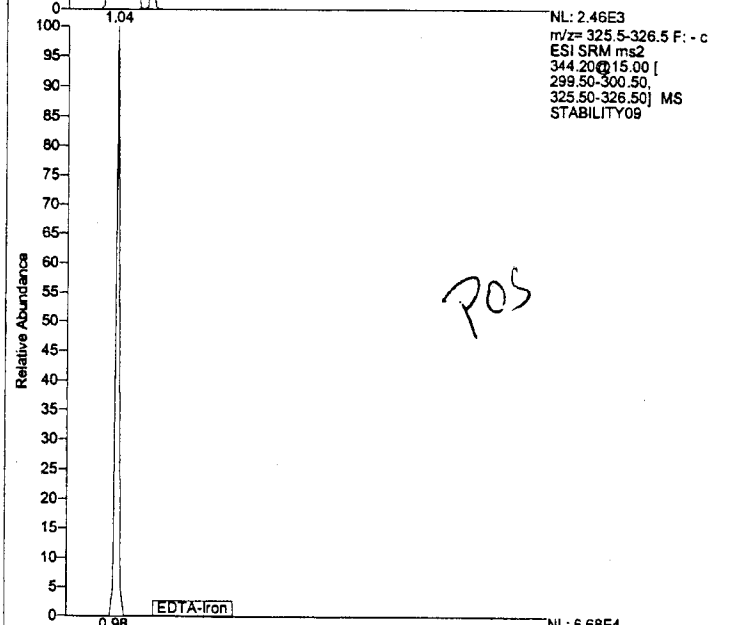
Ex 446
(6)

C:\Xcalibur\...\Stability\Stability09 JDB 02/28/07 06:02:38 Card A extract (EDTA +)









POS

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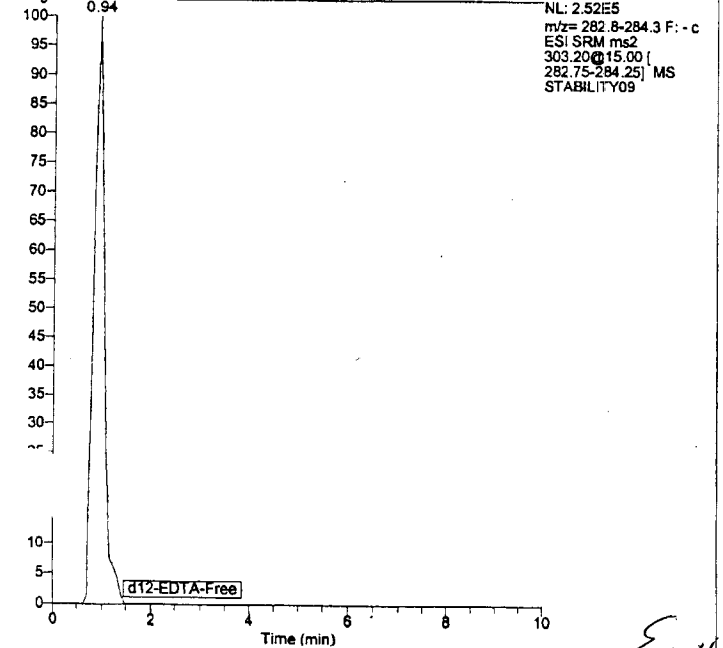

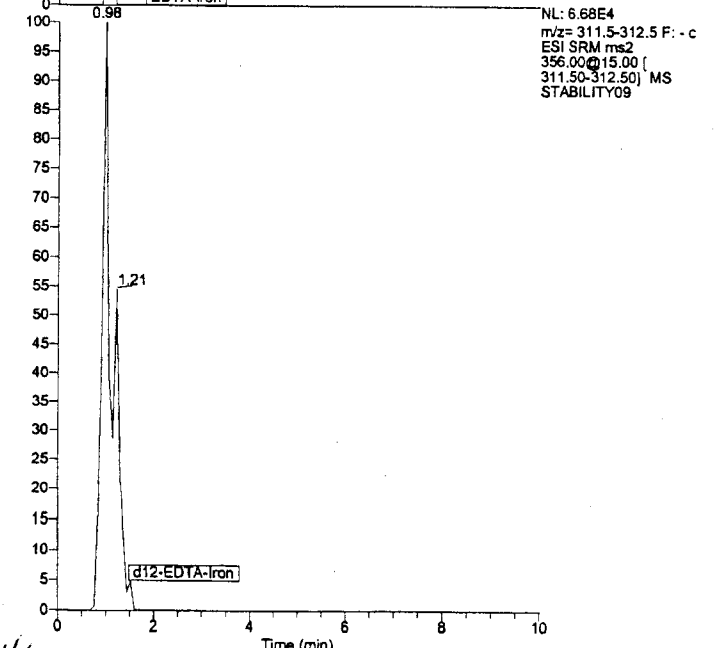

Ex 446

(7)

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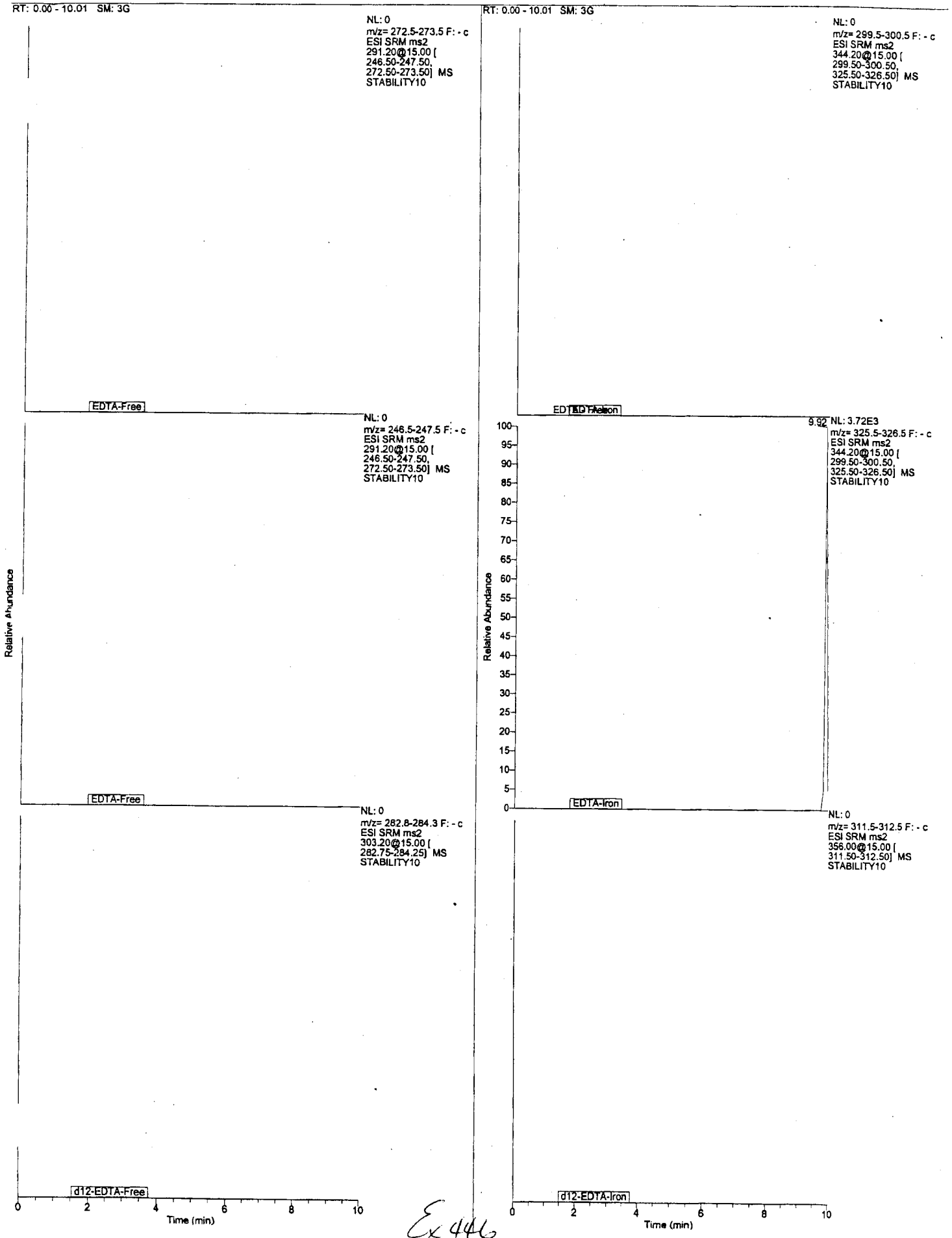

Ex 446
(8)

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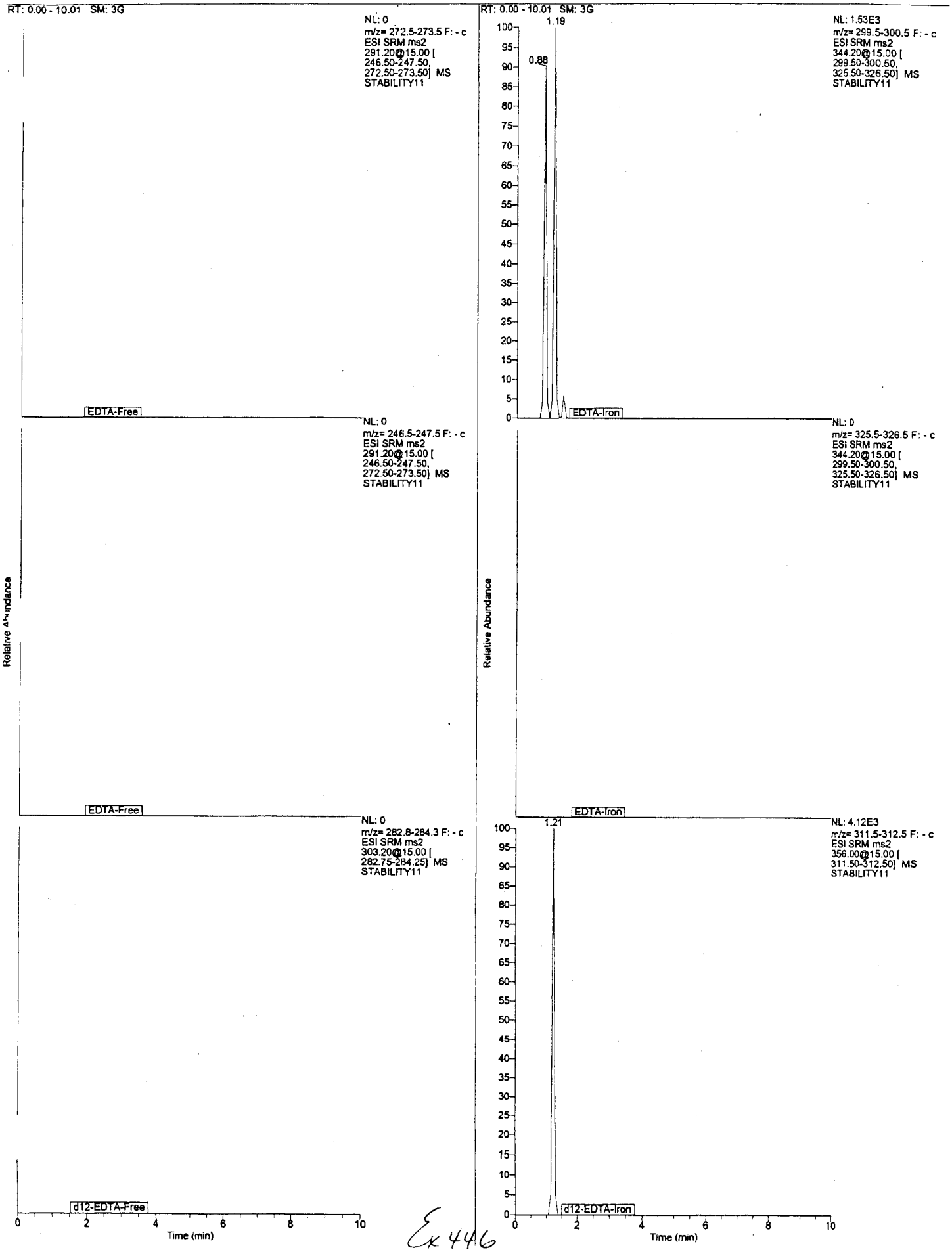

Ex 446

(9)

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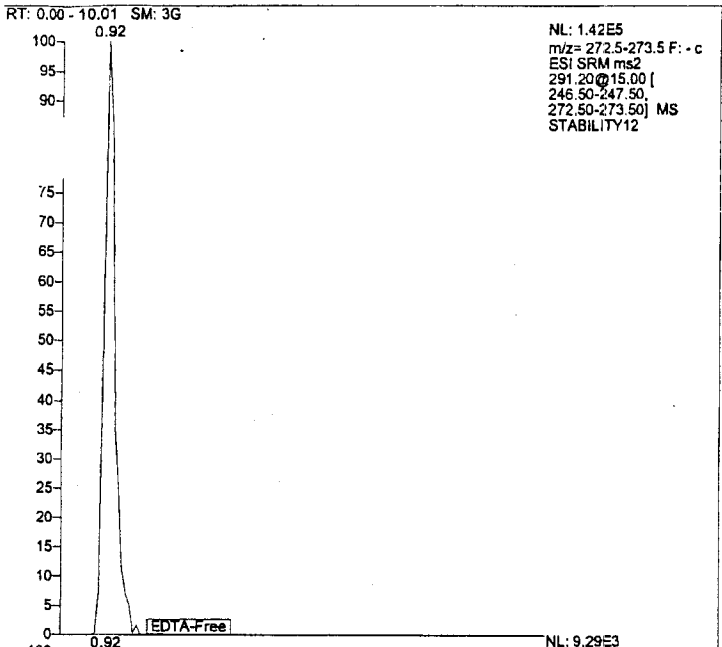

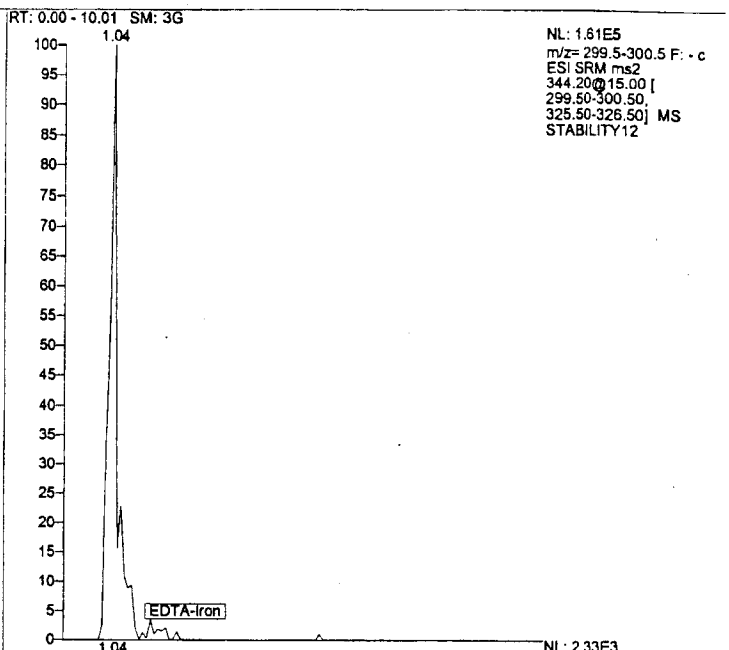

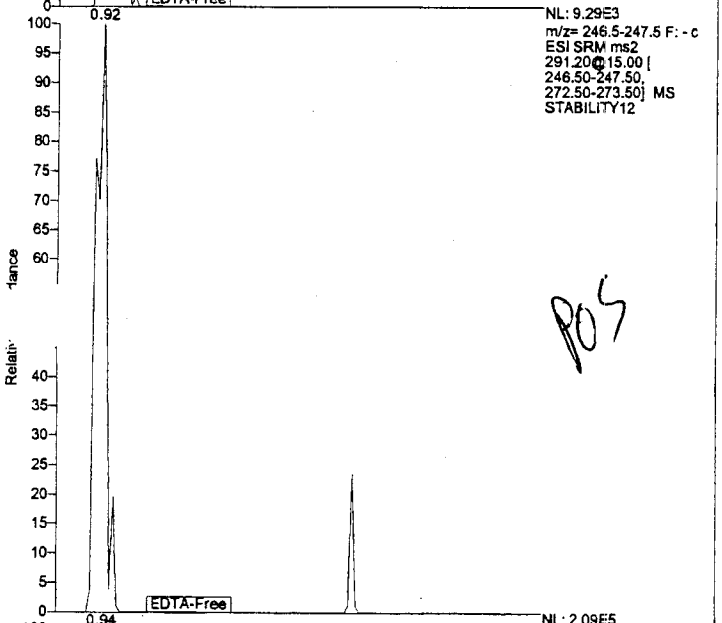

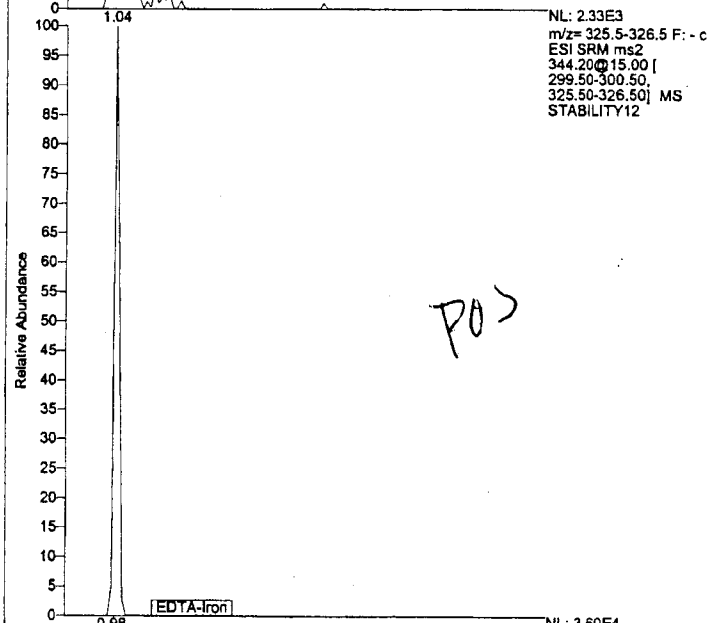

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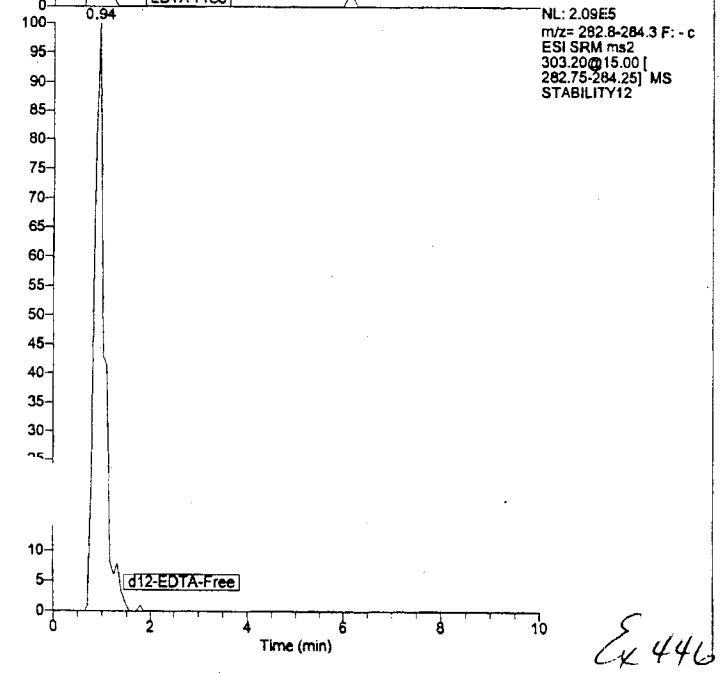

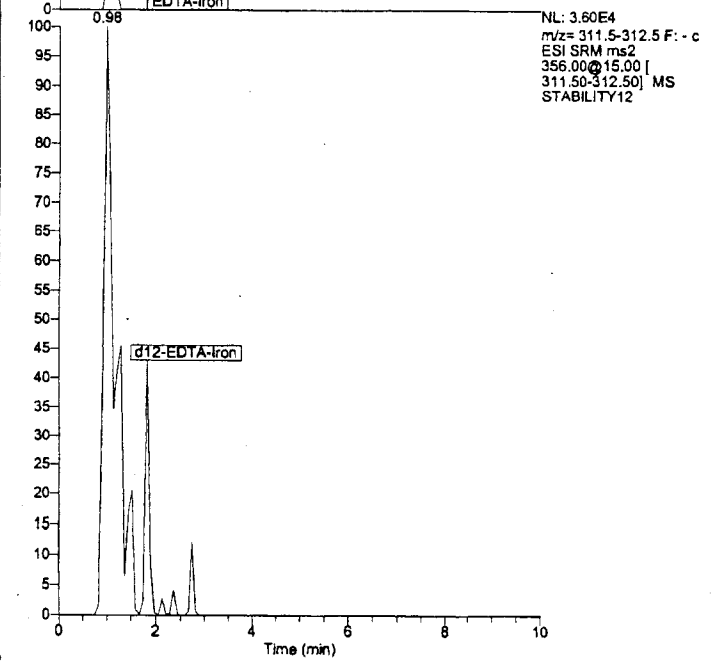

Ex 446
(10)

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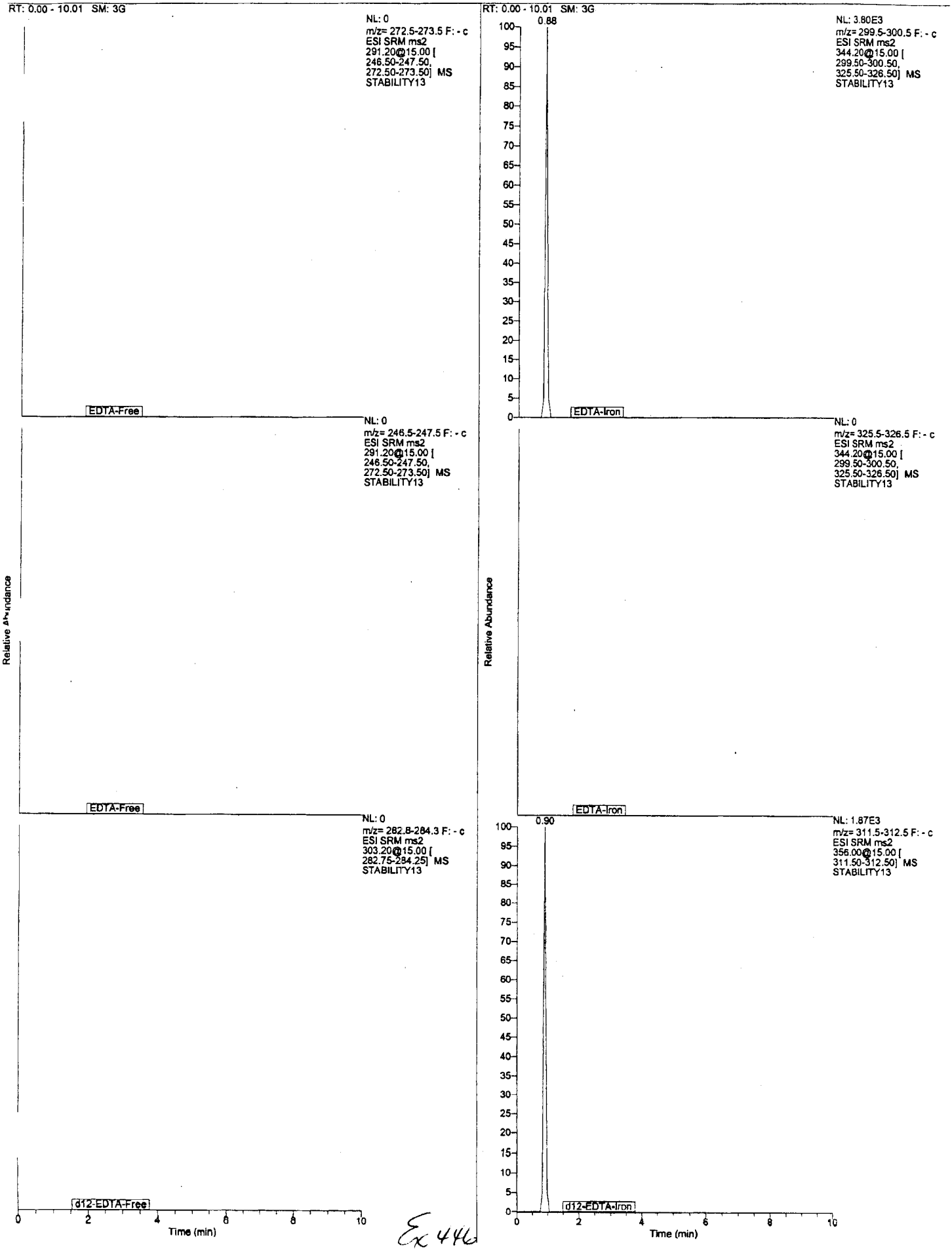

Ex 446
(11)

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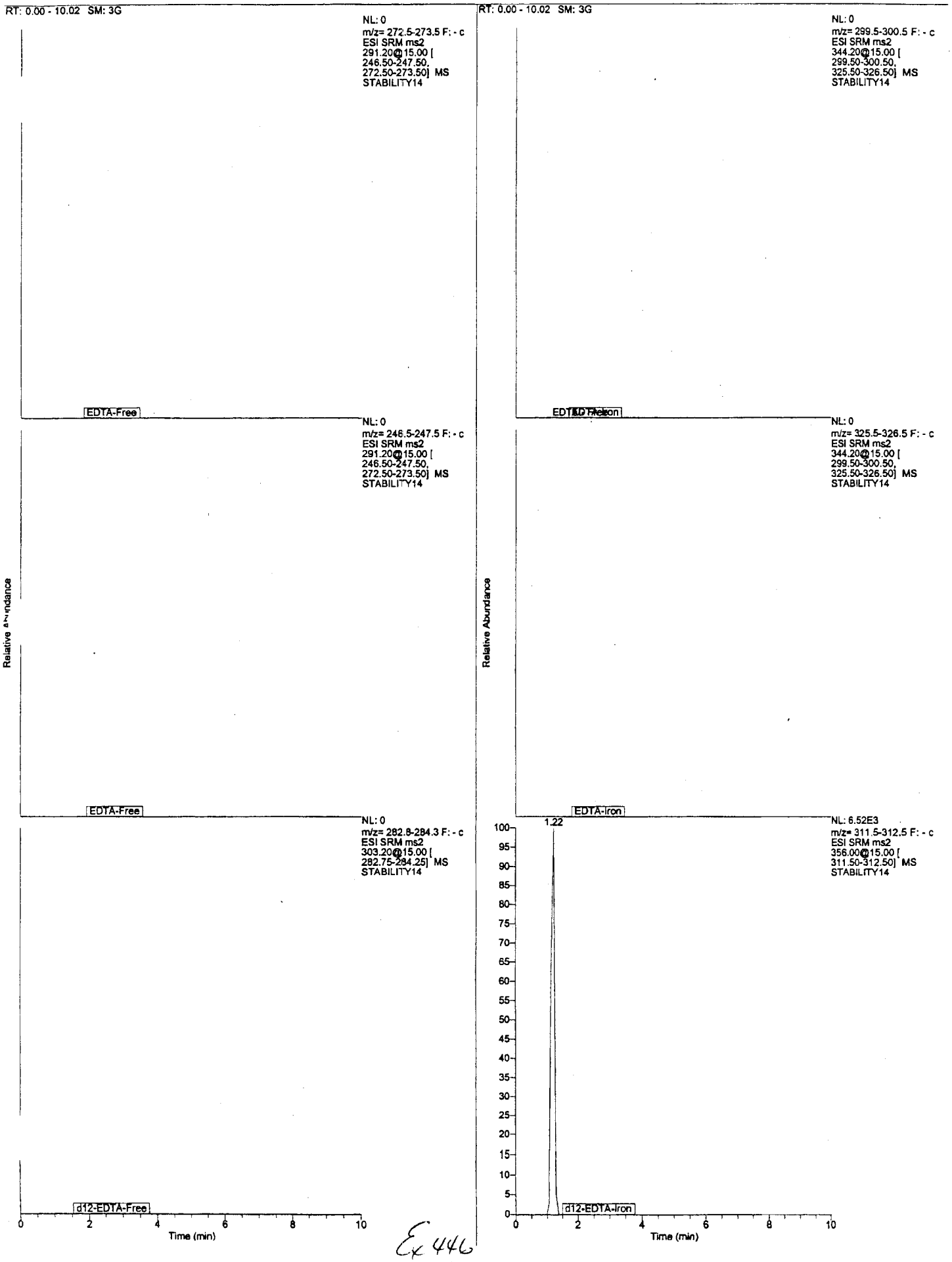
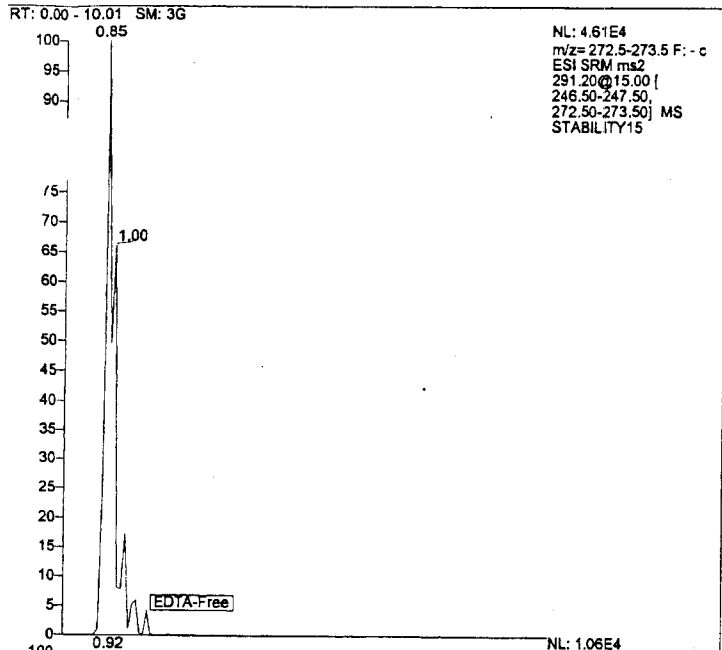
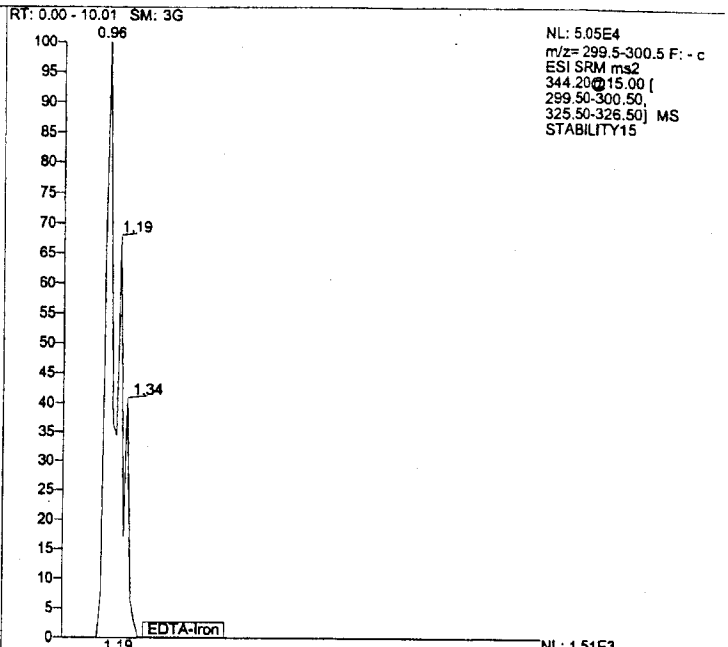
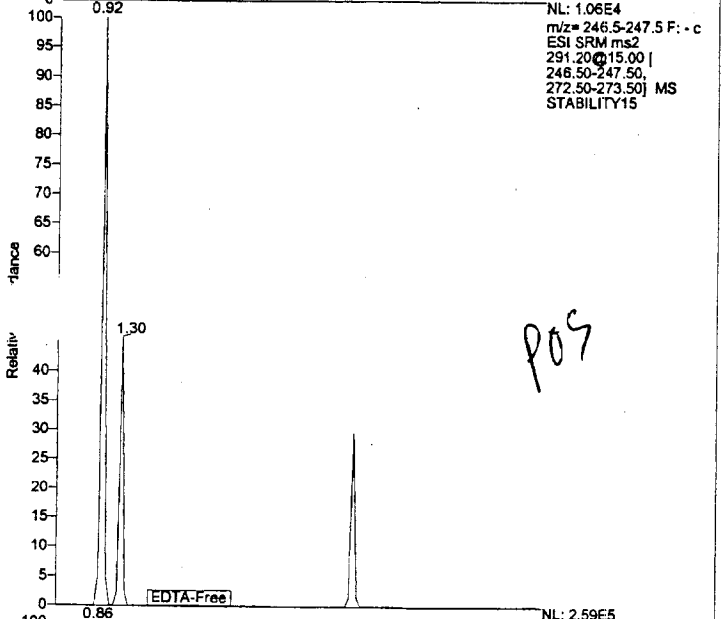


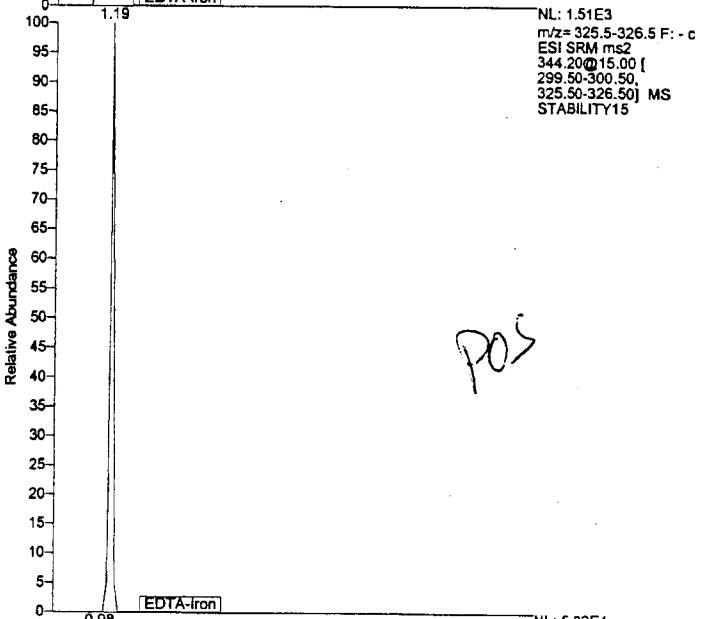
Ex 446
(12)

C:\Xcalibur\...\Stability\Stability15 JDP 02/28/07 07:07:51 Card C extract (EDTA +)









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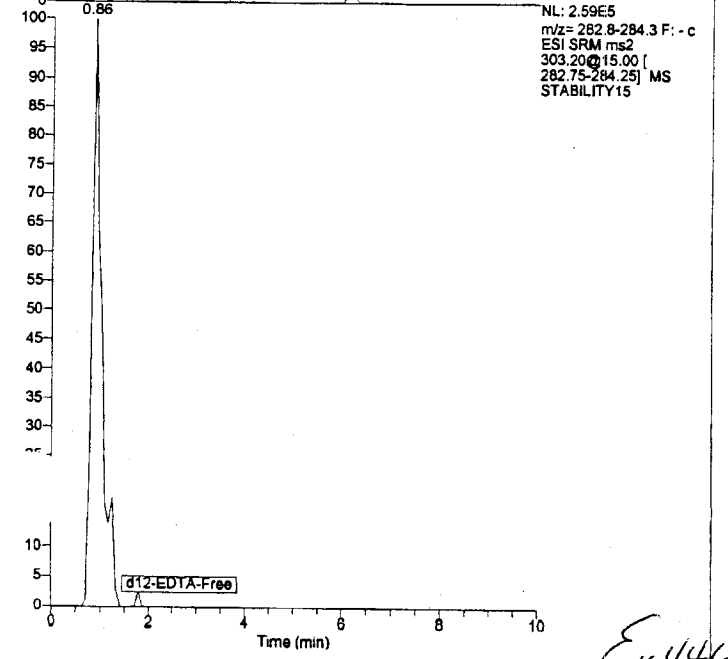

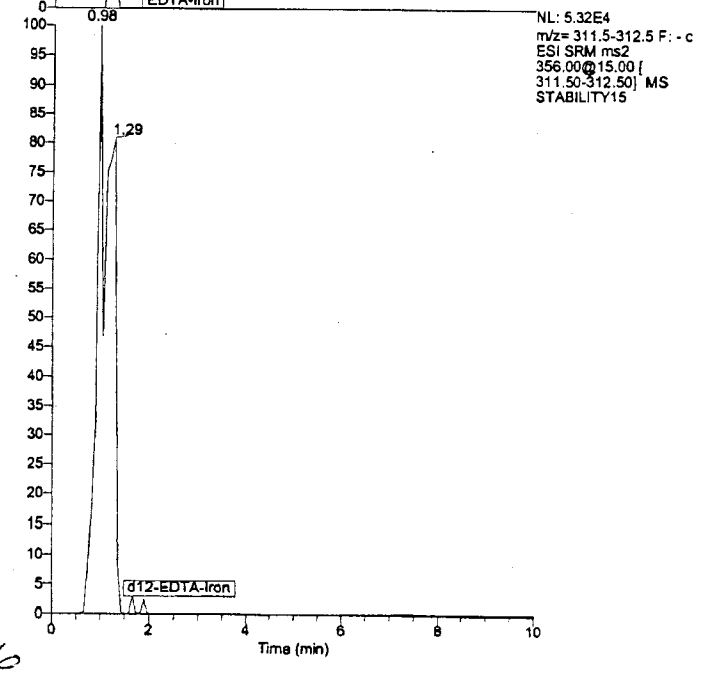

Ex 446
(13)

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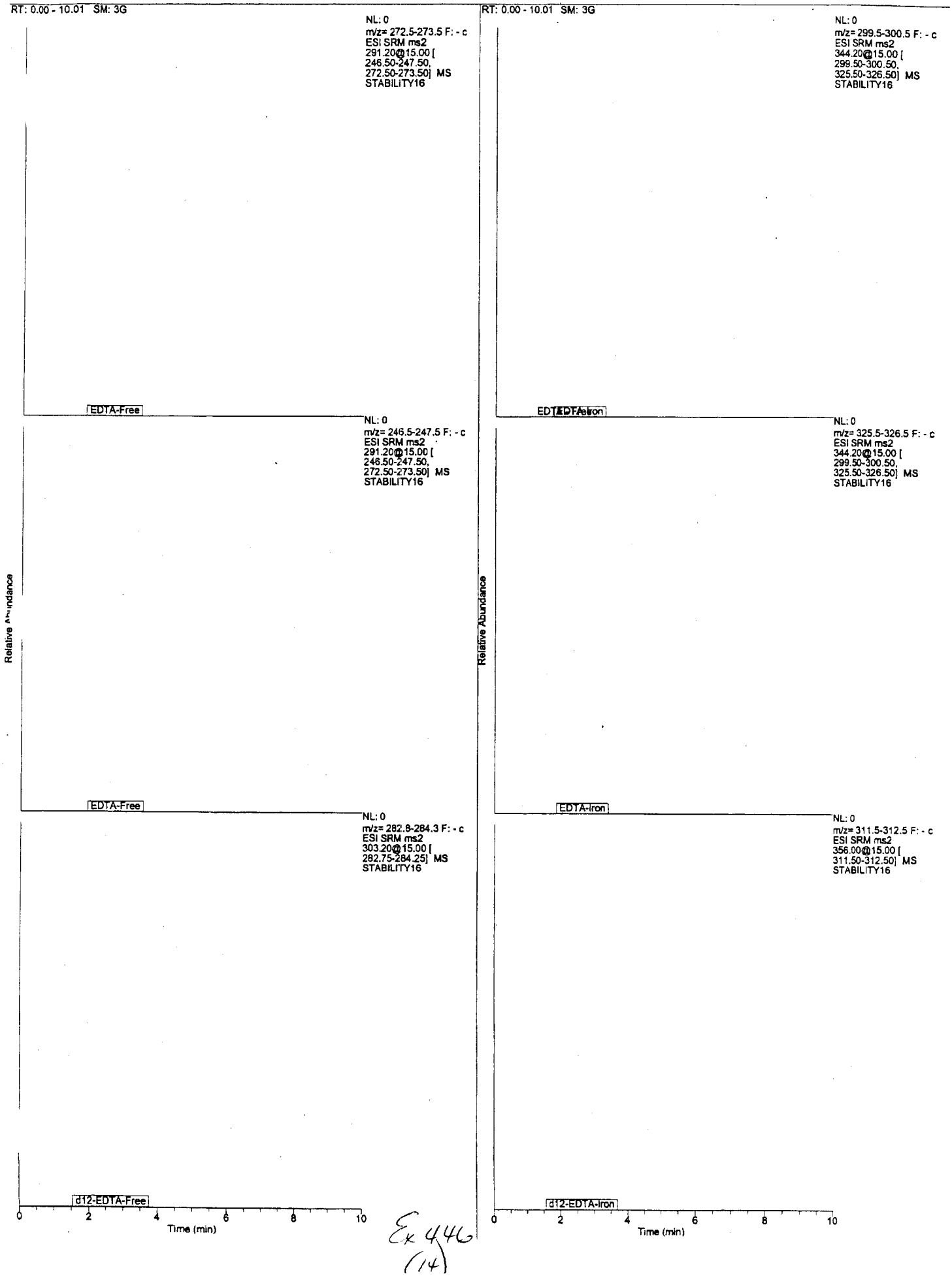

Ex 446
(14)

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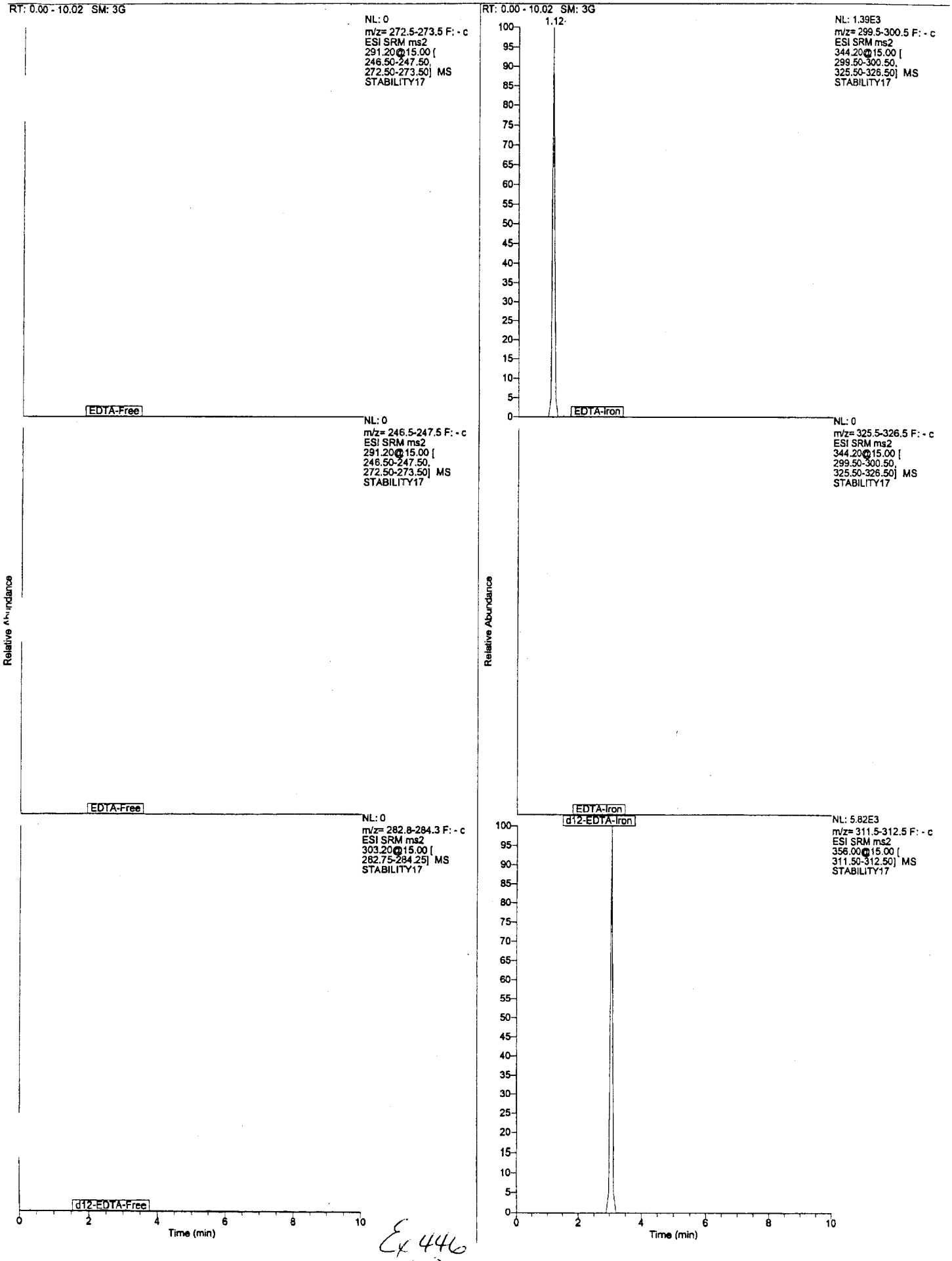
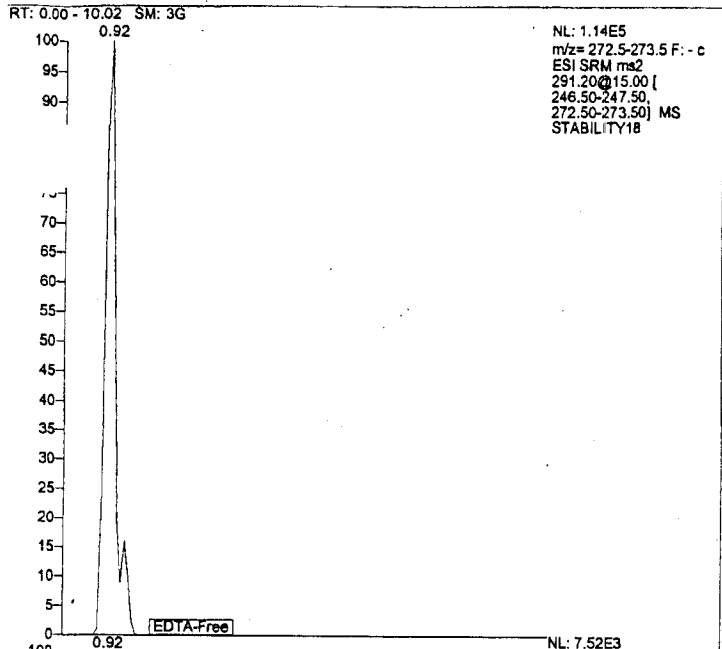
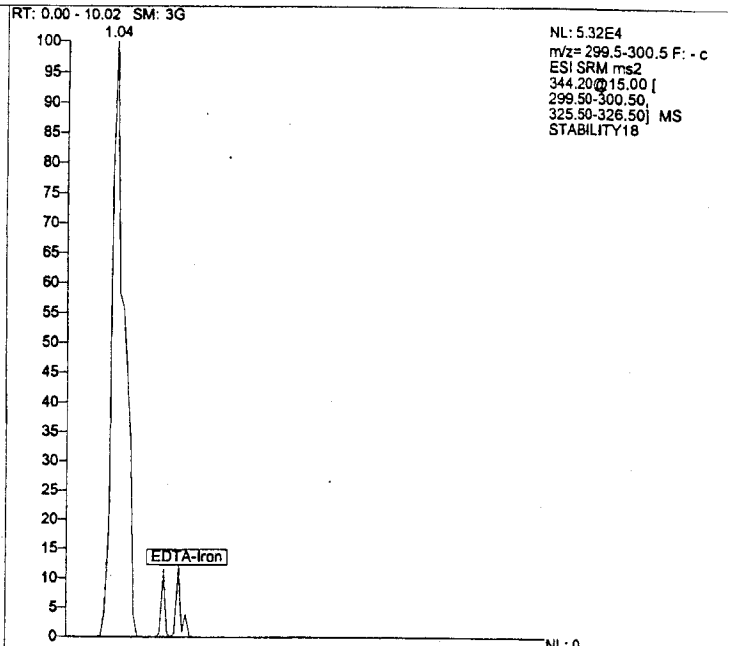
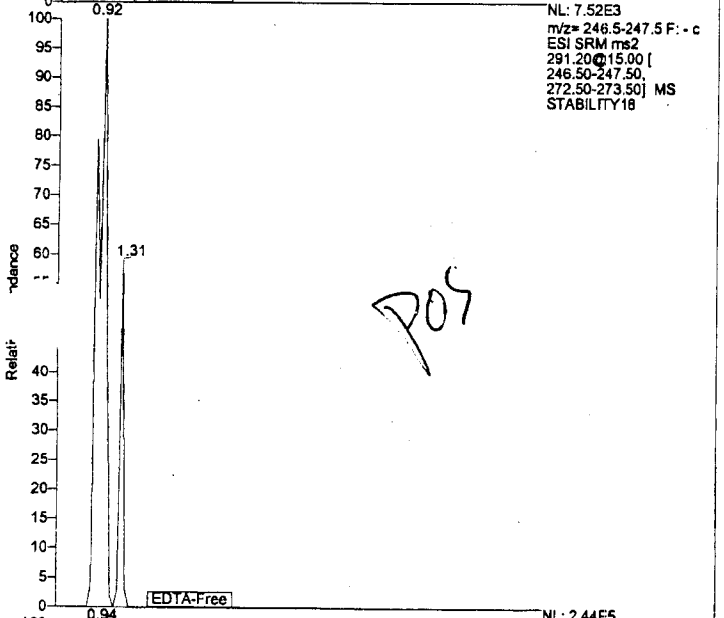


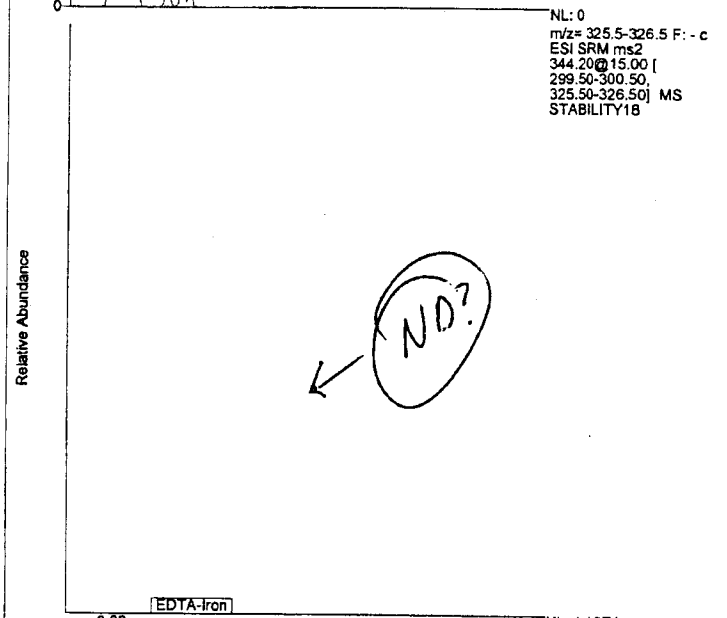
Ex 446
(16)

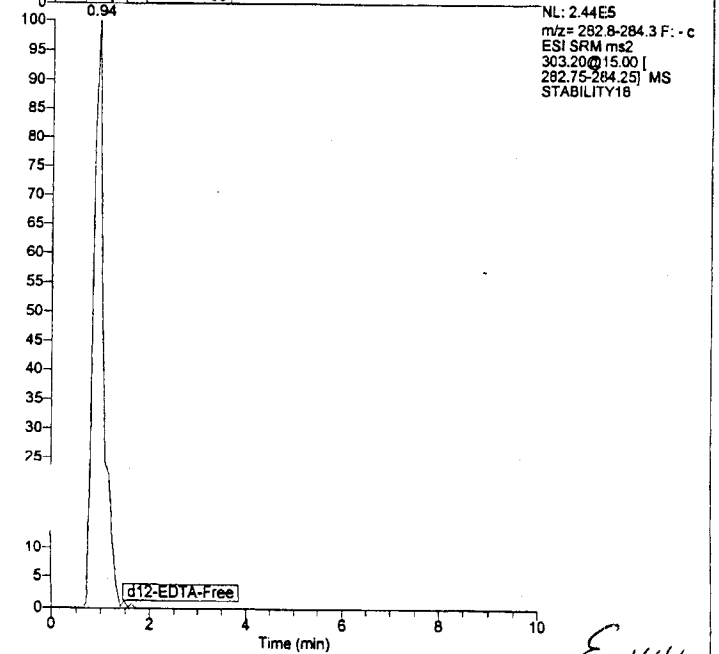
C:\Xcalibur\...\Stability\Stability18 JDP 02/28/07 07:40:31 Card D extract (EDTA +)

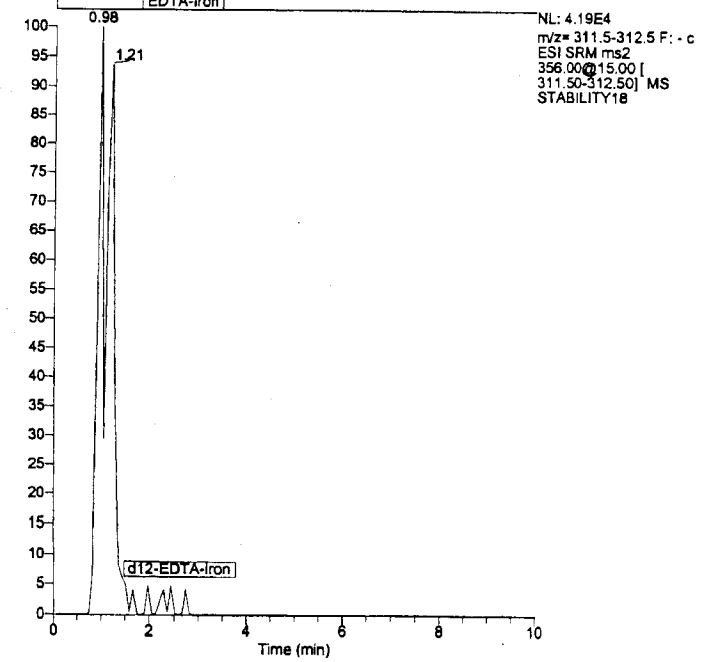












Ex 446
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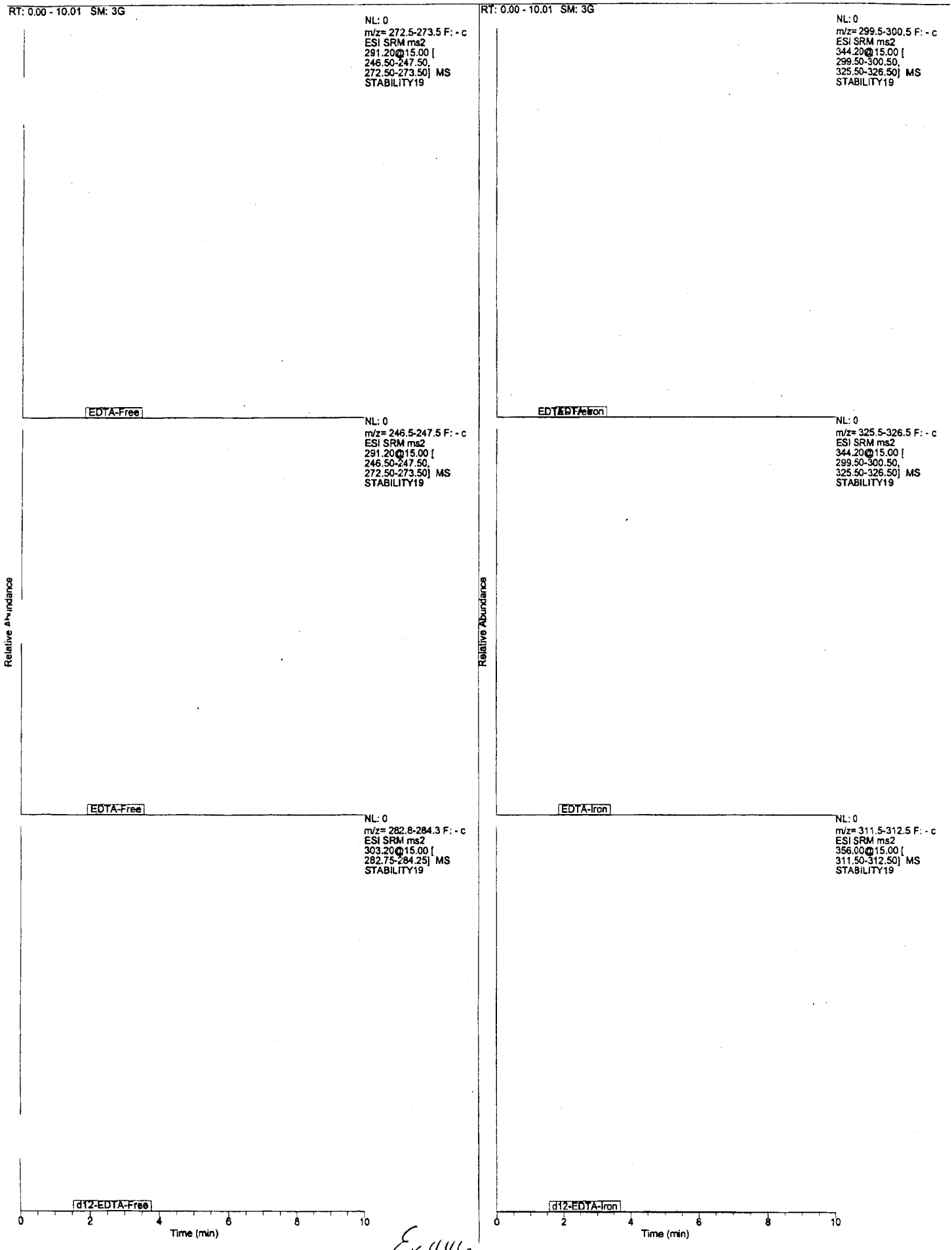
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NL: 0
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
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272.50-273.50] MS
STABILITY19
EDTA-Free
NL: 0
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246.50-247.50,
272.50-273.50] MS
STABILITY19
EDTA-Free
NL: 0
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ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
STABILITY19
d12-EDTA-Free
Relative Abundance
Time (min)
RT: 0.00 - 10.01 SM: 3G
NL: 0
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ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
STABILITY19
EDTA-Iron
NL: 0
m/z= 325.5-326.5 F: - c
ESI SRM ms2
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299.50-300.50,
325.50-326.50] MS
STABILITY19
EDTA-Iron
NL: 0
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
STABILITY19
d12-EDTA-Iron
Relative Abundance
Time (min)

Ex 446
1/7

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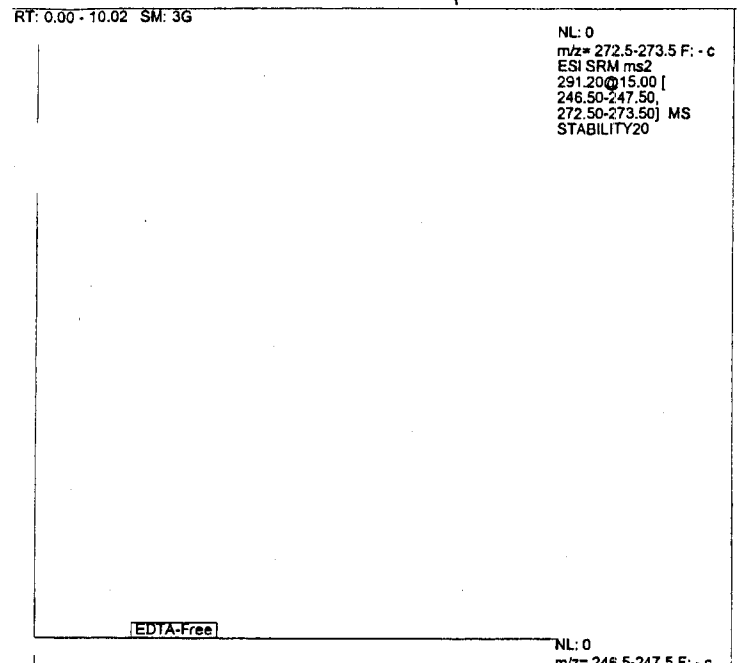

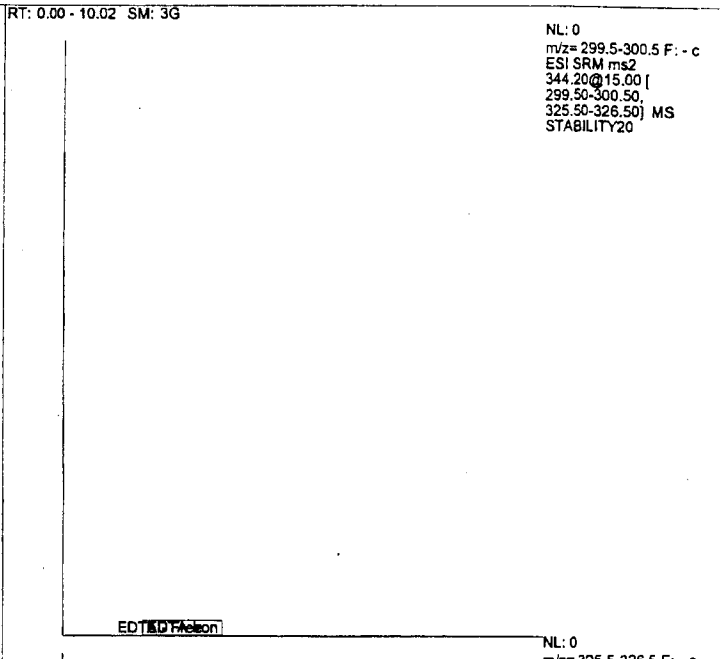

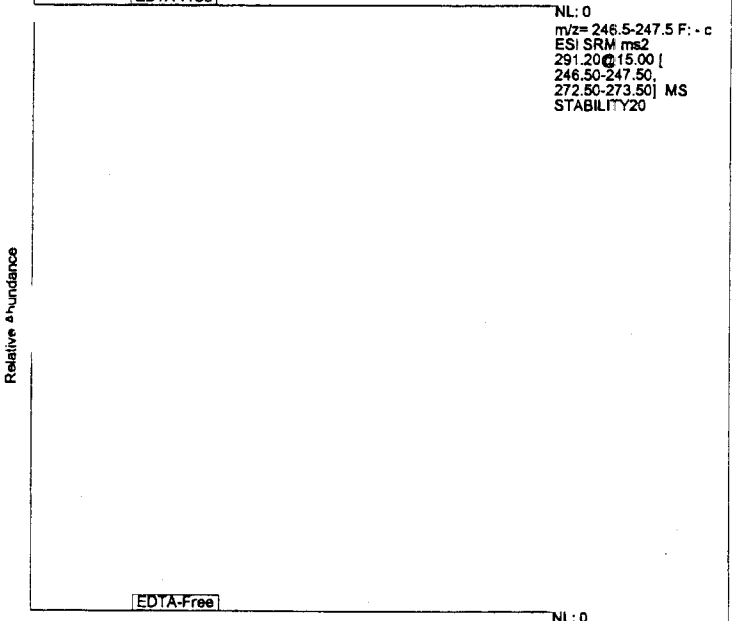

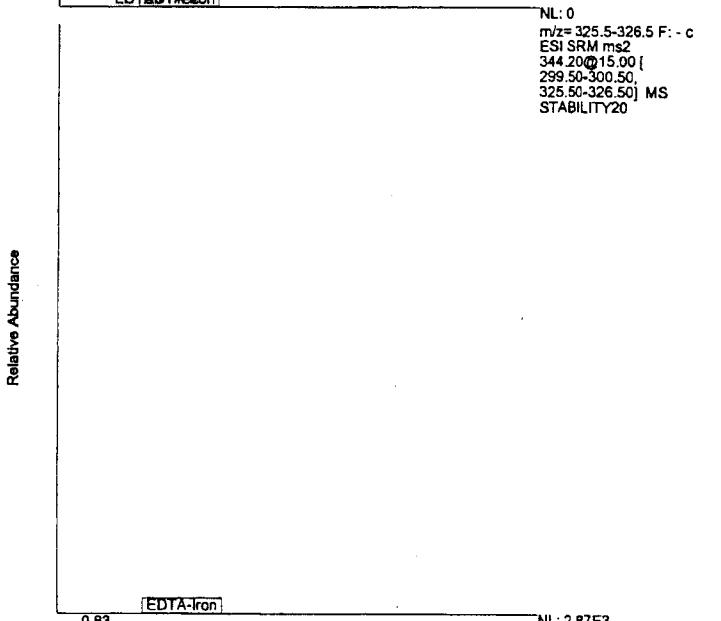

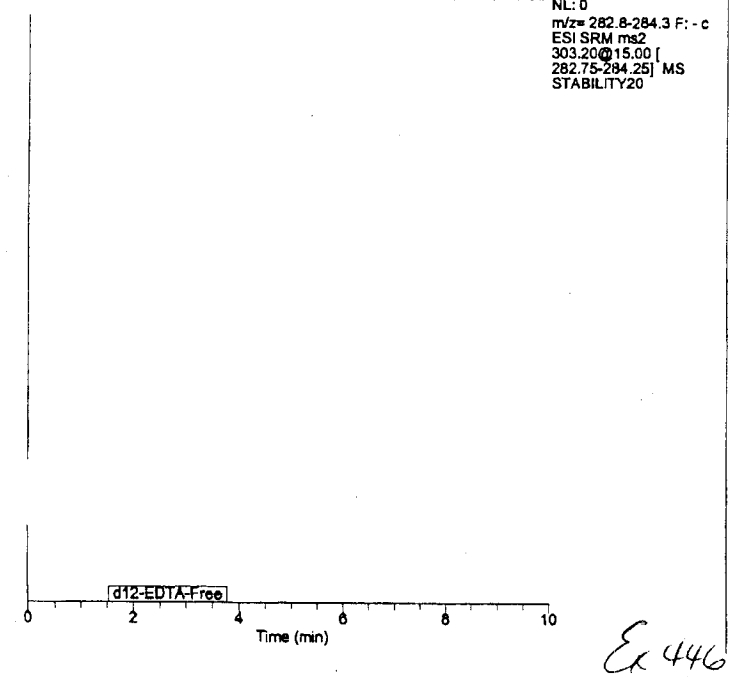

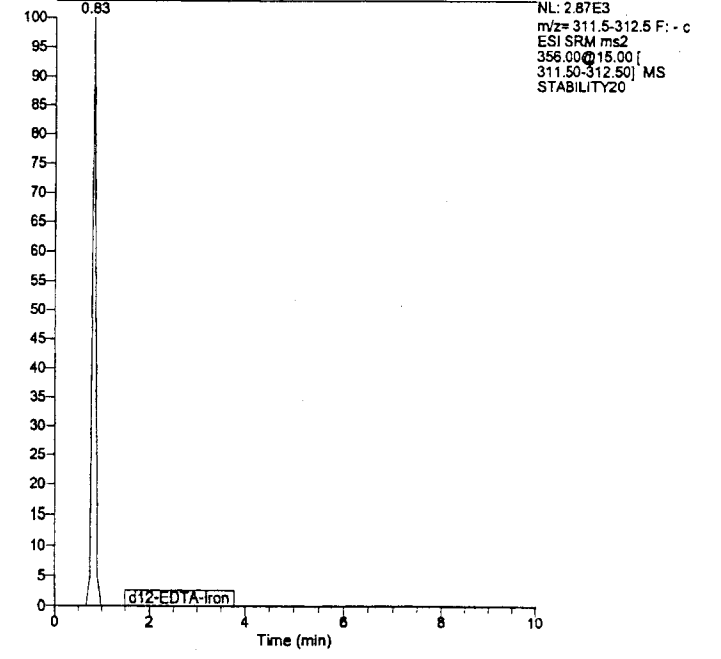

Ex 446
(18)

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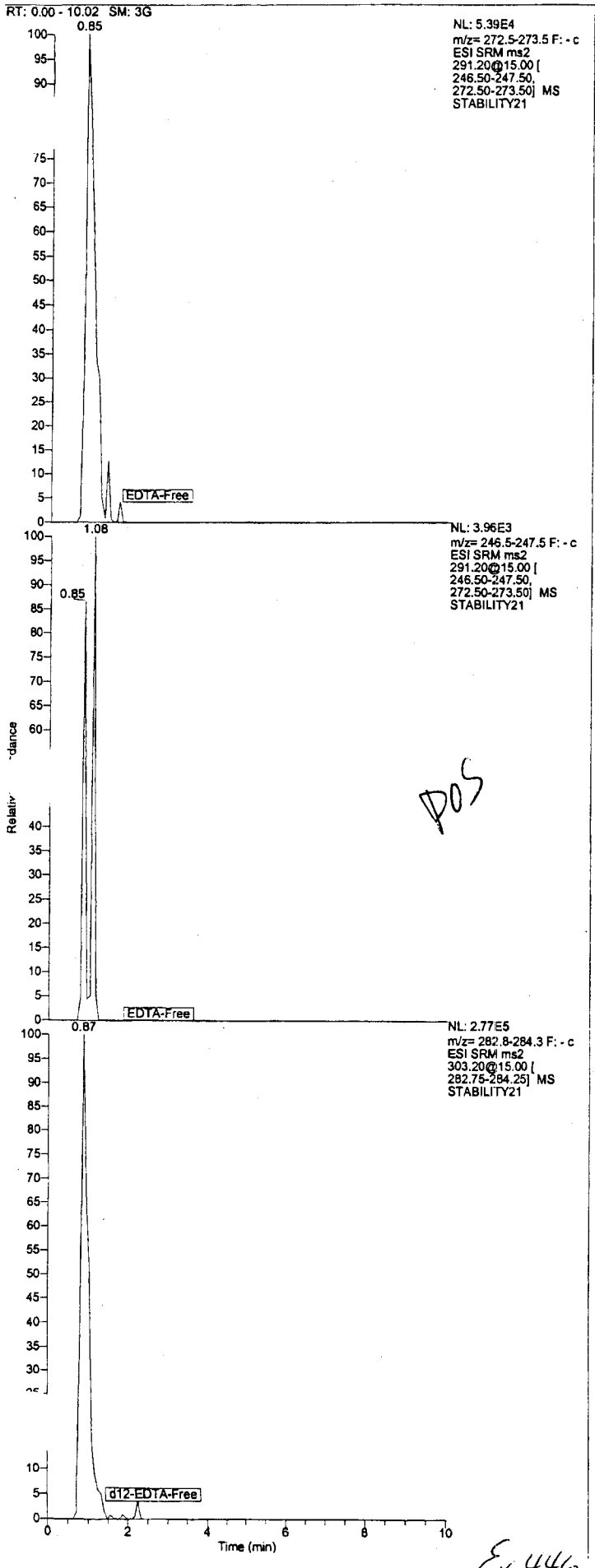

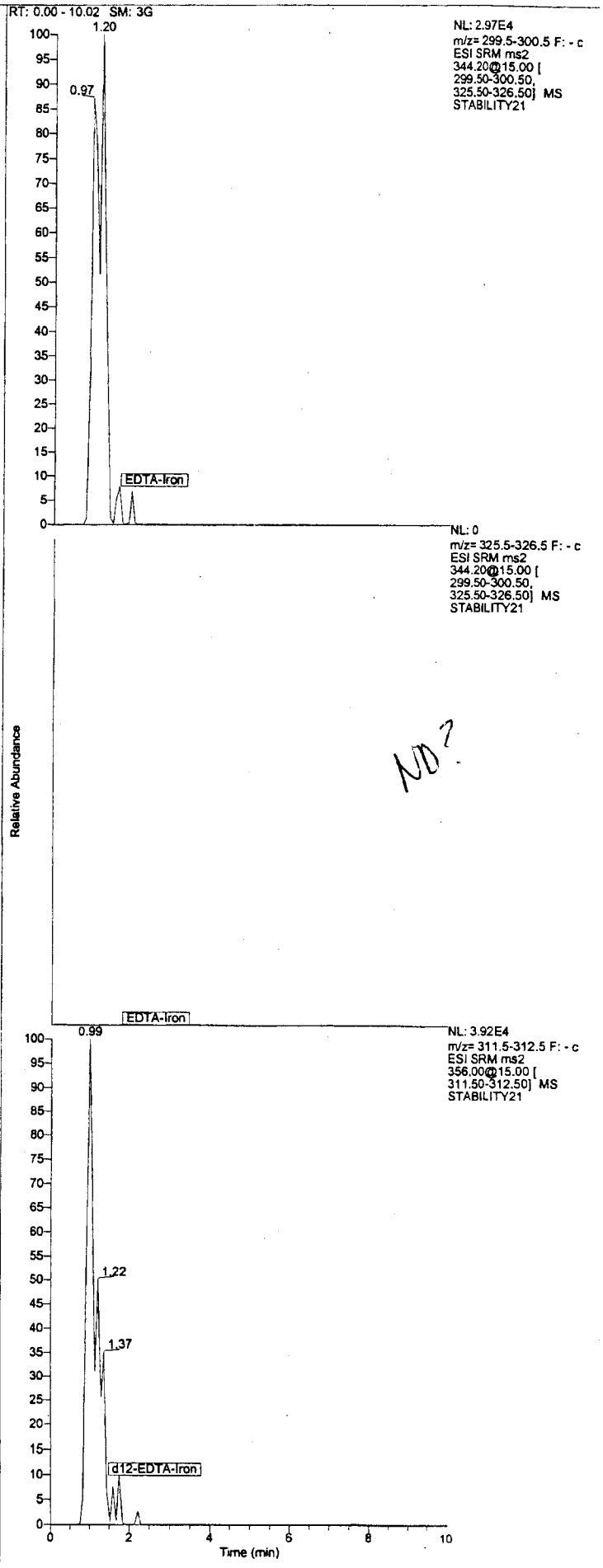

Ex 446
1/19

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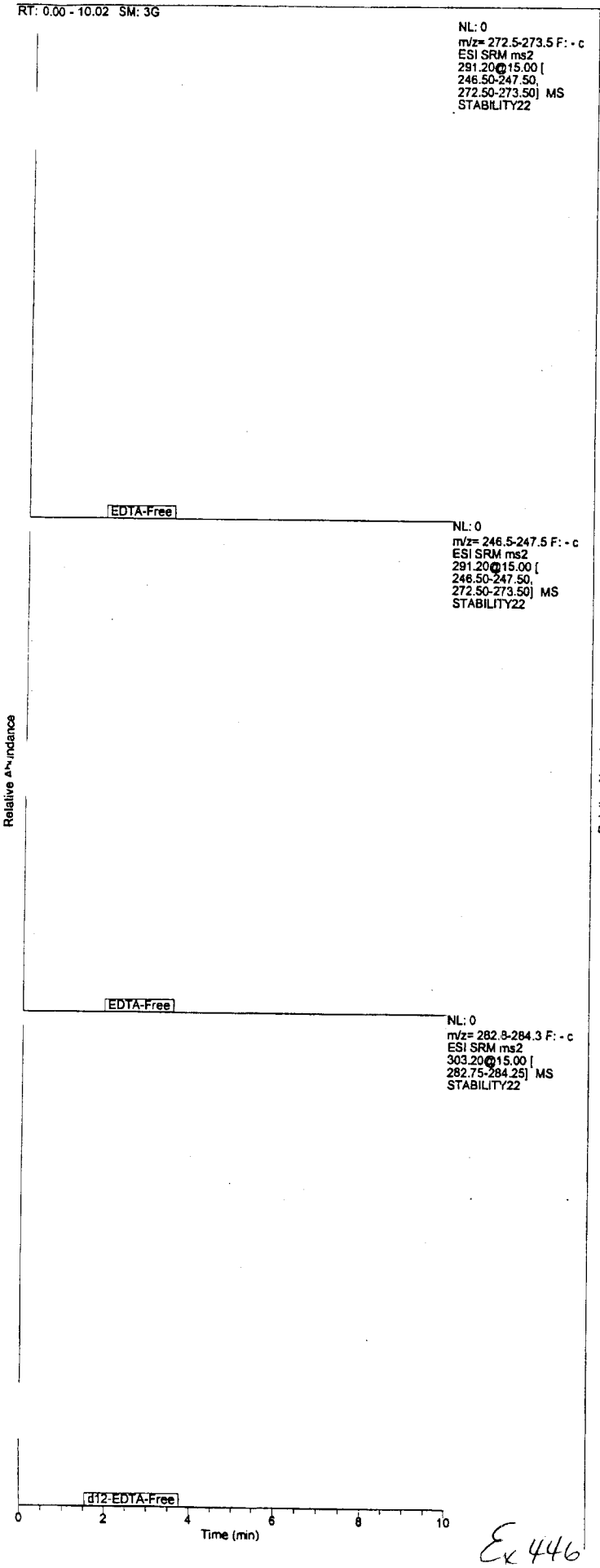
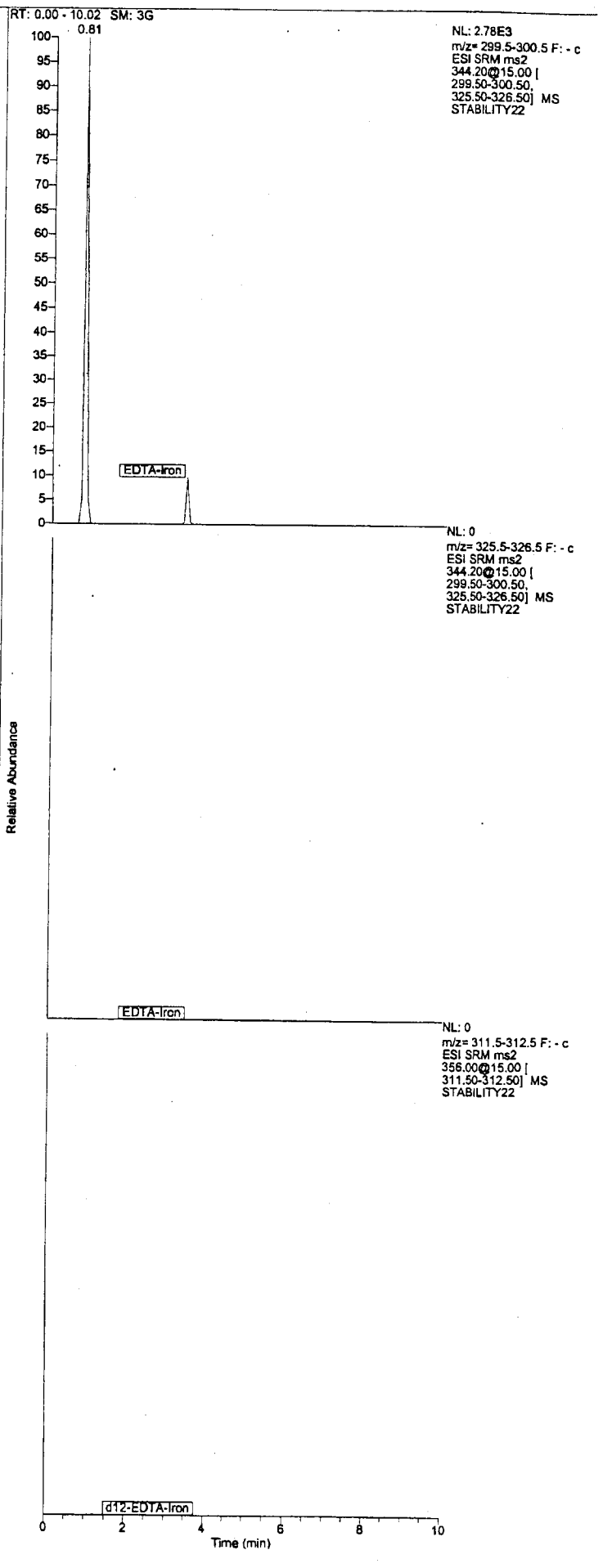
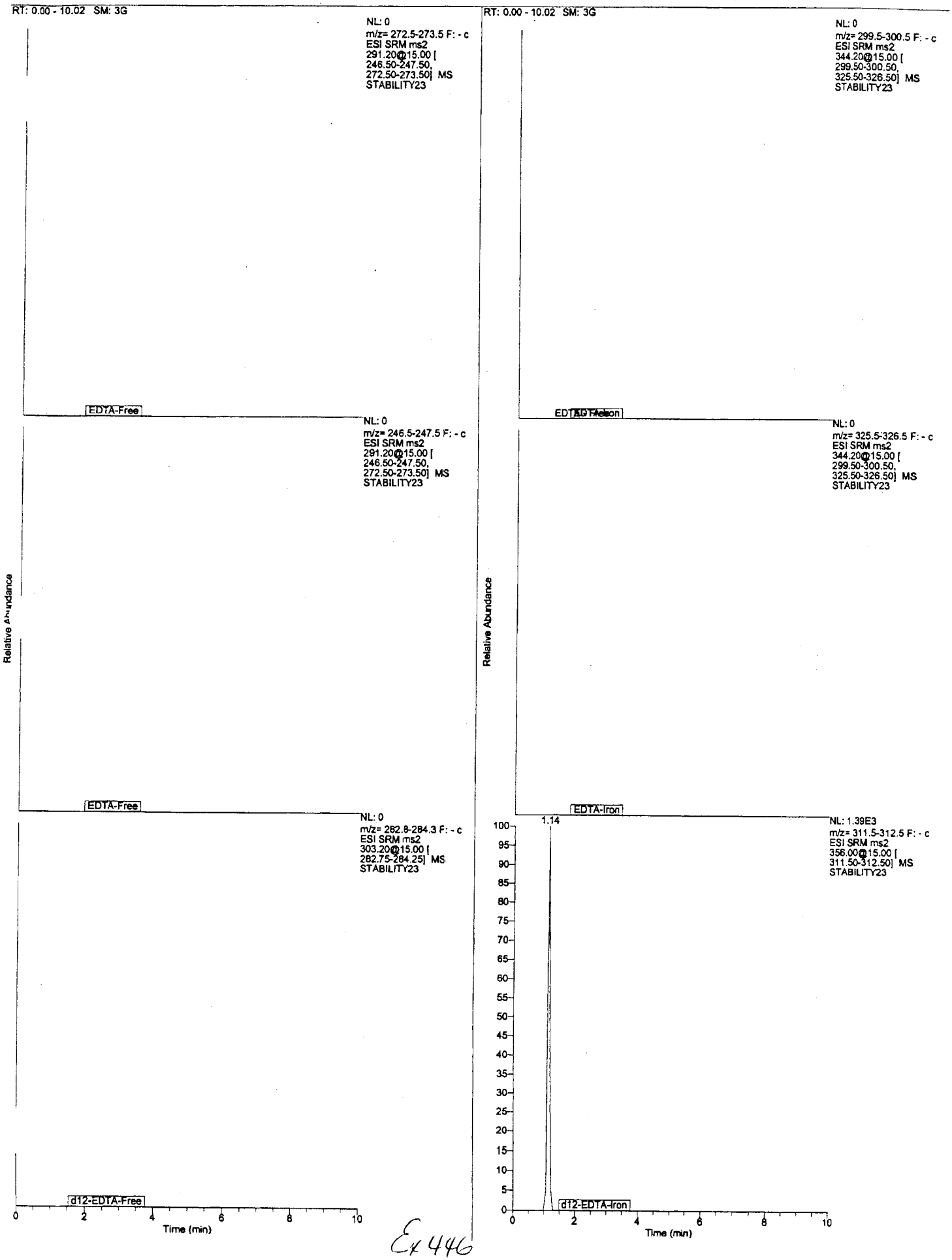




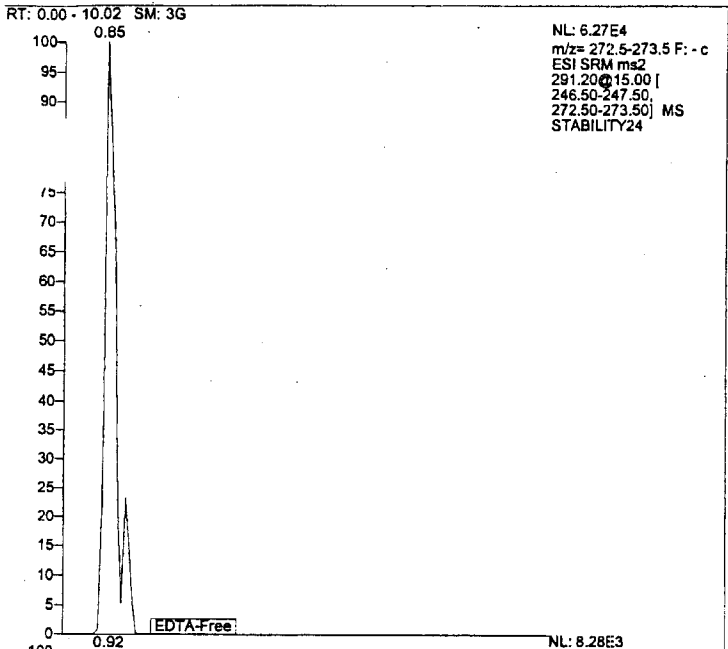
Ex 446
(x)

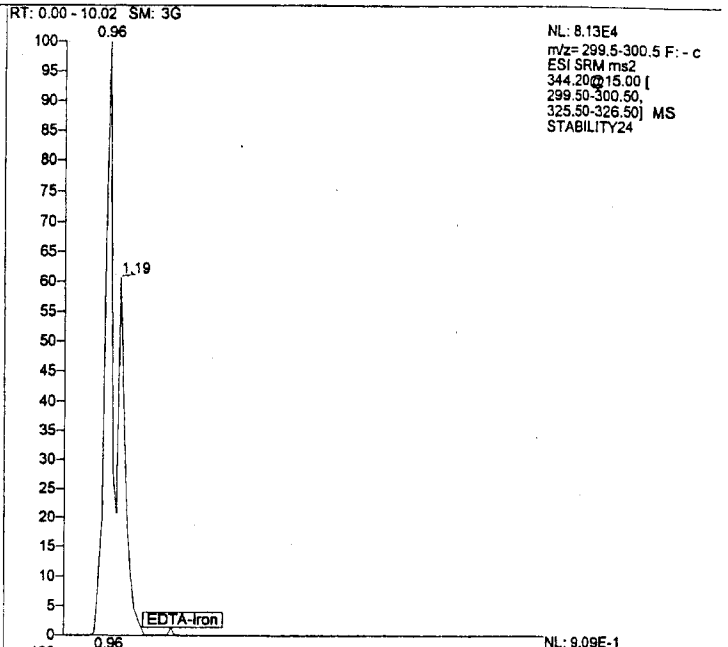
C:\Xcalibur\...\Stability\Stability23 JDB 02/28/07 08:34:58 BLANK (neg blood, DI H2O ext.)

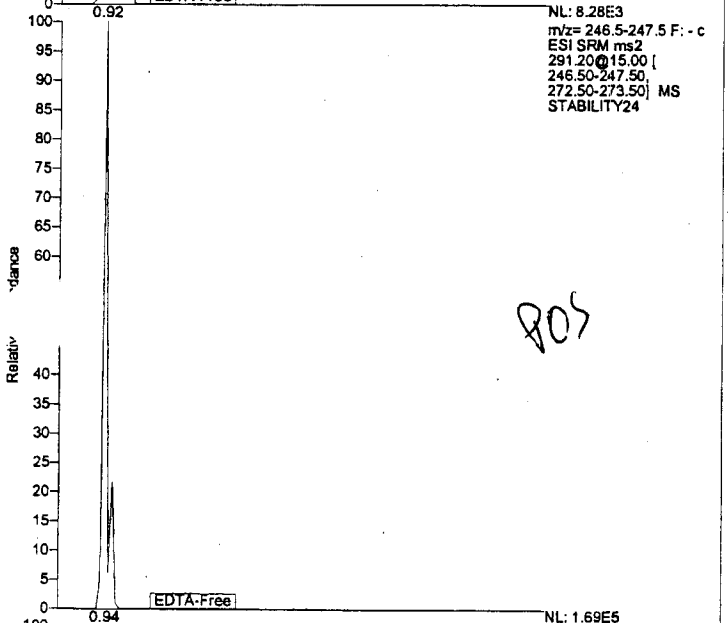


Ex 446

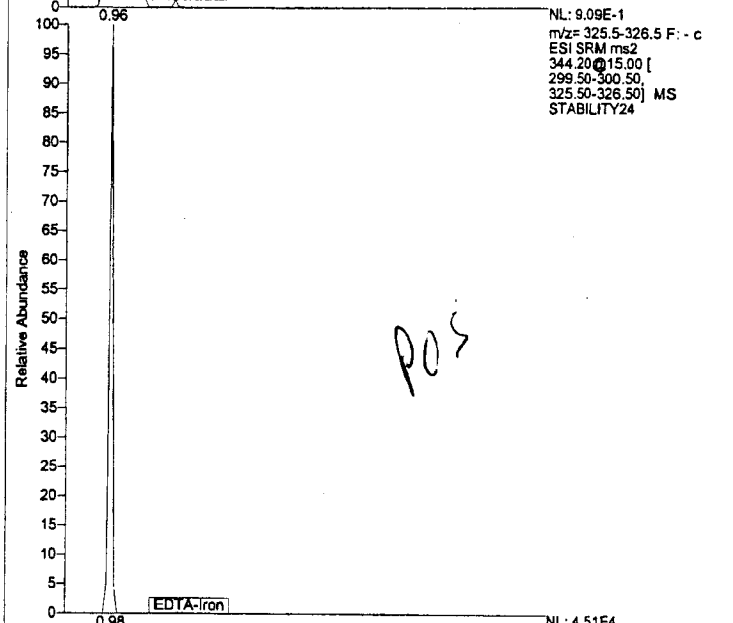
C:\Xcalibur\...\Stability\Stability24 JDB 02/28/07 08:45:49 Card F extract (EDTA +)



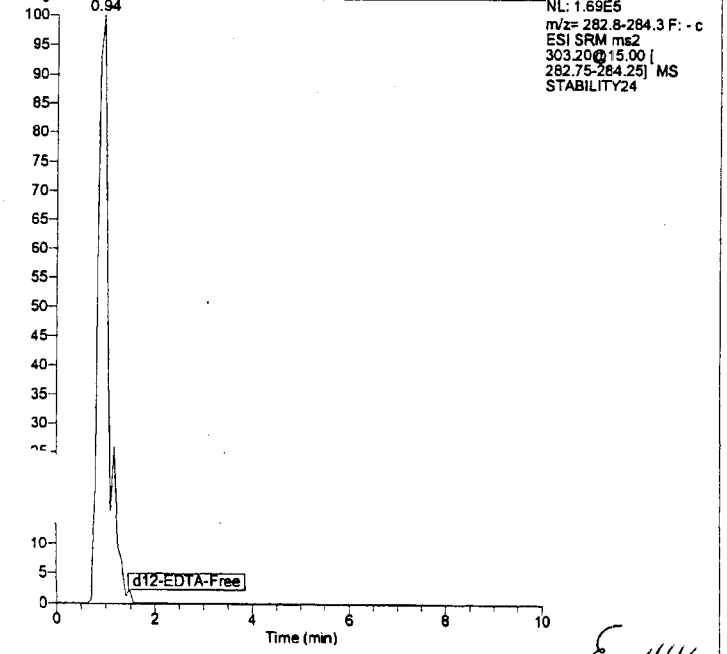


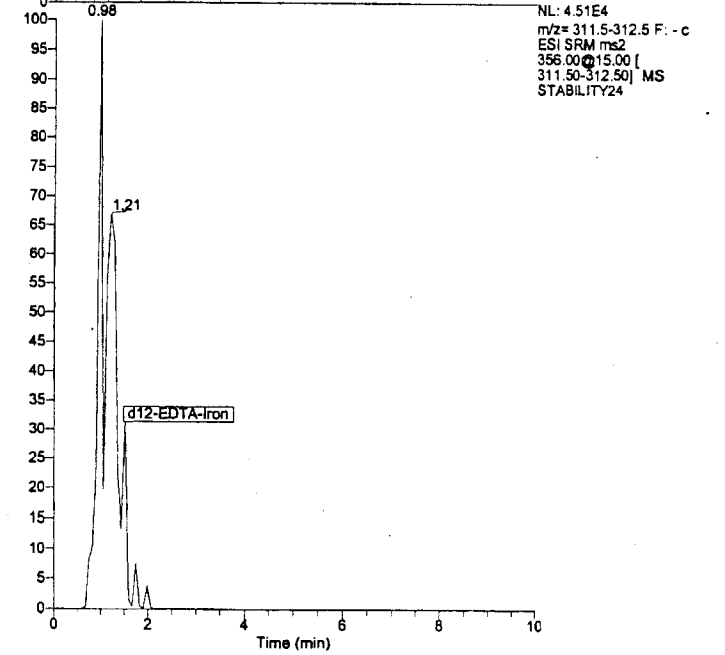


POS



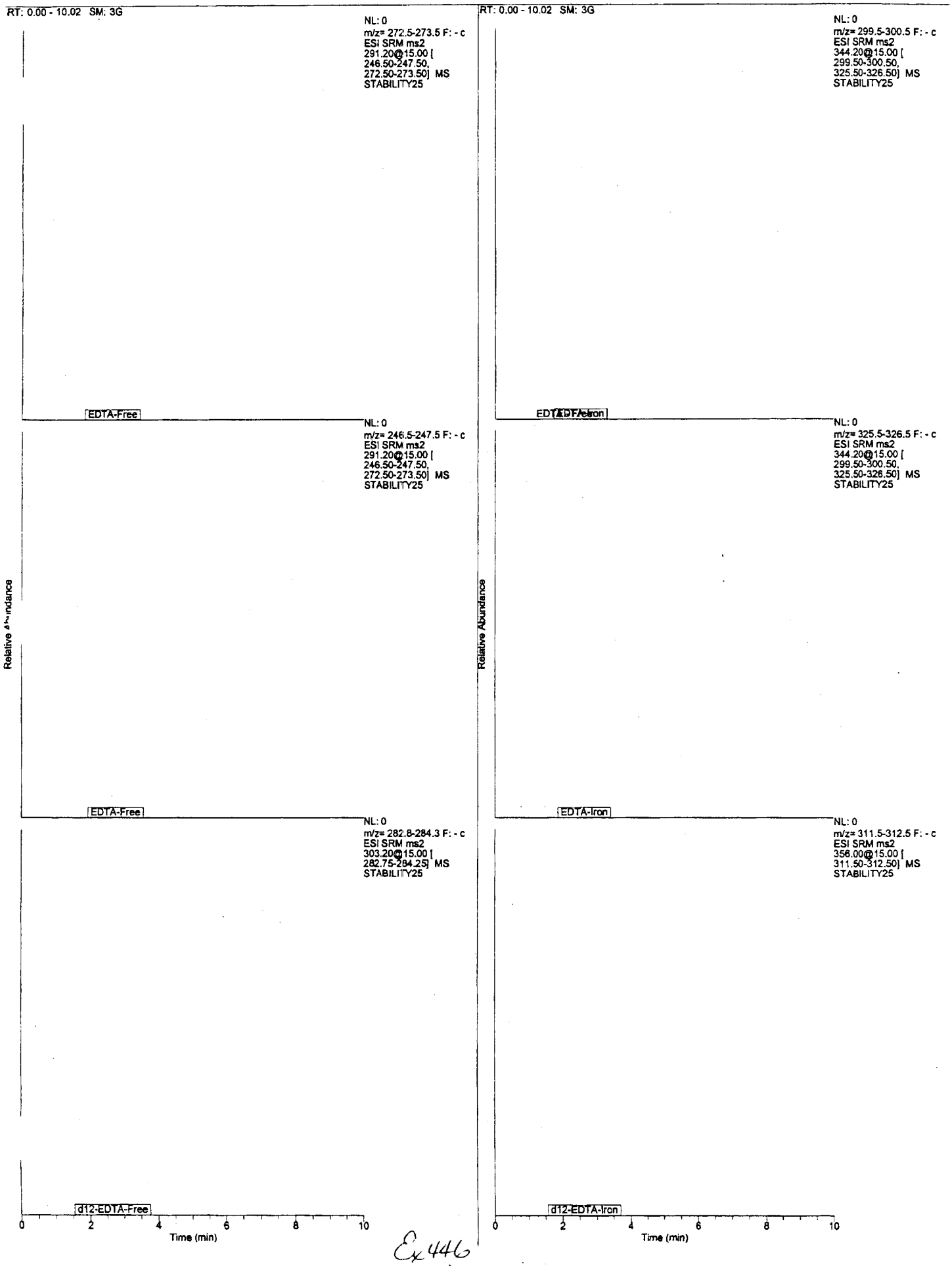
POS





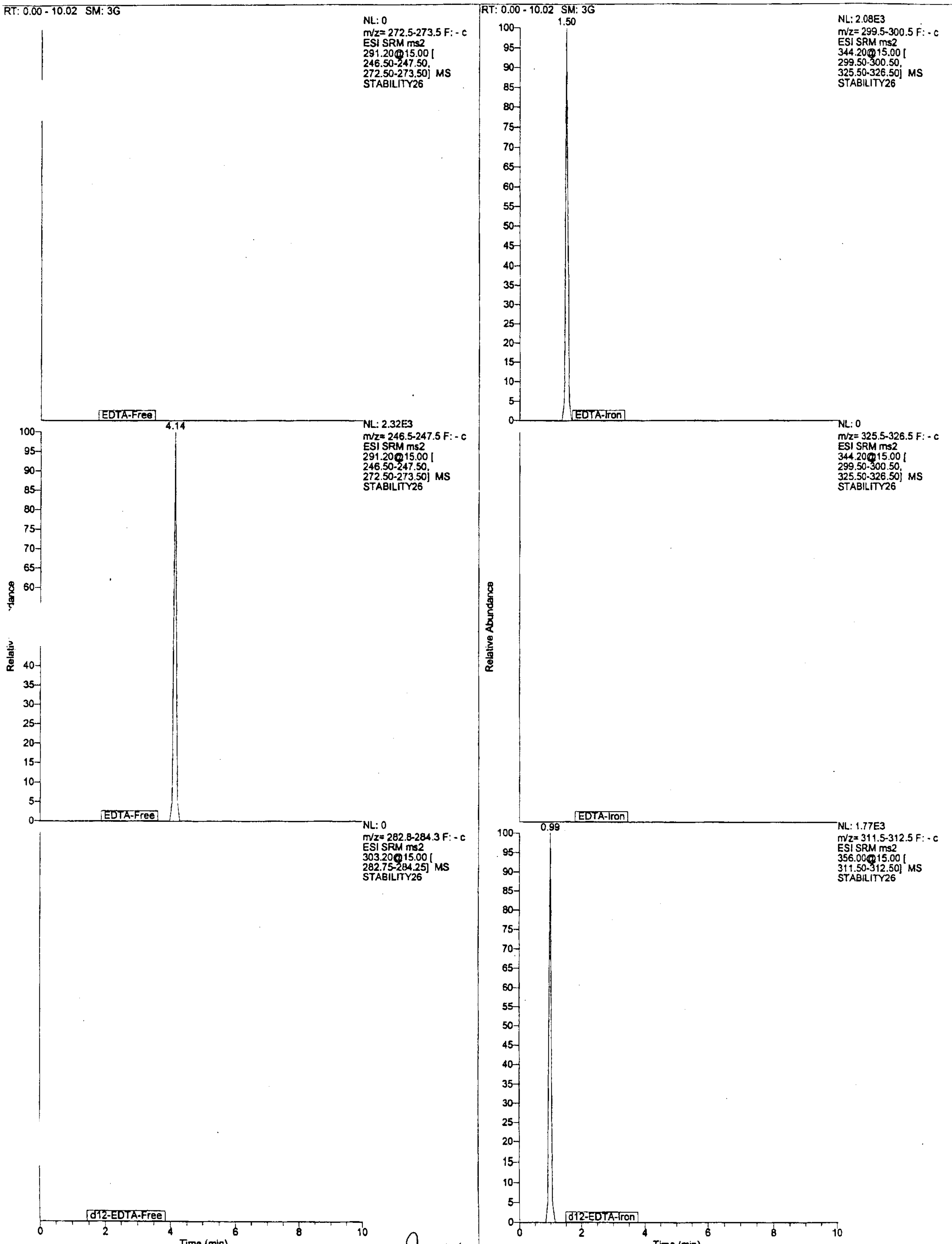
Ex 446
(22)

C:\Xcalibur\...\Stability\Stability25 JPB 02/28/07 08:56:43 BLANK (neg blood, DI H2O ext.)



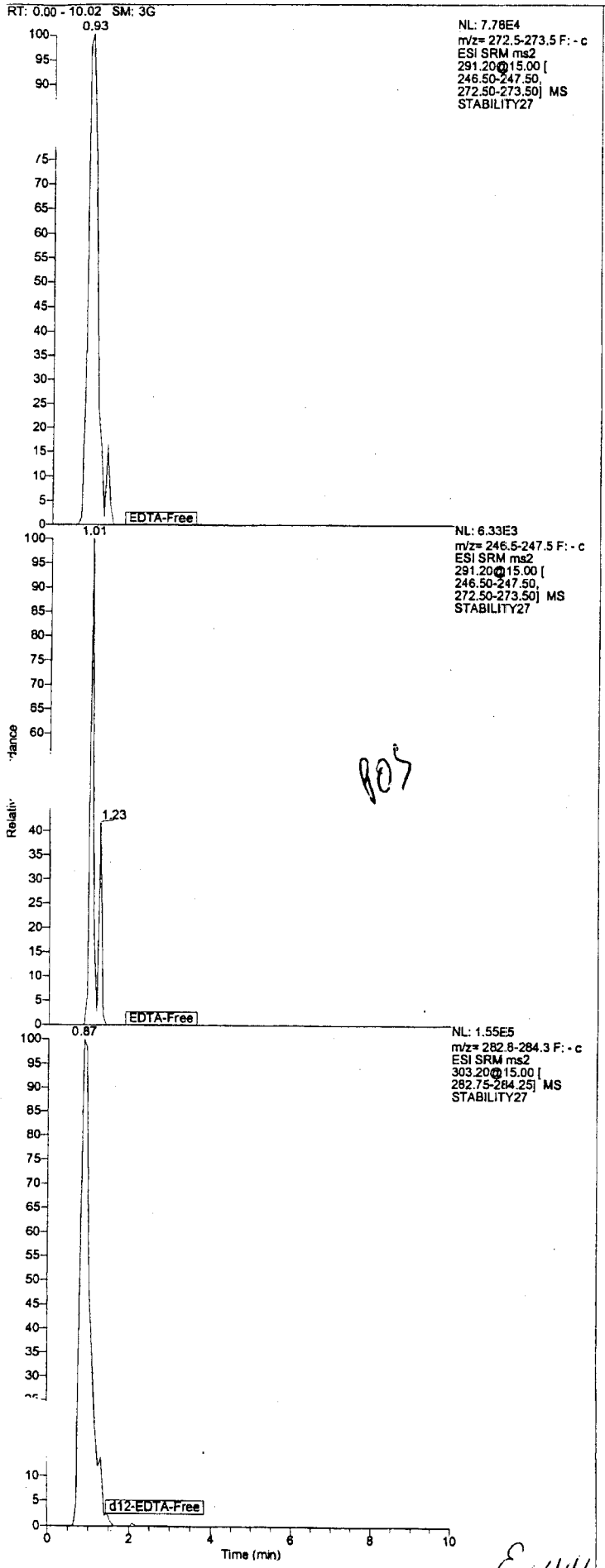
Ex 446
(23)

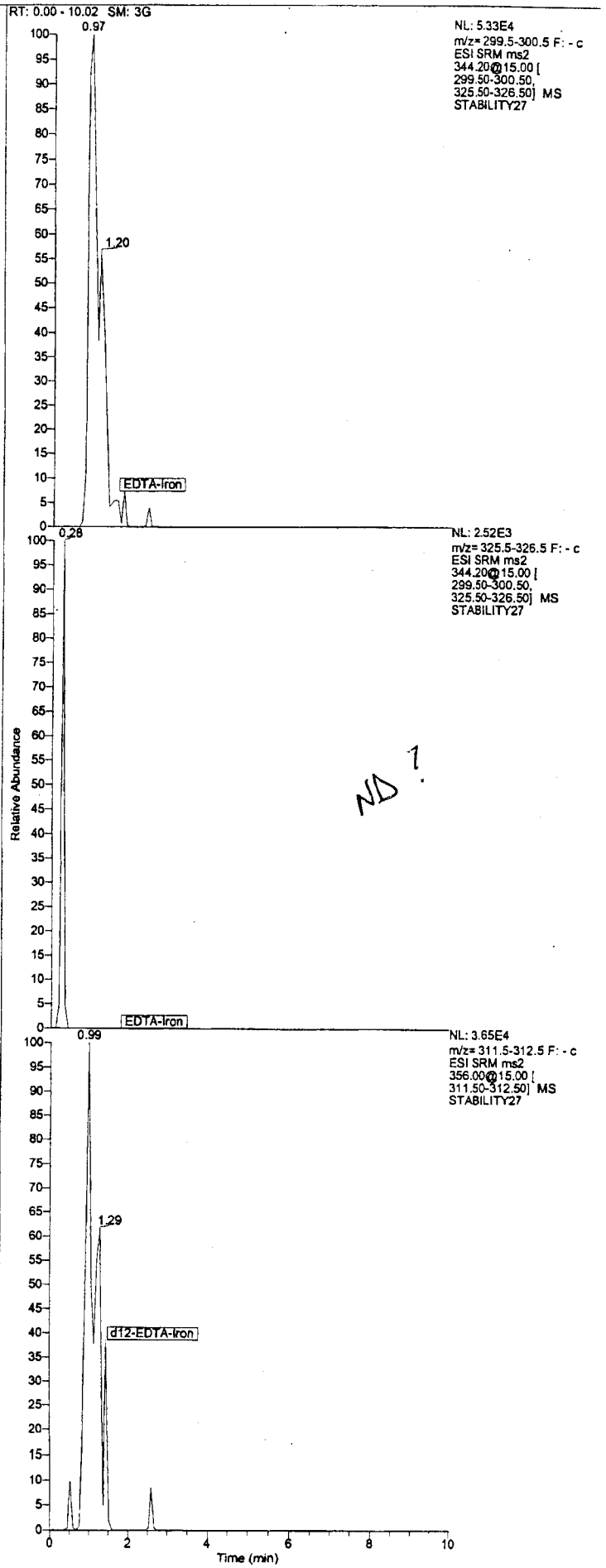
C:\Xcalibur\...\Stability\Stability26 JDP 02/28/07 09:07:38 BLANK (neg blood, DI H2O ext.)



Ex 446
(24)

C:\Xcalibur\...\Stability\Stability27 JBP 02/28/07 09:18:28 Card G extract (EDTA +)



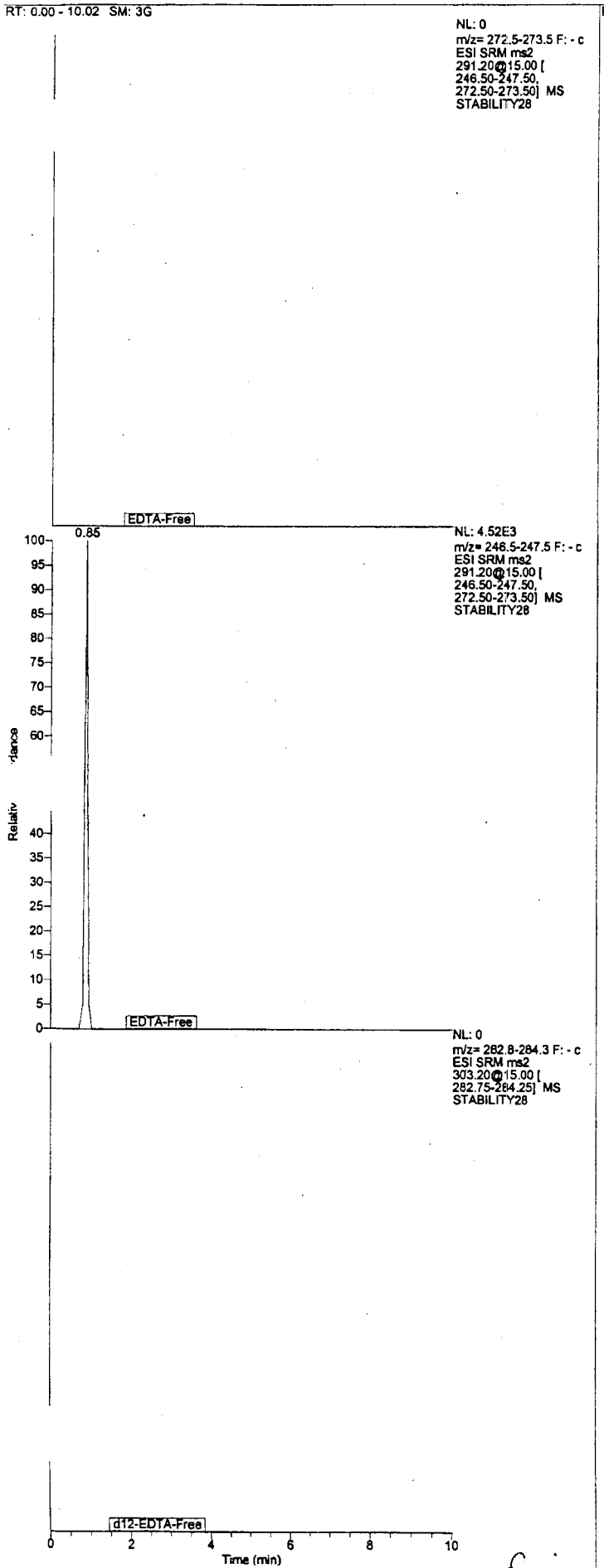


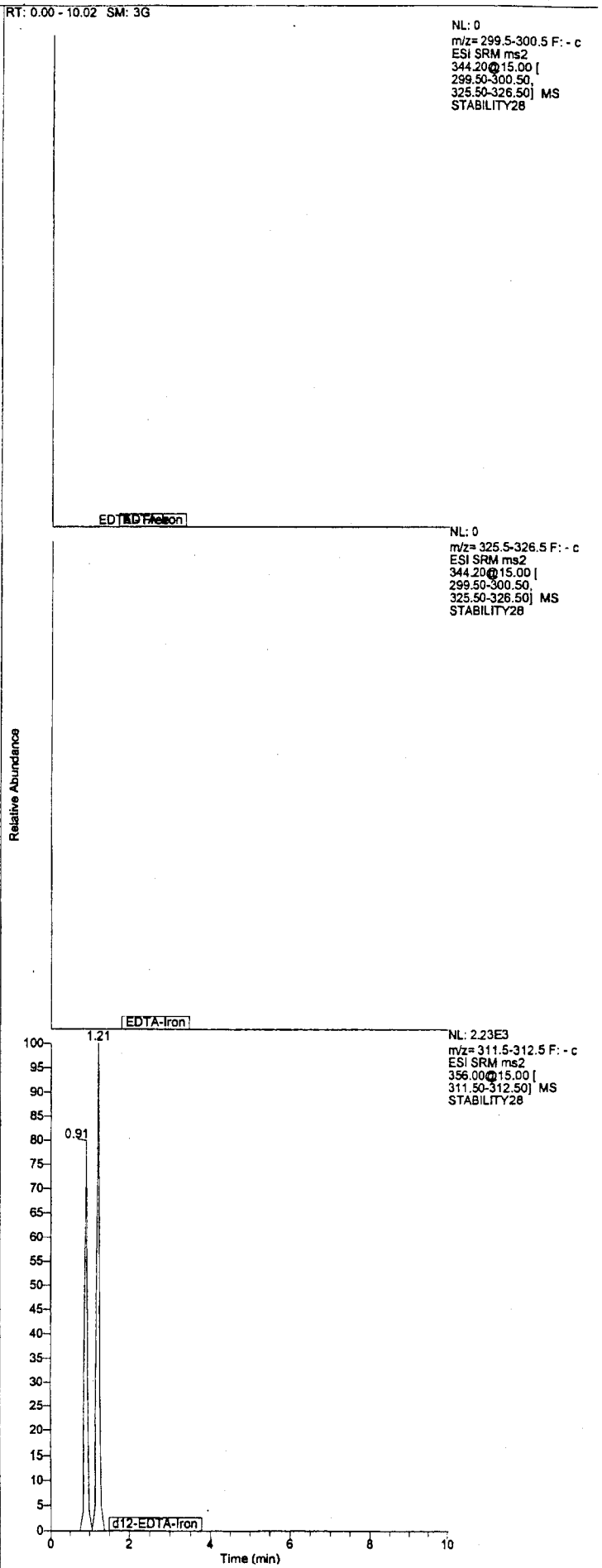
803

ND 1.

Ex 446
(25)

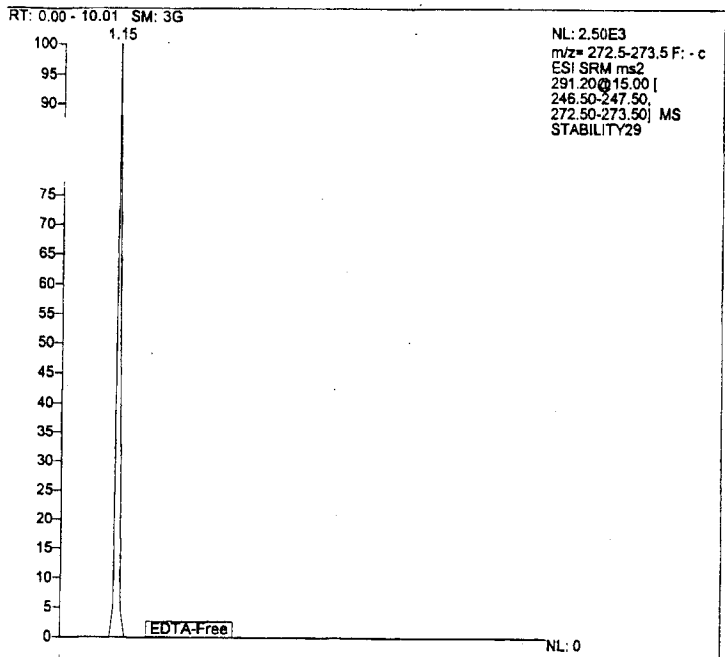
C:\Xcalibur\...\Stability\Stability28 JDB 02/28/07 09:29:23 BLANK (neg blood, DI H2O ext.)

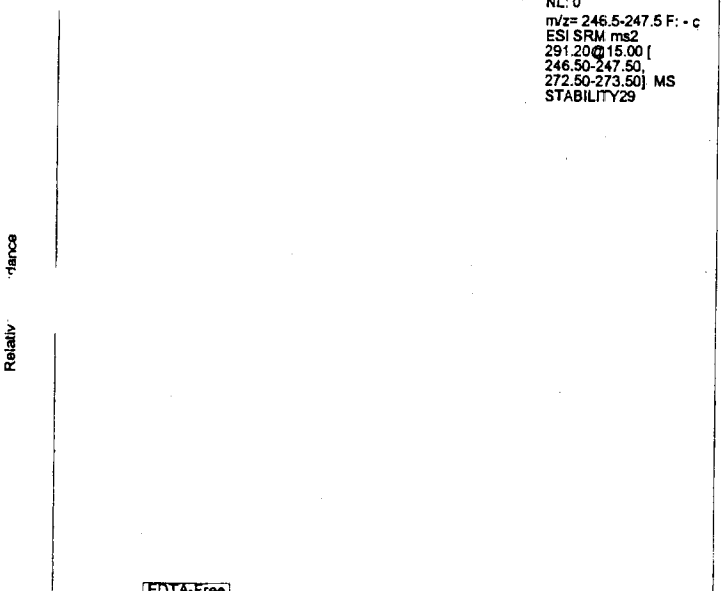


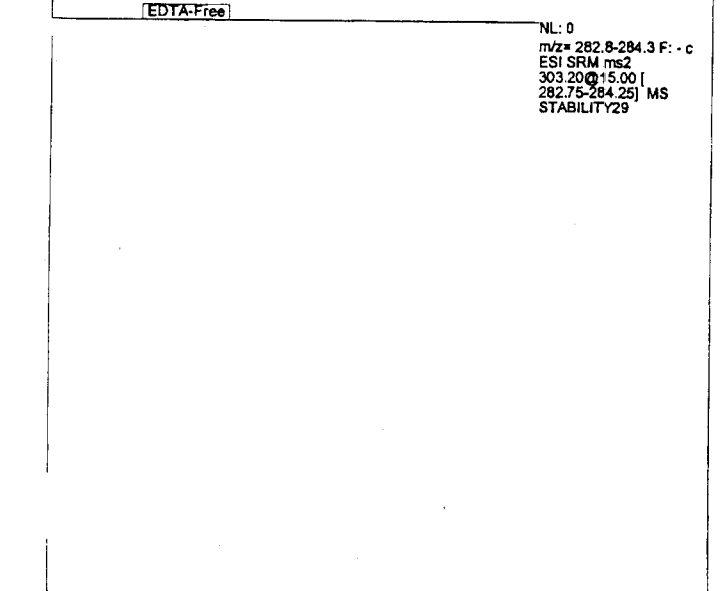


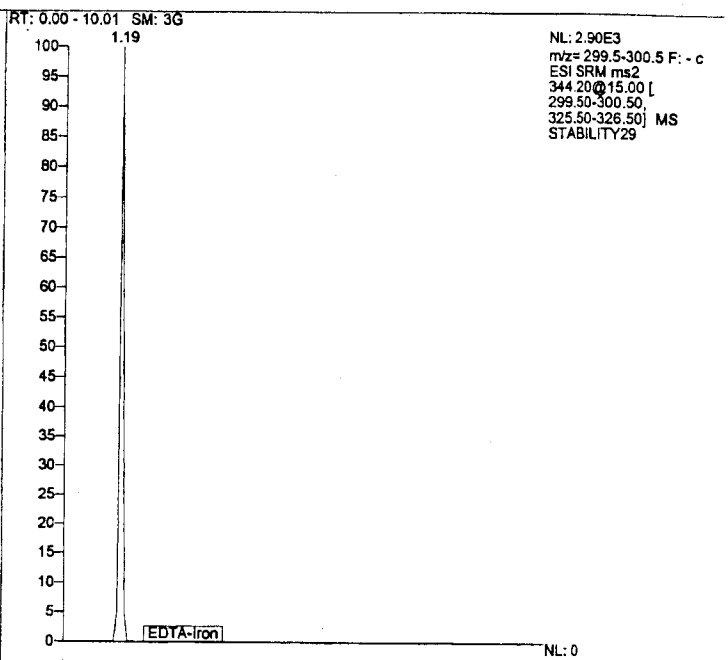
Ex 446
(24)

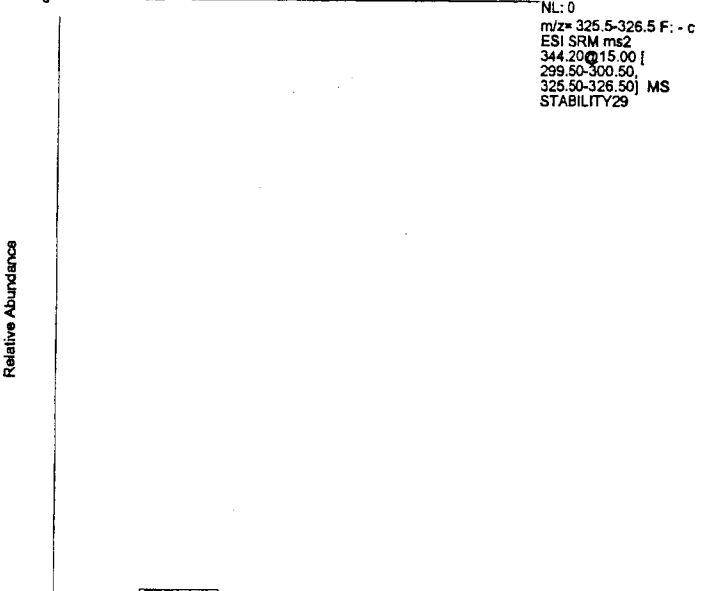
C:\Xcalibur\...\Stability\Stability29 JDR 02/28/07 09:40:17 BLANK (neg blood, DI H2O ext.)

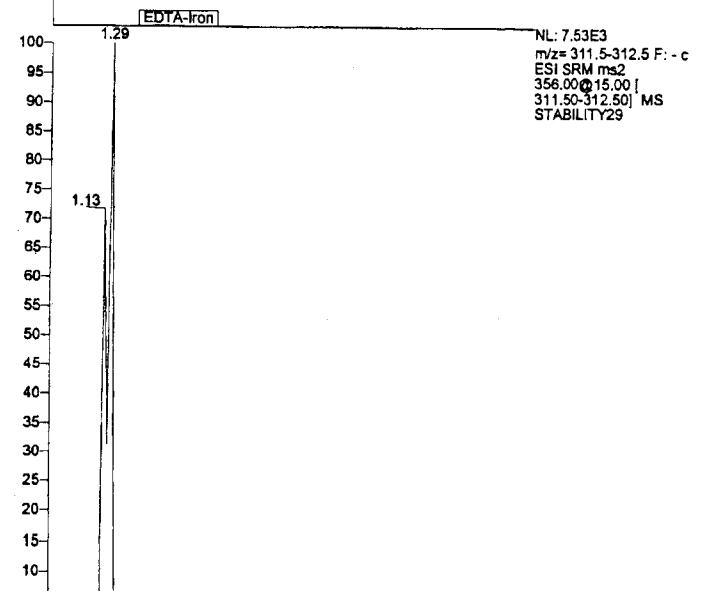








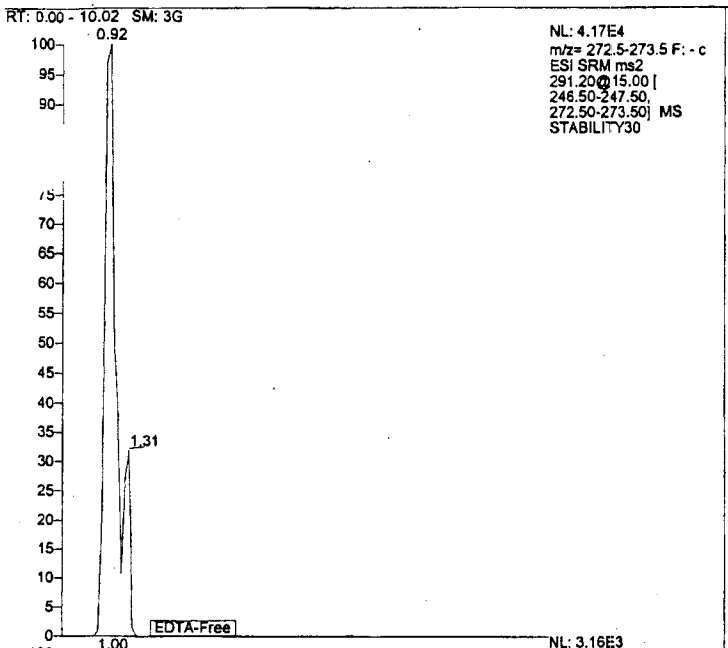


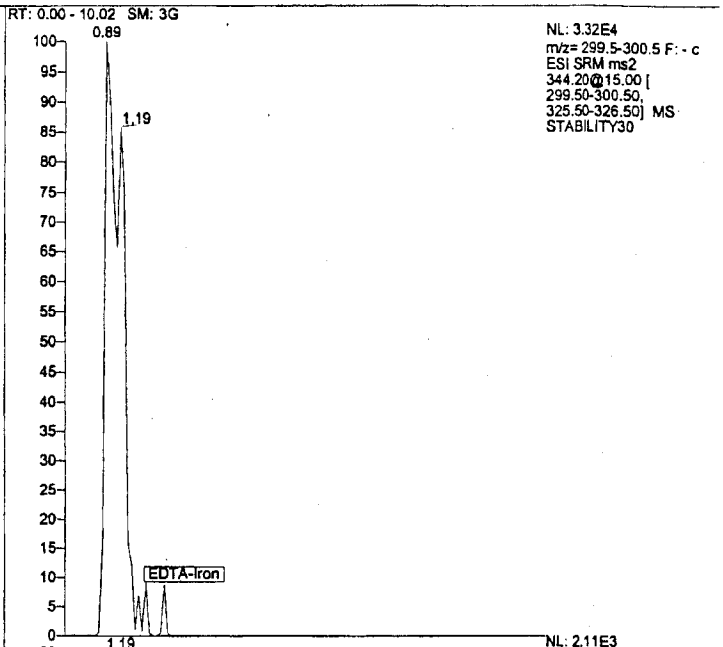


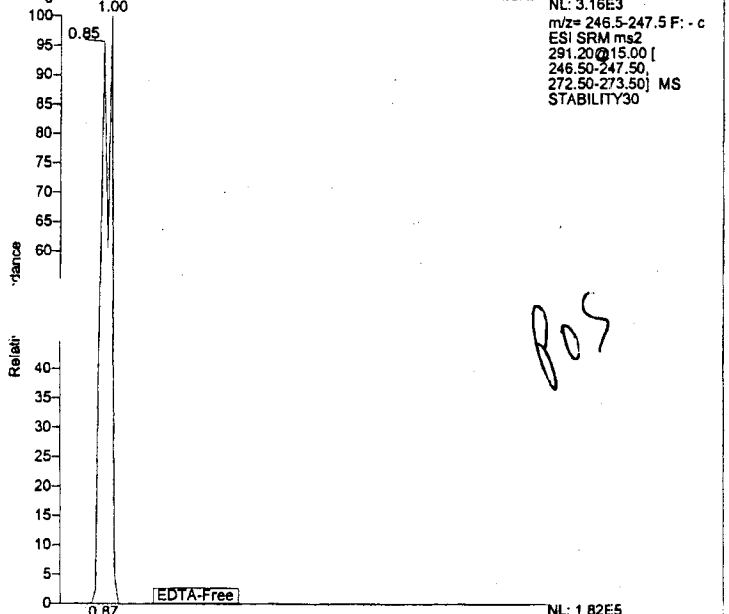
Relative Abundance

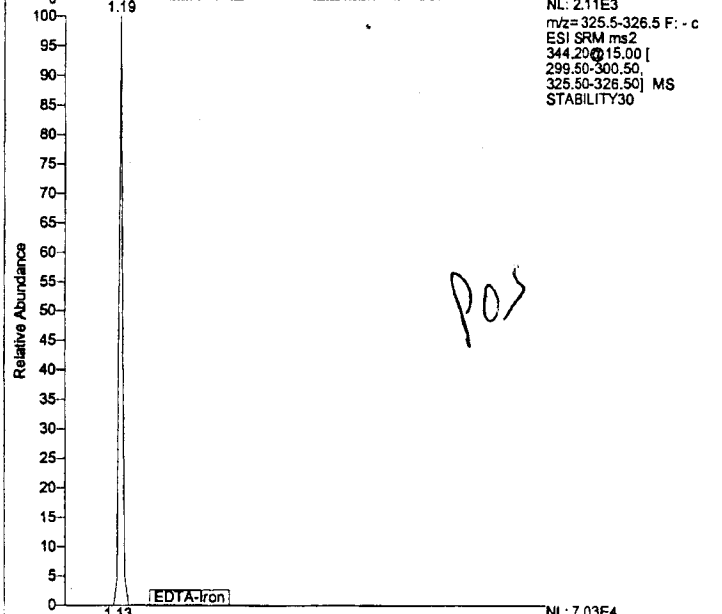
Ck 446
(27)

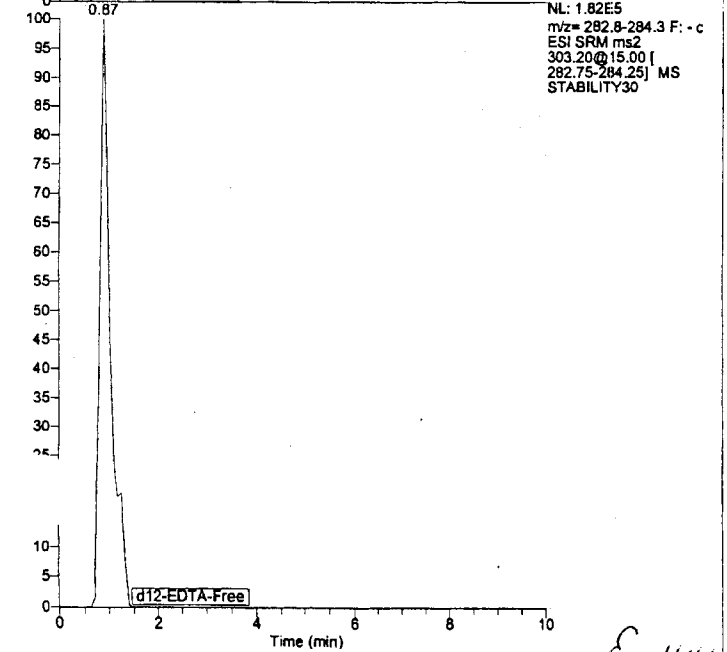
C:\Xcalibur\...\Stability\Stability30 JDβ 02/28/07 09:51:06 Card H extract (EDTA +)

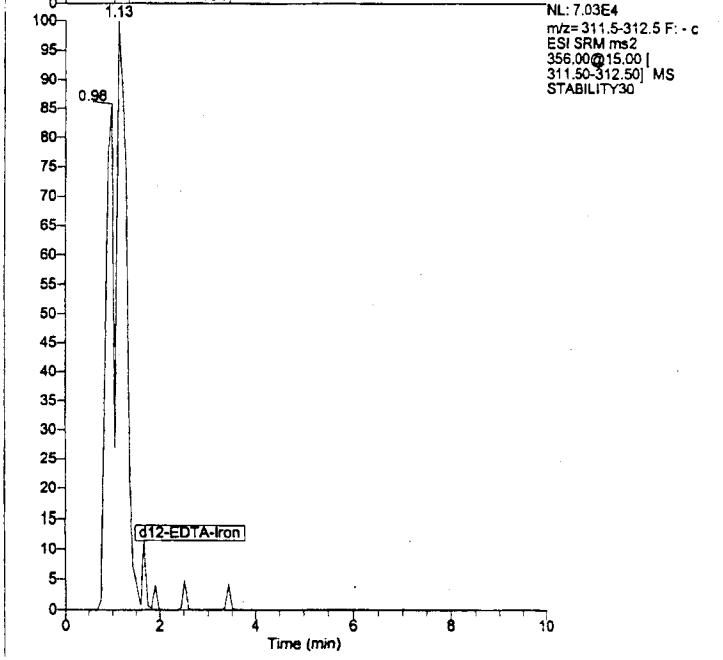






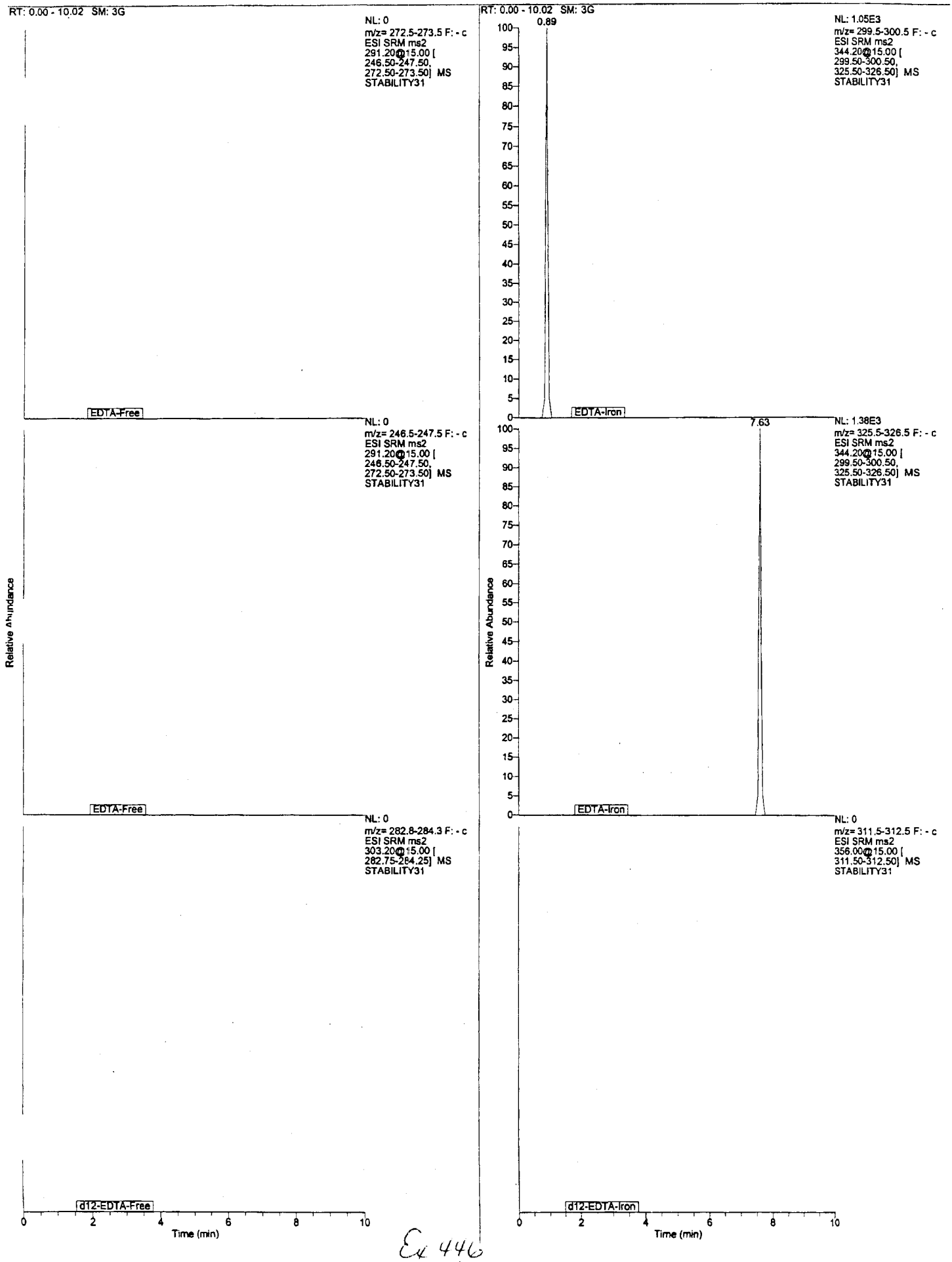






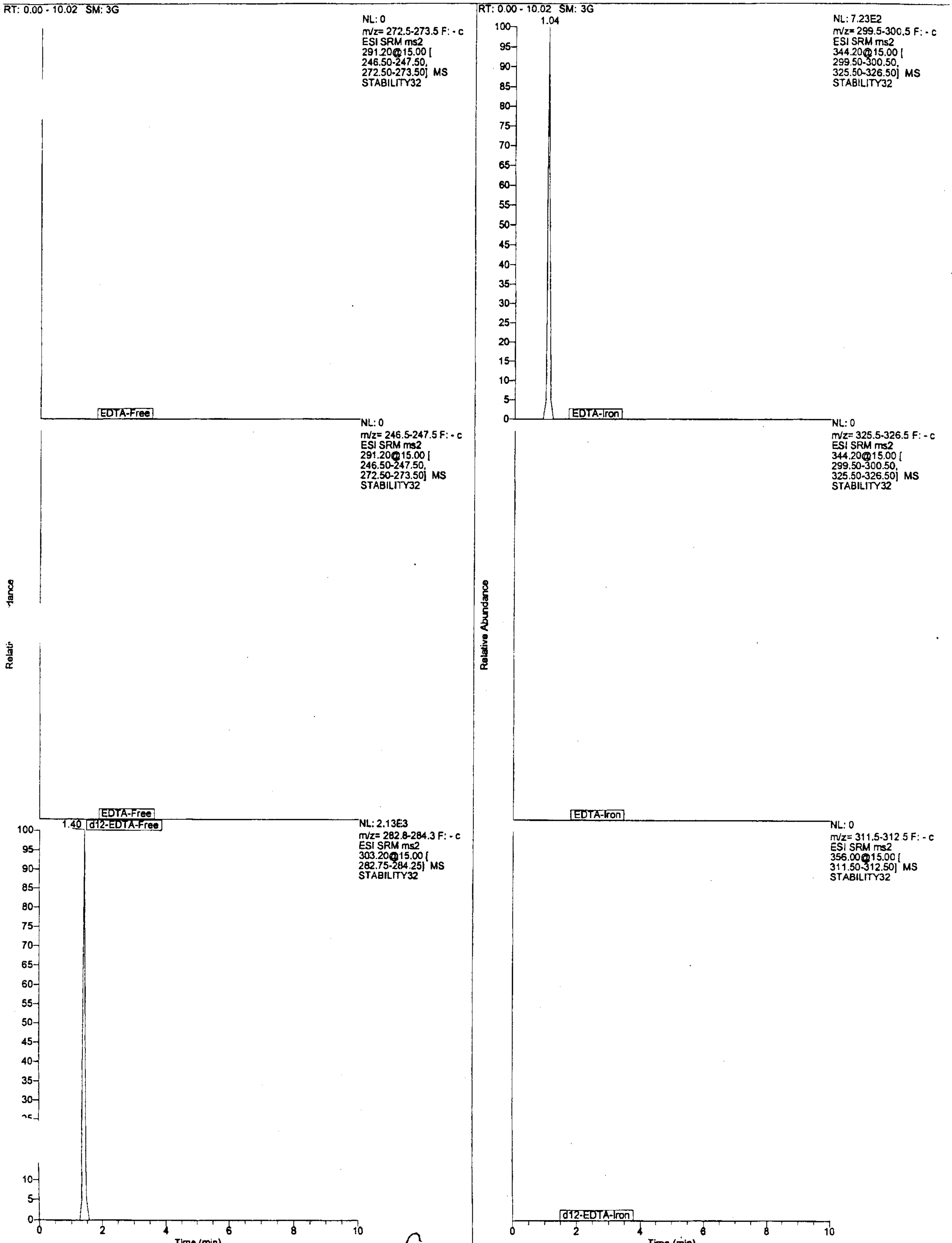
Ex 446
(28)

C:\Xcalibur\...\Stability\Stability31 JDB 02/28/07 10:01:55 BLANK (neg blood, DI H2O ext.)



Ex 446
(2)

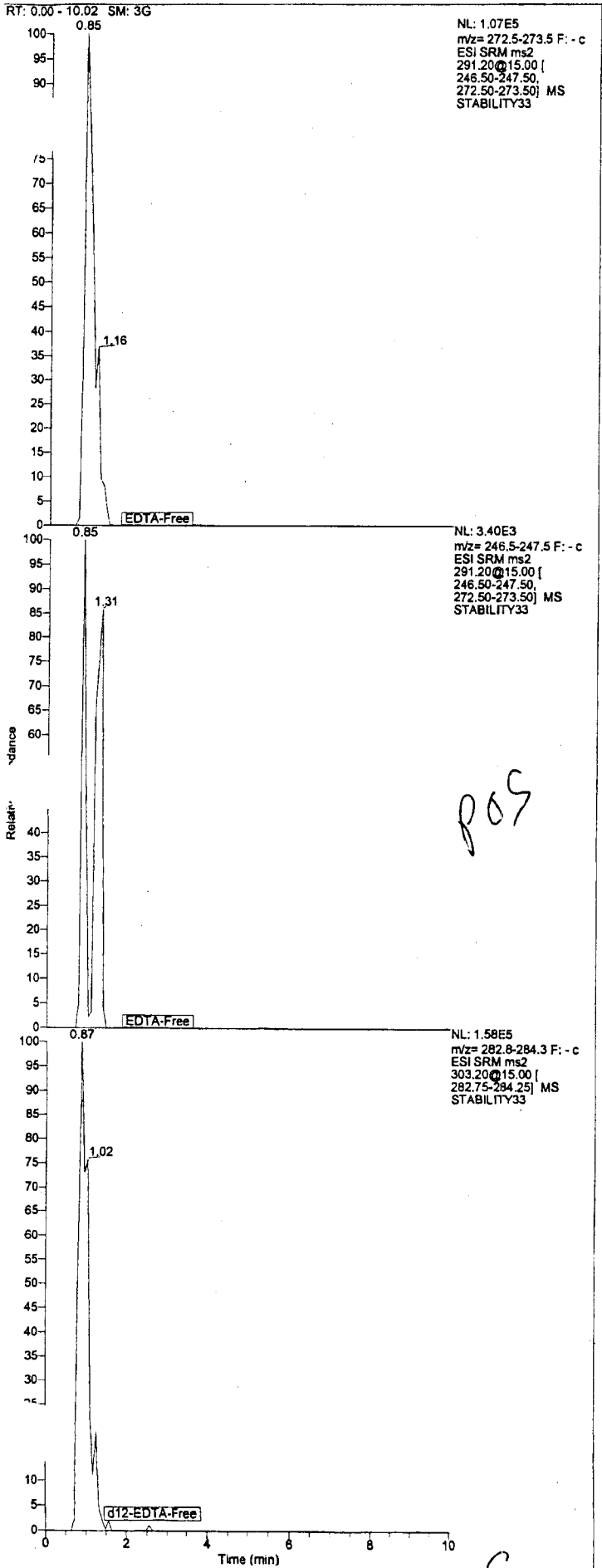
C:\Xcalibur\...\Stability\Stability32 JDP 02/28/07 10:12:51 BLANK (neg blood, DI H2O ext.)

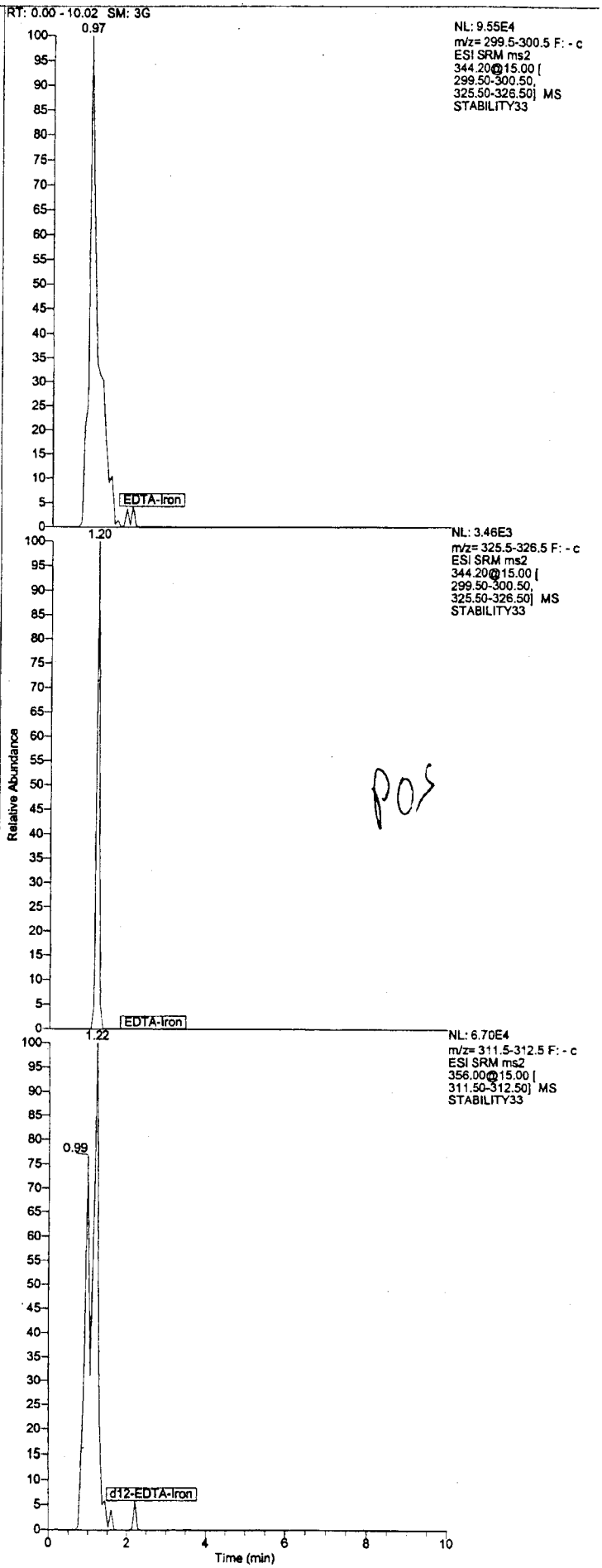


E4 446

(30)

C:\Xcalibur\...\Stability\Stability33 JDB 02/28/07 10:23:40 Card I extract (EDTA +)



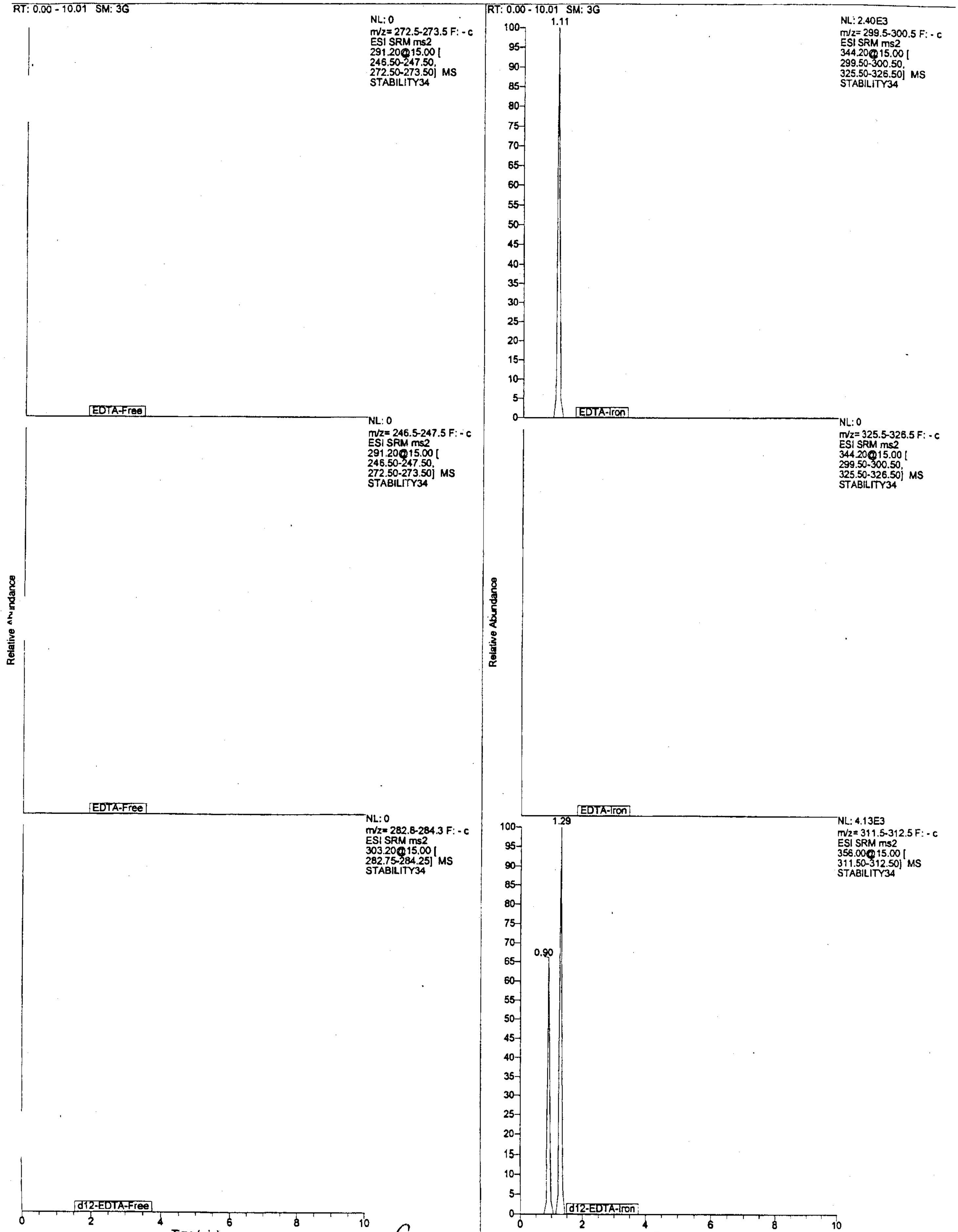


POS

POS

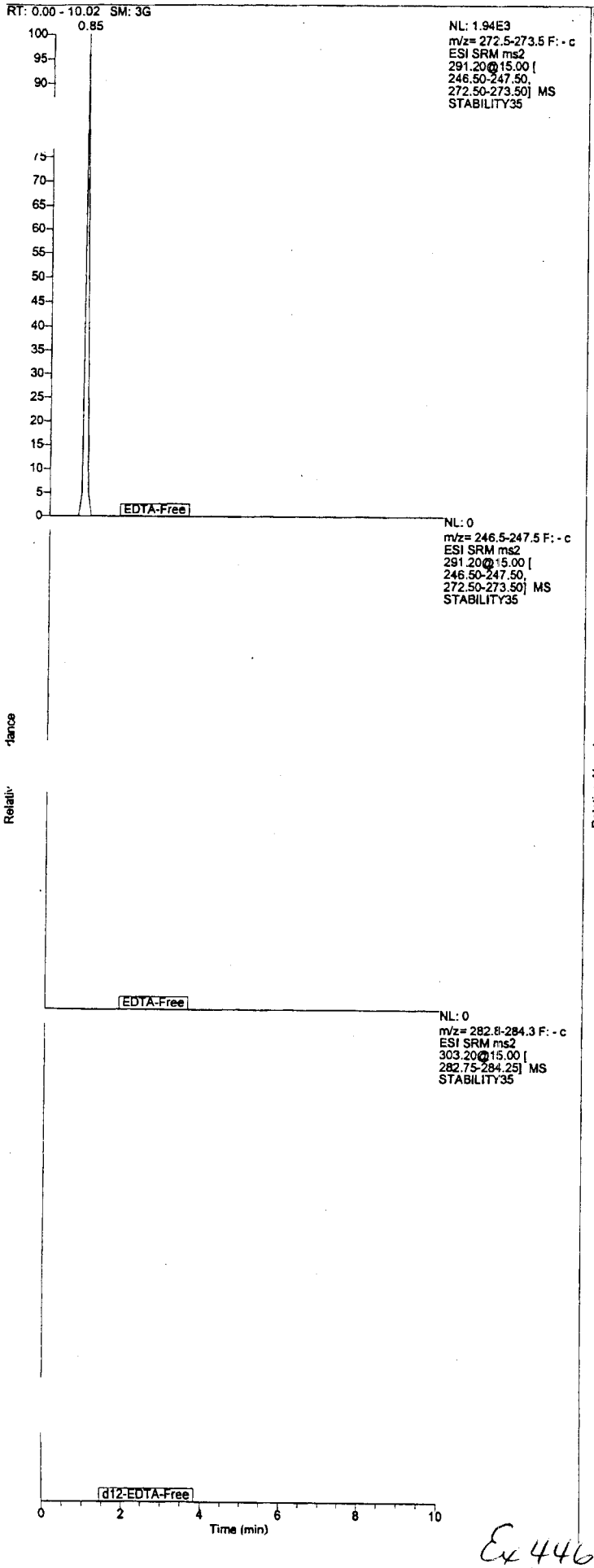
Ex 446
(2)

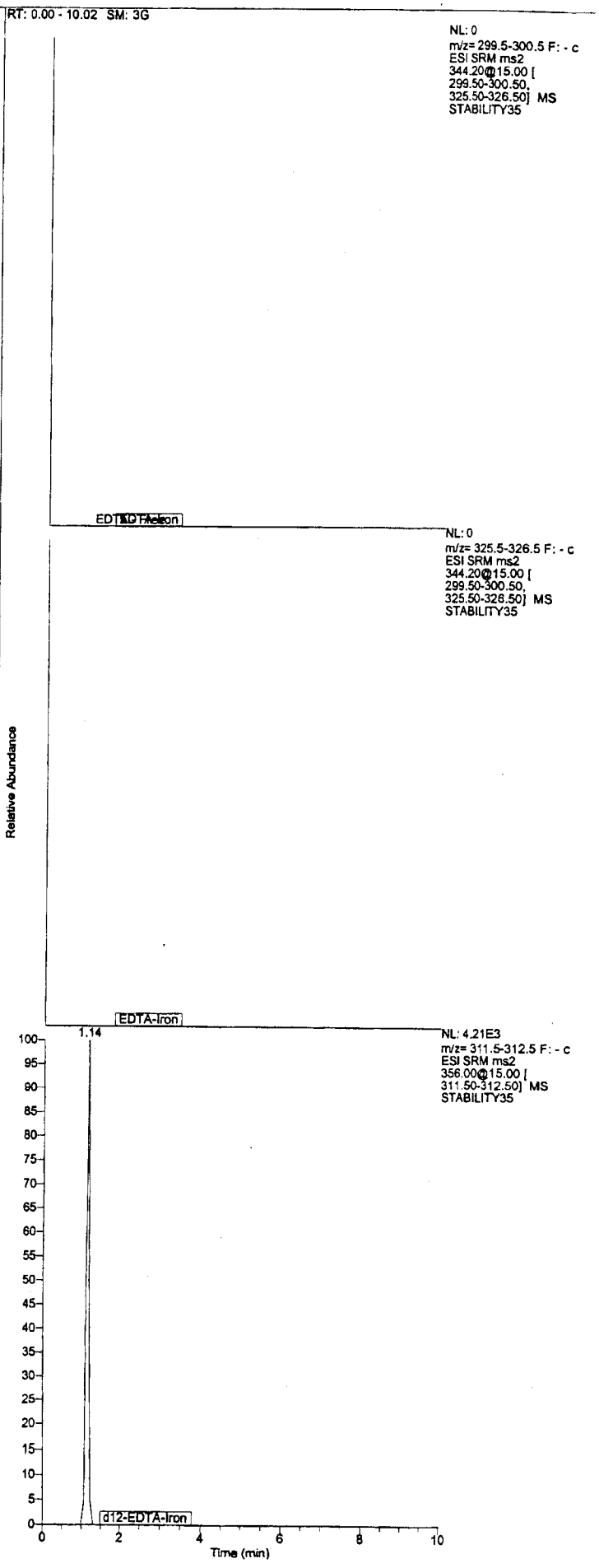
C:\Xcalibur\...\Stability\Stability34 JDβ 02/28/07 10:34:32 BLANK (neg blood, DI H2O ext.)



Ex 446
(32)

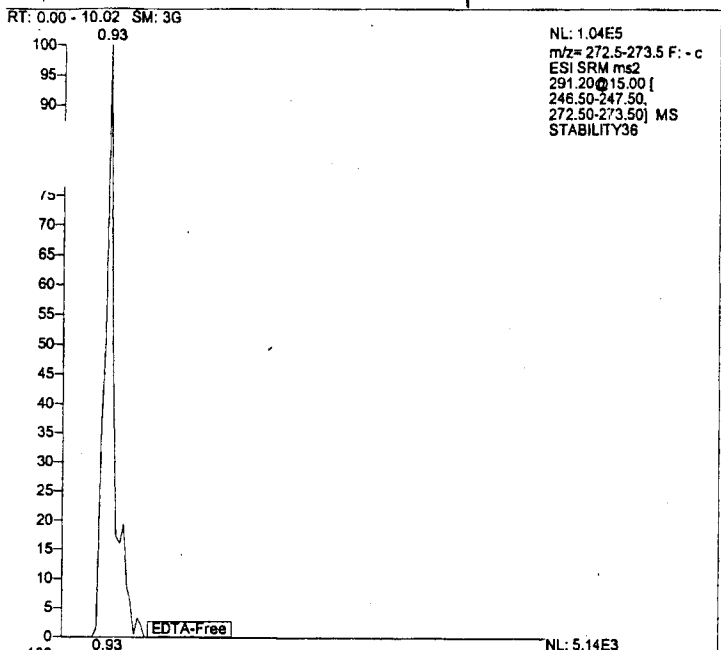
C:\Xcalibur\...\Stability\Stability35 JPB 02/28/07 10:45:25 BLANK (neg blood, DI H2O ext.)

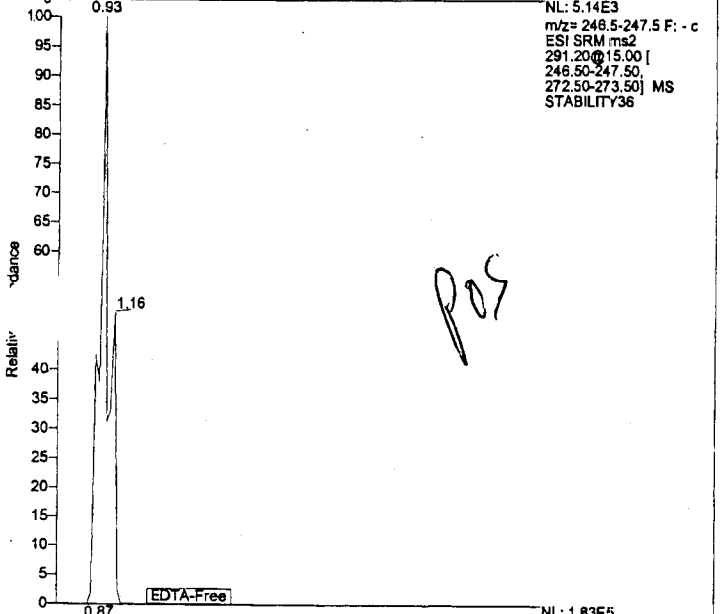




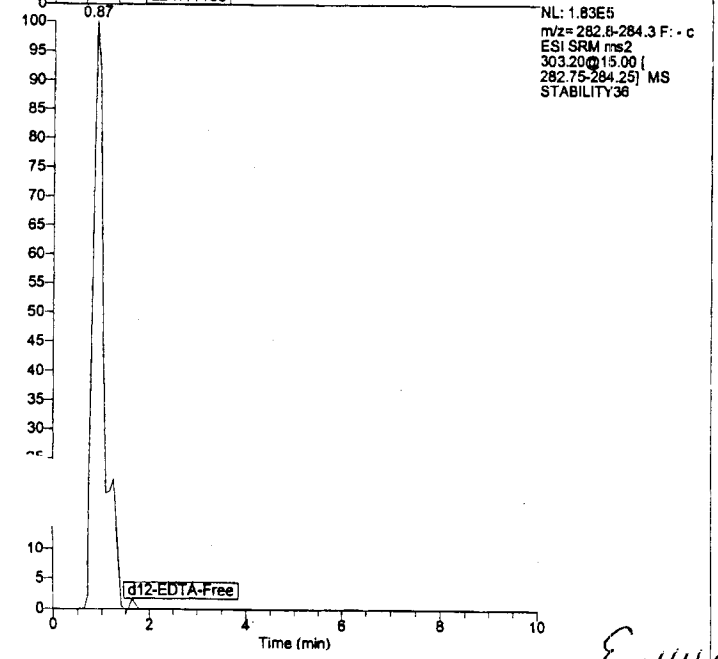
Ex 446

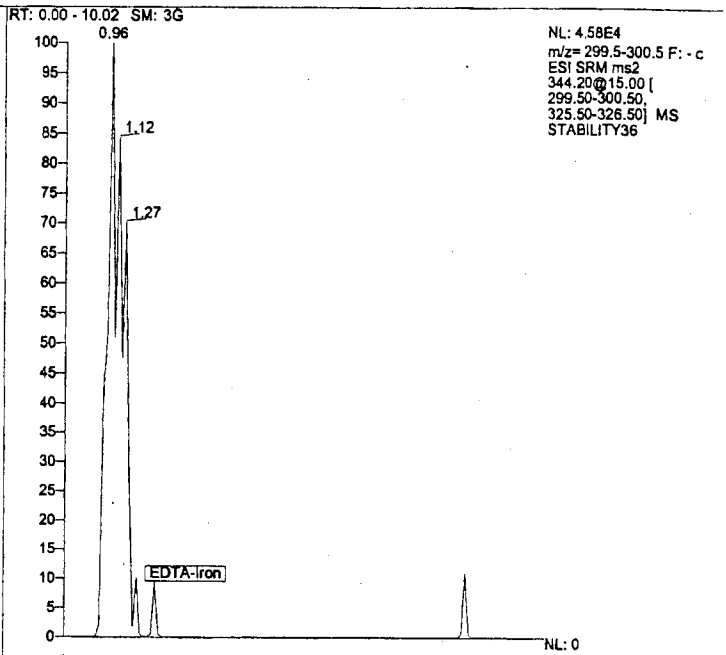
C:\Xcalibur\...\Stability\Stability36 JDB 02/28/07 10:56:17 Card J extract (EDTA +)

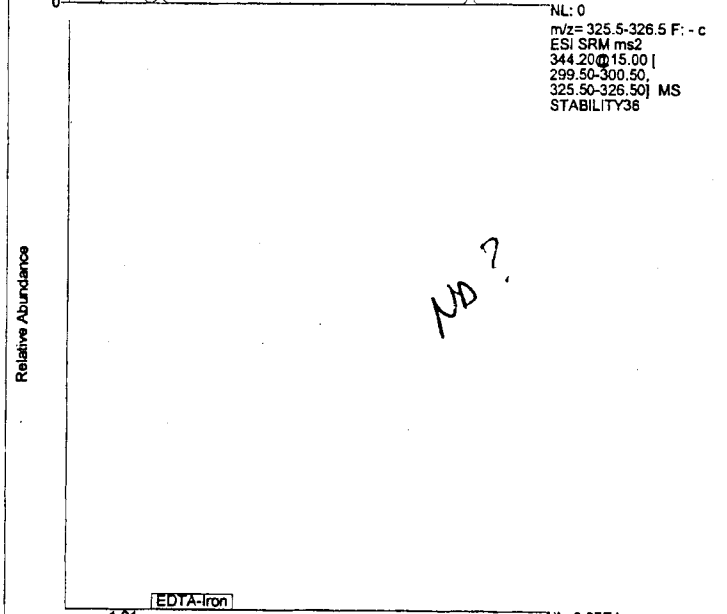




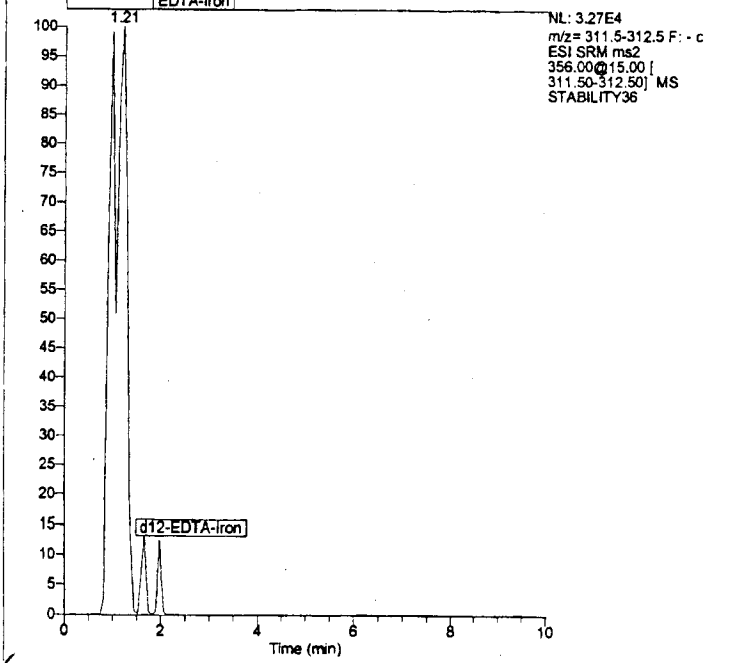
POS





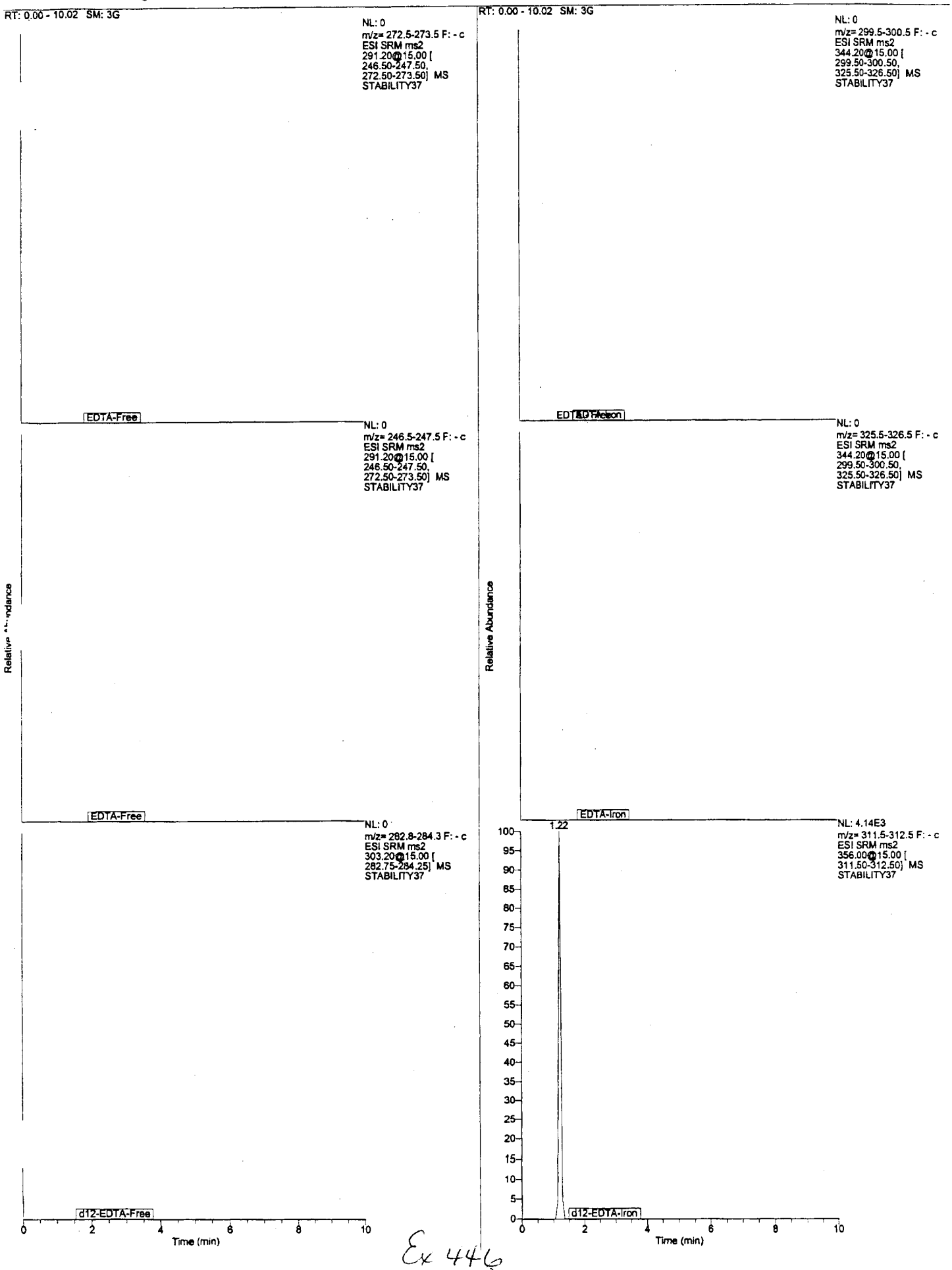


ND ?



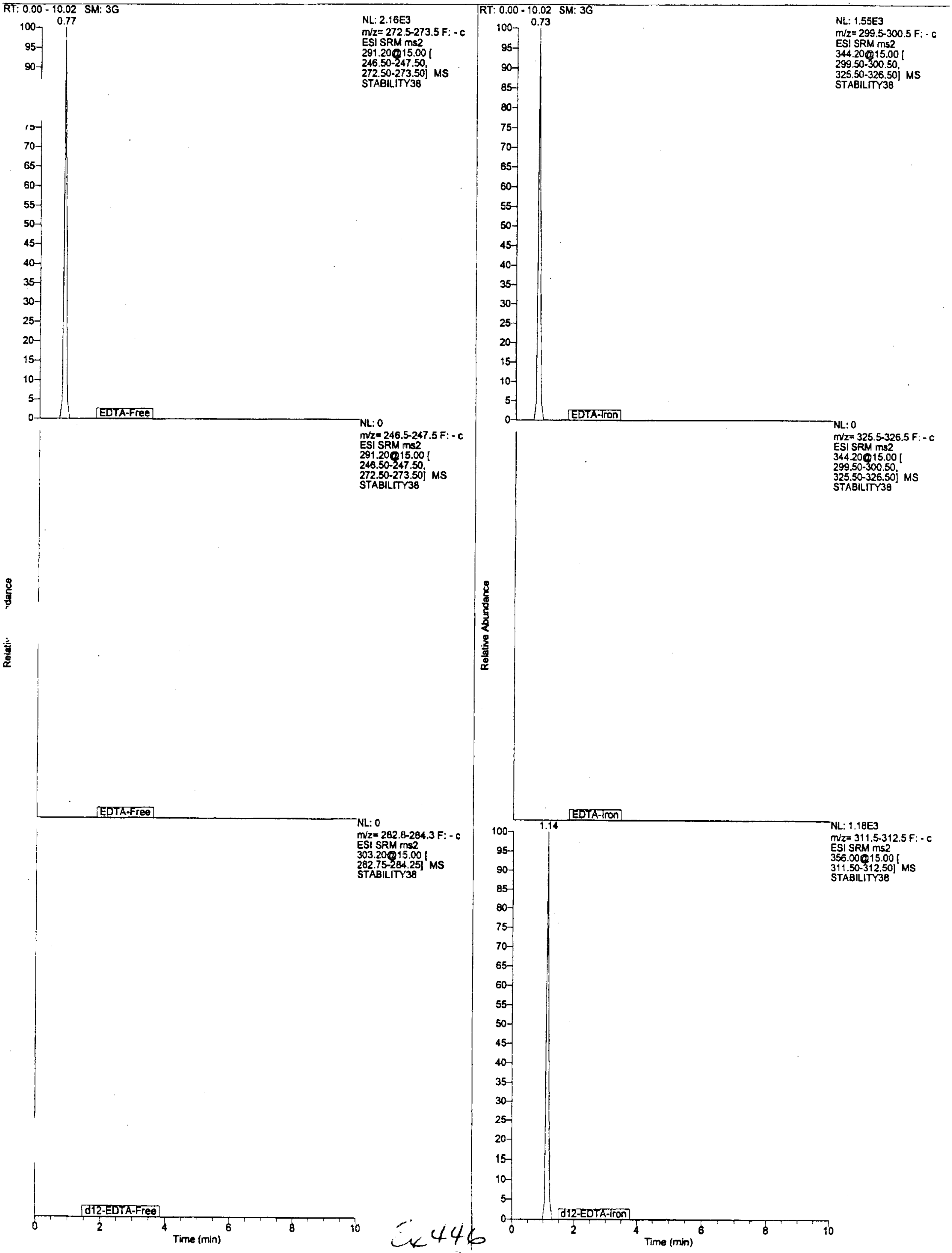
Ex 446

C:\Xcalibur\...\Stability\Stability37 JDP 02/28/07 11:07:10 BLANK (neg blood, DI H2O ext.)



Ex 446

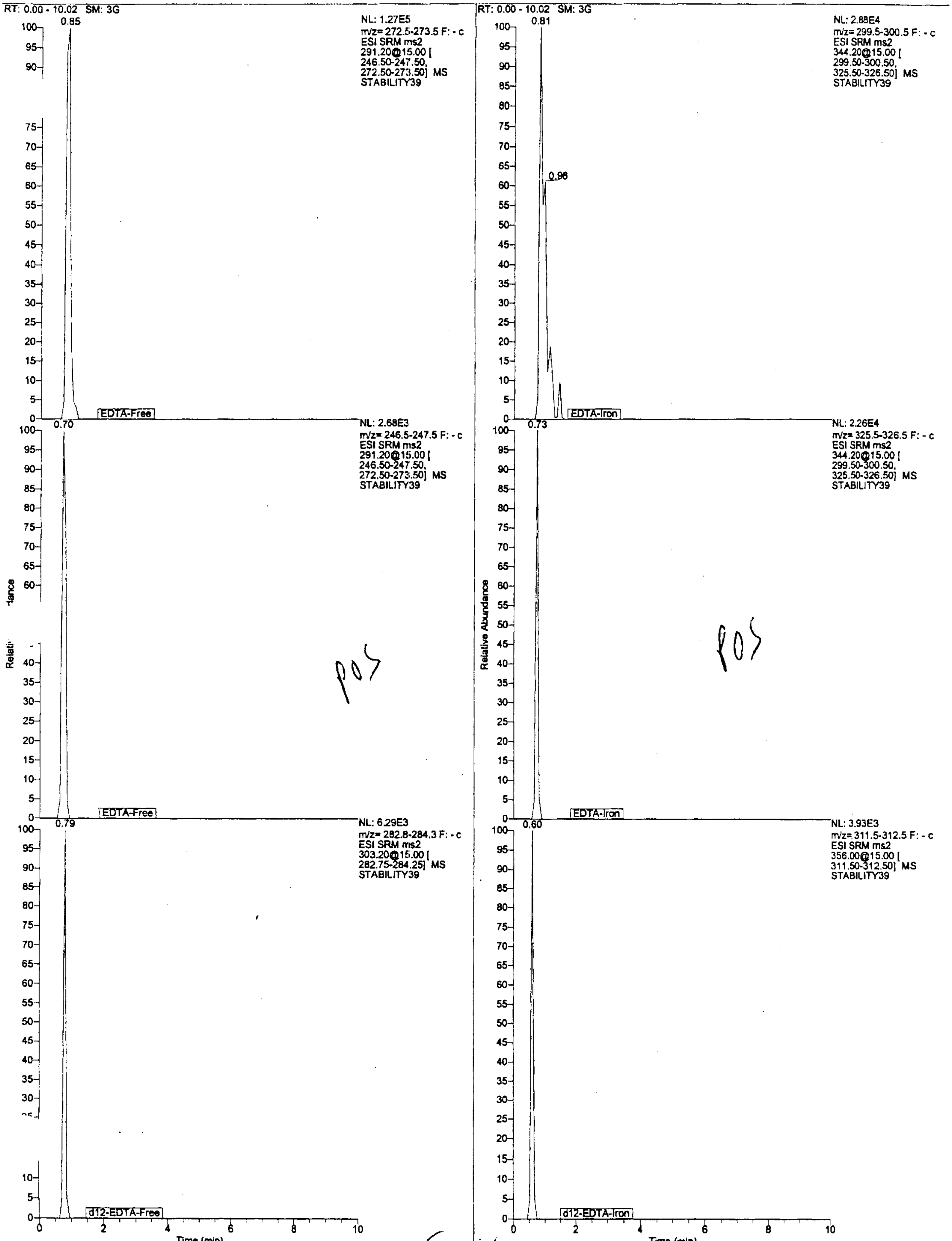
C:\Xcalibur\...\Stability\Stability38 JDB 02/28/07 11:18:05 BLANK (neg blood, DI H2O ext.)



Ex 446

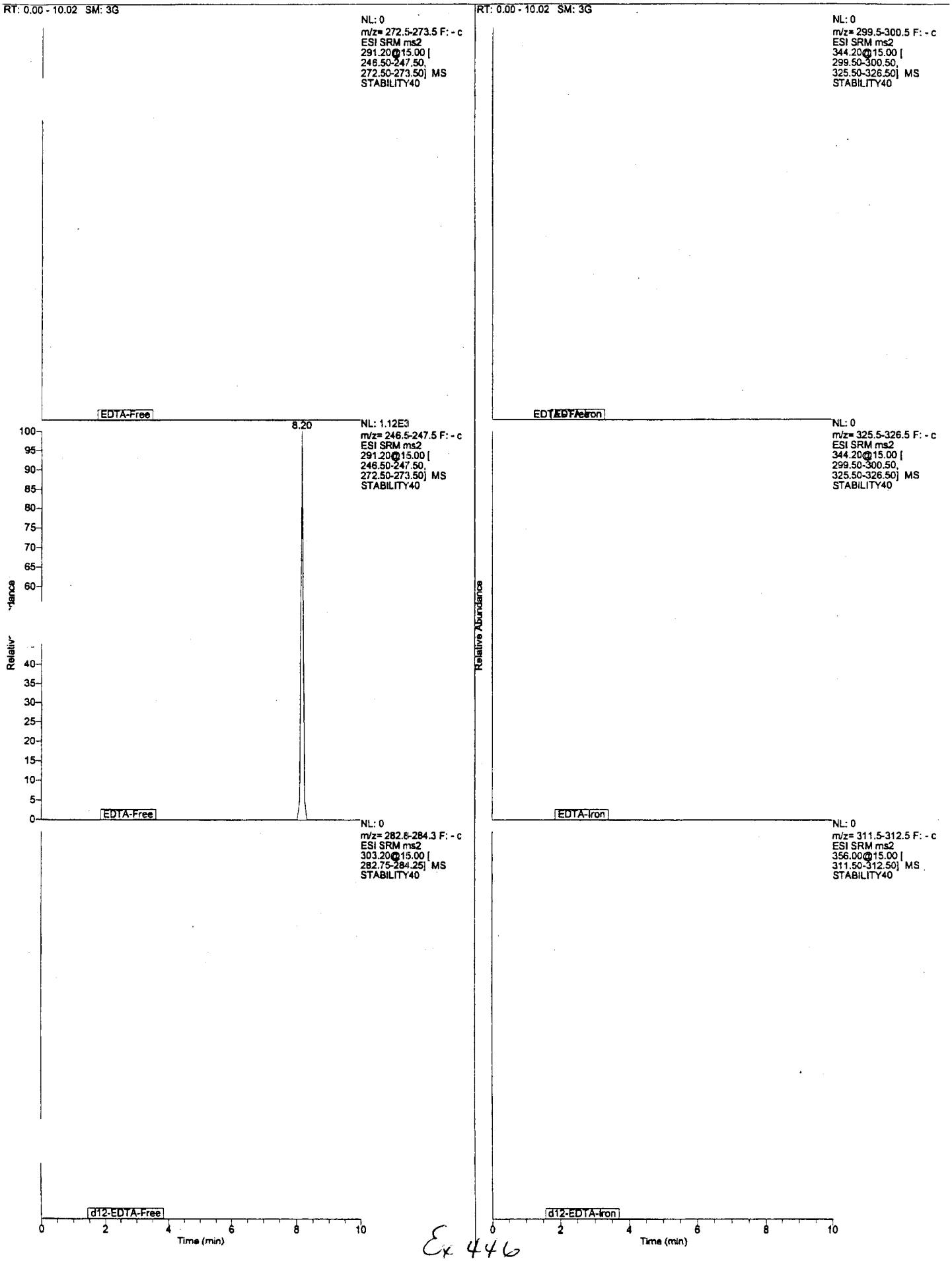
(36)

C:\Xcalibur\...\Stability\Stability39 JDB 02/28/07 11:28:55 100 ppm EDTA



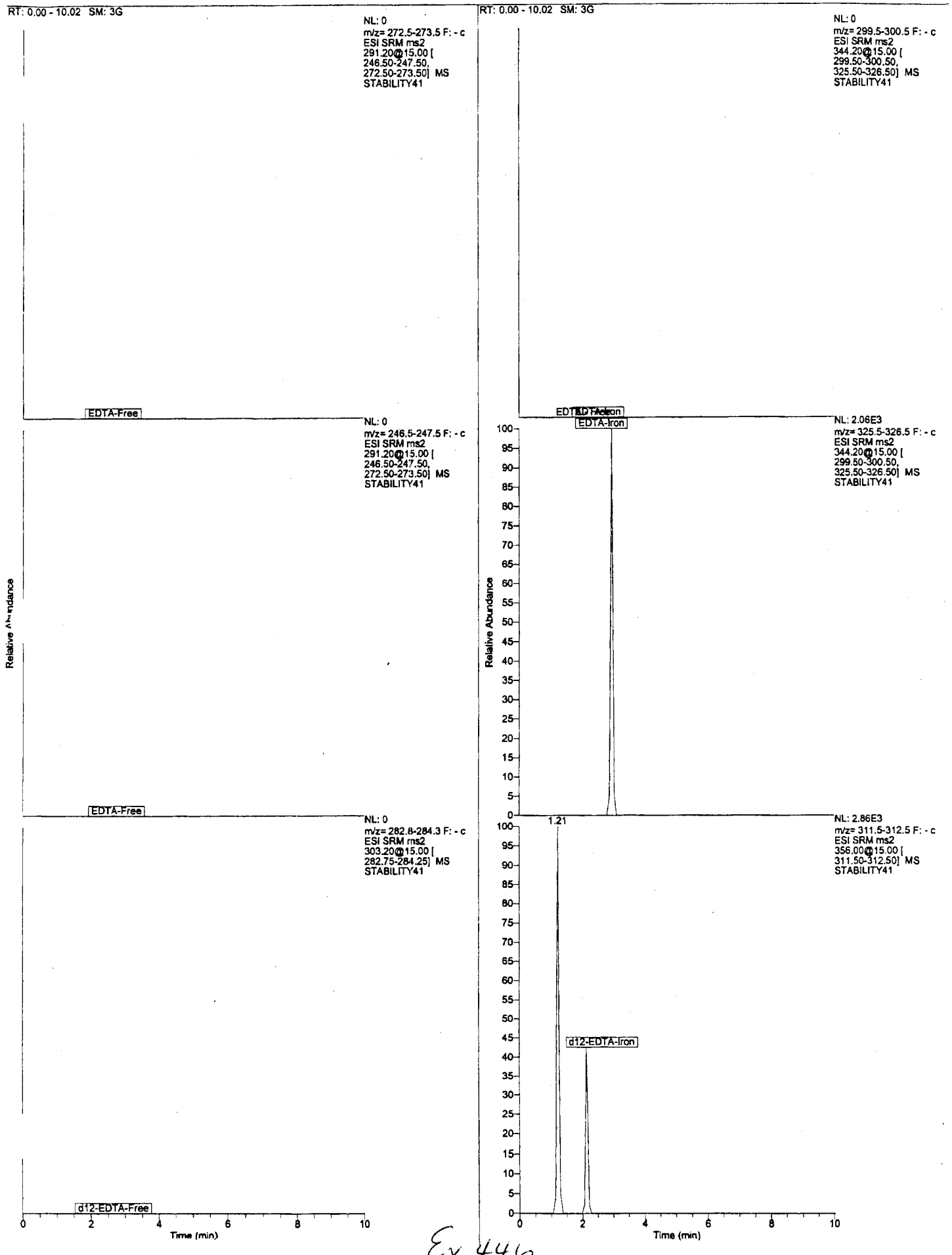
Ex 446
(37)

C:\Xcalibur\...\Stability\Stability40 JDB 02/28/07 11:39:48 BLANK (neg blood, DI H2O ext.)



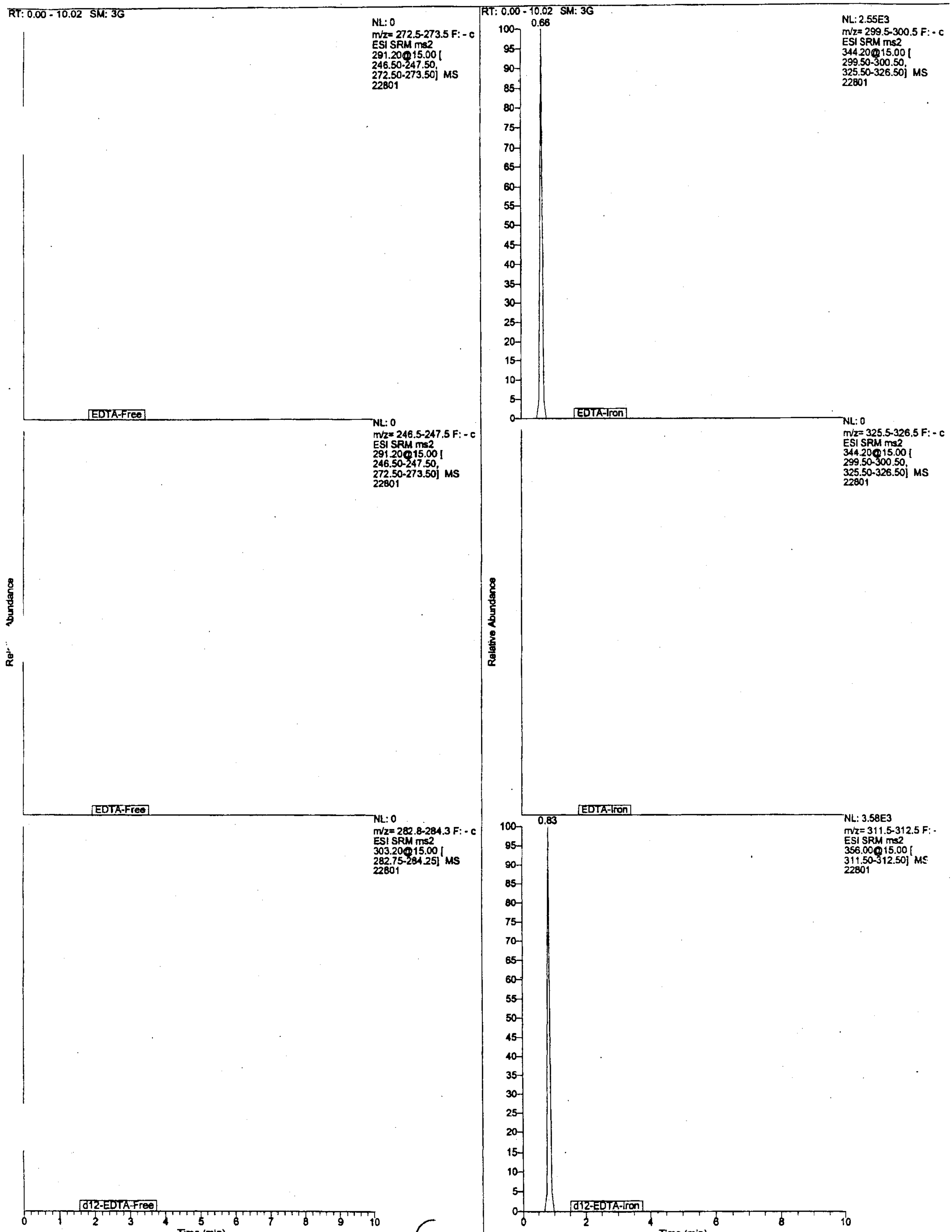
Ex 446
(2c)

C:\Xcalibur\...\Stability\Stability41 JDB 02/28/07 11:50:40 BLANK (neg blood, DI H2O ext.)



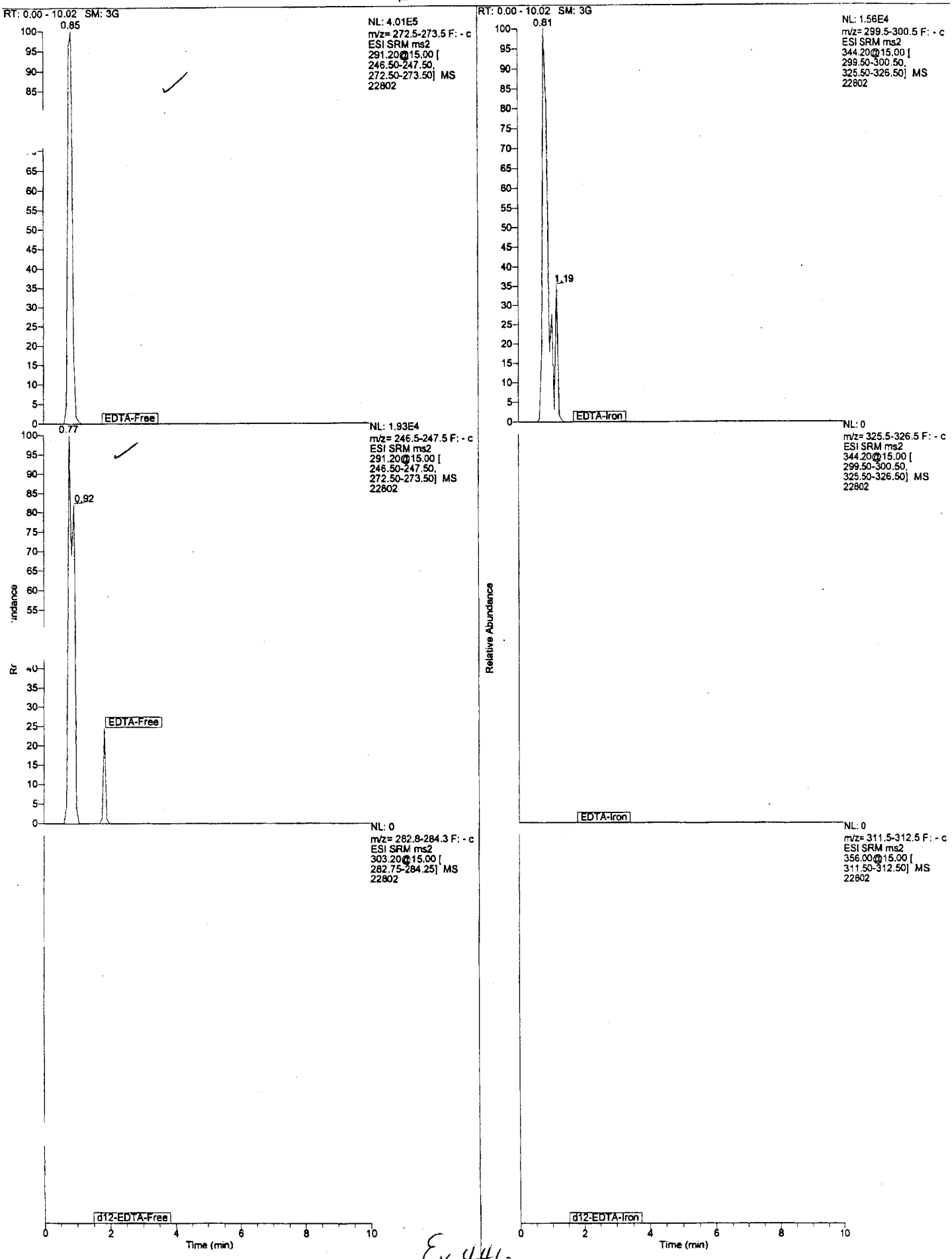
Ex 446
(30)

C:\Xcalibur\...\EDTA\Brewer\022807\22801 02/28/07 03:31:28 EDTA neg blood/H2O extract JOP



Ex 446

C:\Xcalibur\...\EDTA\Brewer\022807\22802 02/28/07 03:42:41 100 ppm EDTA JDP



Ex 446
(41)

Sequence---EDTA_stability_negion.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY01	1	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY02	02	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Ex 446
(42)
page 1

02/28/07
JDB

Sequence---EDTA_stability_negion.sld [Open]

Sample Name: Blank card extract
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY03	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		2	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY04	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Ex 446
(43)

02/28/07
JDB

Sequence---EDTA_stability_negion.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY05	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Sample Name: EDTA neg blood-blank card ext.
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY06	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		3	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Ex 446
(44)

02/28/07
JDP

Sequence---EDTA_stability_negion.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY07	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY08	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Ex 446
(45)

02/28/07
JDB

Sequence---EDTA_stability_negion.sld [Open]

Sample Name: Card A extract (EDTA +)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY09	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		4	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY10	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Ex 446
(46)

02/28/07
JDB

Sequence---EDTA_stability_negion.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY11	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Sample Name: Card B extract (EDTA +)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY12	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		5	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Ex 446
(47)

02/28/07
JDB

Sequence---EDTA_stability_negion.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY13	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY14	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Ex 446
(48)

02/28/07
JOB

Sequence---EDTA_stability_negion.sld [Open]

Sample Name: Card C extract (EDTA +)
Comment:
Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY15	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		6	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:
Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY16	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Ex 446
(49)

02/28/07
JDB

Sequence---EDTA_stability_negion.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY17	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Sample Name: Card D extract (EDTA +)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY18	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		7	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Ex 446
(50)

02/28/07
JDP

Sequence---EDTA_stability_negion.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY19	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	STABILITY20	01	C:\Xcalibur\Data\EDTA\Brewer\Stability	C:\Xcalibur\methods\EDTA_Neg_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	1.000

Dil Factor
1.000

****

Ex 446
(51)

02/28/07
JDB

EDTA Stability Study:

Ten EDTA-preserved blood spot cards were analyzed following approximately 33 months of storage at room temperature. The free acid form of EDTA was detected in all 10 of the spot cards. The EDTA-iron complex was detectable in 6 of the 10 spot cards. Failure to identify the EDTA-iron complex was based on the lack of the less abundant product ion (*m/z* 326). The more abundant *m/z* 300 for EDTA-iron was present in all 10 of the spot cards.

CASE FILE

Ex 446
(53)

FBI Laboratory

2501 Investigation Parkway
Quantico, Virginia 22135

REPORT OF EXAMINATION

To: Milwaukee
Squad 6/GBRA
SA Gerald E. Mullen

Date: February 26, 2007

Case ID No.: 62D-MW-44363-61

Lab No.: 070201013 PM GH

Reference: Communication dated January 30, 2007

Your No.:

Title: STEVEN AVERY;
TERESA HALBACH-VICTIM (DECEASED)
DOMESTIC COOPERATION-HOMICIDE

Date specimens received: February 1, 2007 and February 6, 2007

The following items were examined in the Chemistry Unit:

Q46 Swab (Item 9569)

Q47 Swab (Item 9574)

Q48 Swab (Item 9572)

Q49 Liquid blood sample from STEVEN AVERY (Item 9803)

K2 Two control swabs (Item 9802)

K3 Two control swabs (Item 9801)

K4 Two control swabs (Item 9800)

This report contains the results of the chemistry examinations.

Ex 446
(54)

Results of Examinations:

Specimens Q46-Q49 and K2-K4 were analyzed for the presence of ethylenediamine-tetraacetic acid (EDTA).

Specimen Q49 was listed as a liquid blood sample from STEVEN AVERY in a 10 milliliter (mL) lavender-top blood tube. It contained approximately 5.5 mL of blood. EDTA was identified in specimen Q49.

Specimens Q46-Q48 were reported to be collection swabs of blood stains from the crime scene associated with the death of TERESA HALBACH. Specimens K2-K4 were reported to be control swabs collected in relation to the Q46-Q48 swabs. EDTA, either as the free acid or as the EDTA-iron complex, was not identified on the Q46-Q48 or K2-K4 swabs.

The analysis for EDTA was carried out using liquid chromatography/tandem mass spectrometry in both positive and negative electrospray ionization modes.

Remarks:

EDTA is an anti-coagulant and enzyme inhibitor that is commonly used in blood collection tubes. Blood specimen collection tubes containing EDTA have lavender-colored tops and are the most common collection tube used to collect reference specimens for DNA testing.

The concentration of EDTA in its free acid form in a drawn blood tube is typically 1000-2000 milligrams per liter (mg/L), depending on the volume of blood and the capacity of the tube. At this concentration, the free acid and salt forms of EDTA are soluble in blood. EDTA readily forms water-soluble chelates with nearly all heavy metals, including iron in blood. Aqueous extractions of dried bloodstains allow for the isolation of EDTA (both as the free acid and as the EDTA-iron complex) if present.

Using the procedure employed in this case, EDTA is readily identified at a concentration of 13 mg/L. Additionally, EDTA is also detectable when a 1-microliter drop of EDTA-preserved blood is analyzed.

For questions about the content of this report, please contact Unit Chief Marc LeBeau at (703) 632-7408.

070201013 PM GH

Ex 446
(55)

For questions about the status of remaining forensic examinations, if applicable, please contact Request Coordinator Michael VanArsdale at (703) 632-8809.

The submitted evidence was returned under separate cover of communication.

Marc A. LeBeau, PhD
Chemistry Unit
(703) 632-7408

Technically reviewed and any identifications and associations confirmed by:

Madeline Mnt Date: 2/26/07

Administrative Review: Date: 2/26/07

This report contains the opinions/interpretations of the examiner(s) who issued the report.

070201013 PM GH

For Official Use Only

Ex 446
(56)

7-251 (7-13-98)

LABORATORY DIVISION
SUPPORTING DOCUMENTATION ENVELOPE

Case ID/ Sub 62D-MW-44363

1A Serial # ____________

1 of 1

Please Print Clearly

The Enclosed material for LeBeau (Name) supports Laboratory Report ____________ (Case ID, Sub and Serial)

Lab Number(s) Or W/O Number 070201013

Description of Enclosures

Admin Notes - ~~10~~ 14 Pages
Check In Notes - 12 PAGES
Analysis Notes - 3 PAGES
Photo of Spot Sizes - 1 PAGE
(+) ESI Data - 61 PAGES
(-) ESI Data - 65 PAGES

____ Chain of Custody
____ Communication and Activity Log
____ Notes
____ Instrument Print Outs, Charts, Graphs
____ Negatives
____ Photographs
____ ECS Search Slip
____ Shipping Invoice

Ex 446

(57)

Please Return This Envelope And All Contents To The Laboratory Division File Room

7-1 (Rev. 10-06)

FBI Laboratory

2501 Investigation Parkway
Quantico, Virginia 22135

REPORT OF EXAMINATION

To: Milwaukee
Squad 6/GBRA
SA Gerald E. Mullen

Date: February 26, 2007

Case ID No.: 62D-MW-44363-61

Lab No.: 070201013 PM GH

Reference: Communication dated January 30, 2007

Your No.:

Title: STEVEN AVERY;
TERESA HALBACH-VICTIM (DECEASED)
DOMESTIC COOPERATION-HOMICIDE

Date specimens received: February 1, 2007 and February 6, 2007

The following items were examined in the Chemistry Unit:

Q46 Swab (Item 9569)

Q47 Swab (Item 9574)

Q48 Swab (Item 9572)

Q49 Liquid blood sample from STEVEN AVERY (Item 9803)

K2 Two control swabs (Item 9802)

K3 Two control swabs (Item 9801)

K4 Two control swabs (Item 9800)

This report contains the results of the chemistry examinations.

Ex 446
(58)

Results of Examinations:

Specimens Q46-Q49 and K2-K4 were analyzed for the presence of ethylenediamine-tetraacetic acid (EDTA).

Specimen Q49 was listed as a liquid blood sample from STEVEN AVERY in a 10 milliliter (mL) lavender-top blood tube. It contained approximately 5.5 mL of blood. EDTA was identified in specimen Q49.

Specimens Q46-Q48 were reported to be collection swabs of blood stains from the crime scene associated with the death of TERESA HALBACH. Specimens K2-K4 were reported to be control swabs collected in relation to the Q46-Q48 swabs. EDTA, either as the free acid or as the EDTA-iron complex, was not identified on the Q46-Q48 or K2-K4 swabs.

The analysis for EDTA was carried out using liquid chromatography/tandem mass spectrometry in both positive and negative electrospray ionization modes.

Remarks:

EDTA is an anti-coagulant and enzyme inhibitor that is commonly used in blood collection tubes. Blood specimen collection tubes containing EDTA have lavender-colored tops and are the most common collection tube used to collect reference specimens for DNA testing.

The concentration of EDTA in its free acid form in a drawn blood tube is typically 1000-2000 milligrams per liter (mg/L), depending on the volume of blood and the capacity of the tube. At this concentration, the free acid and salt forms of EDTA are soluble in blood. EDTA readily forms water-soluble chelates with nearly all heavy metals, including iron in blood. Aqueous extractions of dried bloodstains allow for the isolation of EDTA (both as the free acid and as the EDTA-iron complex) if present.

Using the procedure employed in this case, EDTA is readily identified at a concentration of 13 mg/L. Additionally, EDTA is also detectable when a 1-microliter drop of EDTA-preserved blood is analyzed.

For questions about the content of this report, please contact Unit Chief Marc LeBeau at (703) 632-7408.

070201013 PM GH

Ex 446
(59)

For questions about the status of remaining forensic examinations, if applicable, please contact Request Coordinator Michael VanArsdale at (703) 632-8809.

The submitted evidence was returned under separate cover of communication.

Marc A. LeBeau, PhD
Chemistry Unit
(703) 632-7408

Technically reviewed and any identifications and associations confirmed by:

Madeline Mnt Date: 2/26/07

Administrative Review: Date: 2/26/07

This report contains the opinions/interpretations of the examiner(s) who issued the report.

070201013 PM GH

For Official Use Only

Ex 446
(60)

7-251 (7-13-98)

LABORATORY DIVISION
SUPPORTING DOCUMENTATION ENVELOPE

Case ID/ Sub 620-MW-44363

1A Serial # _____

1 of 1

Please Print Clearly

The Enclosed material for LeBeau supports Laboratory Report _____
Name — Case ID, Sub and Serial

Lab Number(s) Or W/O Number 0702010l3

Description of Enclosures

Admin Notes - 14 Pages	_____	Chain of Custody
Check In Notes - 12 PAGES	_____	Communication and Activity Log
Analysis Notes - 3 PAGES	_____	Notes
Photo of Spot Sizes - 1 PAGE	_____	Instrument Print Outs, Charts, Graphs
(+) ESI Data - 61 PAGES	_____	Negatives
(-) ESI Data - 65 PAGES	_____	Photographs
_____	_____	ECS Search Slip
_____	_____	Shipping Invoice

Ex 446
(61)

Please Return This Envelope And All Contents To The

(Rev. 01-31-2003)

FEDERAL BUREAU OF INVESTIGATION

Precedence: DEADLINE 03/09/2007 **Date:** 01/30/2007

To: Laboratory **Attn:** Scientific Analysis Section
Chemistry Unit
Mark Lebeau

From: Milwaukee
Squad 6/GBRA
Contact: SA Gerald E. Mullen, (920) 432-3868

Approved By: Greco Raymond A

Drafted By: Mullen Gerald E:dlg

070201013

Case ID #: 62D-MW-44363 (Pending)-56

Title: STEVEN AVERY;
TERESA HALBACH-VICTIM (DECEASED)
DOMESTIC COOPERATION-HOMICIDE

Synopsis: Summary of captioned matter with request for appropriate examination.

Package Copy: Being forwarded under separate cover are the following items:

1) one box containing vial human blood - To Be sent at later Date -

Barcode
2) envelopes containing swabs from crime scene
BB-E04033640

Details: Teresa Halbach was reported missing by family members on 11/03/2005. She was last seen on 10/31/2005 on the property of Steven Avery who owns a salvage yard with his family. Halbach was taking photographs of a vehicle that was for sale as she worked for the Auto Trader magazine. Numerous search warrants were executed on the Avery property and significant evidence was recovered implicating Steven Avery in the murder of Teresa Halbach. The Calumet County Sheriff, Jerry Pagel, has requested the assistance of the FBI for laboratory examinations.

Steven Avery was released from prison in 2003 after being exonerated for a rape conviction by DNA evidence. A vial of Avery's blood, which was drawn and used to exonerate him in the rape conviction, was maintained at the Manitowoc County Clerk of Courts Office. Avery's defense has alleged the investigators from the Manitowoc County Sheriff's Department used this vial of blood to plant evidence at the crime scene implicating Avery in murder of Teresa Halbach. This vial of blood contains EDTA.

RETURN EVIDENCE
TO MILWAUKEE DIVISION

Ex 446

To: Laboratory From: Milwaukee
Re: 62D-MW-44363, 01/30/2007

The purpose of this request is to establish the presence of EDTA, in the vial of blood, thereby eliminating the allegation that this vial was used to plant evidence.

Avery's trial is scheduled to began on 02/05/2007. Special Prosecutor Calumet County District Attorney Ken Kratz, has requested this examination be completed by 03/09/2007 to be used as rebuttal evidence.

Ex 446
(63)

2/24/17

To: Laboratory From: Milwaukee
Re: 62D-MW-44363, 01/30/2007

LEAD(s):

Set Lead 1: (Action)

LABORATORY

AT QUANTICO, VA

Chemstry Unit conduct appropriate examination of vial of blood to determine the presence of EDTA. Conduct relevant comparisons to swabs from crime scene. Conduct degradation analysis of blood. It is requested this examination be completed by 03/09/2007. The point of contact from the Wisconsin Department of Justice is Assistant Attorney General Norm Gahn at (920) 418-4087.

◆◆

RETURN EVIDENCE
TO MILWAUKEE DIVISION

Ex 446
(64)

2/27/07

070201013

7-243 (Rev. 3-31-04)

FBI Laboratory

Chain-of-Custody Log

COPY

Laboratory No.: 070201013 RF PM **Case ID No.:** 62D-MW-44363

Container(s)	Received Via	Accepted By	Date	Contributor
1 BOX	Fed Ex (Signature) / Agency / Unit	(Signature) ECU Unit	2/1/07	FBI Milwaukee

Tracking No(s).: 7190 4673 4441

Opened for Retrieval of Communication By: (Signature) **Date:** 2/1/07

☐ **Shipping Container Damage**

ECC Comments:

Container(s)	Delivered By	Accepted By	Date	Remarks
1 BOX	(Signature) ECU Unit	**Into ECU Storage** ☑ Refrigerator ☐ Safe ☐ Shelf	2/1/07	
1 BOX	**From ECU Storage** Unit	(Signature) ECU Unit	2/1/07	Q46-Q48, NE2-NE4
Q46-Q48, NE2-NE4	(Signature) ECU Unit	ECU Storage (Signature) Unit	2/1/07	~~For Inventory~~ DMM
Items Received:	Q46-Q48 ~~NOT NE4~~, Q49 K2K4			
Q46-Q48 NE2-NE4	ECU Storage (Signature) Unit	(Signature) ECU Unit	2/2/07	
Q46-Q48 NE2-NE4	(Signature) ECU Unit	TTMeagy (Signature) CU Unit	02-02 2007	
	(Signature) Unit	(Signature) Unit		Chain combined

Ex 446 (65)

COPY

Item(s)	Delivered By	Accepted By	Date	Remarks
	Signature / Unit	Signature / Unit		
	Signature / Unit	Signature / Unit		
	Signature / Unit	Signature / Unit		
	Signature / Unit	Signature / Unit		
	Signature / Unit	Signature / Unit		
	Signature / Unit	Signature / Unit		
	Signature / Unit	Signature / Unit		
	Signature / Unit	Signature / Unit		
	Signature / Unit	Signature / Unit		
	Signature / Unit	Signature / Unit		
	Signature / Unit	Signature / Unit		
	Signature / Unit	Signature / Unit		

Chain Continued

070201013

Ex 446
(66)

Laboratory Number:

(Rev. 3-31-04)

FBI Laboratory

Chain-of-Custody Log

COPY

Laboratory No.: 070901013 Case ID No.: 102D-MW-443603

Container(s)	Received Via	Accepted By	Date	Contributor
1 Box	FEDEX Signature Agency / Unit	Jamie Lewis Signature ECU Unit	2/6/2007	Milwaukee, WI. FBI

Tracking No(s).: 7190 4073 5091

Opened for Retrieval of Communication By: Jamie Lewis Date: 2/6/2007

☐ Shipping Container Damage

ECC Comments: Additional evidence. @ ECU 2/6/2007.

Container(s)	Delivered By	Accepted By	Date	Remarks
1 Box	Jamie Lewis Signature ECU Unit	Into ECU Storage ☑ Refrigerator ☐ Safe ☐ Shelf	2/6/2007	
1 box	From ECU Storage Unit	[signature] Signature Unit	2/7/07	Q49
Q49	[signature] Signature Unit	ECU Storage Signature Unit	2/7/07	For Inventory
Items Received:				
Q49.	ECU Storag Signature Unit	[signature] Signature Unit	2/7/07	
Q49.	[signature] Signature Unit	[signature] Signature CU Unit	02-07 2007	
Q46-Q49, K2-K4	[signature] Signature CU Unit	[signature] Signature ECU Unit	2/16/07	NE2-NE4 changed to K2-K4 by CU

Ex 446
(67)

COPY

Item(s)	Delivered By	Accepted By	Date	Remarks
Q46-Q49, K2-K4	Erin P. MM Signature ECU Unit	TOECU STORAGE Signature Unit	2/16/07	
Q46-Q49 K2-K4	Signature Unit	Signature Unit	02/16/07	1 Box
1 Box	Signature Unit	Signature Unit	02/20/07	
1 box	From ECU Storage Signature Unit	Charles Peters Signature ECU Unit	2/20/07	
1 box	Charles Peters Signature ECU Unit	Joe Blado Signature ASU Unit	2/20/07	
1 box	Joe Blado Signature ASU Unit	"Returned via Fed Ex" Signature Unit	2/20/07	
	Signature Unit	Signature Unit		
	Signature Unit	Signature Unit		
	Signature Unit	Signature Unit		
	Signature Unit	Signature Unit		
	Signature Unit	Signature Unit		

Ex 446
(68)

Laboratory Number: 070201013

7-243a (Rev. 3-31-04)

FBI Laboratory
Chain-of-Custody Log

Laboratory No.: 07020101013 Case ID No.: 62D-MW-44363

☐ Request Coordinator's Log
☑ Intraunit Log
☐ Other

Unit Name: _____
Unit Name: CU
Unit Name: _____

Item(s)	Delivered By	Accepted By	Date	Remarks
Q46-Q48; NE2-NE4.	Peggy Signature CU Unit	EVID. TEMP. STORAGE — rm 4261 Signature CU Unit	02-02 2007	
Q46-Q48; NE2-NE4	EVID. TEMP. STORAGE - RM 4261 Signature CU Unit	[signature] Signature CU Unit	02/05/07	CHECK-IN; CHANGED NE2-NE4 TO K2-K4
Q46-Q48; K2-K4	[signature] Signature CU Unit	EVIDENCE LOCKER Signature CU Unit	02/05/07	
Q49.	Peggy Signature CU Unit	EVID. TEMP. STORAGE — rm 4261 Signature CU Unit	02-07 2007	REFRIG/BioHaz
Q49	EVID TEMP STORAGE REFRIGERATOR RM 4261 Signature CU Unit	[signature] Signature CU Unit	02/08/07	CHECK-IN
Q49	[signature] Signature CU Unit	EVID. TEMP. STORAGE REFRIGERATOR Signature CU Unit	02/08/07	
Q46-~~Q47~~ JB Q48; K2-K4	EVIDENCE LOCKER Signature CU Unit	[signature] Signature CU Unit	02/16/07	RETURNED TO ECU
Q49	EVID. TEMP. STORAGE REFRIGERATOR Signature CU Unit	[signature] Signature CU Unit	02/16/07	RETURNED TO ECU

Ex 446 (69)

2/26/07

7-245 (Rev. 6-19-00)

FBI Laboratory
Activity and Communication Log

Laboratory No.: 070201013 Case ID No.: 62D-MW-44363

Date	Activity/Communication and Outcome
2/1/07 By: CAL	Tel call to SA Gerald Mullen. Left a mess for him to call me back about the swabs and the type of exams needed.
02/01/07 By: m	Voicemail left for SA Mullen informing that blood sample was not submitted – Advised swabs would be sent to [illegible] for exam
02/01/07 By: m	Voicemail from SA Mullen – Judge will not release blood to come to lab yet – hearing on 02/22/07 to discuss – he will advise of results of hearing
2/12/07 By: MM	Spoke w/ Norm Gahn concerning status of exams. Told him method validation was nearly finished and hope to analyze samples this week or next week.
2/16/07 By: MM	Spoke w/ Norm Gahn. Told him preliminary finding on Q46 is negative for EDTA. More tests to run. Results must be reviewed. Report should be ready the week of Feb 26th.
__/__/__ By: ____	
__/__/__ By: ____	

Ex 446
(70)

MM 2/28/07

ECS Search Slip

Violation(s):
estic Police Cooperation

Violation date:
Violation Location:

Victim:
HALBACH, TERESA

Cross Reference:

Remarks:

Lab No: 070201013 PM GH
City: Milwaukee, Wisconsin
Agency: FBI
Form: Electronic Communication, 01/30/2007
CaseID: 62D-MW-44363 **Serial:** 56
Latent Number:
Contributor No: 62D-MW-44363

Subject:
AVERY, STEVEN

ECC Delivered to Unit:_____

Previous Submission(s)

061227012 PM PV Milwaukee, Wisconsin FBI Electronic Comm.:12/19/2006 Qs:15-45 Ks: NE Items:	
114006 PM PV NN waukee, Wisconsin ectronic Comm.:11/07/2006 Qs:13-14 Ks: NE Items:	
061108009 PM PV Milwaukee, Wisconsin FBI Electronic Comm.:11/02/2006 Qs:11-12 Ks: NE Items:	
060118254 QG Milwaukee, Wisconsin FBI Electronic Comm.:01/04/2006 Final:04/11/2006 Qs:3-10 Ks: NE Items:1	
051123009 PM MR Milwaukee, Wisconsin FBI Electronic Comm.:11/16/2005 Qs:1-2 Ks:1 NE Items:	

el Method and No: Federal Express - 7190 4673 4441
xes - 1 Date Received: 02/01/2007 -More
Received in ECC: 02/01/2007
02/07/2007 12:03:28 PM - mvanarsdale

Ex 446
(71)

2/26/07

ECU - 1 (Rev. 7/27/2005)

Evidence Control Unit
Evidence Check-In Notes

Laboratory Number: 070201013 PM Case ID: 62D-MW-44363

Received via FedEx one sealed box, tracking number 7190 4673 4441, containing:
one sealed box containing:
six sealed paper bags:
bag labeled "9569" contained:
one sealed envelope containing:
Q46 Swab (Item 9569)
bag labeled "9574" contained:
one sealed envelope containing:
Q47 Swab (Item 9574)
bag labeled "9572" contained:
one sealed envelope containing:
Q48 Swab (Item 9572)
bag labeled "9801" contained:
one unsealed envelope containing: *envelope sealed in ECU*
one unsealed box containing: *box sealed in ECU*
NE2 Two control swabs (Item 9801)
bag labeled "9802" contained:
one unsealed envelope containing: *envelope sealed in ECU*
one unsealed box containing: *box sealed in ECU*
NE3 Two control swabs (Item 9802)
bag labeled "9800" contained:
one unsealed envelope containing: *envelope sealed in ECU*
one unsealed box containing: *box sealed in ECU*
NE4 Two control swabs (Item 9800)

Initials of Preparer: DAH
Date: 2/1/07
Page 1 of 12

Ex 446
(72)

ECU - 1 (Rev. 7/27/2005)

Evidence Control Unit
Evidence Check-In Notes

Laboratory Number: 070201013 PM

Case ID: 62D-MW-44363

* indicates unopened in ECU to preserve examinations, descriptions taken from packaging/EC

Received one sealed FedEx box TRK# 7190 4673 5091 (discarded in ECU) containing one sealed box containing one sealed container containing one closed vial containing one purple top tube of liquid blood:

Q49 Liquid blood sample from STEVEN AVERY (Item 9803)

Initials of Preparer: JE
Date: 2/7/07
Page 1 of 1

Ex 446
(73)

2nd 2nd

2/7/07

7-2 (Rev. 7-10-06)

Federal Bureau of Investigation
Laboratory Division

LABORATORY WORK SHEET

To: Milwaukee
Squad 6/GBRA
SA Gerald E. Mullen

Date: February 1, 2007

Case ID No.: 62D-MW-44363

Lab No.: 070201013 PM

Reference: Communication dated January 30, 2007

Your No.:

Title: STEVEN AVERY;
TERESA HALBACH-VICTIM (DECEASED)
DOMESTIC COOPERATION-HOMICIDE

Date specimens received: February 1, 2007

Specimens:

Q46 Swab (Item 9569)

Q47 Swab (Item 9574)

Q48 Swab (Item 9572)

K3 ~~NE2~~ JP Two control swabs (Item 9801)

K2 ~~NE3~~ JP Two control swabs (Item 9802)

K4 ~~NE4~~ JP Two control swabs (Item 9800)

Ex 446
(74)

2/26/07

2 (Rev. 04-10-06)

FBI Laboratory
Examination Plan

boratory Number: 070201013

Case ID Number: 62D-MW-44363

quest Coordinator: Van Arsdale

Contributor: M. Wacker

tal Items Received: Q46-Q48, Q49 N02-N04

Evidence Storage Area:

Items	Unit	Examiner/Symbols	Delivered	Remarks	Returned
Q46-Q48, Q49 N02-N04	CV	Lobon			☐ Evidence ☐ Secondary evidence ☐ File Copy ☐ IA
					☐ Evidence ☐ Secondary evidence ☐ File Copy ☐ IA
					☐ Evidence ☐ Secondary evidence ☐ File Copy ☐ IA
					☐ Evidence ☐ Secondary evidence ☐ File Copy ☐ IA
					☐ Evidence ☐ Secondary evidence ☐ File Copy ☐ IA
					☐ Evidence ☐ Secondary evidence ☐ File Copy ☐ IA

ECU Notations:

Date: 02/01/07
02/01/07 mw

Initials: mw

Page: 1 of 1

Chemistry Unit
Evidence Check-In Sheet

Check-In Date: 02/05/2007 Case ID #: 623-MW-44363 Examiner: LE BEAU
CHEMIST: BREWER

Mode Received:
☐ USPS ☐ FedEx ☑ Delivered By: ECU
Tracking #: _____

Condition of External Container(s):
☑ Proper Seal(s)/Label(s) ☐ Other: (Comment Below)

Evidence Initially Received In:
☑ Boxes (# /) ☐ Paper Bags (#)
☐ Plastic Bags (#) ☐ Envelopes (#)
☐ Cans (#)
☐ Other: _____

GENERAL DESCRIPTION OF EVIDENCE AND CONTAINERS:

ONE BOX SEALED W/ YELLOW TAPE W/ INITIALS, MARKED "070201013, Q46-Q48, NE2-NE4."

INSIDE IS ONE BOX SEALED W/ YELLOW TAPE W/ INITIALS, MARKED 070201013, Q46-Q48, NE2-NE4."

INSIDE ARE SIX PAPER BAGS. EACH BAG IS SEALED W/ RED EVIDENCE TAPE W/ INITIALS, AND EACH BAG CONTAINS AN ORANGE "CALUMET COUNTY SHERIFF'S DEPARTMENT" EVIDENCE LABEL. IN ADDITION, EACH BAG IS MARKED "070201013."

BAG #1 IS ALSO MARKED "Q46" AND LABELED "LEDGER 06-119, 05-0157-955, DATE: 05/25/06, EVIDENCE TAG # 9569, RECEIVED FROM: STATE CRIME LAB MADISON, LOCATION: DASHBOARD NEAR IGNITION SWITCH, OFFICER: BALDWIN 1819, DESCRIPTION/INFORMATION: SWABS OF STAIN."

INSIDE BAG #1 IS A SMALL MANILLA ENVELOPE SEALED W/ RED EVIDENCE TAPE W/ INITIALS AND W/ CLEAR TAPE W/ INITIALS, MARKED "070201013, Q46, MO?-2467, A(8), 11-7-05." LABELED "MADISON LAB, MO5-2467, #A8, 05-0157-955"

Lab #: 070201013 Initials: JDB
Date: 02/05/2007 Page 1 of 5

Ex 446
(76)

GENERAL DESCRIPTION OF EVIDENCE AND CONTAINERS (continued):

INSIDE THE ENVELOPE IS ONE COTTON SWAB W/ A DARK BROWNISH STAIN.

BAG #2 IS ALSO MARKED "Q47" AND LABELED "LEDGER # 06-119, 05-0157-955, DATE: 05/25/06, EVIDENCE TAG # 9574, RECEIVED FROM: STATE CRIME LAB MADISON, LOCATION: METAL ? DOOR ENTRANCE AREA (PASS SIDE), OFFICER: BALDWIN 1819, DESCRIPTION/INFORMATION: SWAB OF STAIN."

INSIDE BAG # 2 IS A SMALL MANILLA ENVELOPE SEALED W/ RED EVIDENCE TAPE W/ INITIALS, MARKED "07020103, Q47, 11-7-05, 4-3-06, 5/25/06." LABELED "MADISON LAB, M05-2467, #A12, 05-0157-955."

INSIDE THE ENVELOPE IS ONE COTTON SWAB W/ A DARK BROWNISH STAIN.

BAG #3 IS ALSO MARKED "Q48" AND LABELED "LEDGER # 06-119, 05-0157-955, DATE: 05/25/06, EVIDENCE TAG # 9572, RECEIVED FROM: ATE CRIME LAB MADISON, LOCATION: BLK CD CASE ON FRONT PASSENGER SEAT, OFFICER: BALDWIN 1819, DESCRIPTION/INFORMATION: SWAB OF STAIN."

INSIDE BAG # 3 IS A SMALL MANILLA ENVELOPE SEALED W/ RED EVIDENCE TAPE W/ INITIALS, MARKED "07020103, Q48, A(10), 11-7-05, 5/25/06, 4-3-06." LABELED "MADISON LAB, M05-2467, #A10, 05-0157-955."

INSIDE THE ENVELOPE IS ONE COTTON SWAB W/ A SMALL LIGHT BROWNISH STAIN.

BAG #4 IS ALSO MARKED "NE2" AND LABELED "05-0157-955, DATE: 01/31/07, EVIDENCE TAG # 9801, RECEIVED FROM: RAV 4, LOCATION: METAL REAR DOOR AREA PASSENGER SIDE, OFFICER: HAWKINS 1826, DESCRIPTION/INFORMATION: CONTROL SWAB."

INSIDE BAG #4 IS A MANILLA ENVELOPE SEALED W/ RED EVIDENCE TAPE W/ INITIALS, MARKED "07020103, NE2, 05-0157-955,

Ex 446 (77)

GENERAL DESCRIPTION OF EVIDENCE AND CONTAINERS (continued):

ITEM # 9801, X CONTROL SWAB, 2 # OF SWABS, BY: HAWKINS 1826, DATE: 01/31/07, TIME: 0943 am, LOCATION: BY LABEL ON METAL PIECE."

INSIDE THE ENVELOPE IS A WHITE BOX SEALED W/ RED EVIDENCE TAPE W/ INITIALS, MARKED "070201013, NE2, CONTROL, 01/31/07, TAG # 9801."

INSIDE ARE TWO COTTON SWABS. ONE OF THE SWABS APPEARS TO HAVE A SMALL AMT. OF DIRT/STAIN PRESENT, THE OTHER SWAB APPEARS CLEAN.

BAG #5 IS SIMILAR TO BAG #4 W/ THE FOLLOWING EXCEPTIONS, MARKED "NE3", LABELED "EVIDENCE TAG # 9802, LOCATION: BY IGNITION SWITCH DASHBOARD."

INSIDE BAG #5 IS A MANILLA ENVELOPE SEALED W/ RED EVIDENCE TAPE W/ INITIALS. IT IS MARKED SIMILARLY TO THE ENVELOPE IN BAG #4 W/ THE FOLLOWING EXCEPTIONS, "NE3, ITEM # 9802, TIME: 0938 am, LOCATION: BY IGNITION SWITCH DASHBOARD."

INSIDE THE ENVELOPE IS A WHITE BOX SEALED W/ RED EVIDENCE TAPE W/ INITIALS, MARKED "070201013, NE3, CONTROL, 01/31/07, TAG # 9802."

INSIDE ARE TWO COTTON SWABS.

BAG #6 IS SIMILAR TO BAGS #4 & #5 W/ THE FOLLOWING EXCEPTIONS, MARKED "NE4", LABELED "EVIDENCE TAG # 9800, RECEIVED FROM: BLACK CD CASE, LOCATION: NEXT TO LABEL."

INSIDE BAG #6 IS A MANILLA ENVELOPE SEALED W/ RED EVIDENCE TAPE W/ INITIALS. IT IS MARKED SIMILARLY TO THE ENVELOPES IN BAGS #4 & #5 W/ THE FOLLOWING EXCEPTIONS, "NE4, ITEM # 9800, TIME: 0840 am, LOCATION: NEXT TO LABEL ON BOTTOM."

INSIDE THE ENVELOPE IS A WHITE BOX SEALED W/ RED EVIDENCE TAPE W/ INITIALS, MARKED "070201013, NE4, TAG # 9800, CD CASE."

Ev 446 (78)

GENERAL DESCRIPTION OF EVIDENCE AND CONTAINERS (continued):

INSIDE ARE TWO COTTON SWABS W GRAYISH STAINS.

JDB

Ex 446
(79)

Lab #: 07020103	Initials: JDB
Date: 02/05/2007	Page 4 of 5

Chemistry Unit
Evidence Check-In Sheet

Specimen #	Contributor #	Description:
Q46	ITEM 9569	ONE COTTON SWAB W/ DARK BROWNISH STAIN, FROM DASHBOARD NEAR IGNITION SWITCH
Q47	ITEM 9574	ONE COTTON SWAB W/ DARK BROWNISH STAIN, FROM METAL DOOR ENTRANCE AREA (PASS. SIDE)
Q48	ITEM 9572	ONE COTTON SWAB W/ LIGHT BROWNISH STAIN, FROM BLK CD CASE ON FRONT PASSENGER SEAT
K3 ~~NE2~~ JB	ITEM 9801	TWO COTTON CONTROL SWABS. FROM "METAL DOOR AREA PASSENGER SIDE"
K2 ~~NE3~~ JB	ITEM 9802	TWO COTTON CONTROL SWABS. FROM "BY IGNITION SWITCH DASHBOARD".
K4 ~~NE4~~ JB	ITEM 9800	TWO COTTON CONTROL SWABS W/ GRAYISH STAINS. FROM "NEXT TO LABEL ON BOTTOM OF CD CASE"

Ex 446 (50) MM

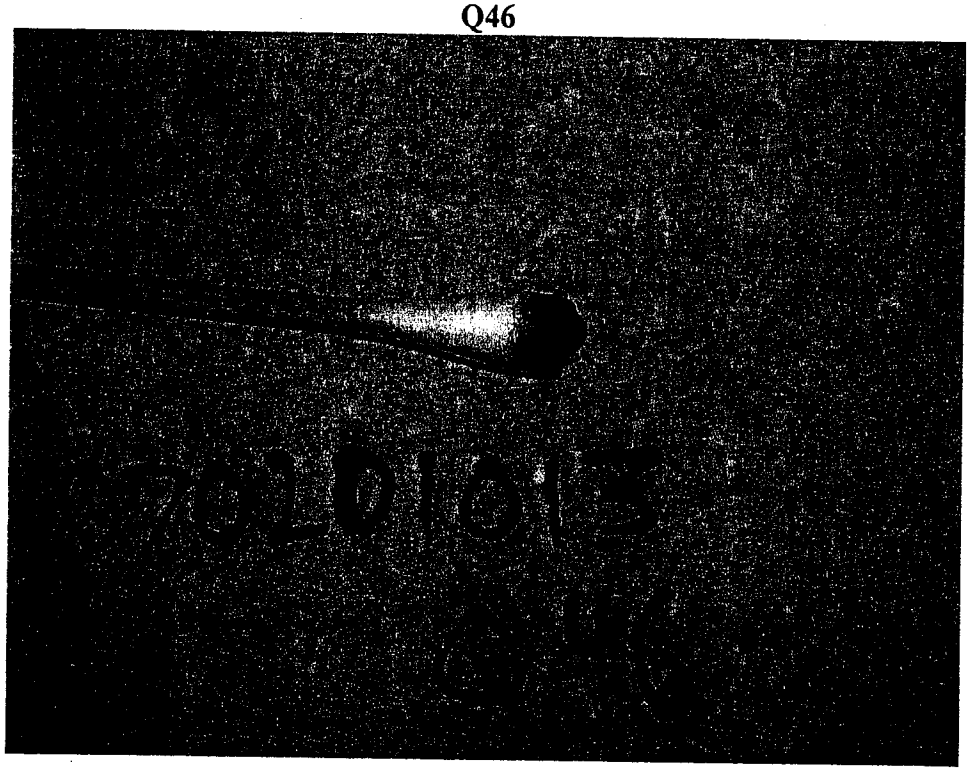
Lab #: 07020103 Initials: JDB

Date: 02/05/2007 Page 5 of 5

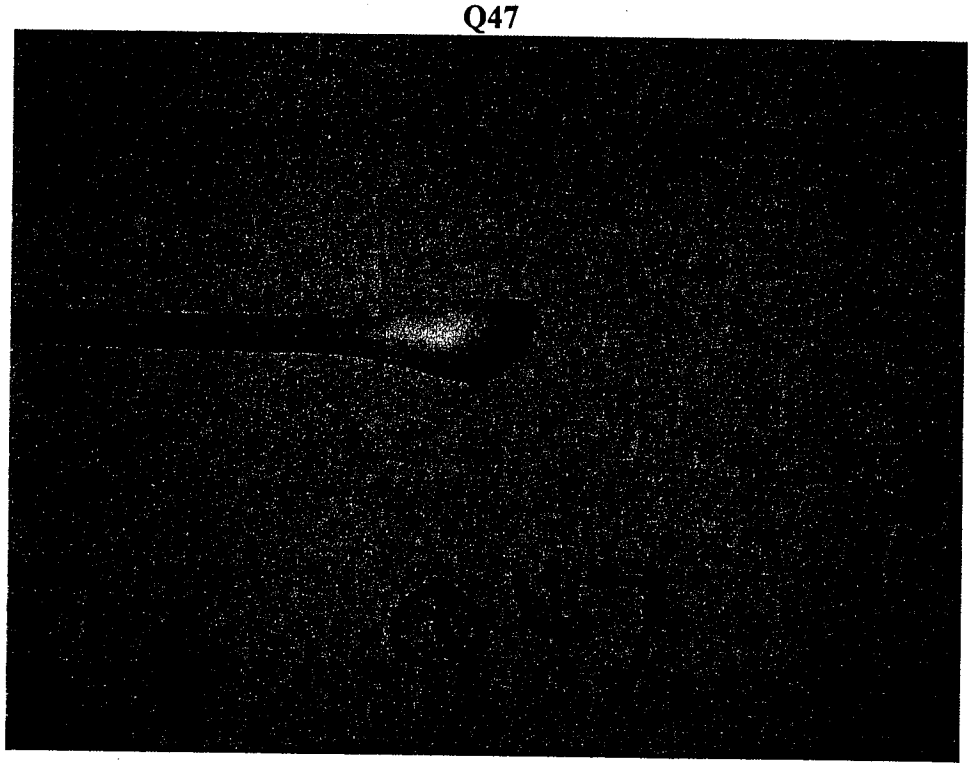
070201013
02/05/2007

JDB

Q46



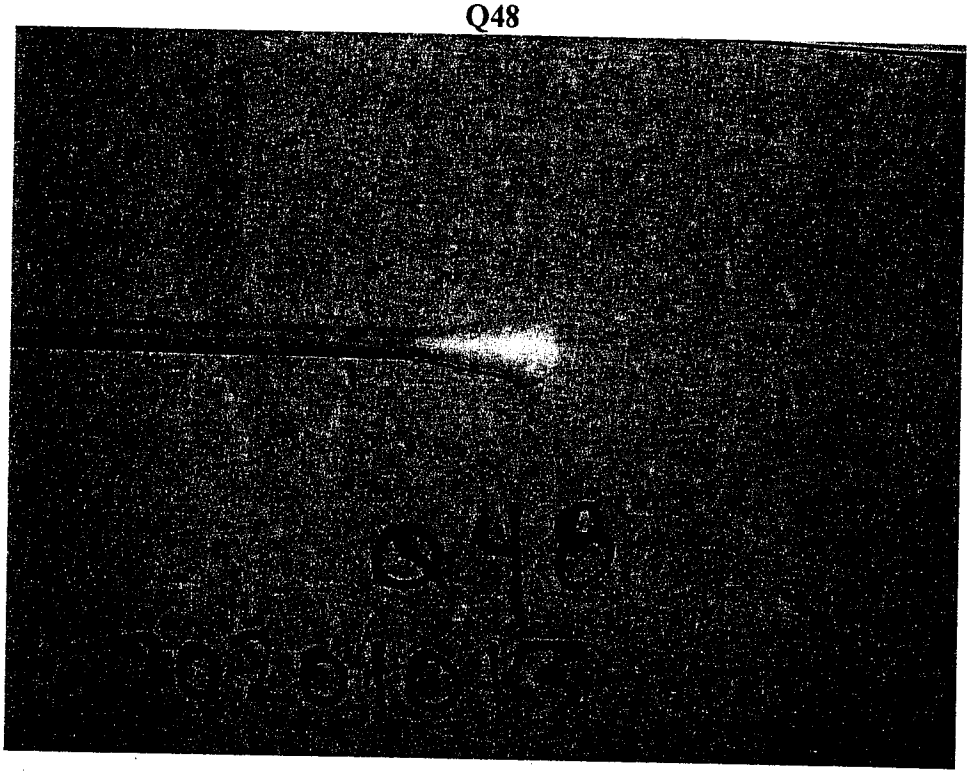
Q47



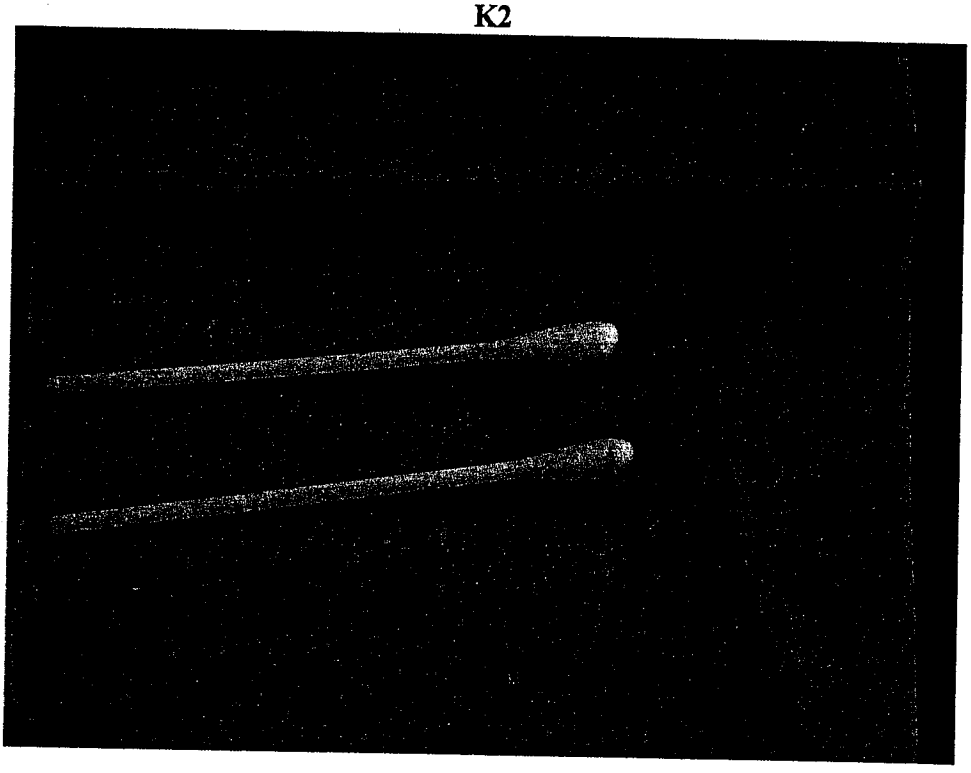
Ex 446
(81)

070201013
02/05/2007
JDB

Q48



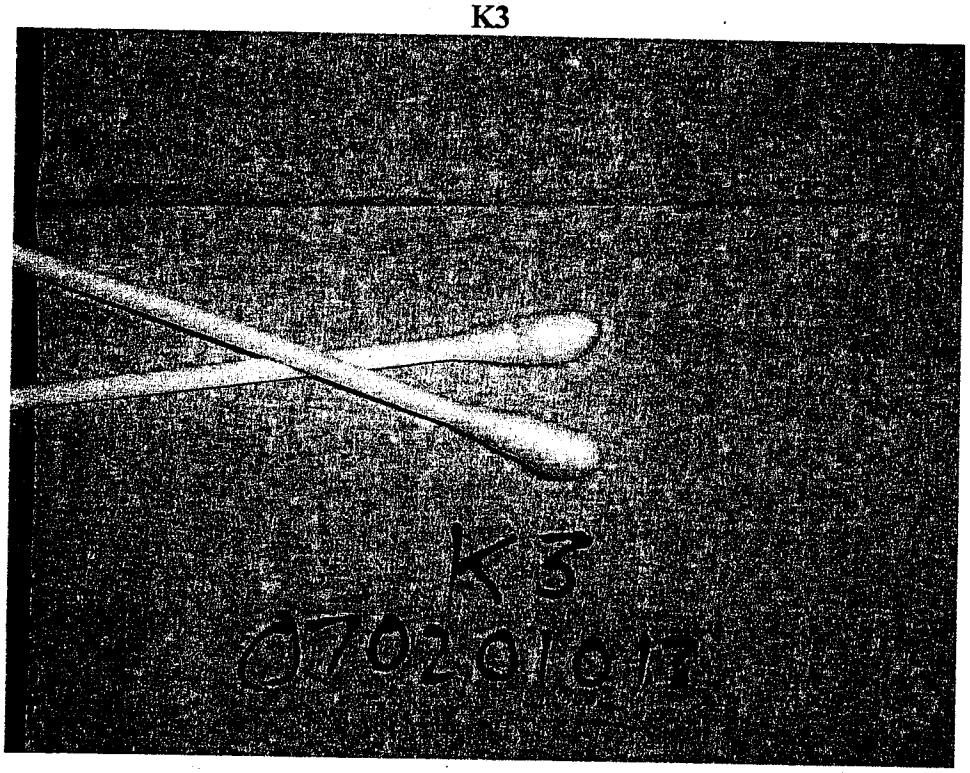
K2



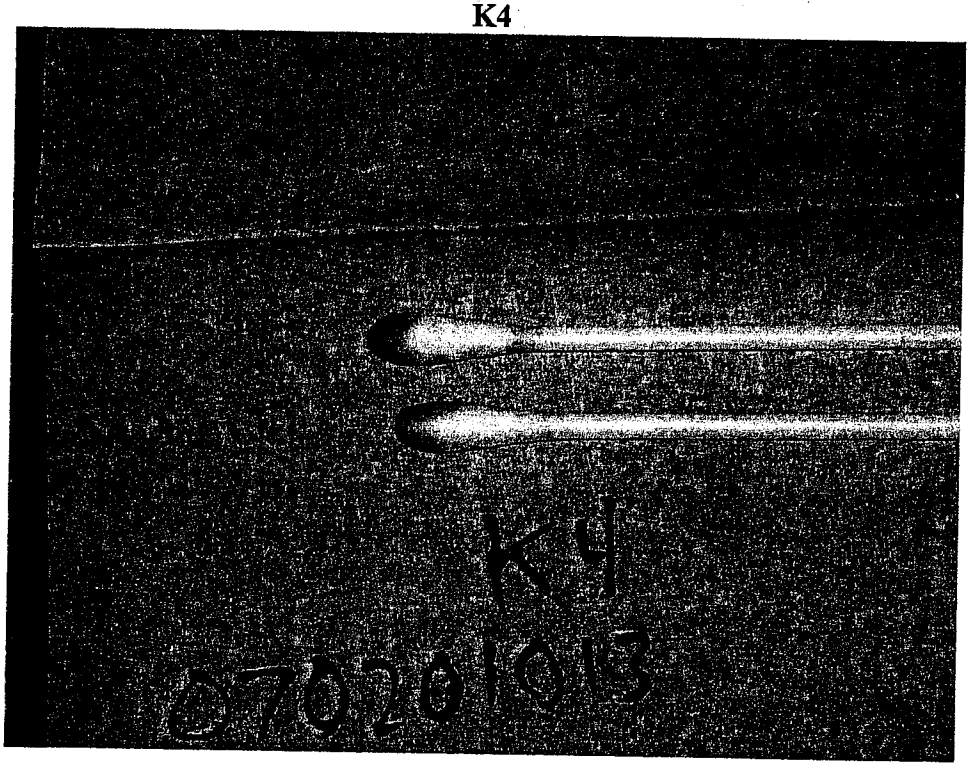
Ex 446
(82)

070201013
02/05/2007
JMB

K3



K4



Ex 446
(83)

Chemistry Unit
Evidence Check-In Sheet

Check-In Date: 02/08/2007 Case ID #: 62D-MW-44363 Examiner: LE BEAU
CHEMIST: BREWER

Mode Received:
☐ USPS ☐ FedEx ☑ Delivered By: ECU
Tracking #: _____

Evidence Initially Received In:
☑ Boxes (# 1) ☐ Paper Bags (#)
☐ Plastic Bags (#) ☐ Envelopes (#)
☐ Cans (#)
☐ Other: _____

Condition of External Container(s):
☑ Proper Seal(s)/Label(s) ☐ Other: (Comment Below)

GENERAL DESCRIPTION OF EVIDENCE AND CONTAINERS:

ONE BOX SEALED W/ YELLOW TAPE W/ INITIALS MARKED "070201013, Q49."
INSIDE IS ONE RED PLASTIC CYLINDRICAL CONTAINER SEALED W/ RED EVIDENCE TAPE W/ INITIALS, MARKED "070201013" AND LABELED W/ CALUMET COUNTY SHERIFF'S DEPARTMENT LABEL " CASE REPORT # 05-157-955, DATE: 2-05-07, EVIDENCE TAG # 9803, RECEIVED FROM: MANITOWAC ? CLERK C75, LOCATION: 1010 S. 8TH ST # 105 MANITOWOC, OFFICER: WIEGERT, OFFICER WITNESSING: WIEGERT, DESCRIPTION/INFORMATION: 1 - VILE OF STEVE AVERYS BLOOD."
INSIDE IS ONE PLASTIC CONTAINER W/ A BLUE SCREW TOP (SEALED) ~~LABELED~~ JB MARKED "070201013, Q49." (PLASTIC TOP HAS A TAMPER EVIDENT SEAL, HAD TO BREAK SEAL TO OPEN).
INSIDE IS A LAVENDER TOP "VACUTAINER" TUBE ~ 60% FILLED W/ A DARK RED LIQUID, LABELED " EDTA (K_3), AVERY STEVEN, 122987" AND " 004-983, 0001, S.A., 01/04/96, 95-624."

Lab #: 070201013 Initials: JDB
Date: 02/08/2007 Page 1 of 2

Ex 446
(84)

Chemistry Unit
Evidence Check-In Sheet

Specimen #	Contributor #	Description:
Q49	ITEM 9803	LIQUID BLOOD SAMPLE FROM STEVEN AVERY (LAVENDER TOP TUBE, "EDTA K_2")

Ex 446
(85)

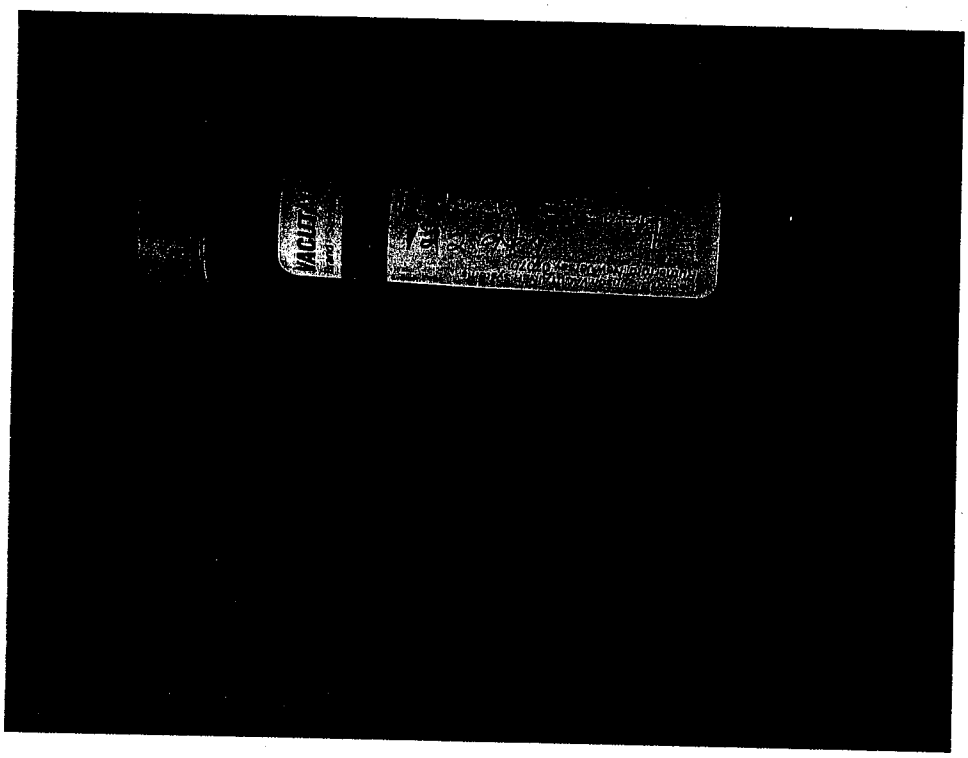
Lab #: 07020101B Initials: JDB

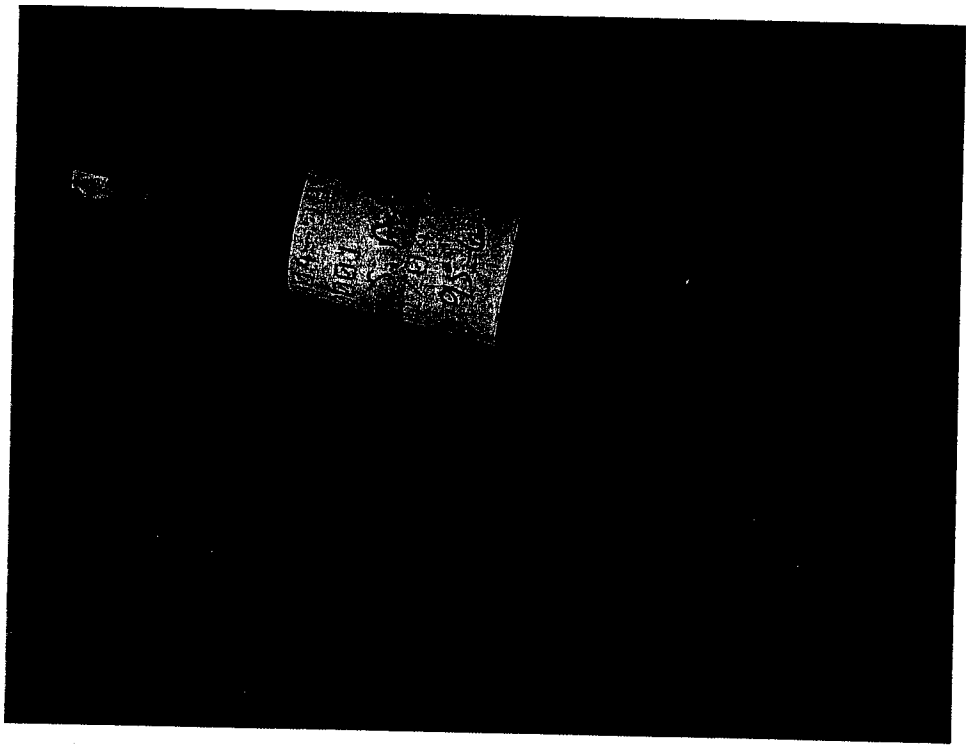
Date: 02/08/2007 Page 2 of 2

070201013
02/08/2007

Q49

JDB

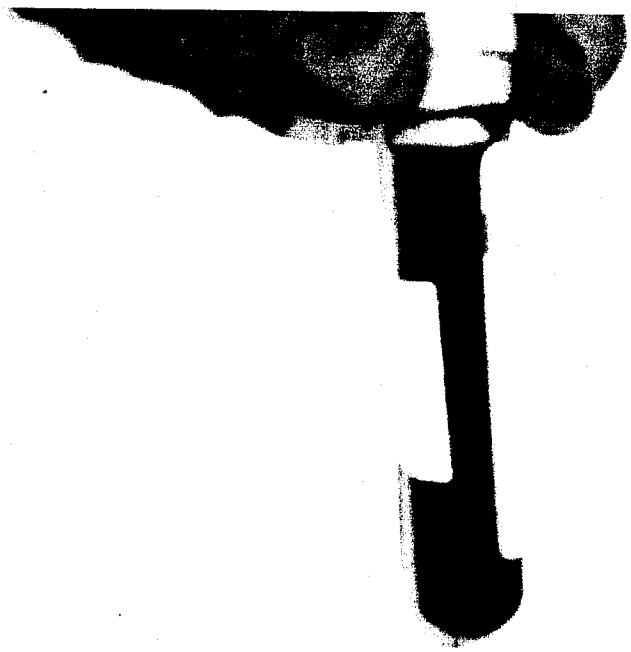




Ex 446
(84)

070201013
02/08/2007
JDB

Q49 (cont'd)



Ex 446
(87)

070201013
PG 1 OF 3

02/16/2007
JDB

VISUAL

- Q46 - ONE COTTON SWAB, PARTIALLY CUT. HAS A DARK BROWNISH-RED STAIN PRESENT. (LOCATION- DASHBOARD NEAR IGNITION).
- Q47 - ONE COTTON SWAB, PARTIALLY CUT. HAS A DARK BROWNISH-RED STAIN PRESENT. (LOCATION - DOOR ENTRANCE AREA, PASS. SIDE).
- Q48 - ONE COTTON SWAB, PARTIALLY CUT. VERY SMALL AREA HAS AN OFF-WHITE/YELLOWISH DISCOLORATION. (LOCATION- CD CASE)
- K2 - TWO COTTON SWABS, PACKAGED TOGETHER. SLIGHT DISCOLORATION. (LOCATION - DASHBOARD IGNITION SWITCH, NEAR).
- K3 - TWO COTTON SWABS, PACKAGED TOGETHER. SLIGHT DISCOLORATION. (LOCATION- DOOR AREA, PASS. SIDE).
- K4 - TWO COTTON SWABS, BLACKISH STAINS PRESENT ON EACH. (LOCATION- CD CASE). PACKAGED TOGETHER.

Q49 - LAVENDER TOP "VACUTAINER, EDTA (K_3)" BLOOD TUBE. ~50-60% FULL. TUBE CAPACITY ESTIMATED TO BE 10 mL, BLOOD VOLUME ESTIMATED TO BE 5.5 mL.

SAMPLING

- Q46 - CUT 1/2 OF THE STAINED AREA W/ CLEAN SCISSORS AND PLACED DIRECTLY INTO A LABELED CENTRIFUGE FILTER DEVICE (CFD) AND CAPPED.
- Q47 - CUT 1/2 OF THE STAINED AREA W/ CLEAN SCISSORS AND PLACED DIRECTLY INTO A LABELED CFD AND CAPPED.
- Q48 - CUT 1/2 OF THE DISCOLORED AREA W/ CLEAN SCISSORS AND PLACED DIRECTLY INTO A LABELED CFD AND CAPPED.
- K2 - CUT 1/2 OF 1 OF THE SWABS AND PLACED DIRECTLY INTO A LABELED CFD AND CAPPED.
- K3 - CUT 1/2 OF 1 OF THE SWABS AND PLACED DIRECTLY INTO A LABELED CFD AND CAPPED.
- K4 - CUT 1/2 OF 1 OF THE SWABS AND PLACED DIRECTLY INTO A LABELED CFD AND CAPPED. Ex 446

02/16/2007
JDB

SAMPLING (CONT'D)

- NEGATIVE CONTROL – 5 uL OF NON-EDTA PRESERVED BLOOD (YELLOW TOP, MAL, 2/2/07) WAS PIPETTED ONTO A CLEAN COTTON SWAB AND AIR DRIED. THE SWAB WAS PLACED IN A LABELED CFD AND CAPPED.
- POSITIVE CONTROL A – 5 uL OF EDTA PRESERVED BLOOD (LAVENDER TOP, MAL, 2/1/07) WAS PIPETTED ONTO A CLEAN COTTON SWAB AND AIR DRIED. THE SWAB WAS PLACED IN A LABELED CFD AND CAPPED.
- POSITIVE CONTROL B – 5 uL OF Q49 BLOOD WAS PIPETTED ONTO A CLEAN COTTON SWAB AND AIR DRIED. THE SWAB WAS PLACED IN A LABELED CFD AND CAPPED.
- SPOT SIZE LOD A – 1 uL OF Q49 BLOOD WAS PIPETTED ONTO A CLEAN GLASS SLIDE AND AIR DRIED. A CLEAN COTTON SWAB WAS WETTED w/ DI H_2O AND USED TO SWAB THE Q49 BLOOD. THE SWAB WAS AIR DRIED THEN PLACED IN A LABELED CFD AND CAPPED.
- SPOT SIZE LOD B – ABOVE PROCEDURE FOR 'LOD A' WAS REPEATED USING 2 uL OF Q49 BLOOD.
- BLANK – PREPARED 02/15/2007 (JDB) – 800 uL OF DI H_2O AND 20 uL OF NON-EDTA PRESERVED BLOOD (YELLOW TOP, MAL, 2/2/07) WERE ADDED TO ~~AC~~ JDB A LABELED CFD, CAPPED, THEN CENTRIFUGED @ 2500 RPM FOR 10 MIN. THE FILTRATE WAS TRANSFERRED TO A VIAL AND CRIMPED.

EXTRACTION

- 200 uL OF 500 ppm d12-EDTA (aq) (d12-EDTA – CAMBRIDGE ISOTOPE LABORATORIES, LOT P-5921, PREP'D 02/09/07 – JDB) WAS ADDED TO EACH SWAB/SWAB CUTTING WHILE IN ITS RESPECTIVE CFD. (NOTE – d12-EDTA WAS NOT ADDED TO THE 'BLANK').

CENTRIFUGE/FILTRATION

Ex 446 (89)

- AFTER EXTRACTING FOR 45 MIN, EACH CFD WAS CENTRIFUGED (10 MIN, 2500 RPM).
- EACH FILTRATE WAS TRANSFERRED TO A LABELED AUTOSAMPLER VIAL AND

070201013
PG 3 OF 3

02/16/2007
JDB

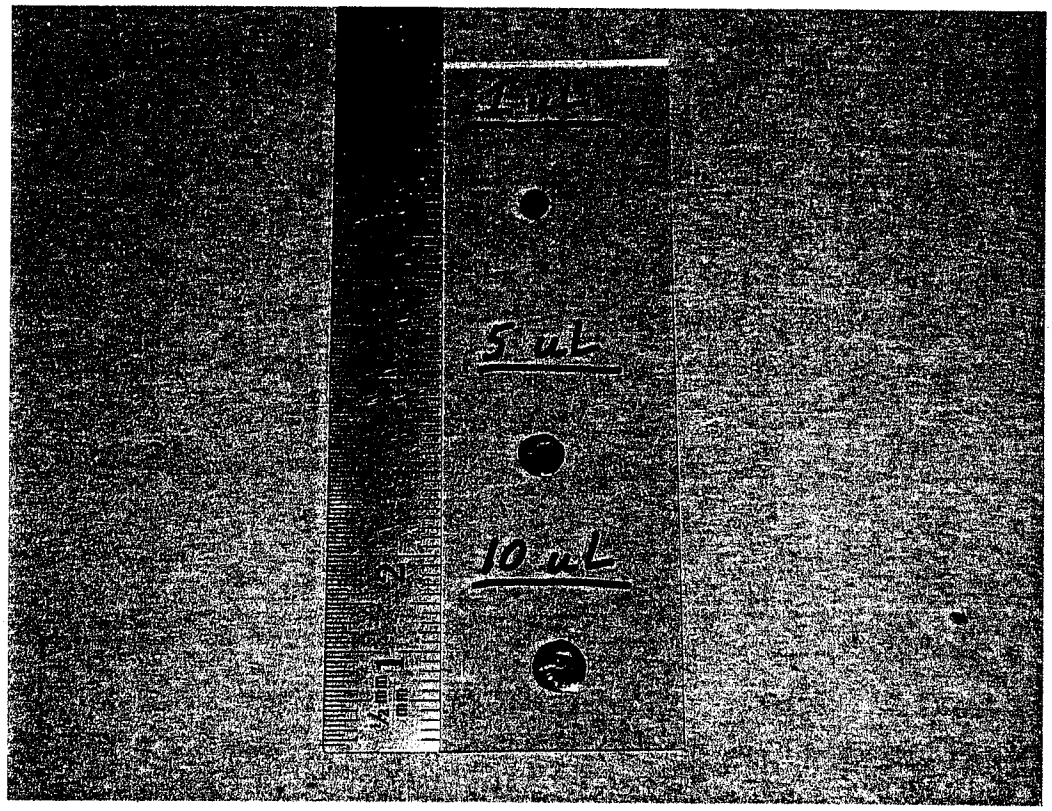
LC/MS-MS

- FILTRATES WERE FIRST ANALYZED IN POSITIVE IONIZATION MODE ON THE 'LCQ-1' (WATERS 2695 LC COUPLED W/ A FINNIGAN MAT LCQ). THE METHOD ('EDTA_Pos_Swabs.meth') AND SEQUENCE ('070201013_Pos.sld') WERE PRINTED.
- THE FILTRATES (SAME VIALS) WERE NEXT ANALYZED IN NEGATIVE IONIZATION MODE ON 'LCQ-1.' THE METHOD ('EDTA_Neg_Swabs.meth') AND SEQUENCE ('070201013_Neg.sld') WERE PRINTED.

02/15/2007

070201013
JDB

Blood spot sizes (1 uL, 5 uL, 10 uL)



Ex 446
(91)

Positive ESI Mode

TANDEM MASS SPECTRA
POS ESI MODE

		EDTA		
		Diagnostic Ions (m/z)		
Specimen		160 m/z	132	247
Pos Control A	response	91367.6	13928	7492.5
	ion ratio	100.0	15.2	8.2
Pos Control B	response	67724.8	9790.4	6929.4
	ion ratio	100.0	14.5	10.2
	pass / fail	PASS	PASS	PASS

		Diagnostic Ions (m/z)		
Specimen		160 m/z	132	247
Pos Control A	response	91367.6	13928	7492.5
	ion ratio	100.0	15.2	8.2
Q49 LOD - 1uL	response	4349.5	0	1803.5
	ion ratio	100.0	0.0	41.5
	pass / fail	PASS	***FAIL***	***FAIL***

		Diagnostic Ions (m/z)		
Specimen		160 m/z	132	247
Pos Control A	response	91367.6	13928	7492.5
	ion ratio	100.0	15.2	8.2
Q49 LOD - 2uL	response	9865	1807.7	698.7
	ion ratio	100.0	18.3	7.1
	pass / fail	PASS	PASS	PASS

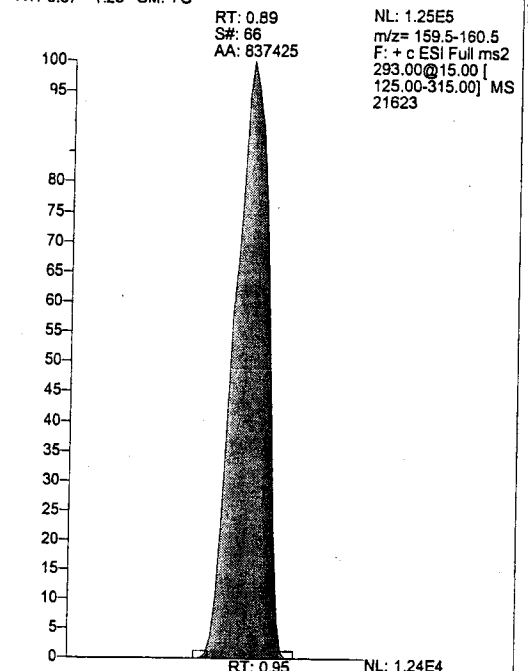
Extra Fragments – Ruled ND

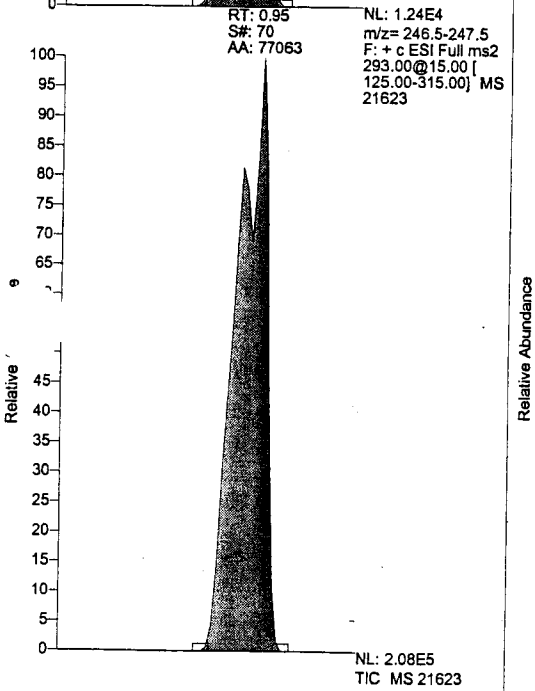
2/26/07
070201013

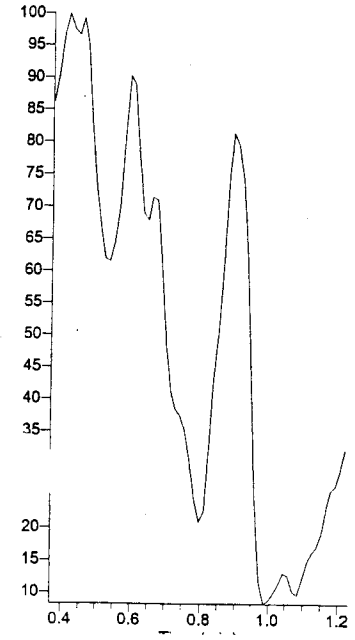
Ex 446
(92)

C:\Xcalibur\data\Brewer\070201013\21623 02/16/07 04:02:32 Pos. Cont. A (MAL EDTA ext.)

RT: 0.37 - 1.23 SM: 7G







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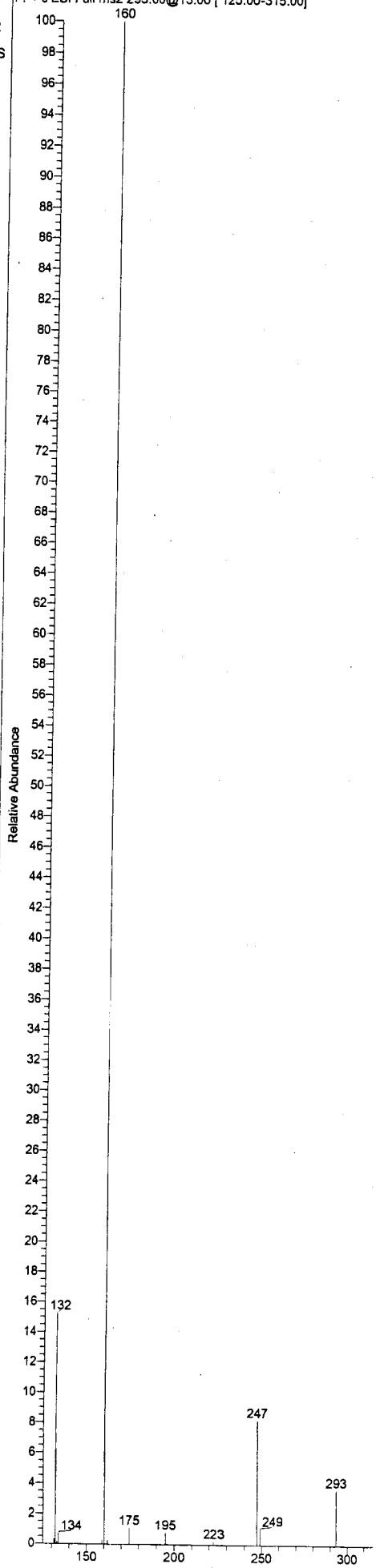


21623#60-70 RT: 0.81-0.95 AV: 11
F: + c ESI Full ms2 293.00@15.00 [125 ...

m/z	Intensity	Relative
131.38	280.5	0.31
132.07	13928.0	15.24
133.02	133.5	0.15
133.98	649.3	0.71
158.96	244.9	0.27
160.04	91367.6	100.00
160.80	0.1	0.00
161.93	253.4	0.28
174.64	965.5	1.06
195.13	709.4	0.78
222.82	192.3	0.21
247.14	7492.5	8.20
249.00	986.0	1.08
293.24	3323.7	3.64

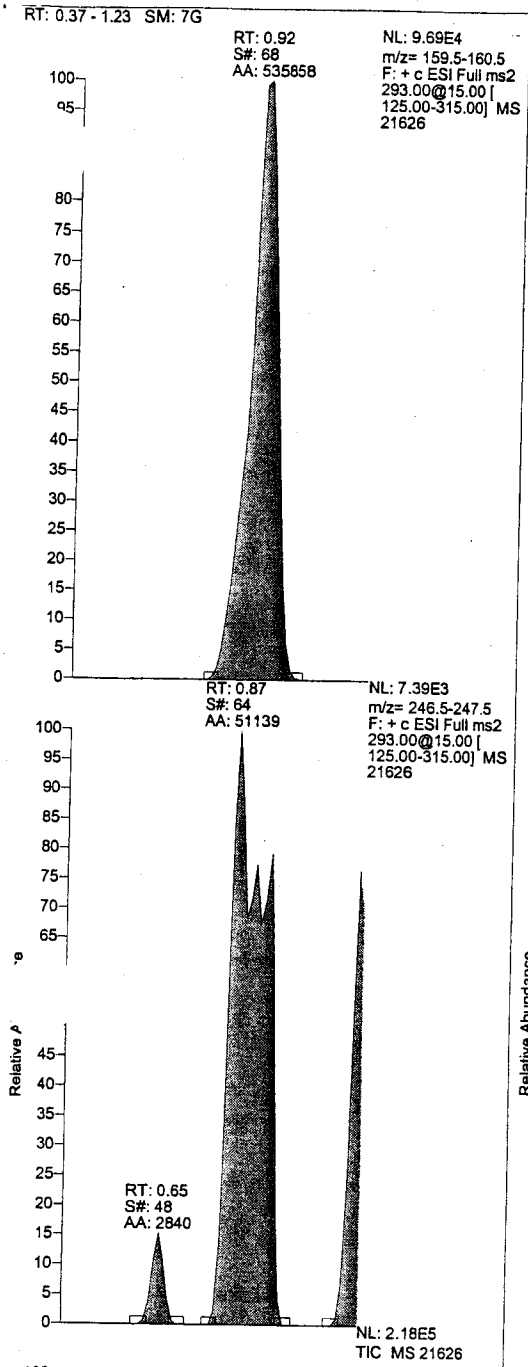
2/26/07 mm

Ex 446
(82)

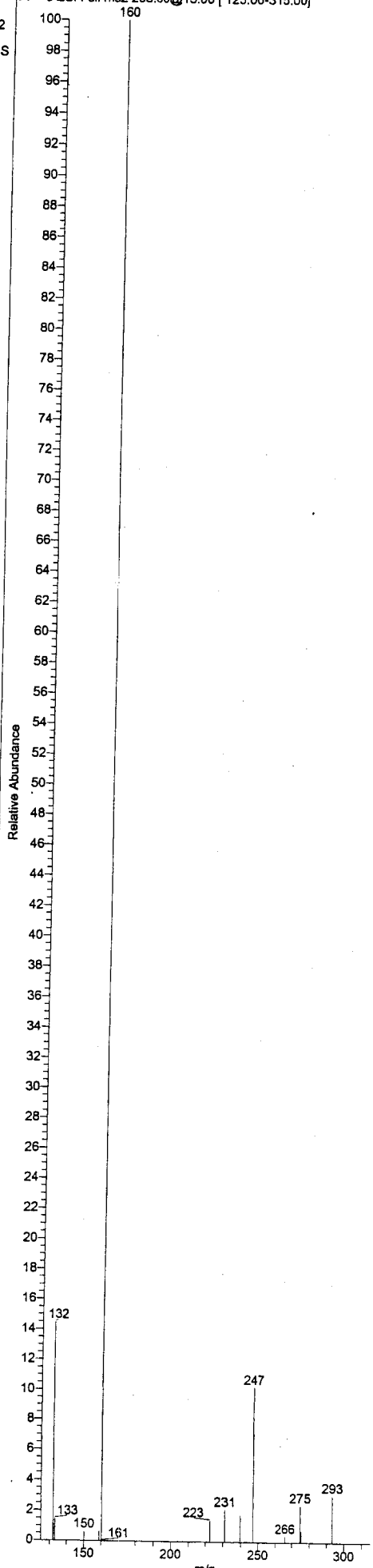
070201013

C:\Xcalibur\data\Brewer\070201013\21626 02/16/07 04:35:06 Pos. Cont. B (Q49 ext.)

RT: 0.37 - 1.23 SM: 7G



21626#63-71 RT: 0.85-0.96 AV: 9 NL: 6.77E4
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



21626#63-71 RT: 0.85-0.96 AV: 9
F: + c ESI Full ms2 293.00@15.00 [125 ...

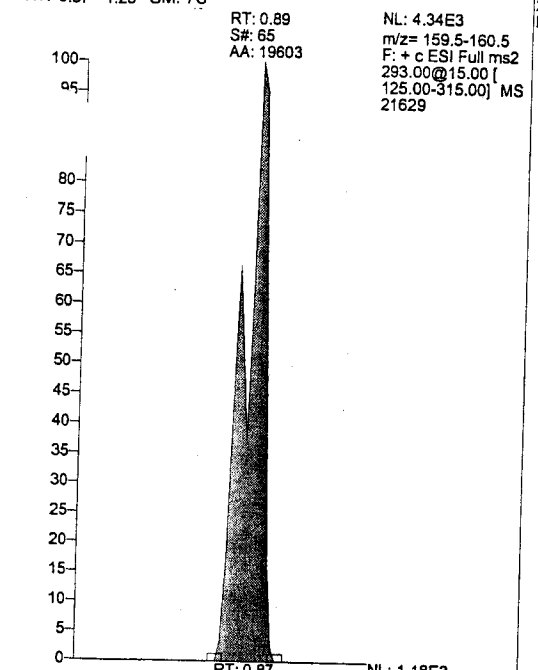
m/z	Intensity	Relative
132.01	9790.4	14.46
133.11	986.1	1.46
150.17	413.8	0.61
158.79	442.9	0.65
160.02	67724.8	100.00
160.55	0.4	0.00
222.50	965.3	1.43
230.96	1431.1	2.11
239.72	1213.1	1.79
246.93	6929.4	10.23
265.95	271.3	0.40
274.80	1631.9	2.41
275.48	499.9	0.74
293.28	2089.4	3.09

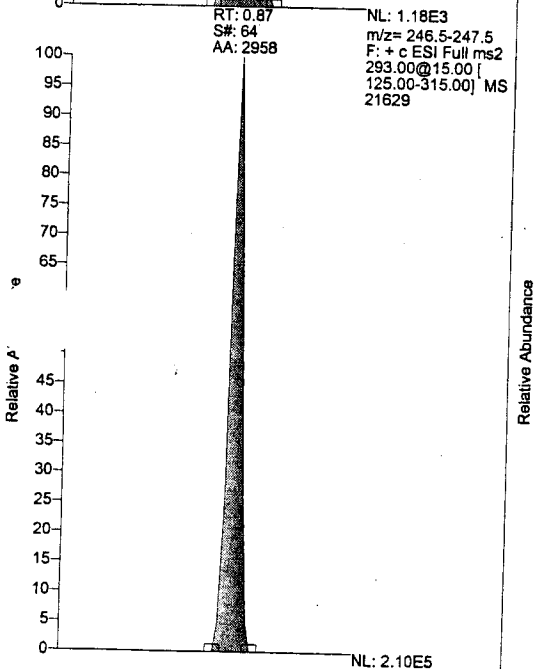
Ex 446
(84)

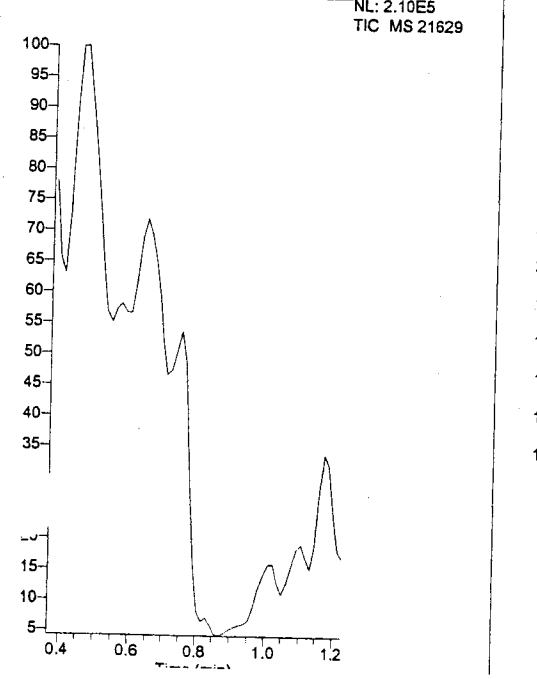
2/26/07

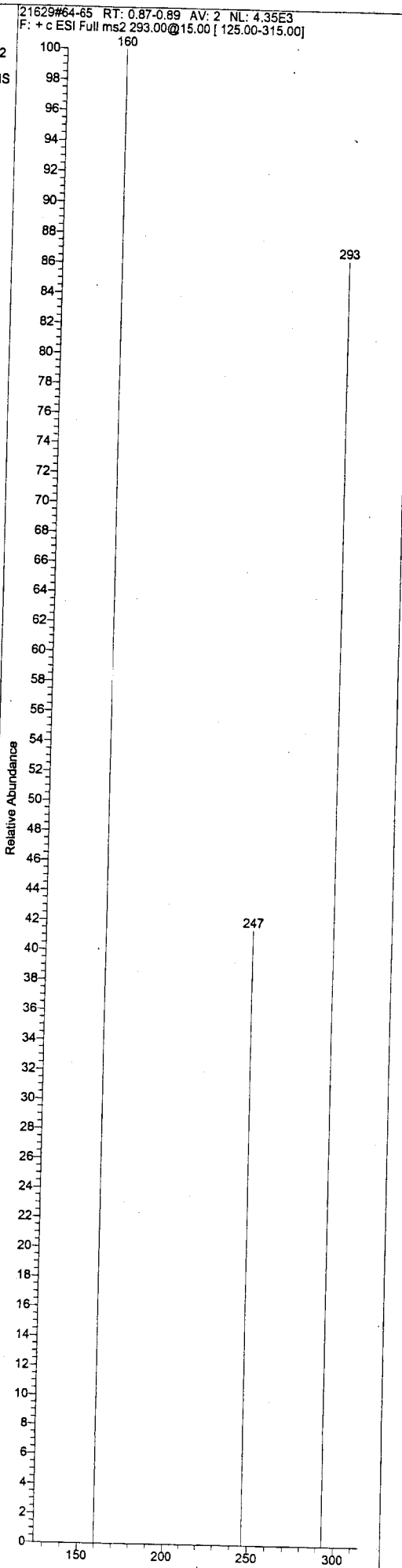
C:\Xcalibur\data\Brewer\070201013\21629 02/16/07 05:07:38 Spot LOD, 1uL Q49

RT: 0.37 - 1.23 SM: 7G









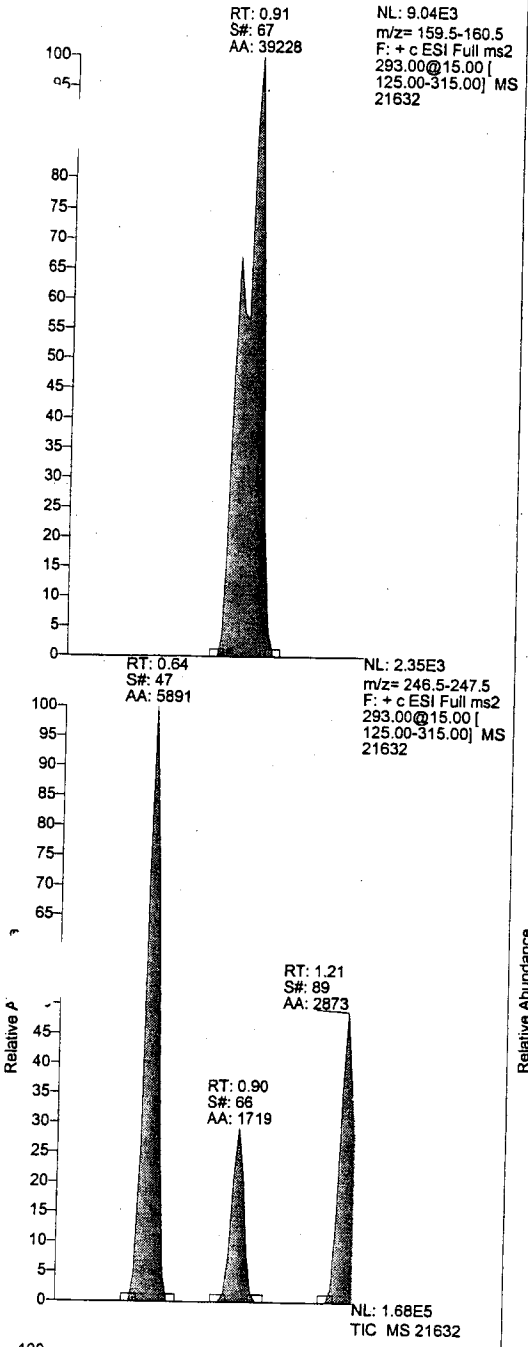
21629#64-65 RT: 0.87-0.89 AV: 2
F: + c ESI Full ms2 293.00@15.00 [125 ...

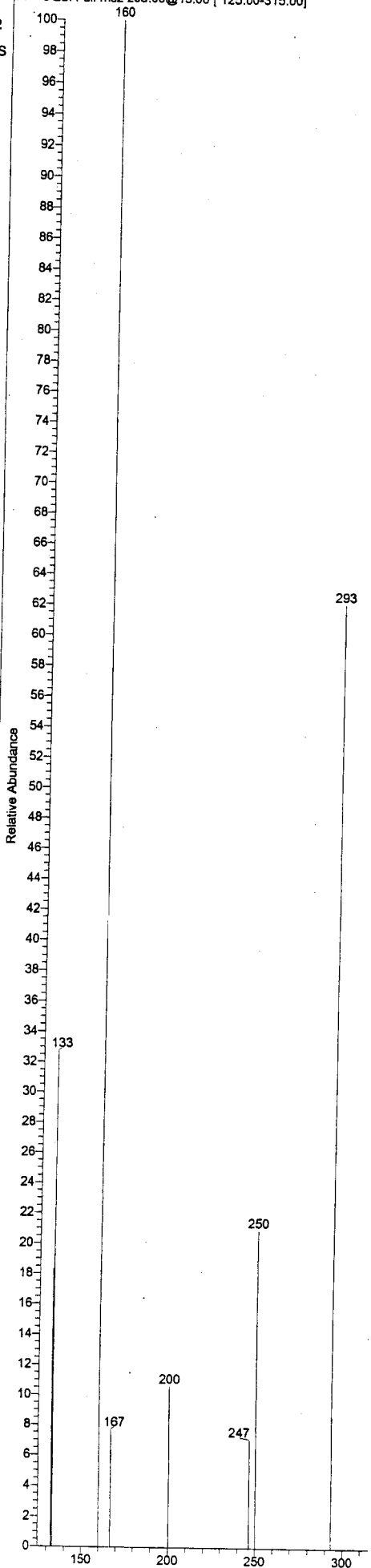
m/z	Intensity	Relative
159.96	4349.5	100.00
246.95	1803.5	41.46
293.33	3758.0	86.40

Ex 446 (65) 2/26/07

C:\Xcalibur\data\Brewer\070201013\21632 02/16/07 05:40:13 Spot LOD, 2 uL Q49

RT: 0.37 - 1.23 SM: 7G





21632#65-67 RT: 0.88-0.91 AV: 3
F: + c ESI Full ms2 293.00@15.00 [125 ...

m/z	Intensity	Relative
132.64	1807.7	18.32
133.16	3227.3	32.71
160.01	9865.0	100.00
167.08	758.7	7.69
200.30	1044.3	10.59
246.56	698.7	7.08
250.08	2064.3	20.93
293.11	6131.0	62.15

Week - ND

Ex 446 (6) 2/26/07

Instrument Method: EDTA_Pos_Swabs.meth

LCQ Instrument Method

Creator: Administrator
Last modified: 2/16/07 by Administrator

MS Run Time (min): 10.00

Divert Valve: not used during run

MS Detector Settings:

Segment 1 Information

Duration (min): 10.00
Number of Scan Events: 1
Tune Method: EDTA_Pos_CID

Scan Event Details:
1: Pos ·(293.0)->o(125.0-315.0)
MS/MS: CE 15.0% IsoW 1.0

Ex 446
(97)

070201013
02/16/2007

Instrument Method: EDTA_Pos_Swabs.meth

Waters 2690 LC System

Injector parameters:
Syringe draw rate (µl/sec):2.50
Injection volume (µl):5

Pump settings:
Solvent A:5:95 ACN:Water + 0.06% NH4OH
Solvent B:B
Solvent C:C
Solvent D:D
Min pressure (PSI):0
Max pressure (PSI):5000
Chart output:Pressure
Chart output:Normal

Gradient program:

Time(min)	Flow(ml/min)	A(%)	B(%)	C(%)	D(%)	Curve
0.00	0.30	100.0	0.0	0.0	0.0	Linear - 6
3.00	0.30	100.0	0.0	0.0	0.0	Linear - 6

Timed events:
Initial states:
Switch 1:No change
Switch 2:No change
Switch 3:No change
Switch 4:No change

Time(min)	Event	Action	Parameter
0.00	Switch1	No change	

Ex 446
(a)

07020101013
02/16/2007

Sequence---070201013_Pos.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21601	1	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: Neg. Control (-EDTA blood swab ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21602	02	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		2	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JDP

Ex 446
(99)

Sequence---070201013_Pos.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21603	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21604	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JDB

Ex 446
(100)

Sequence---070201013_Pos.sld [Open]

Sample Name: K2 extract
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21605	03	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		3	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21606	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JDB

Ex 446
(101)

Sequence---070201013_Pos.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21607	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: Q46 extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21608	04	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		4	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JDB

Ex 446
(102)

Sequence---070201013_Pos.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21609	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21610	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JDB

Ex 446
(103)

Sequence---070201013_Pos.sld [Open]

Sample Name: K3 extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21611	05	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		5	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21612	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JDB

Ex 446
(104)

Sequence---070201013_Pos.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21613	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: Q47 extract

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21614	06	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		6	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JDB

Ex 446
(105)

Sequence---070201013_Pos.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21615	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21616	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JDB

Ex 446
(106)

Sequence---070201013_Pos.sld [Open]

Sample Name: K4 extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21617	07	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		7	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21618	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JDB

Ex 446
(107)

Sequence---070201013_Pos.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21619	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: Q48 extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21620	08	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		8	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JDB

Ex 446
(108)

Sequence---070201013_Pos.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21621	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21622	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JOP

Ex 446
(109)

Sequence---070201013_Pos.sld [Open]

Sample Name: Pos. Cont. A (MAL EDTA ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21623	09	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		9	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21624	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

MM

070201013
02/16/2007
JDB

Ex 446
(110)

Sequence---070201013_Pos.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21625	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: Pos. Cont. B (Q49 ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21626	10	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		10	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JDB

Ex 446
(111)

Sequence---070201013_Pos.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21627	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21628	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JDB

Ex 446
(112)

Sequence---070201013_Pos.sld [Open]

Sample Name: Spot LOD, 1uL Q49

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21629	11	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		11	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21630	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JMB

Ex 446
(113)

Sequence---070201013_Pos.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21631	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: Spot LOD, 2 uL Q49
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21632	12	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		12	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

070201013
02/16/2007
JDB

Ex 446
(114)

Sequence---070201013_Pos.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	21633	01	C:\Xcalibur\Data\Brewer\070201013	C:\Xcalibur\methods\EDTA_Pos_Swabs

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(115)

070201013.
02/16/2007
JDB

Vial List---C:\Xcalibur\methods\brewer\070201013\070201013_Pos.sld

Position	Sample Type	Level	Sample ID	Sample Name
1	Unknown		1	BLANK (neg blood, DI H2O ext.)
	Unknown		02	Neg. Control (-EDTA blood swab ext.)
3	Unknown		03	K2 extract
4	Unknown		04	Q46 extract
5	Unknown		05	K3 extract
6	Unknown		06	Q47 extract
7	Unknown		07	K4 extract
8	Unknown		08	Q48 extract
9	Unknown		09	Pos. Cont. A (MAL EDTA ext.)
10	Unknown		10	Pos. Cont. B (Q49 ext.)
11	Unknown		11	Spot LOD, 1uL Q49
12	Unknown		12	Spot LOD, 2 uL Q49

070201013
02/16/2007
JDB

Ex 446
(116)

TANDEM MASS SPECTRA
NEG ESI MODE

Specimen		EDTA (Free Acid) Diagnostic Ions (m/z) 273 m/z	247	EDTA (Iron Complex) Diagnostic Ions (m/z) 300 m/z	326
Pos Control A	response	105085	8271	23366	2413.5
	ion ratio	100.0	7.9	100.0	10.3
Pos Control B	response	43718.3	2165.3	114448.8	8802
	ion ratio	100.0	5.0	100.0	7.7
	pass / fail	PASS	PASS	PASS	PASS

Specimen		Diagnostic Ions (m/z) 273 m/z	247	Diagnostic Ions (m/z) 300 m/z	326
Pos Control A	response	105085	8271	23366	2413.5
	ion ratio	100.0	7.9	100.0	10.3
Q49 LOD - 1uL	response	3548.4	531.7	11927.5	1017
	ion ratio	100.0	15.0	100.0	8.5
	pass / fail	PASS	PASS	PASS	PASS

Specimen		Diagnostic Ions (m/z) 273 m/z	247	Diagnostic Ions (m/z) 300 m/z	326
Pos Control A	response	105085	8271	23366	2413.5
	ion ratio	100.0	7.9	100.0	10.3
K3 Control	response	0	0	963.5	4031
	ion ratio	#DIV/0!	#DIV/0!	100.0	418.4
	pass / fail	#DIV/0!	#DIV/0!	PASS	***FAIL***

Specimen		Diagnostic Ions (m/z) 273 m/z	247	Diagnostic Ions (m/z) 300 m/z	326
Pos Control A	response	105085	8271	23366	2413.5
	ion ratio	100.0	7.9	100.0	10.3
Q48 Swab	response	0	0	733.8	1561
	ion ratio	#DIV/0!	#DIV/0!	100.0	212.7
	pass / fail	#DIV/0!	#DIV/0!	PASS	***FAIL***

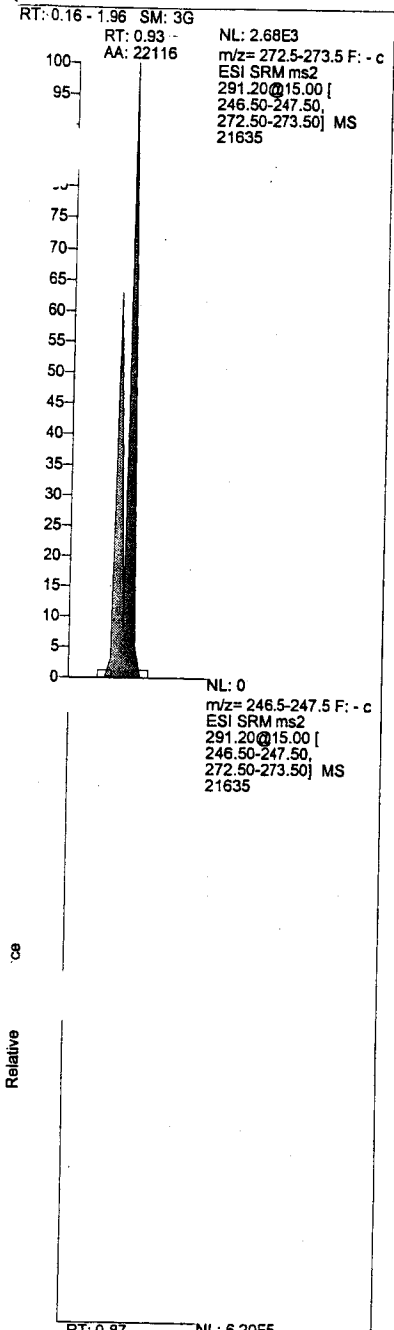
Specimen		Diagnostic Ions (m/z) 273 m/z	247	Diagnostic Ions (m/z) 300 m/z	326
Pos Control A	response	105085	8271	23366	2413.5
	ion ratio	100.0	7.9	100.0	10.3
Q49 LOD - 2uL	response	12717.7	0	28697.5	262.5
	ion ratio	100.0	0.0	100.0	0.9
	pass / fail	PASS	***FAIL***	PASS	PASS

070201013

2/26/07

Ex 446
(11)

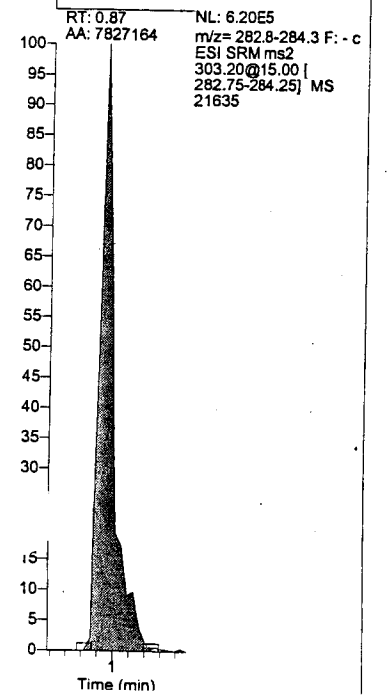
C:\Xcalibur\...\Negative Ion\21635 02/16/07 06:53:36 Neg. Control (-EDTA blood swab ext.)



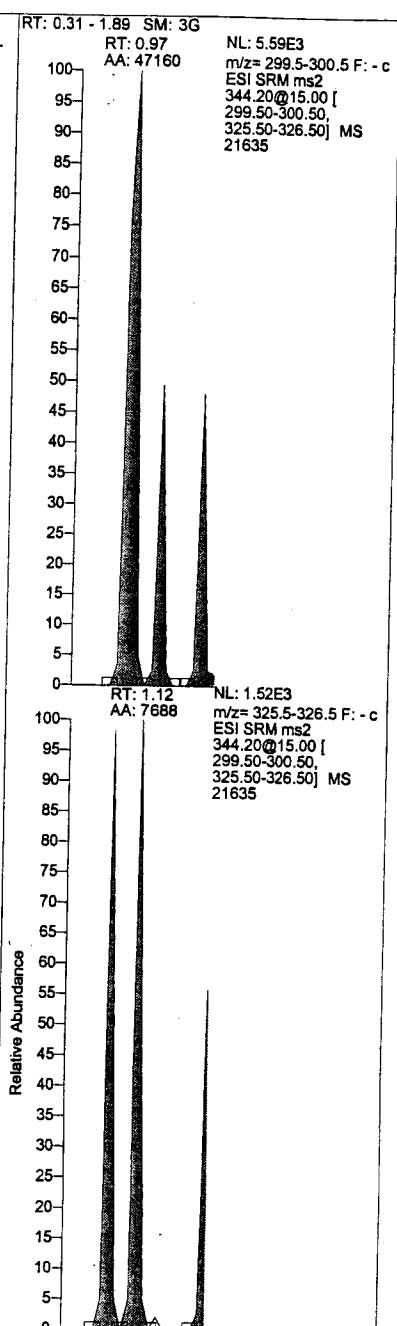
21635#41-49 RT: 0.78-0.93 AV: 3
F: - c ESI SRM ms2 291.20@15.00 [2 ...
m/z= 246.50-326.50

m/z	Intensity	Relative
272.70	620.7	63.05
273.27	984.3	100.00

(ND)



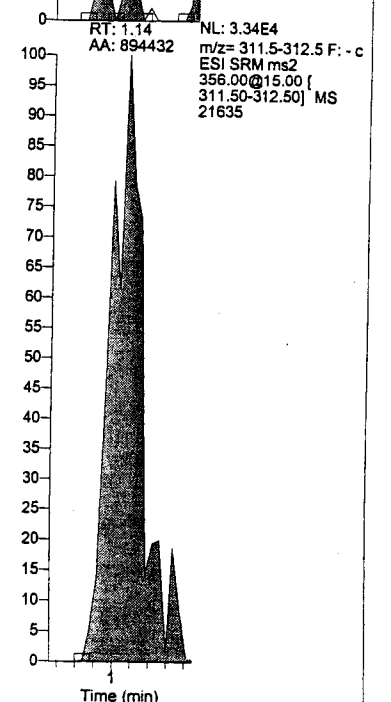
Ex 446 (118)



21635#47-51 RT: 0.89-0.97 AV: 2
F: - c ESI SRM ms2 344.20@15.00 [2 ...
m/z= 298.00-326.50

m/z	Intensity	Relative
299.70	5134.0	100.00

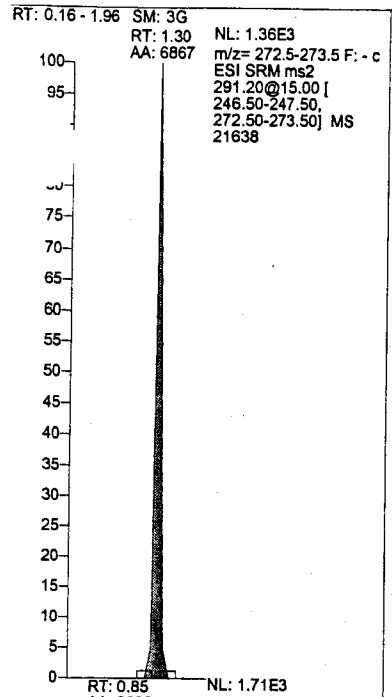
(ND)

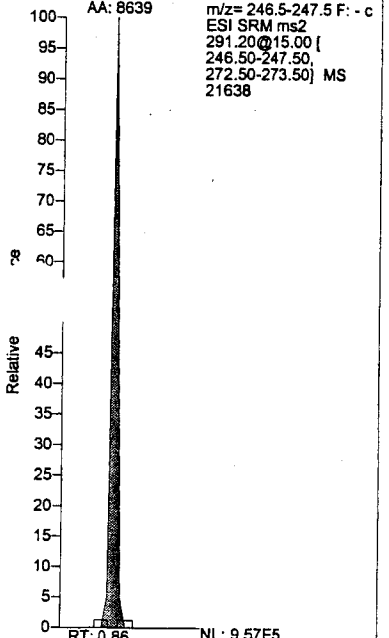


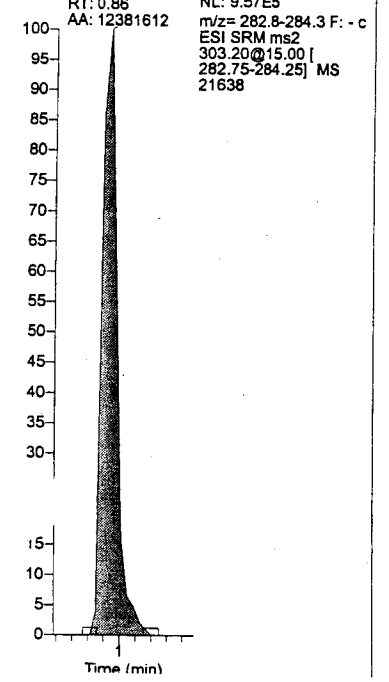
070201013
2/26/07

C:\Xcalibur\...\Negative Ion\21638 02/16/07 07:26:12 K2 extract

RT: 0.16 - 1.96 SM: 3G







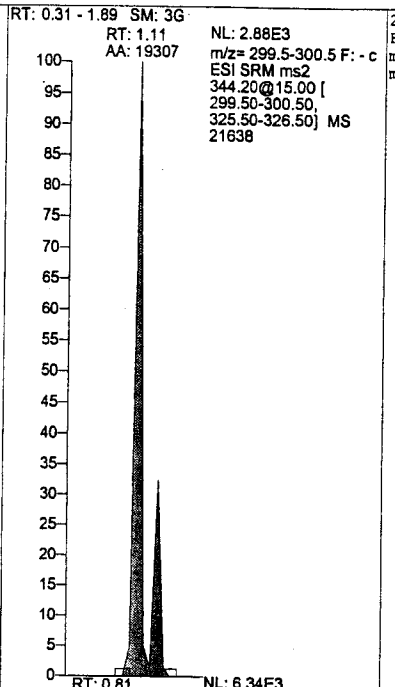
21638#41-45 RT: 0.77-0.85 AV: 2
F: - c ESI SRM ms2 291.20@15.00 [2 ...
m/z= 246.50-326.50

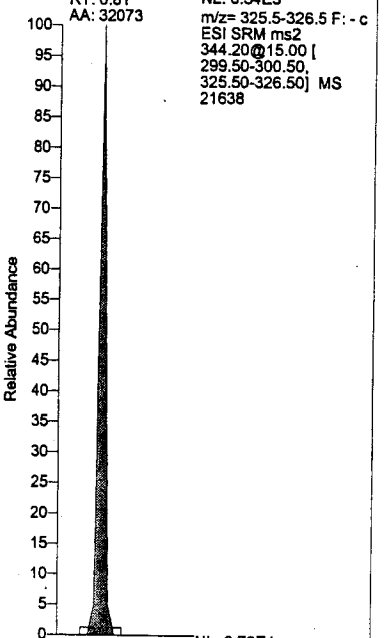
m/z	Intensity	Relative
246.81	940.0	100.00

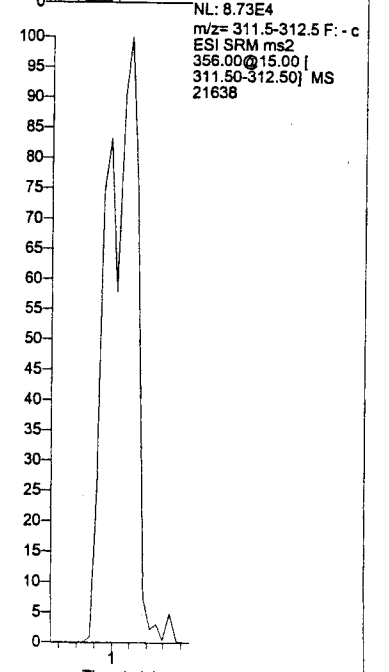
ND

Ex 446
(119)

RT: 0.31 - 1.89 SM: 3G







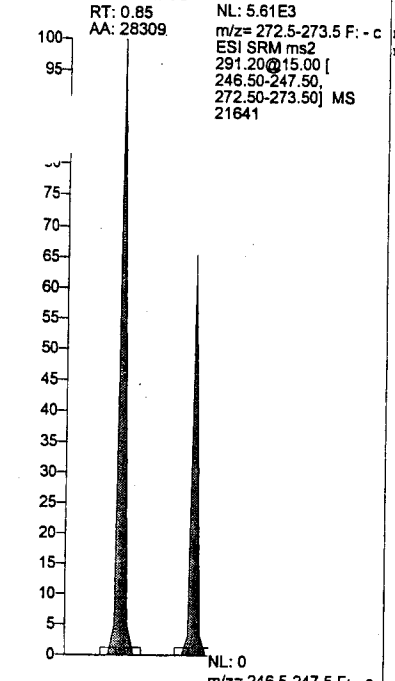
21638#51-59 RT: 0.96-1.11 AV: 3
F: - c ESI SRM ms2 344.20@15.00 [2 ...
m/z= 298.00-326.50

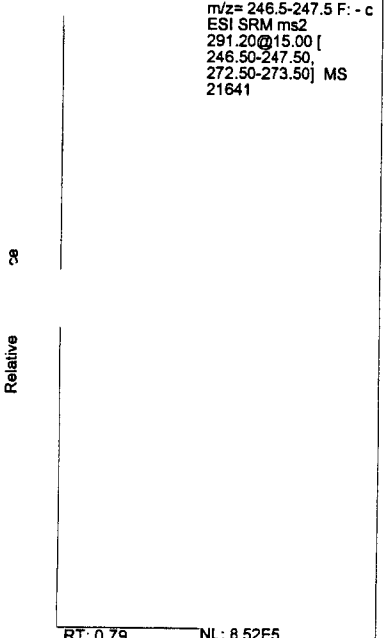
m/z	Intensity	Relative
300.13	1057.3	100.00

ND

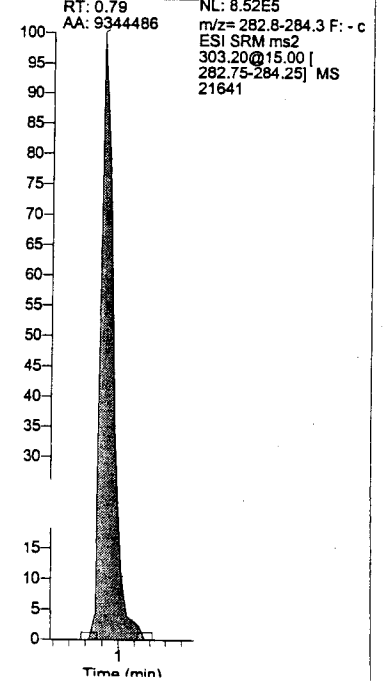
070201013
2/26/07

RT: 0.16 - 1.96 SM: 3G





Relative



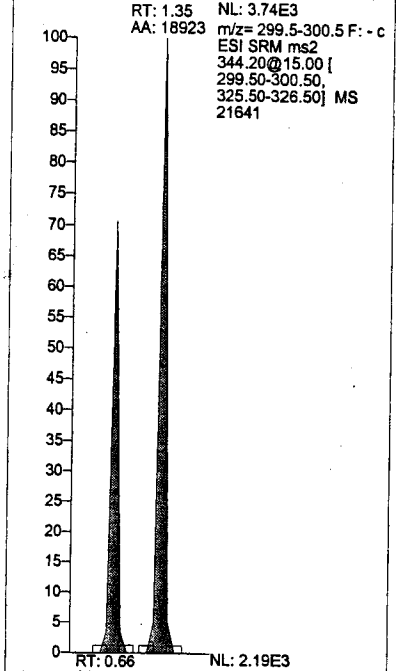
21641#41-45 RT: 0.78-0.85 AV: 2
F: - c ESI SRM ms2 291.20@15.00 [2 ...
m/z= 246.50-326.50

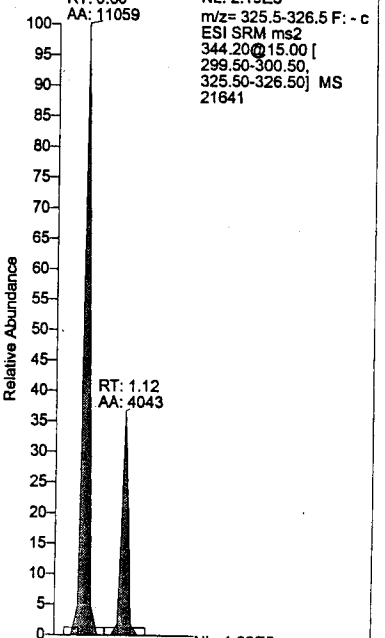
m/z	Intensity	Relative
273.19	3083.5	100.00

ND

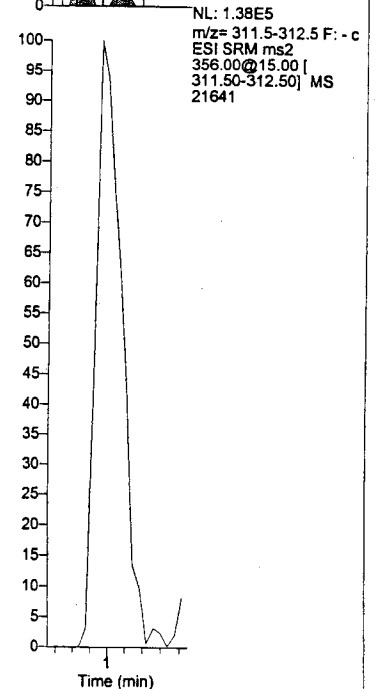
Ex 446
(120)

RT: 0.31 - 1.89 SM: 3G





Relative Abundance



21641#43 RT: 0.81
F: - c ESI SRM ms2 344.20@15.00 [2 ...
m/z= 298.00-326.50

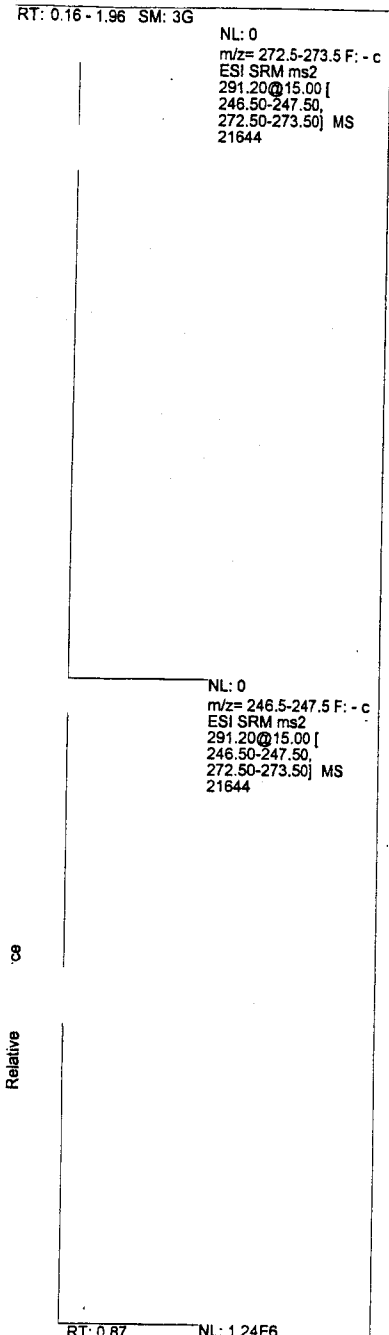
m/z	Intensity	Relative
299.79	2907.0	100.00

ND

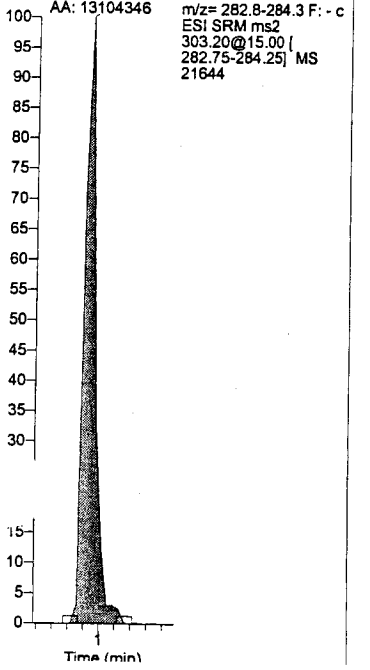
0702201013
2/26/07

C:\Xcalibur\...\Negative Ion\21644 02/16/07 08:31:35 K3 extract

RT: 0.16 - 1.96 SM: 3G



RT: 0.87
AA: 13104346

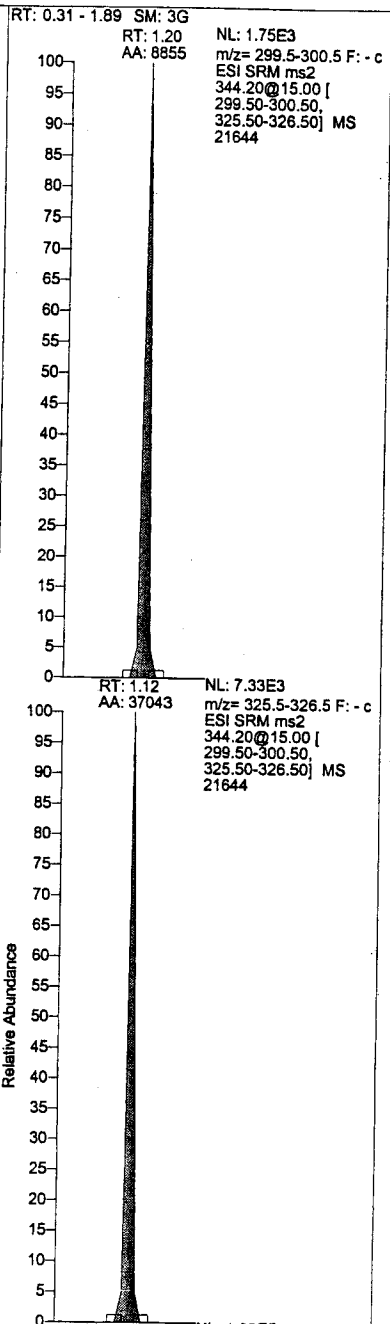


21644#41-49 RT: 0.78-0.93 AV: 3
F: - c ESI SRM ms2 291.20@15.00 [2 ...
m/z= 246.50-326.50
Scan contains no data.

ND

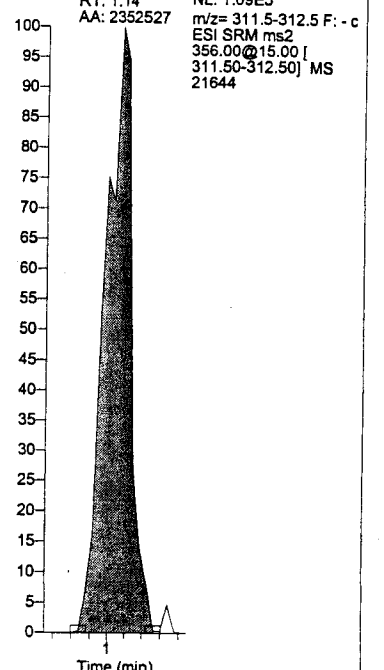
RT: 0.31 - 1.89 SM: 3G

RT: 1.20
AA: 8855



RT: 1.12
AA: 37043

RT: 1.14
AA: 2352527



21644#59-63 RT: 1.12-1.20 AV: 2
F: - c ESI SRM ms2 344.20@15.00 [2 ...
m/z= 298.00-326.50

m/z	Intensity	Relative
299.66	963.5	23.90
326.41	4031.0	100.00

ND - Ratios Fail

Ex 446 (21)

070201013
2/26/07

RT: 0.16 - 1.96 SM: 3G

NL: 0
m/z= 272.5-273.5 F: - c ESI SRM ms2 291.20@15.00 [246.50-247.50, 272.50-273.50] MS 21647

NL: 0
m/z= 246.5-247.5 F: - c ESI SRM ms2 291.20@15.00 [246.50-247.50, 272.50-273.50] MS 21647

RT: 0.79
AA: 8017102
NL: 7.51E5
m/z= 282.8-284.3 F: - c ESI SRM ms2 303.20@15.00 [282.75-284.25] MS 21647

Relative Abundance

Time (min)

21647#41-49 RT: 0.77-0.93 AV: 3
F: - c ESI SRM ms2 291.20@15.00 [2-...
m/z= 246.50-326.50
Scan contains no data.

ND

RT: 0.31 - 1.89 SM: 3G

RT: 1.81
AA: 7866
NL: 1.56E3
m/z= 299.5-300.5 F: - c ESI SRM ms2 344.20@15.00 [299.50-300.50, 325.50-326.50] MS 21647

RT: 1.19
AA: 4347

NL: 0
m/z= 325.5-326.5 F: - c ESI SRM ms2 344.20@15.00 [299.50-300.50, 325.50-326.50] MS 21647

NL: 2.10E5
m/z= 311.5-312.5 F: - c ESI SRM ms2 356.00@15.00 [311.50-312.50] MS 21647

Relative Abundance

Time (min)

21647#47-51 RT: 0.89-0.96 AV: 2
F: - c ESI SRM ms2 344.20@15.00 [2 ...
m/z= 298.00-326.50
Scan contains no data.

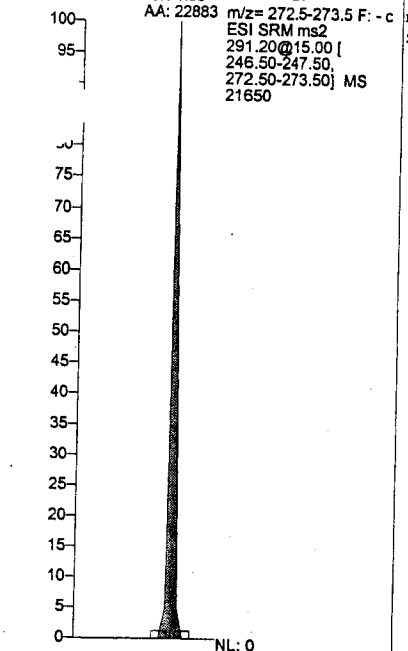
ND

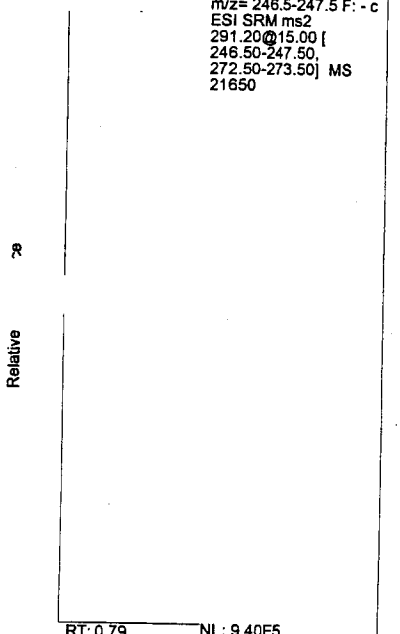
Ex 446
(122)

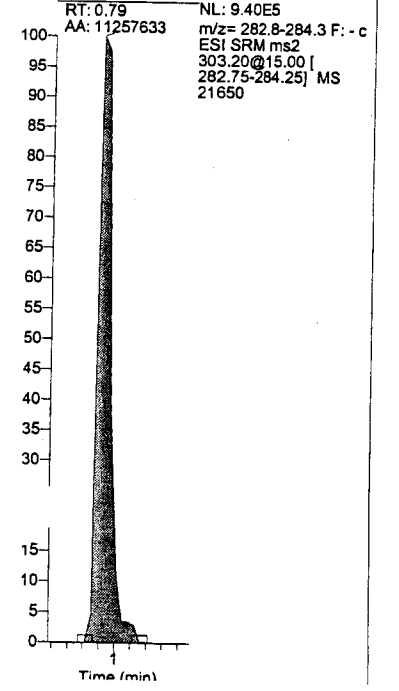
070201013
2/26/07

C:\Xçalibur\...\Negative Ion\21650 02/16/07 09:36:53 K4 extract

RT: 0.16 - 1.96 SM: 3G



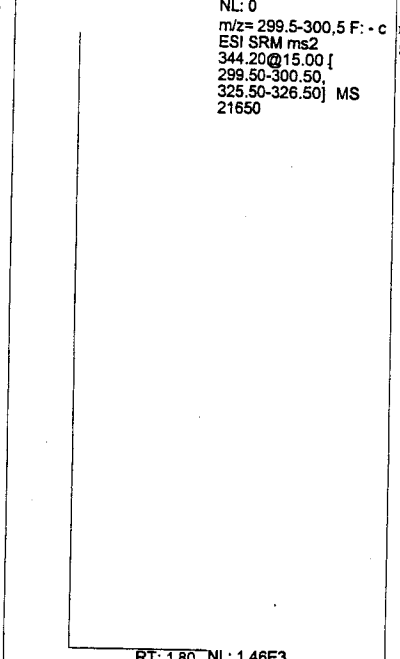


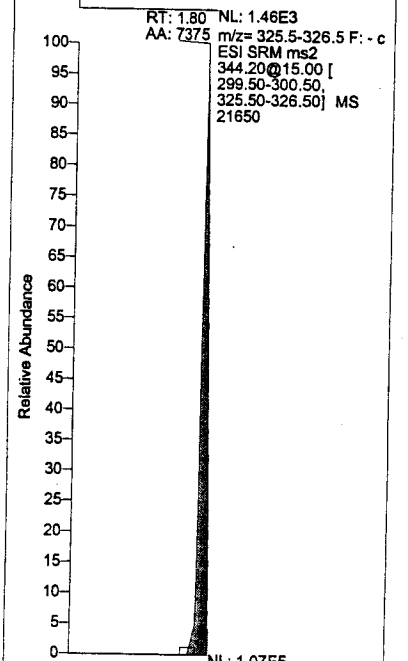


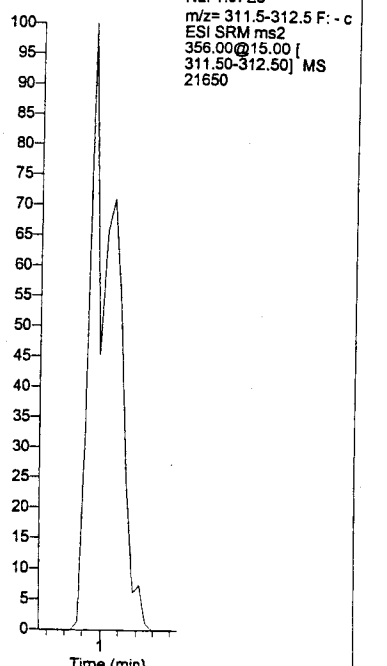
21650#41-49 RT: 0.77-0.92 AV: 3
F: - c ESI SRM ms2 291.20@15.00 [2 ...
m/z= 246.50-326.50
Scan contains no data.

ND

RT: 0.31 - 1.89 SM: 3G







21650#47-51 RT: 0.88-0.96 AV: 2
F: - c ESI SRM ms2 344.20@15.00 [2 ...
m/z= 298.00-326.50
Scan contains no data.

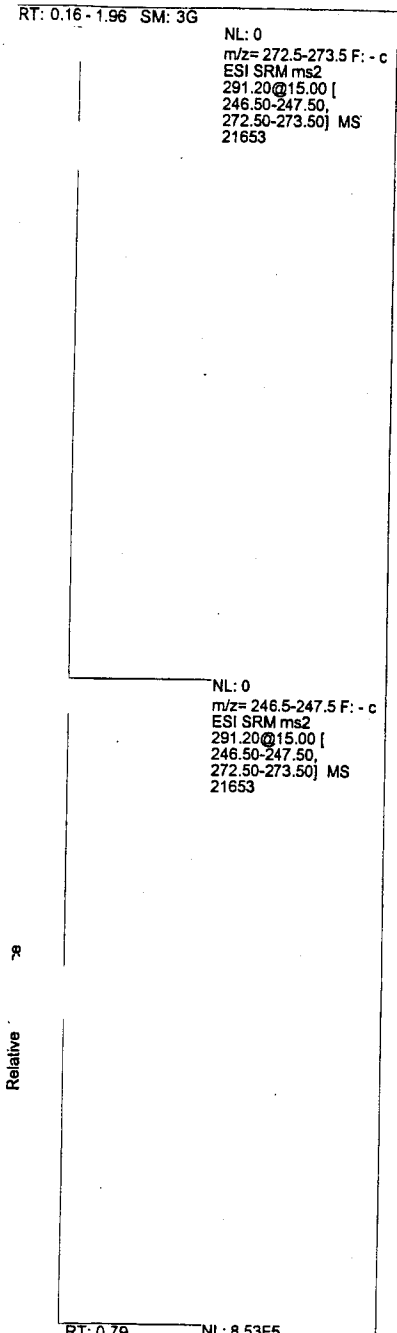
ND

Ex 446
(123)

070201013
02/26/07

C:\Xcalibur\...\Negative Ion\21653 02/16/07 10:09:33 Q48 extract

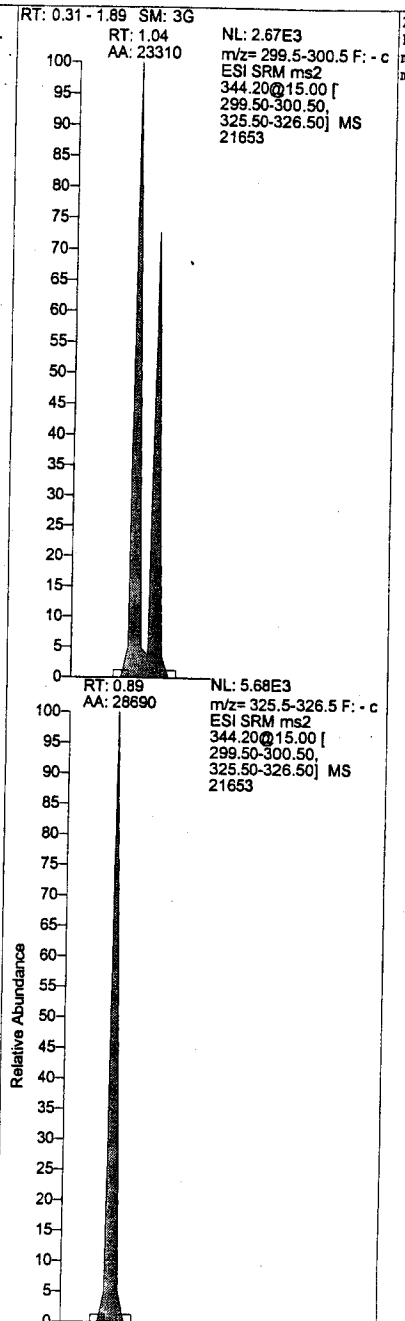
RT: 0.16 - 1.96 SM: 3G



21653#41-49 RT: 0.77-0.92 AV: 3
F: - c ESI SRM ms2 291.20@15.00 [2 ...
m/z= 246.50-326.50
Scan contains no data.

ND

RT: 0.31 - 1.89 SM: 3G

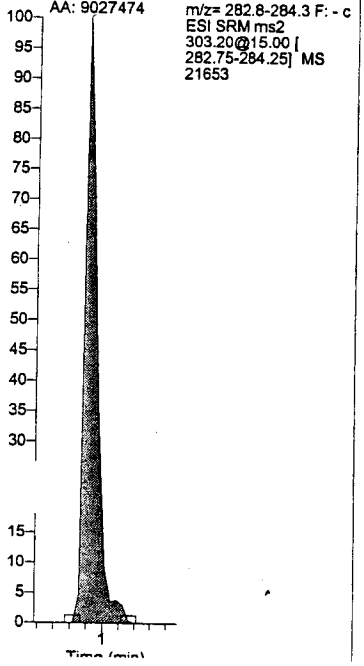


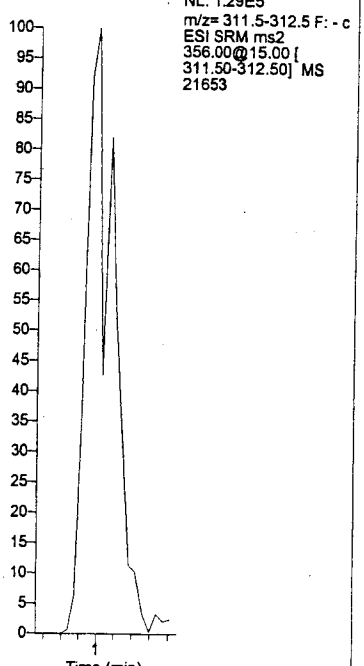
21653#43-55 RT: 0.81-1.04 AV: 4
F: - c ESI SRM ms2 344.20@15.00 [2 ...
m/z= 298.00-326.50

m/z	Intensity	Relative
300.25	733.8	47.01
326.11	1561.0	100.00

ND - Ratios Fail

RT: 0.79
AA: 9027474



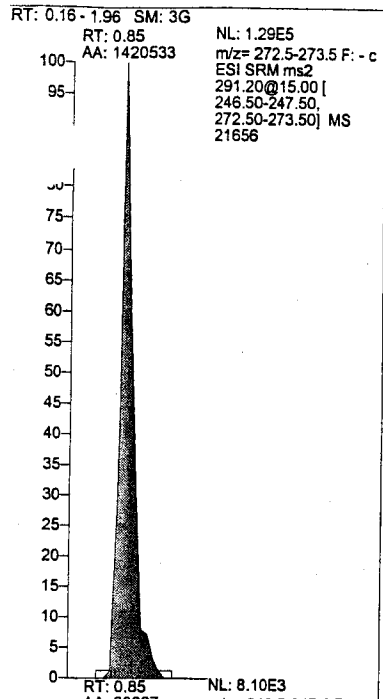


Ex 446 (124)

070201013

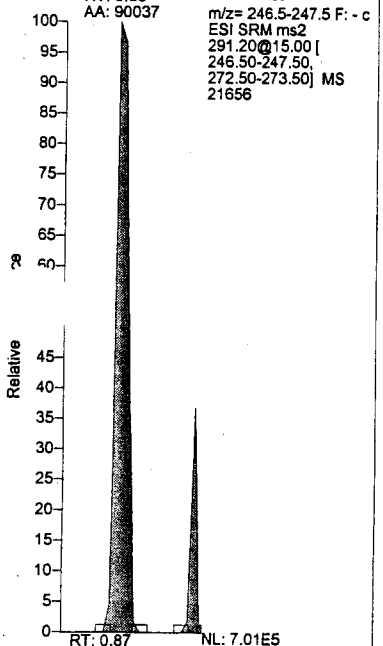
2/26/07

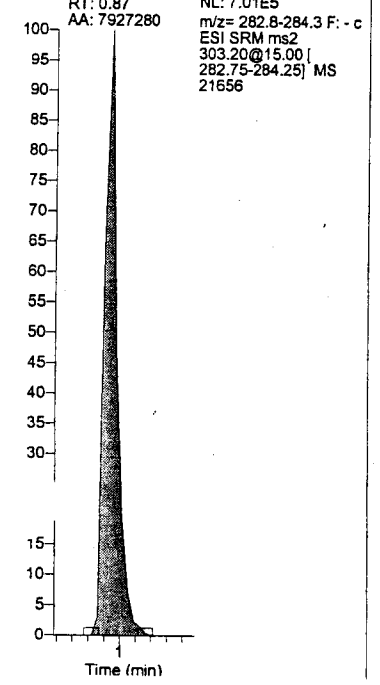
RT: 0.16 - 1.96 SM: 3G



21656#45-49 RT: 0.85-0.93 AV: 2
F: - c ESI SRM ms2 291.20@15.00 [2 ...
m/z= 246.50-326.50

m/z	Intensity	Relative
247.16	8271.0	7.87
273.08	105085.0	100.00



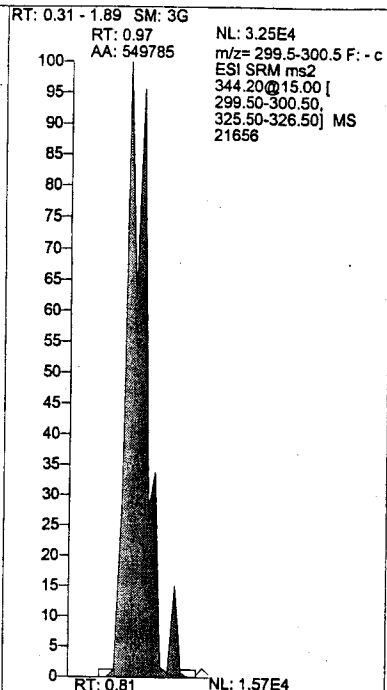


POS

NM

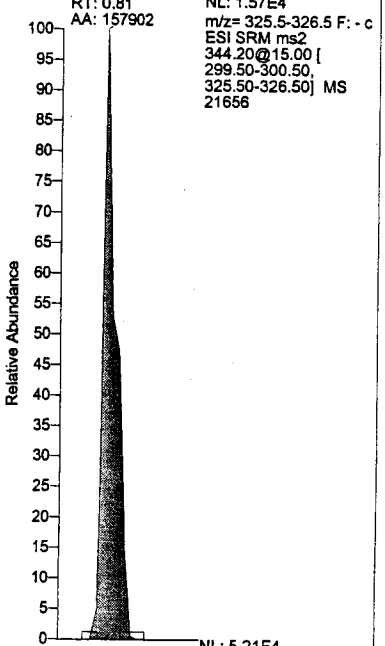
Ex 446 (125)

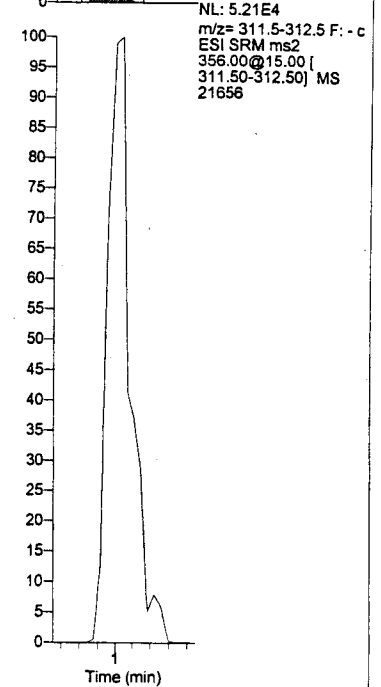
RT: 0.31 - 1.89 SM: 3G



21656#51-63 RT: 0.97-1.20 AV: 4
F: - c ESI SRM ms2 344.20@15.00 [2 ...
m/z= 298.00-326.50

m/z	Intensity	Relative
300.02	23366.0	100.00
326.05	2413.5	10.33

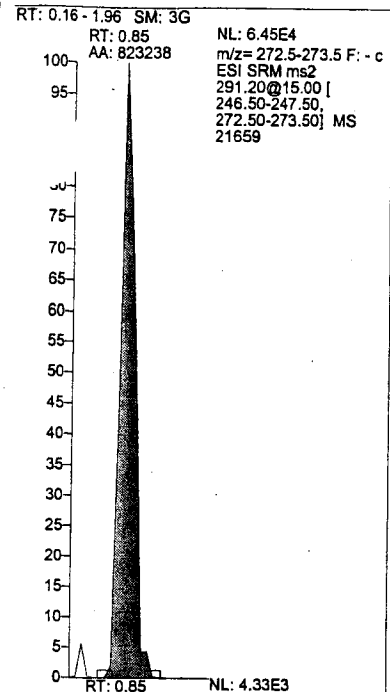




POS

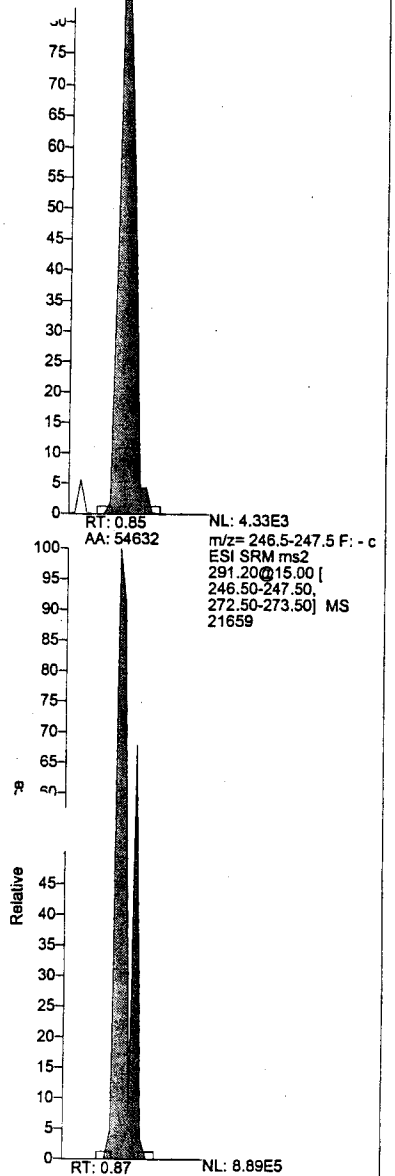
NM

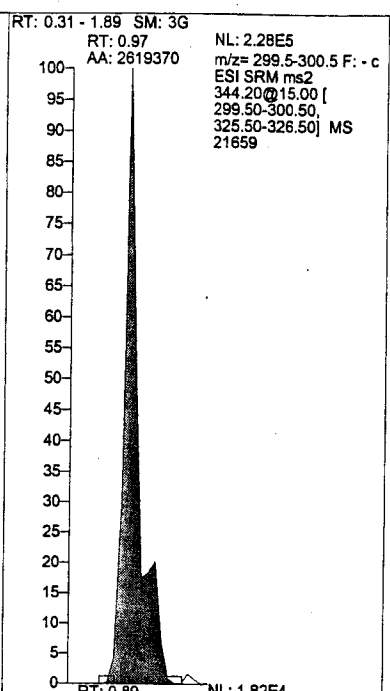
070201013
2/26/07



21659#41-53 RT: 0.77-1.00 AV: 4
F: - c ESI SRM ms2 291.20@15.00 [2 ...
m/z= 246.50-326.50

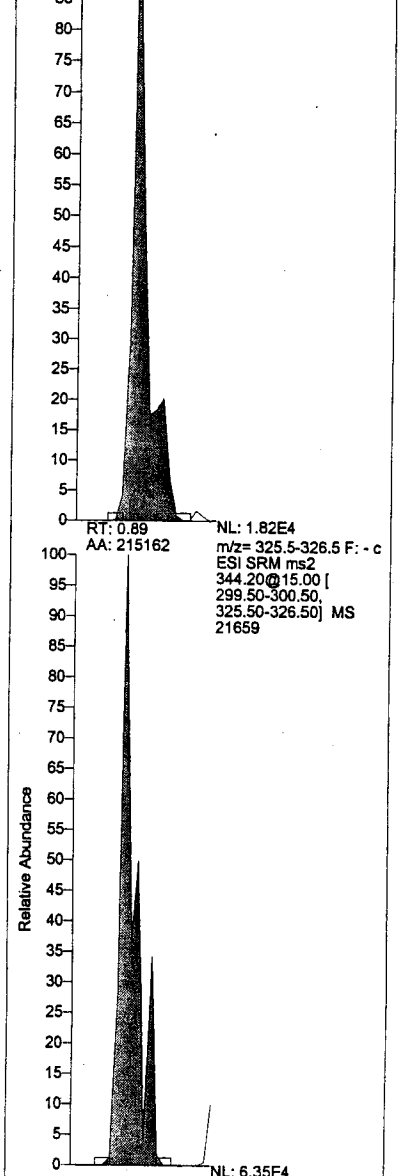
m/z	Intensity	Relative
247.29	2165.3	4.95
273.06	43718.3	100.00





21659#47-59 RT: 0.89-1.12 AV: 4
F: - c ESI SRM ms2 344.20@15.00 [2 ...
m/z= 298.00-326.50

m/z	Intensity	Relative
299.97	114448.8	100.00
326.04	8802.0	7.69

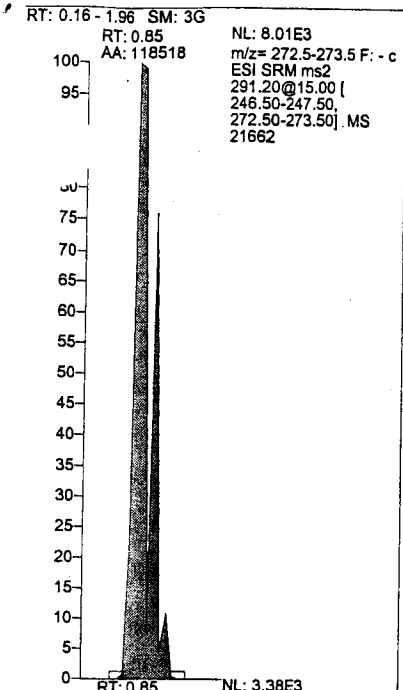


POS

POS

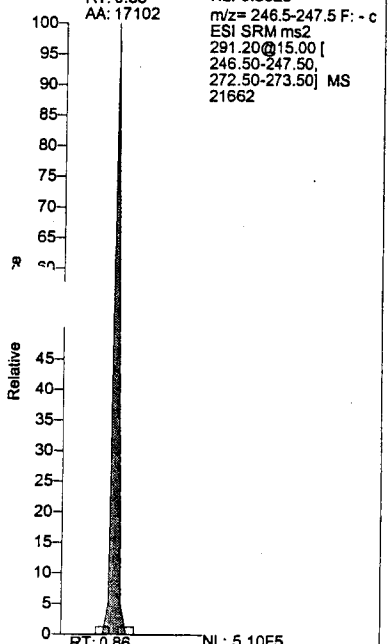
Ex 446
(126)

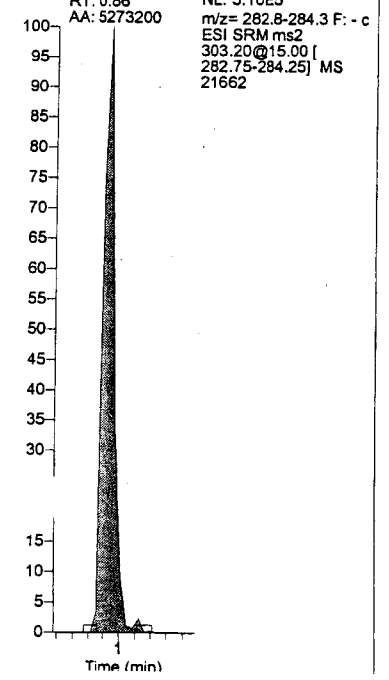
070201013
2/26/07



21662#37-61 RT: 0.69-1.15 AV: 7
F: - c ESI SRM ms2 291.20@15.00 [2 ...
m/z= 246.50-326.50

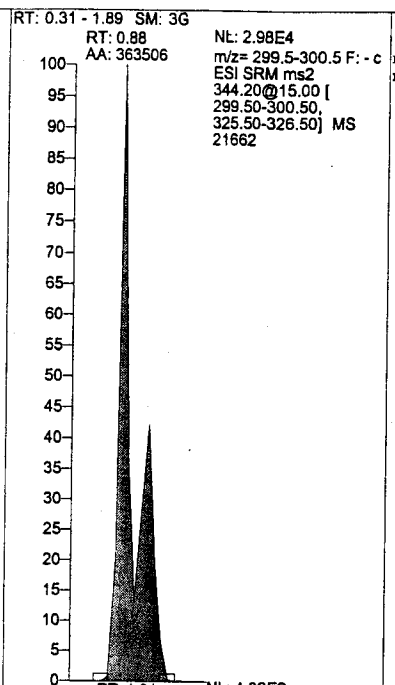
m/z	Intensity	Relative
247.16	531.7	14.98
273.08	3548.4	100.00





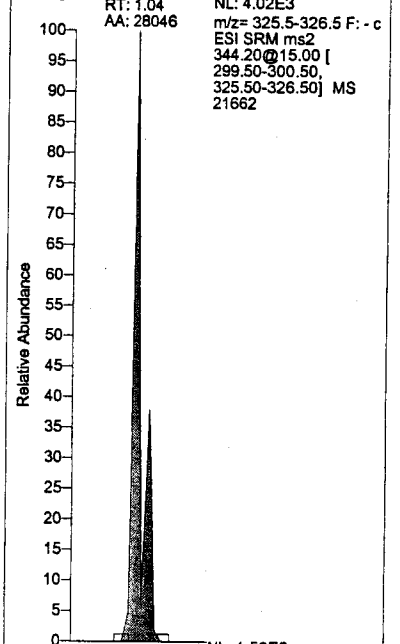
POS

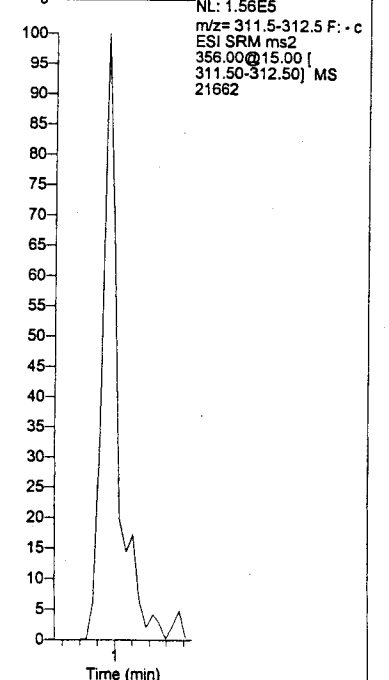
Ex 446
(127)



21662#43-63 RT: 0.81-1.19 AV: 6
F: - c ESI SRM ms2 344.20@15.00 [2 ...
m/z= 298.00-326.50

m/z	Intensity	Relative
299.92	11927.5	100.00
326.13	1017.0	8.53

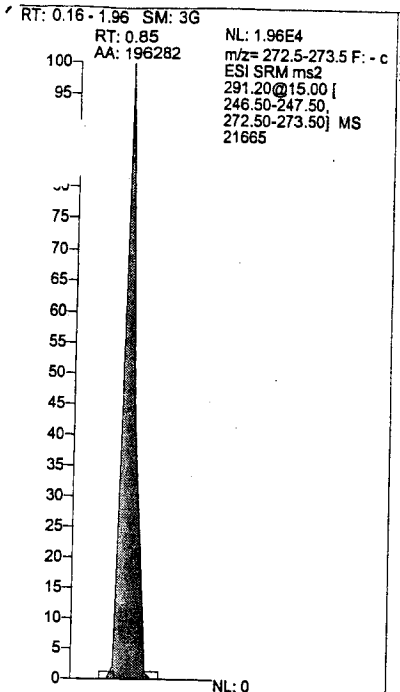




POS

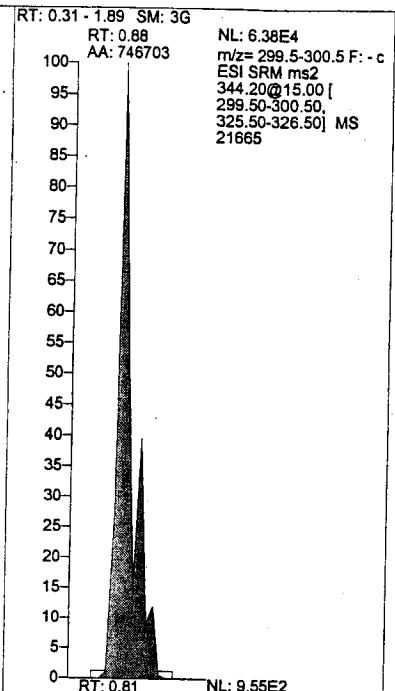
070201017
2/26/07

C:\Xcalibur\...\Negative Ion\21665 02/17/07 12:20:10 Spot LOD, 2 uL Q49



21665#41-49 RT: 0.77-0.92 AV: 3
F: - c ESI SRM ms2 291.20@15.00 [2 ...
m/z= 246.50-326.50

m/z	Intensity	Relative
273.09	12717.7	100.00



21665#39-51 RT: 0.73-0.96 AV: 4
F: - c ESI SRM ms2 344.20@15.00 [2 ...
m/z= 298.00-326.50

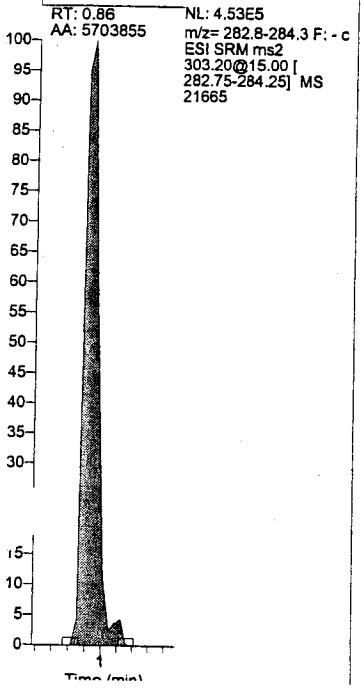
m/z	Intensity	Relative
299.88	28697.5	100.00
326.28	262.5	0.91

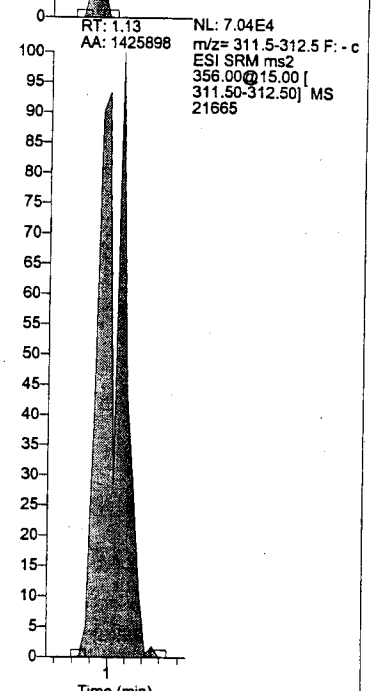
Relative

Relative Abundance

ND ? Weak Inject?

FDS

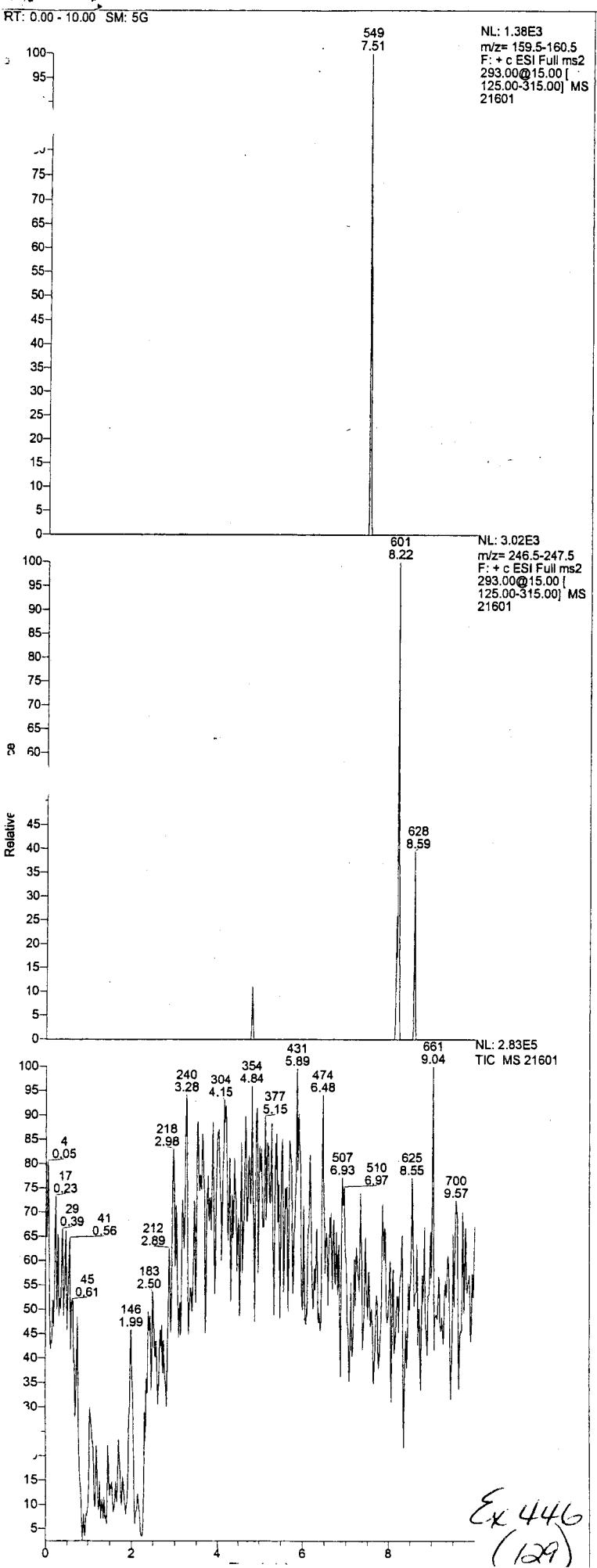




Ex 446 (128)

070201013
2/26/07

C:\Xcalibur\data\Brewer\070201013\21601 JDB 02/16/07 12:03:08 BLANK (neg blood, DI H2O ext.)



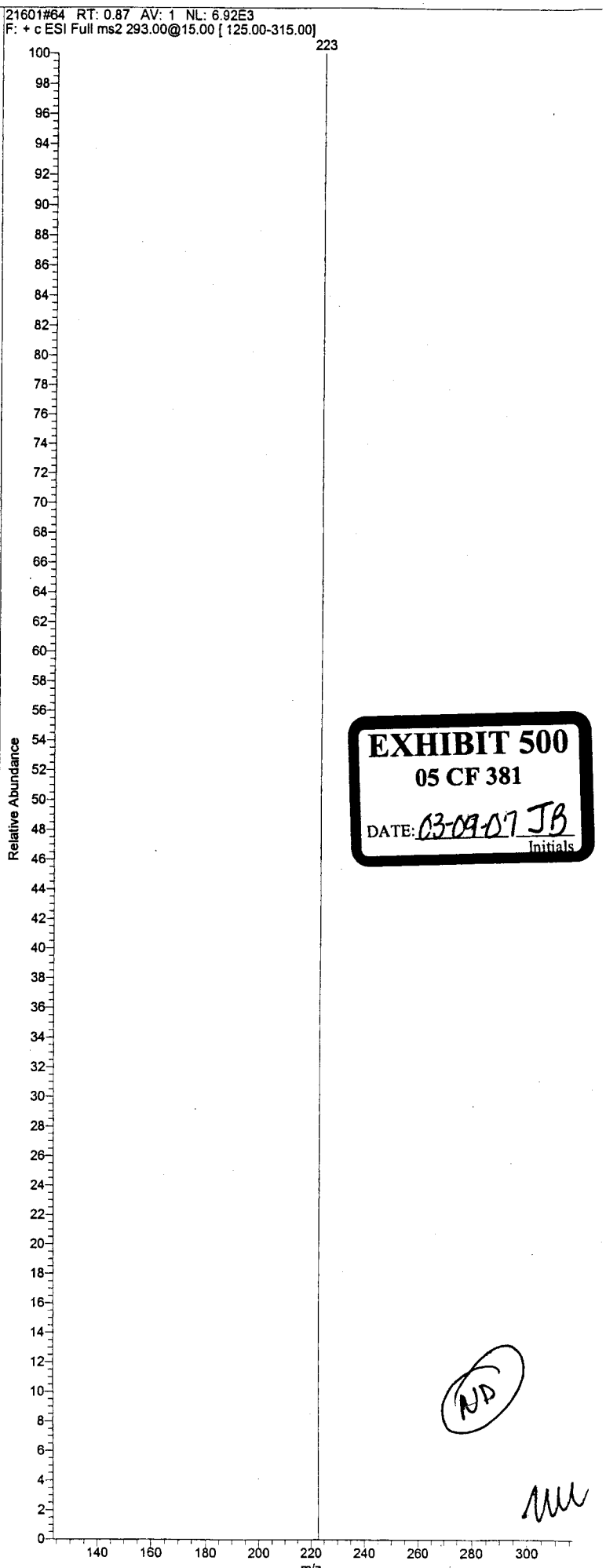


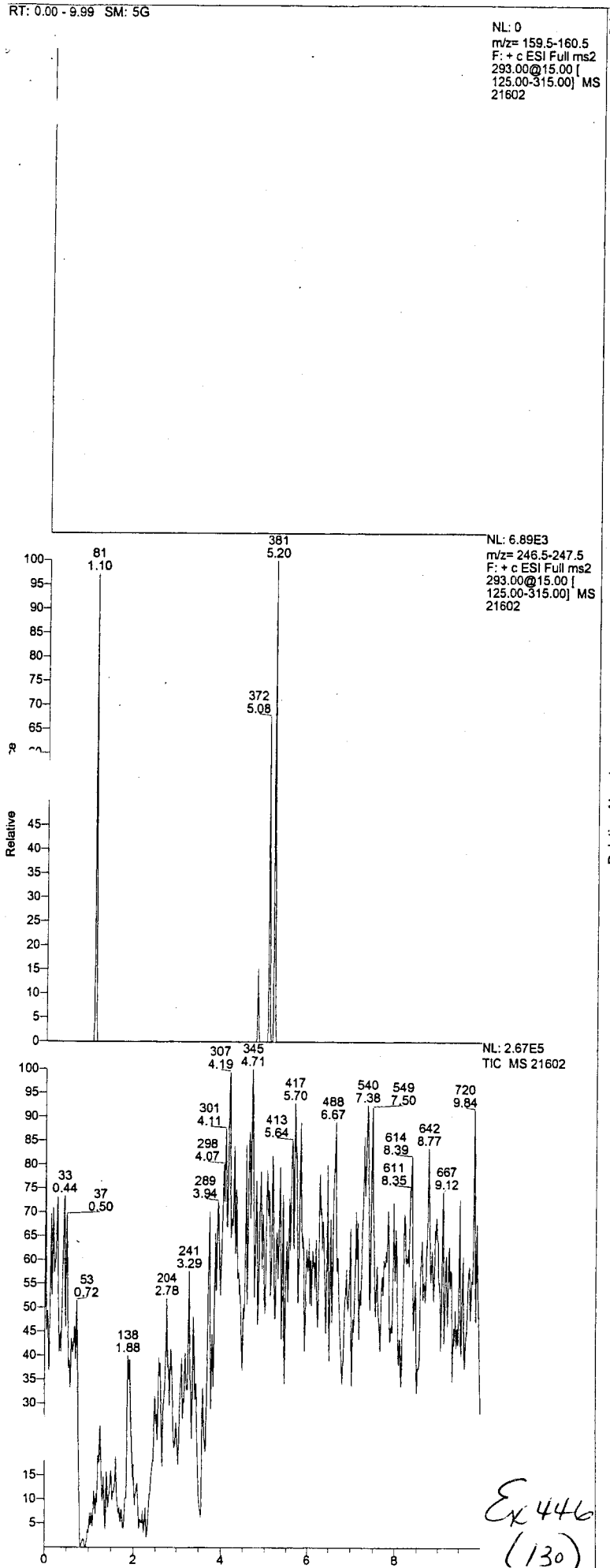
EXHIBIT 500
05 CF 381
DATE: 03-09-07 JB
Initials

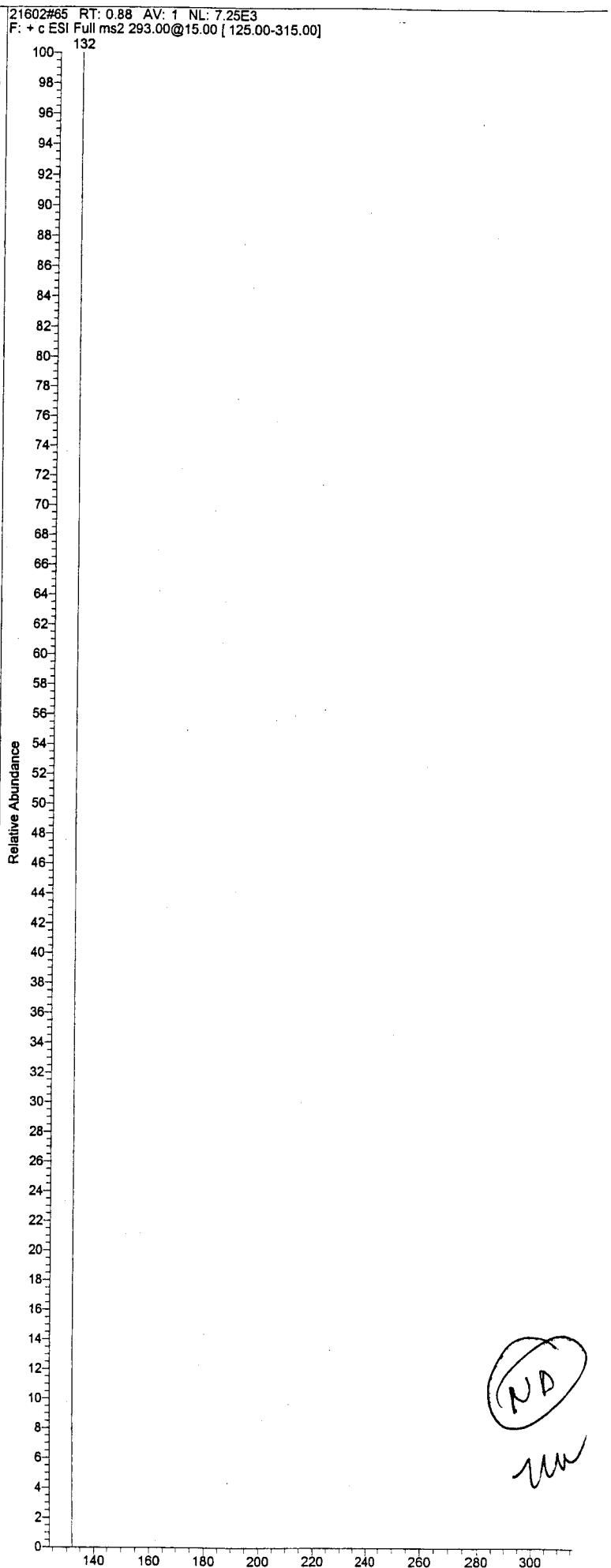
Ex 446
(129)

ND

MW

C:\Xcalibur\data\Brewer\070201013\21602 JDB 02/16/07 12:14:29 Neg. Control (-EDTA blood swab ext.)





Ex 446
(130)

ND

C:\Xcalibur\data\Brewer\070201013\21603 JDB 02/16/07 12:25:21 BLANK (neg blood, DI H2O ext.)

RT: 0.00 - 10.00 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21603

NL: 3.43E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21603

518
7.07
268
3.65
6
0.07
2
0.02

NL: 1.90E5
TIC MS 21603

21
0.28
31
0.41
34
0.46
52
0.70
153
2.08
245
3.34
299
4.08
333
4.54
342
4.67
404
5.51
416
5.68
490
6.69
512
6.99
552
7.54
567
7.74
579
7.91
597
8.15
689
9.41
715
9.77
728
9.94

Relative Abundance

21603#65 RT: 0.88 AV: 1 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

Relative Abundance

Ex 446
(43)

ND

C:\Xcalibur\data\Brewer\070201013\21604 JDB 02/16/07 12:36:16 BLANK (neg blood, DI H2O ext.)

RT: 0.00 - 10.00 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21604

NL: 0
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21604

NL: 1.19E5
TIC MS 21604

Relative Abundance

2 0.02
8 0.10
108 1.47
148 2.02
196 2.67
203 2.77
293 4.00
355 4.85
397 5.42
426 5.82
459 6.27
496 6.77
542 7.40
558 7.62
612 8.36
616 8.41
647 8.84
650 8.88
679 9.27
719 9.82

21604#65 RT: 0.88 AV: 1 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

Relative Abundance

Ex 446
(132)

ND

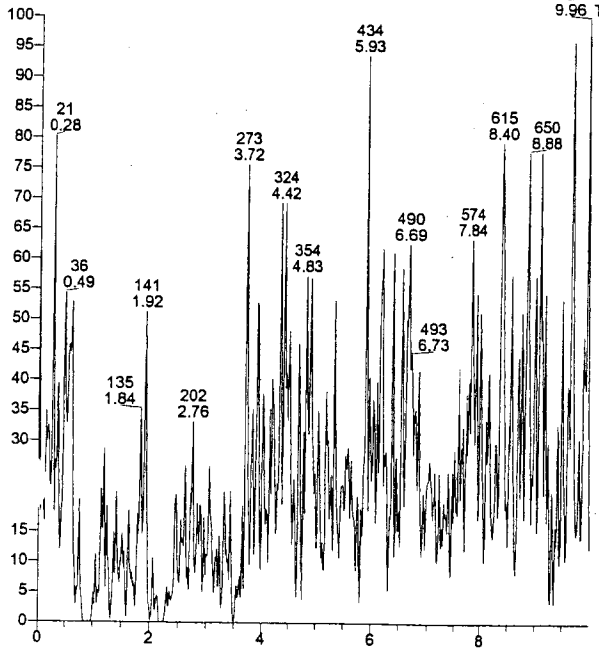
C:\Xcalibur\data\Brewer\07020101\3\21605 JDB 02/16/07 12:47:04 K2 extract

RT: 0.00 - 10.00 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21605

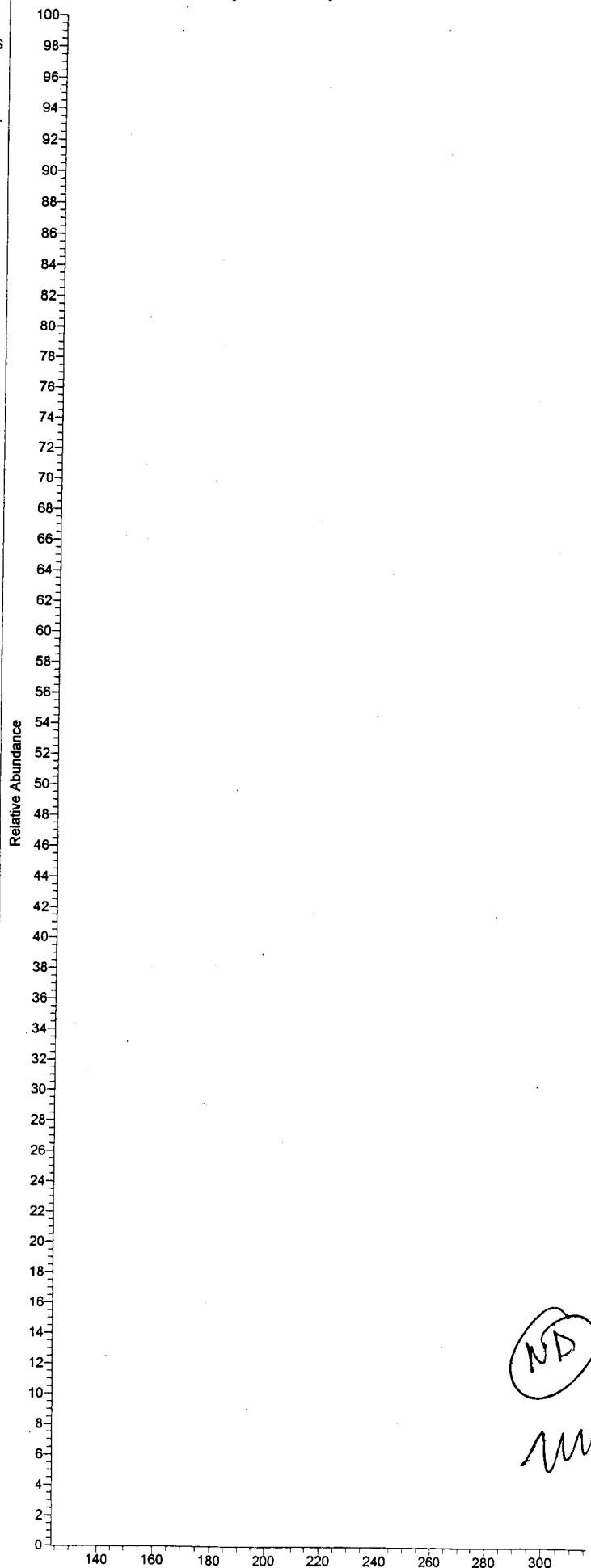
NL: 0
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21605

729 NL: 9.45E4
9.96 TIC MS 21605



Relative Abundance

21605#65 RT: 0.88 AV: 1 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



Ex 446
(133)

ND

C:\Xcalibur\data\Brewer\070201013\21606 JDB 02/16/07 12:57:54 BLANK (neg blood, DI H2O ext.)

RT: 0.00 - 9.99 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21606

504
6.88

NL: 2.81E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21606

33
0.44

621
8.48

55
0.74

NL: 2.27E5
TIC MS 21606

Relative Abundance

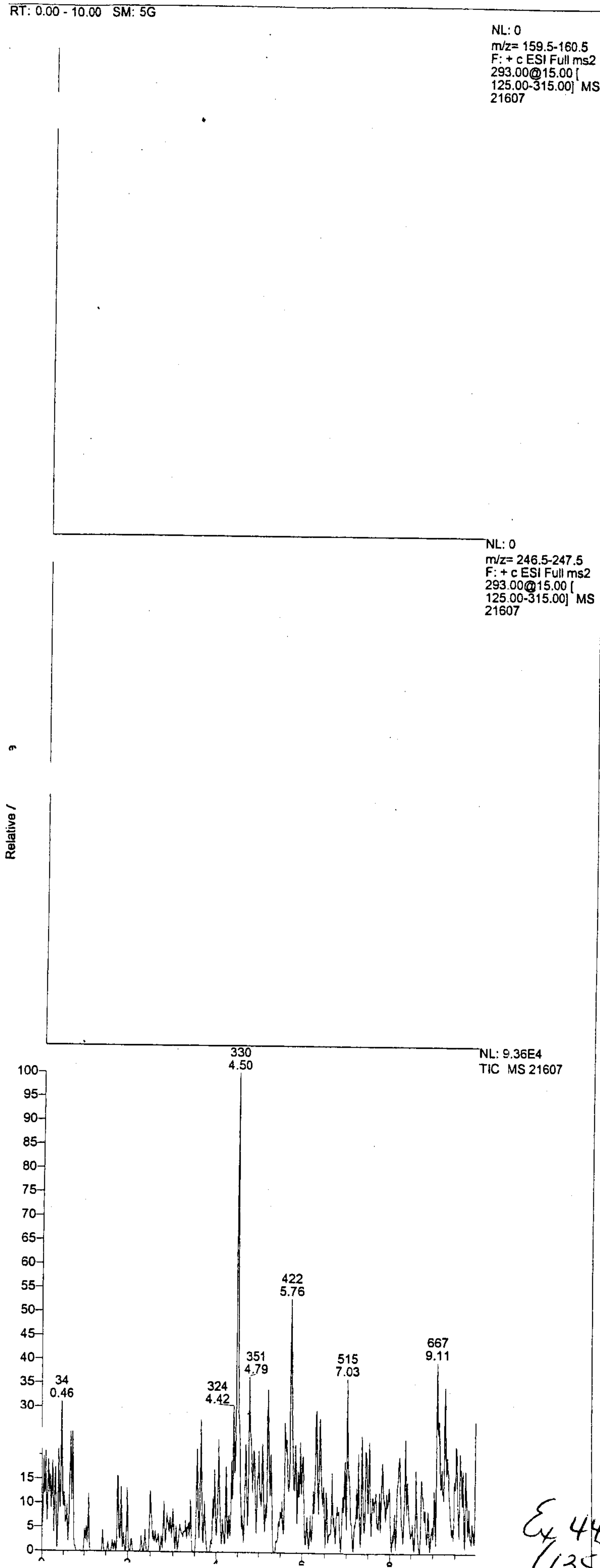
21606#65 RT: 0.88 AV: 1 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

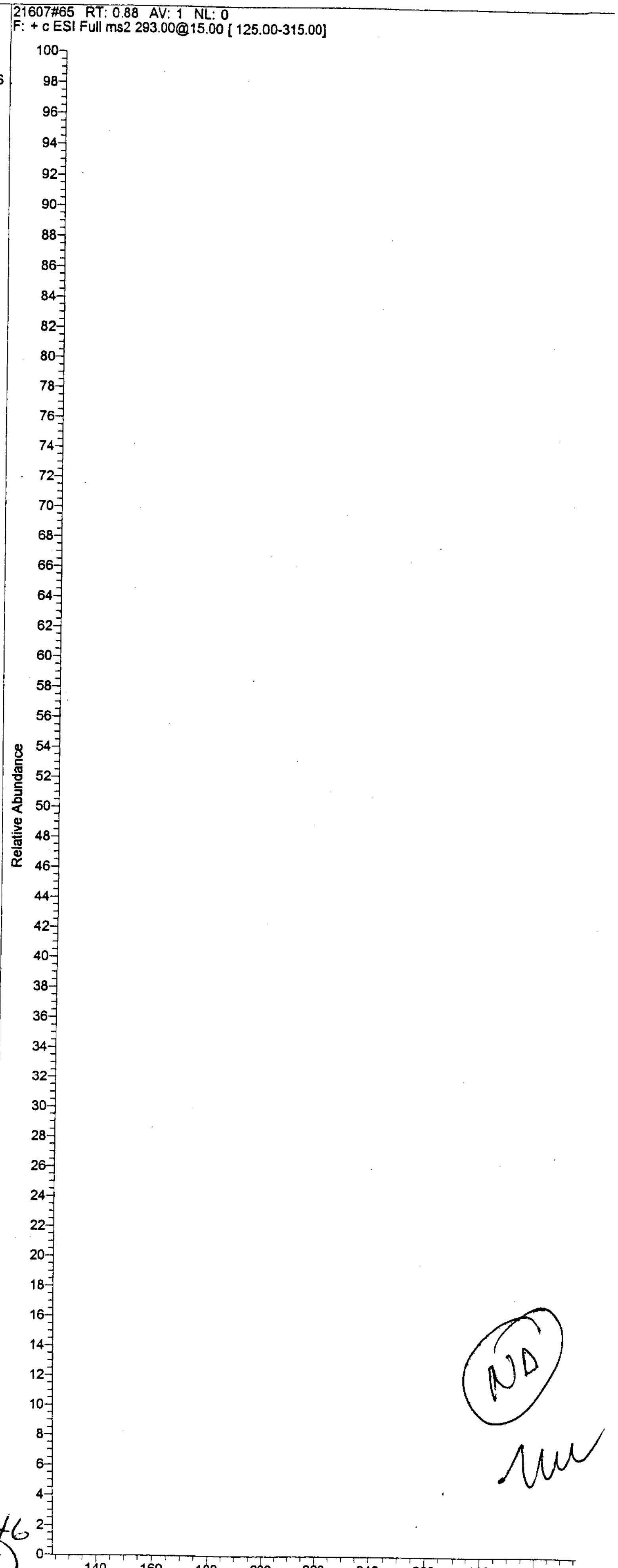
Relative Abundance

ND

Ex 446
(134)

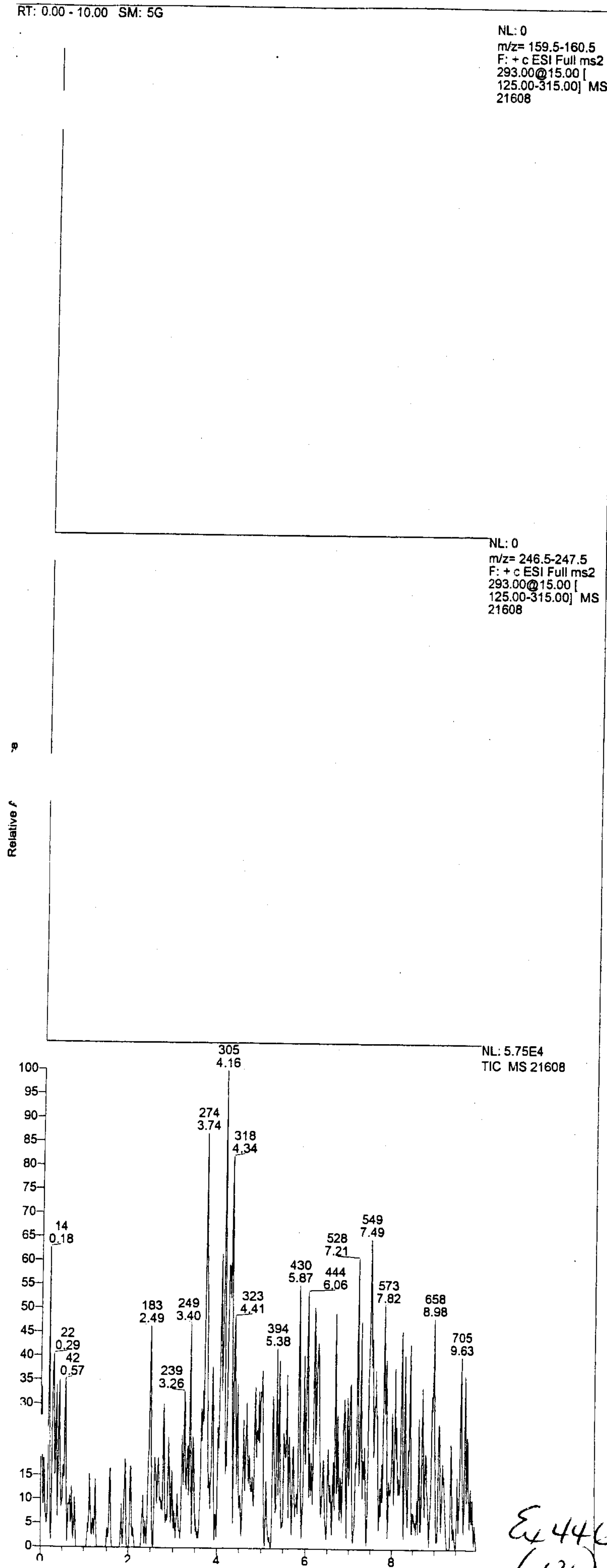
C:\Xcalibur\data\Brewer\070201013\21607 JDB 02/16/07 01:08:51 BLANK (neg blood, DI H2O ext.)

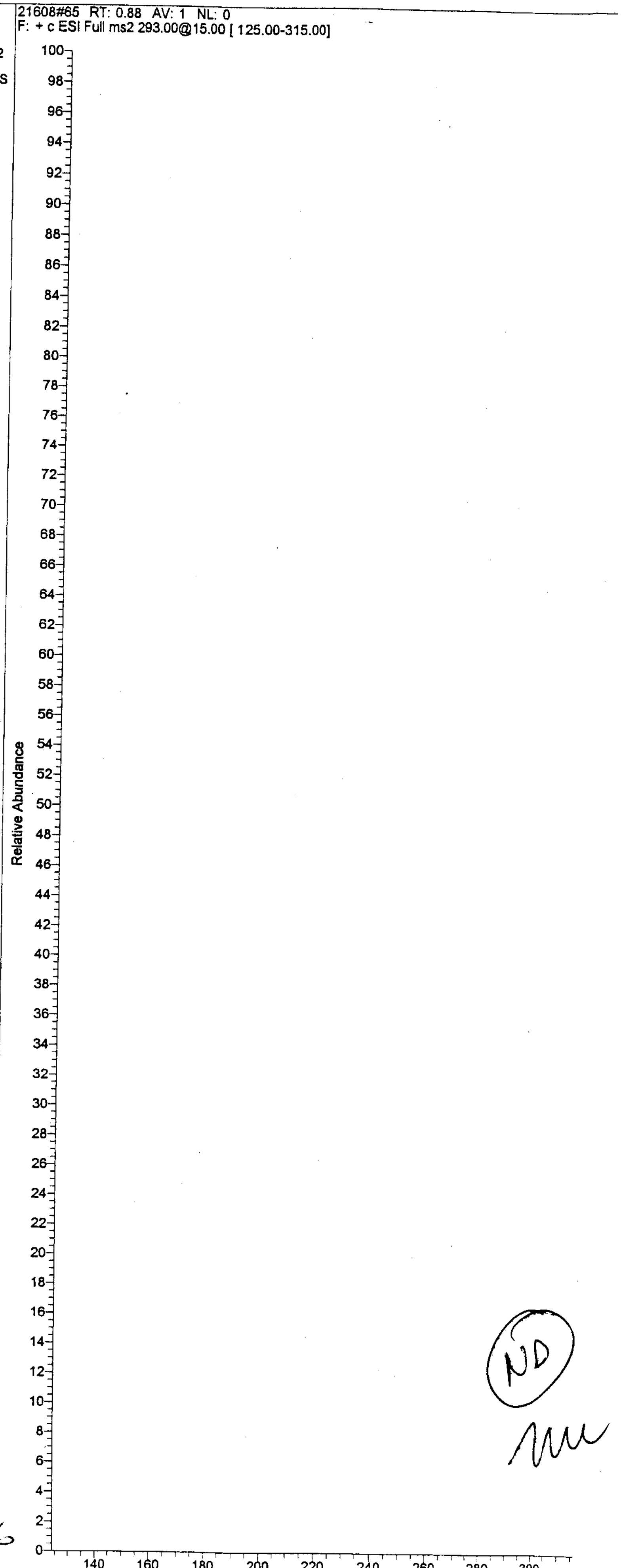




Ex 446
(135)

C:\Xcalibur\data\Brewer\070201013\21608 JDB 02/16/07 01:19:41 Q46 extract





Ex 446

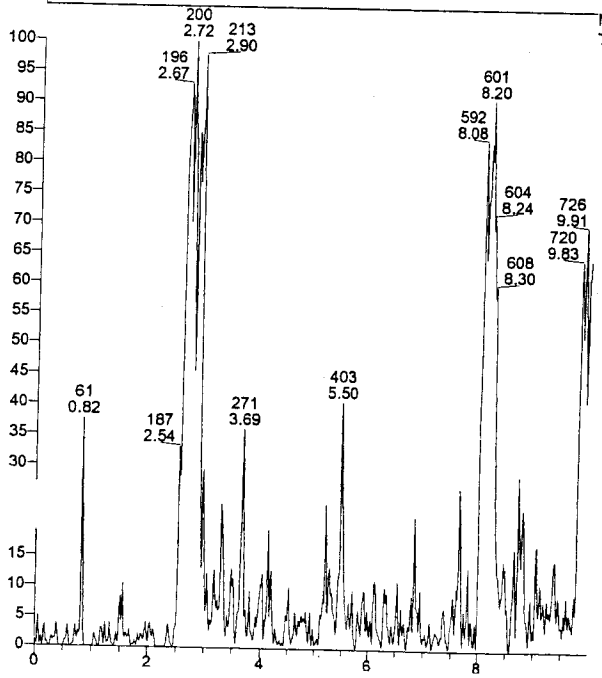
C:\Xcalibur\data\Brewer\070201013\21609 JDB 02/16/07 01:30:31 BLANK (neg blood, DI H2O ext.)

RT: 0.00 - 9.99 SM: 5G

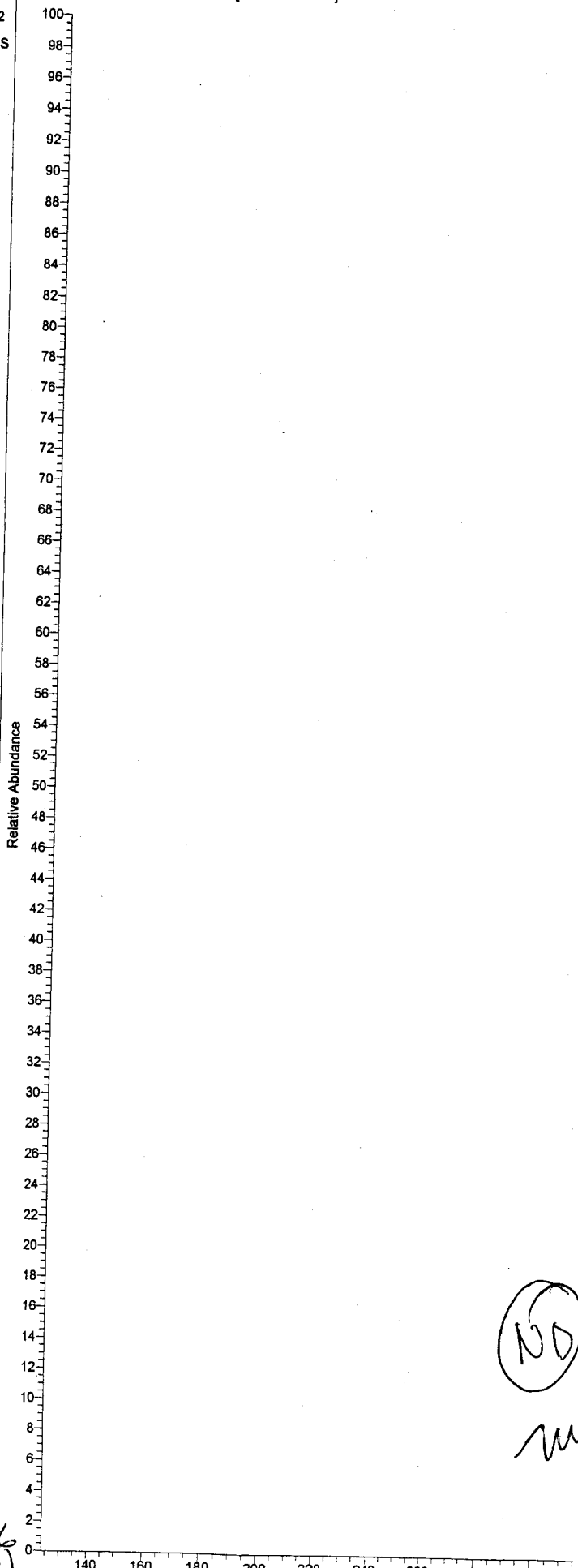
NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21609

NL: 0
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21609

NL: 3.02E5
TIC MS 21609



21609#65 RT: 0.88 AV: 1 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



ND

Ex 446
(437)

C:\Xcalibur\data\Brewer\070201013\21610 JDB 02/16/07 01:41:28 BLANK (neg blood, DI H2O ext.)

RT: 0.00 - 9.99 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21610

NL: 4.11E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21610

23
0.31
354
4.83
365
4.99
450
6.15
597
8.16

Relative

NL: 3.16E5
TIC MS 21610

6
0.08
16
0.21
30
0.40
38
0.51
55
0.75
186
2.54
209
2.85
219
2.99
225
3.07
283
3.86
291
3.97
308
4.21
364
4.97
379
5.18
476
6.50
515
7.04
522
7.13
599
8.19
634
8.67
658
9.00
701
9.58

21610#65 RT: 0.88 AV: 1 NL: 6.32E3
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

159

Relative Abundance

ND

Ex 44

C:\Xcalibur\data\Brewer\070201013\21611 TDB 02/16/07 01:52:18 K3 extract

RT: 0.00 - 9.99 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21611

NL: 4.71E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21611

526
7.19

41
0.55

NL: 3.13E5
TIC MS 21611

14
0.18
21
0.28
34
0.46
202
2.75
220
3.00
270
3.68
303
4.14
345
4.71
369
5.04
375
5.12
433
5.91
497
6.79
508
6.94
556
7.60
587
8.02
660
9.02
711
9.72

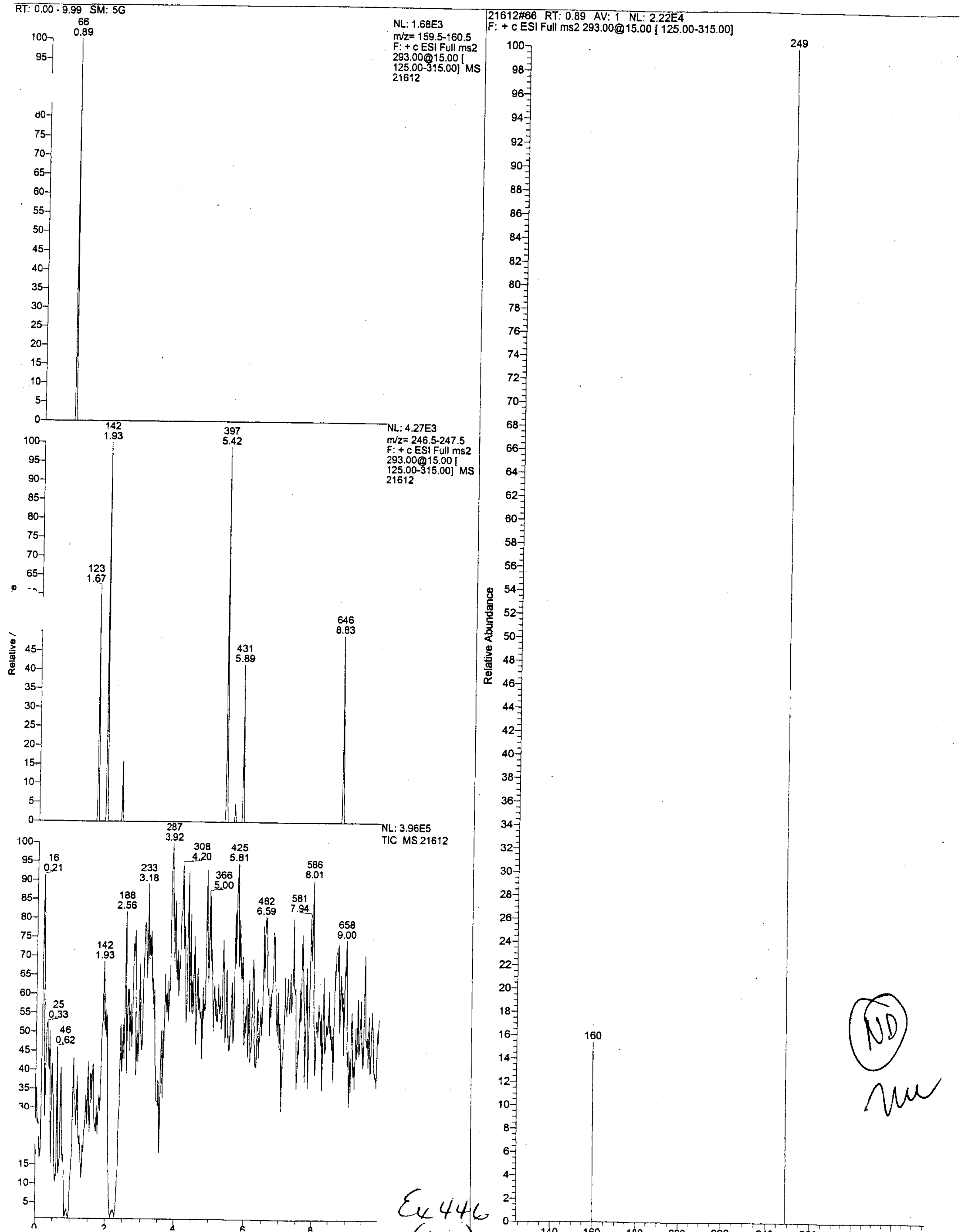
Relative Abundance

21611#65 RT: 0.88 AV: 1 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

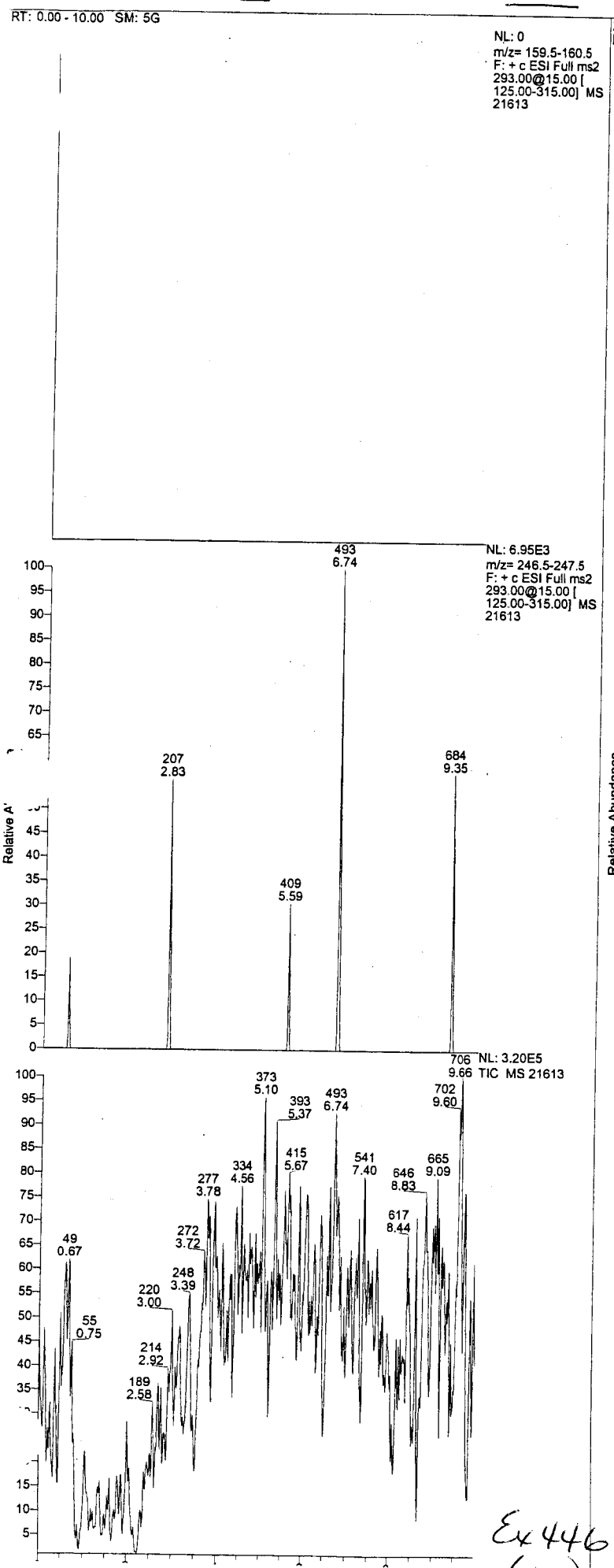
ND

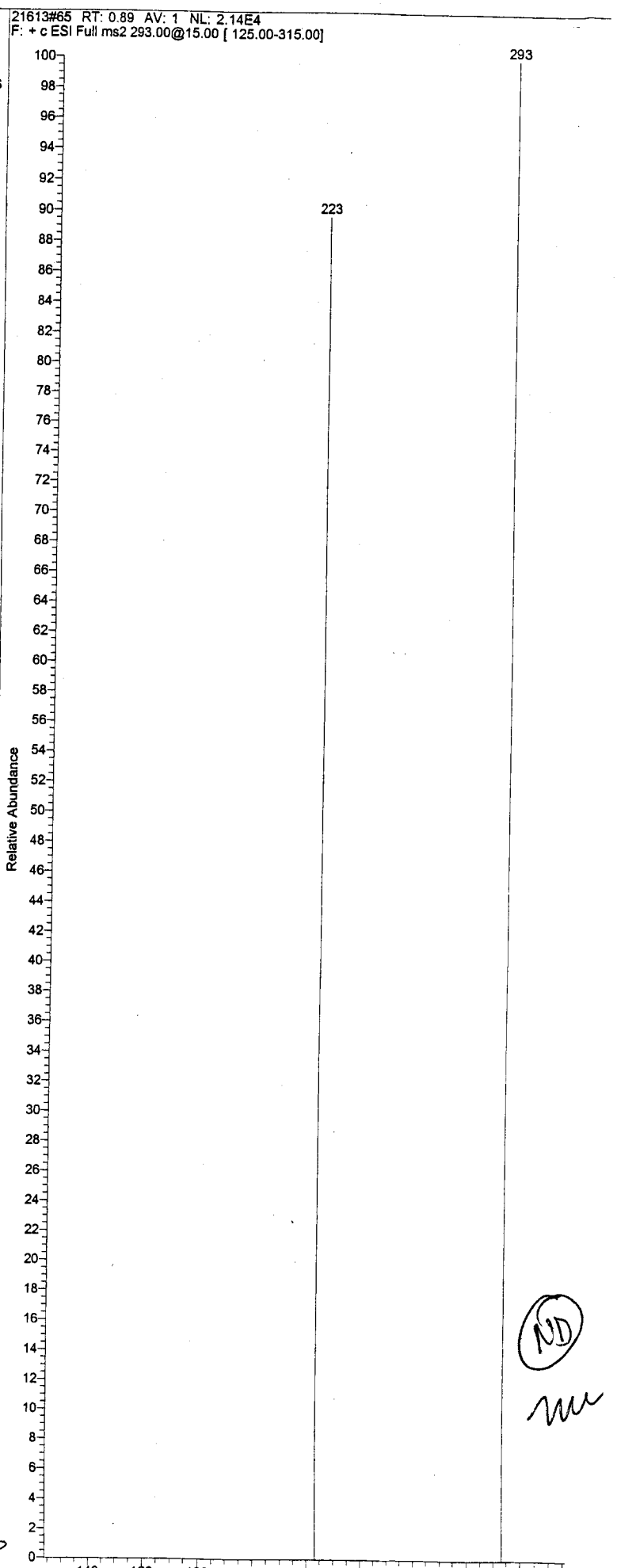
Ex 446
(430)

C:\Xcalibur\data\Brewer\070201013\21612 Job 02/16/07 02:03:10 BLANK (neg blood, DI H2O ext.)



C:\Xcalibur\data\Brewer\070201013\21613 JDB 02/16/07 02:14:01 BLANK (neg blood, DI H2O ext.)





Ex 446

ND

C:\Xcalibur\data\Brewer\070201013\21614 JDB 02/16/07 02:24:52 Q47 extract

RT: 0.00 - 9.99 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21614

NL: 7.94E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21614

300
4.09
163
2.22
250
3.41
323
4.41
426
5.82

NL: 3.86E5
TIC MS 21614

337
4.60
419
5.72
454
6.20
468
6.39
347
4.74
312
4.26
286
3.90
475
6.49
508
6.94
236
3.22
572
7.82
220
3.00
652
8.91
34
0.46
593
8.11
214
2.92
41
0.55
698
9.54
133
1.81

Relative Abundance

21614#65 RT: 0.88 AV: 1 NL: 5.95E3
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

275

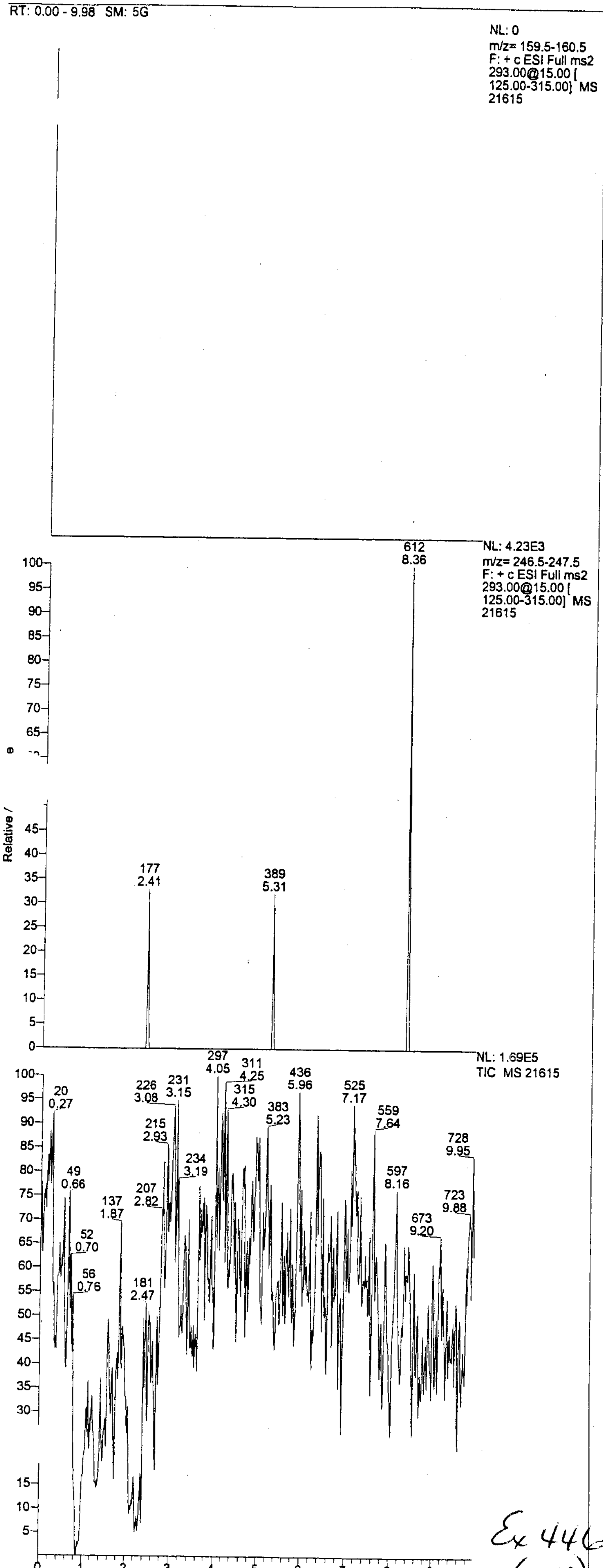
Relative Abundance

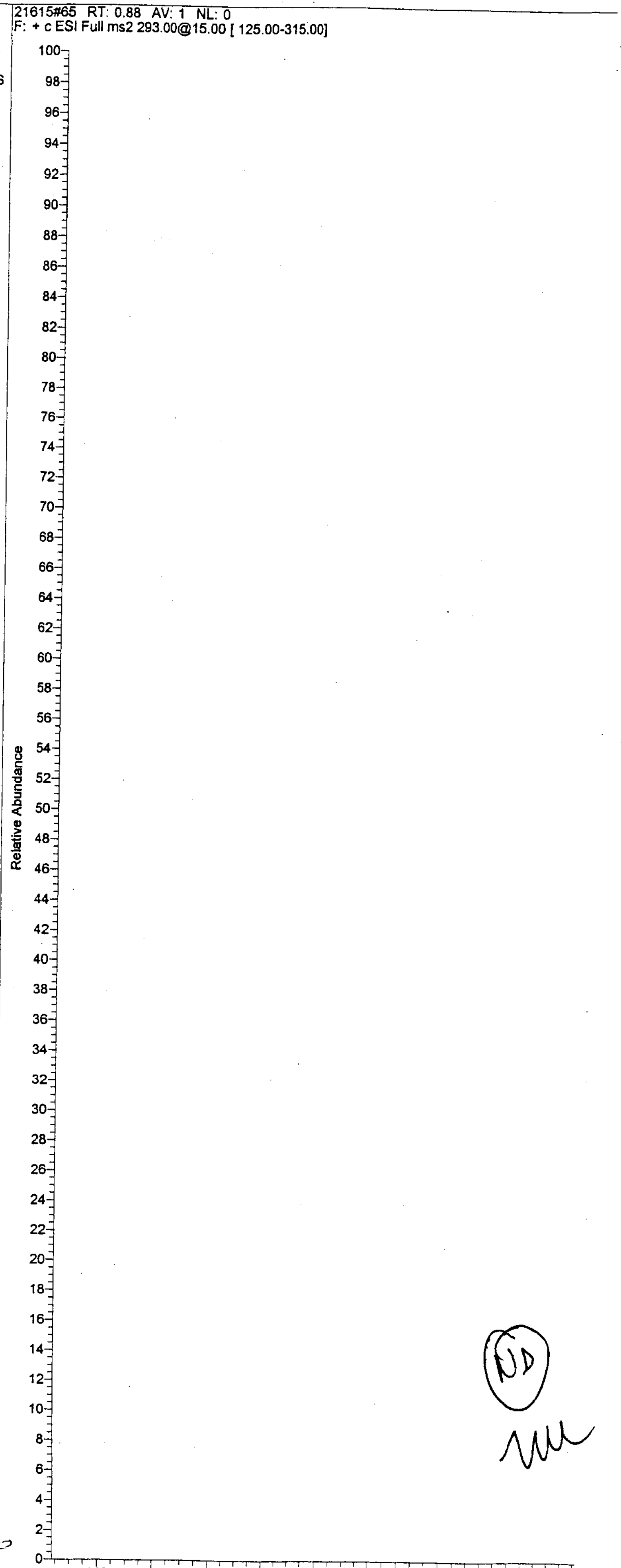
m/z

Ex 446

ND

C:\Xcalibur\data\Brewer\070201013\21615 JDβ 02/16/07 02:35:44 BLANK (neg blood, DI H2O ext.)

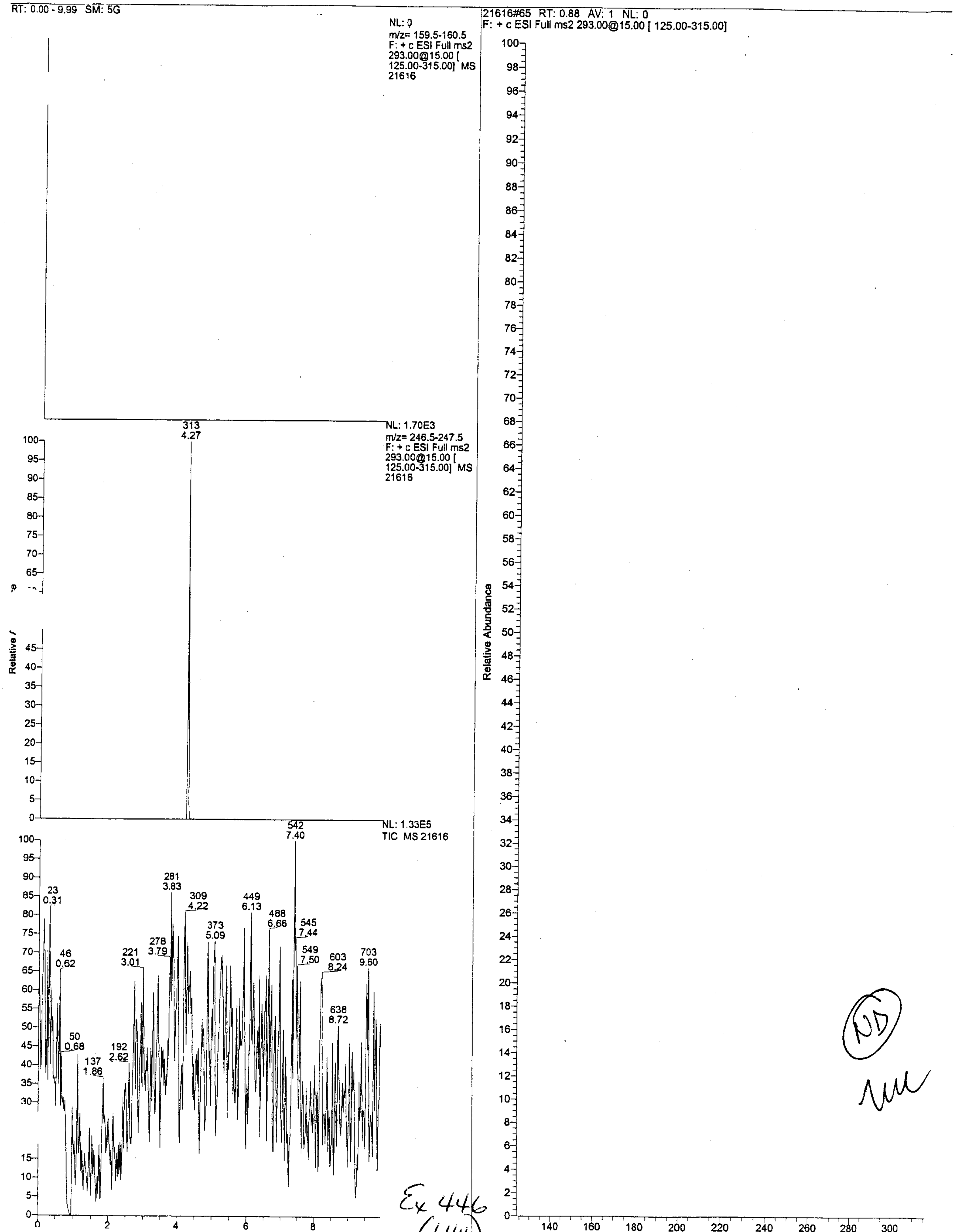




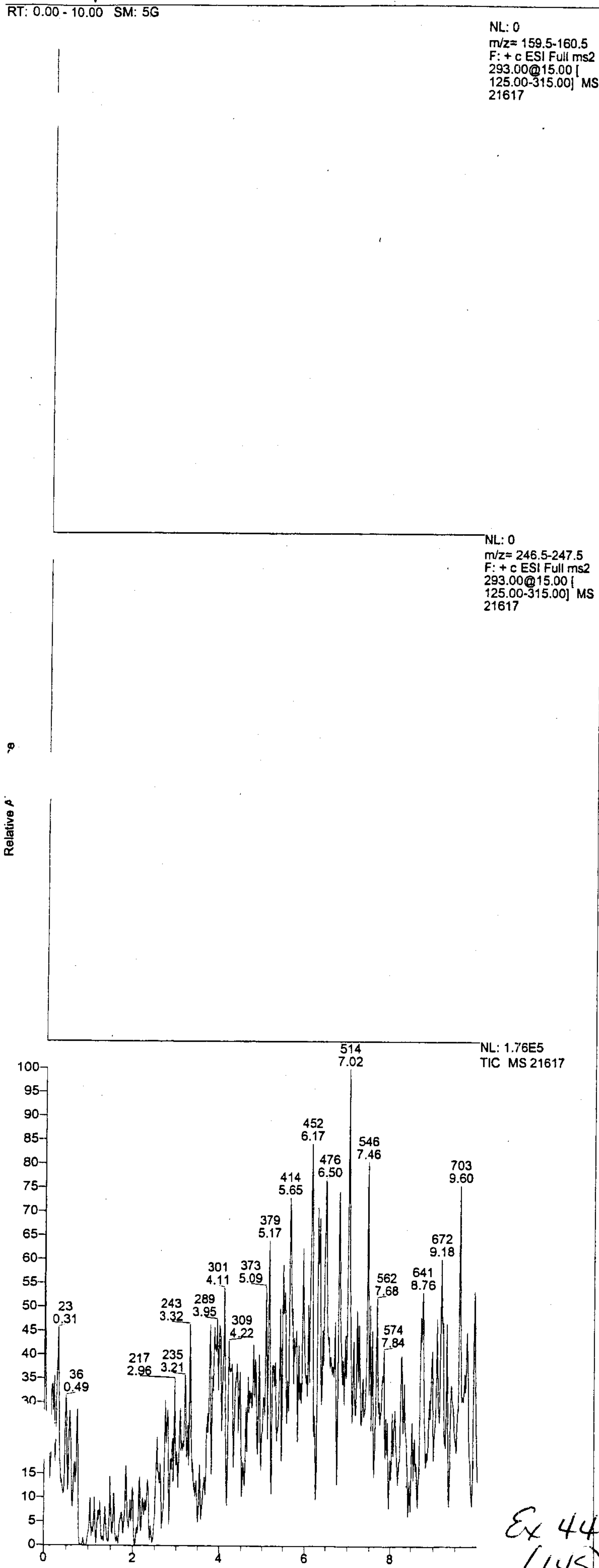
Ex 446

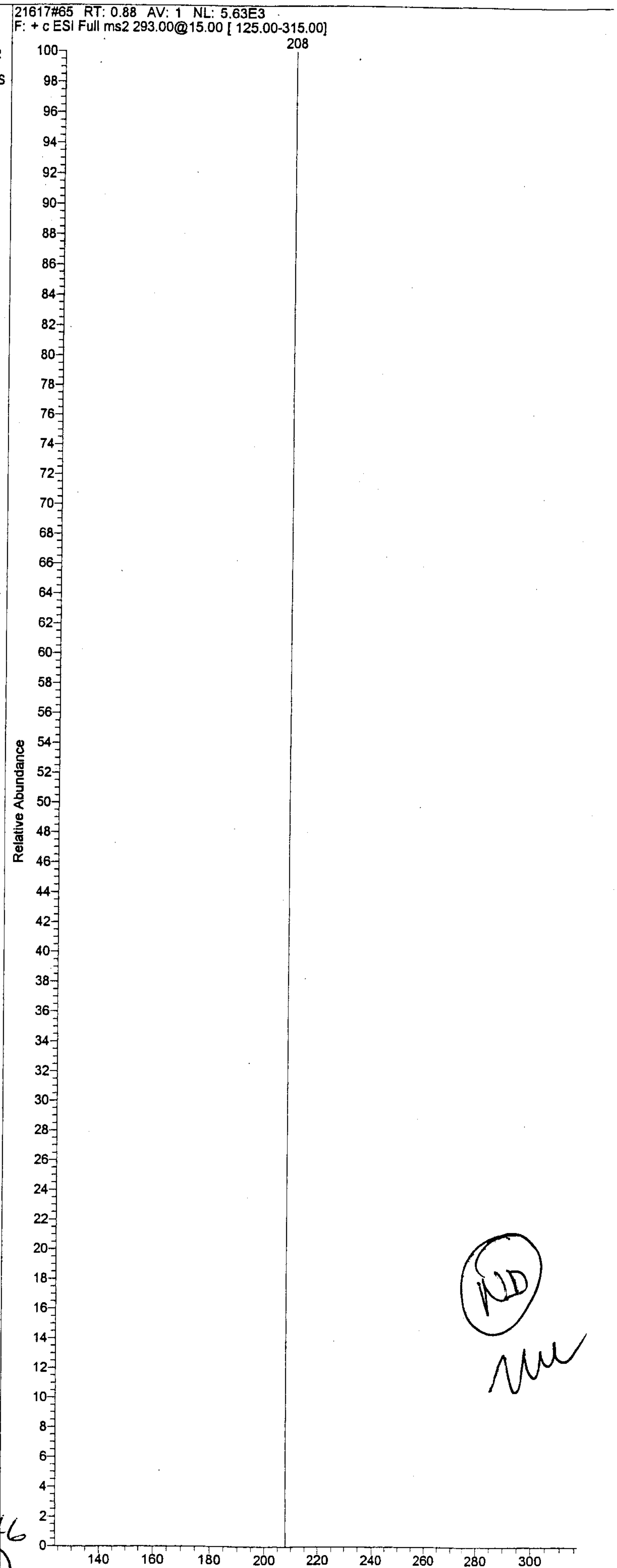
ND

C:\Xcalibur\data\Brewer\070201013\21616 Jbβ 02/16/07 02:46:33 BLANK (neg blood, DI H2O ext.)



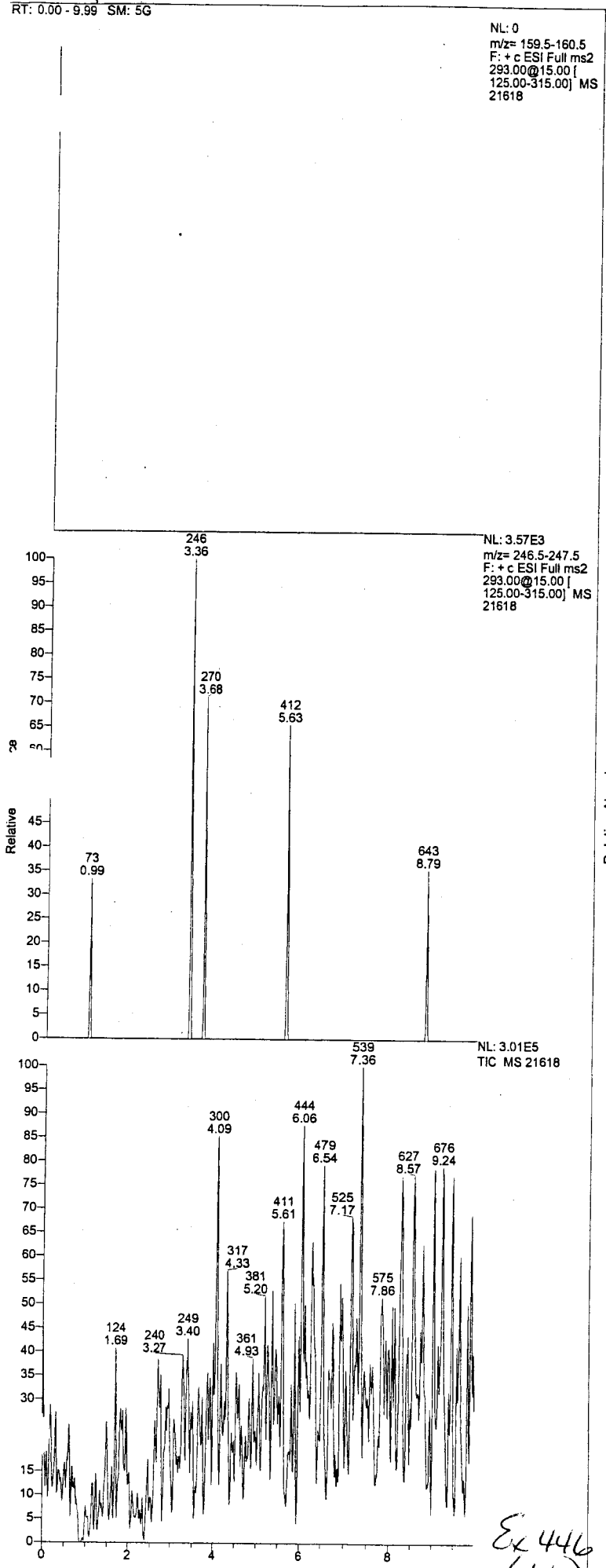
C:\Xcalibur\data\Brewer\070201013\21617 Jvβ 02/16/07 02:57:25 K4 extract

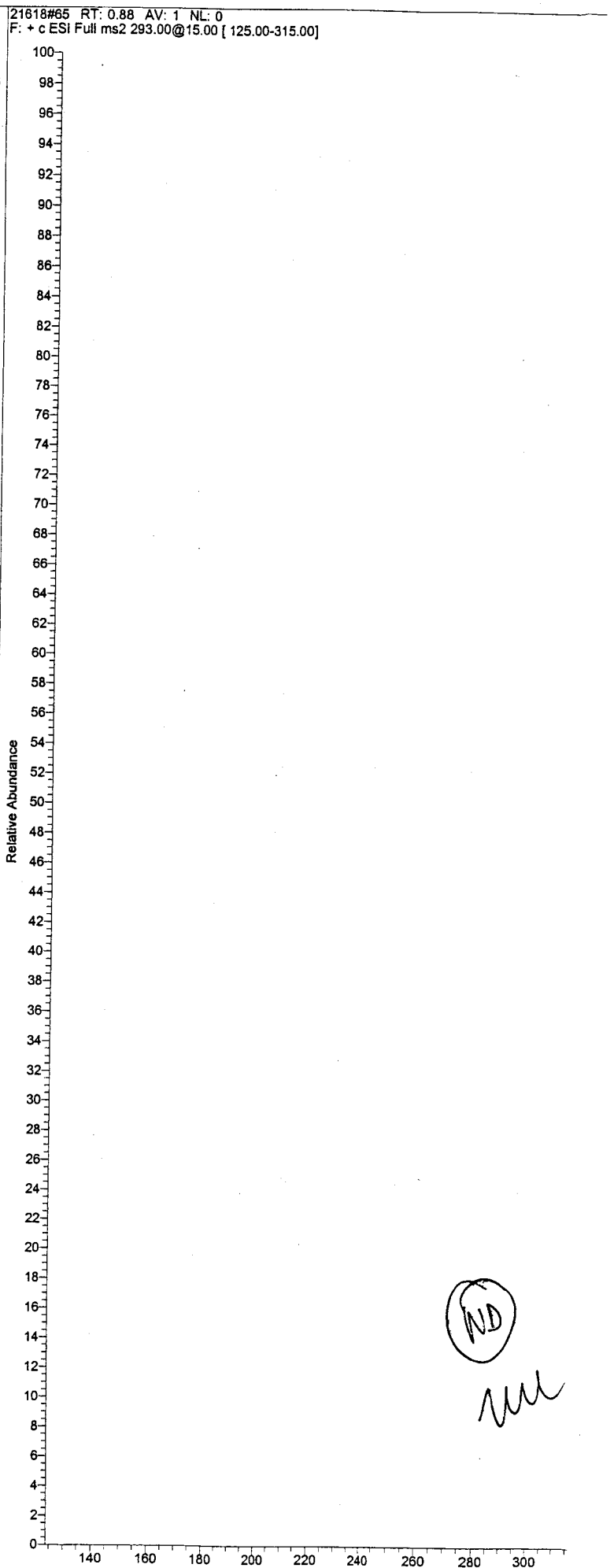




Ex 446 (145)

C:\Xcalibur\data\Brewer\070201013\21618 JDB 02/16/07 03:08:17 BLANK (neg blood, DI H2O ext.)





Ex 446

ND

C:\Xcalibur\data\Brewer\070201013\21619 JNB 02/16/07 03:19:12 BLANK (neg blood, DI H2O ext.)

RT: 0.00 - 9.98 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21619

NL: 6.69E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21619

382
5.22
443
6.05
611
8.35

Relative Abundance

NL: 3.77E5
TIC MS 21619

519
7.09
428
5.85
569
7.78
508
6.94
697
9.53
292
3.99
319
4.36
242
3.31
397
5.43
214
2.92
332
4.54
693
9.47
614
8.39
192
2.62
182
2.48
11
0.15
29
0.39
41
0.56
57
0.78
150
2.05

21619#64 RT: 0.87 AV: 1 NL: 1.35E4
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

290
221

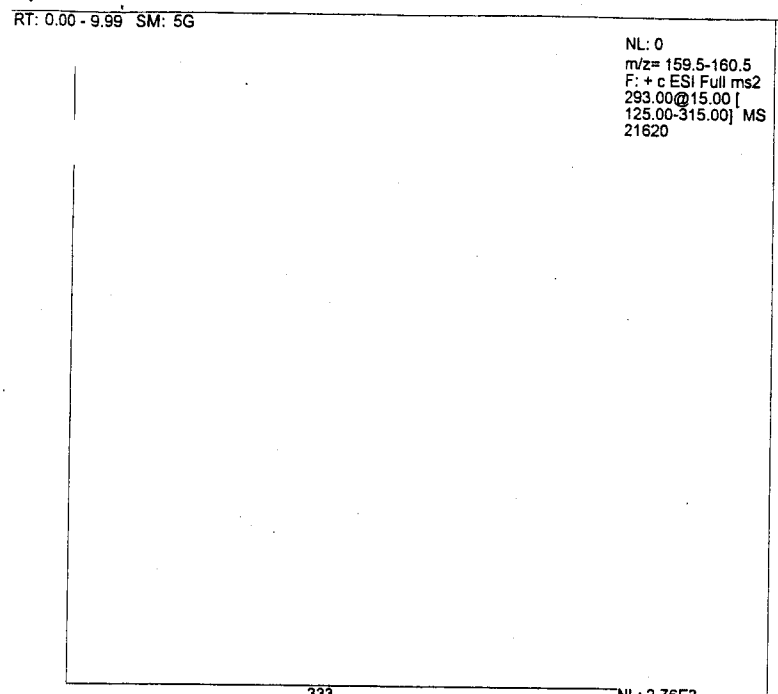
Relative Abundance

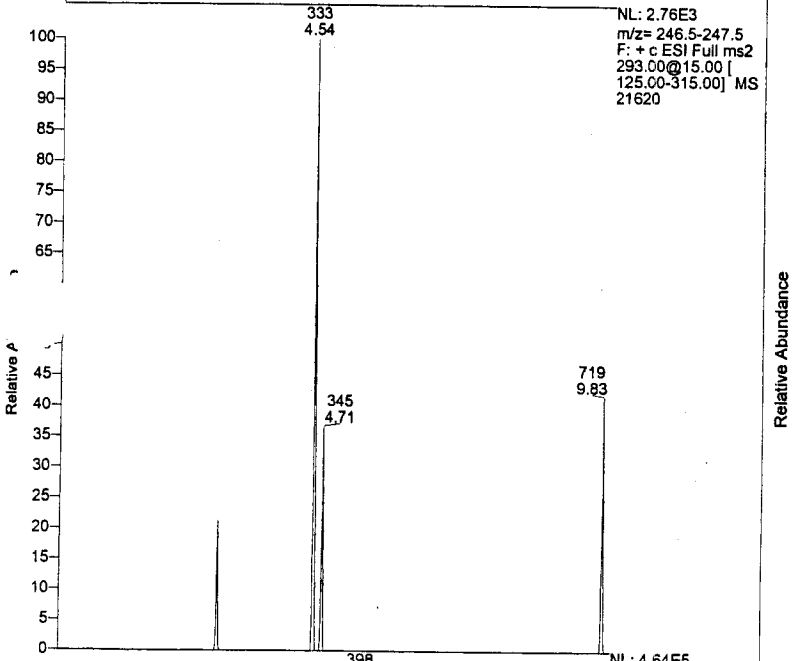
ND

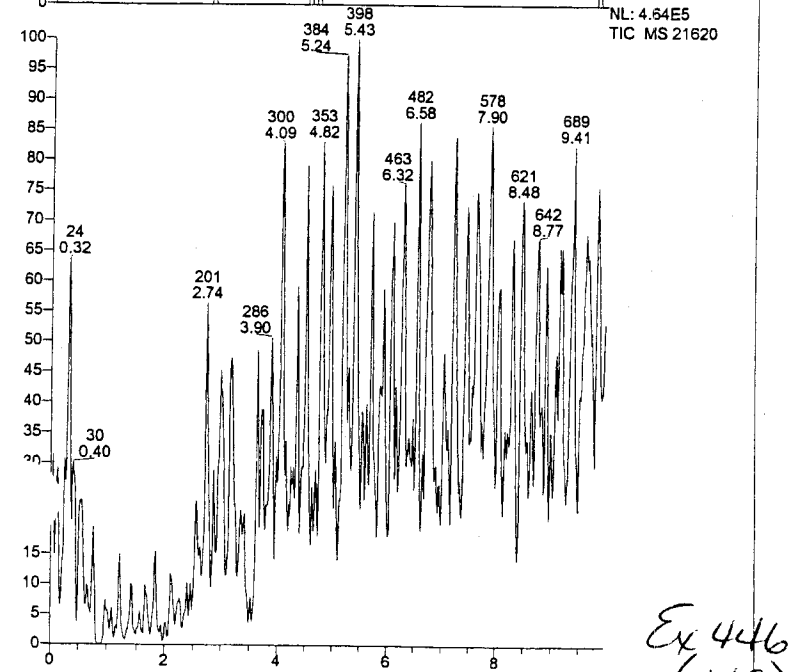
Ex 446

C:\Xcalibur\data\Brewer\070201013\21620 JDB 02/16/07 03:29:57 Q48 extract

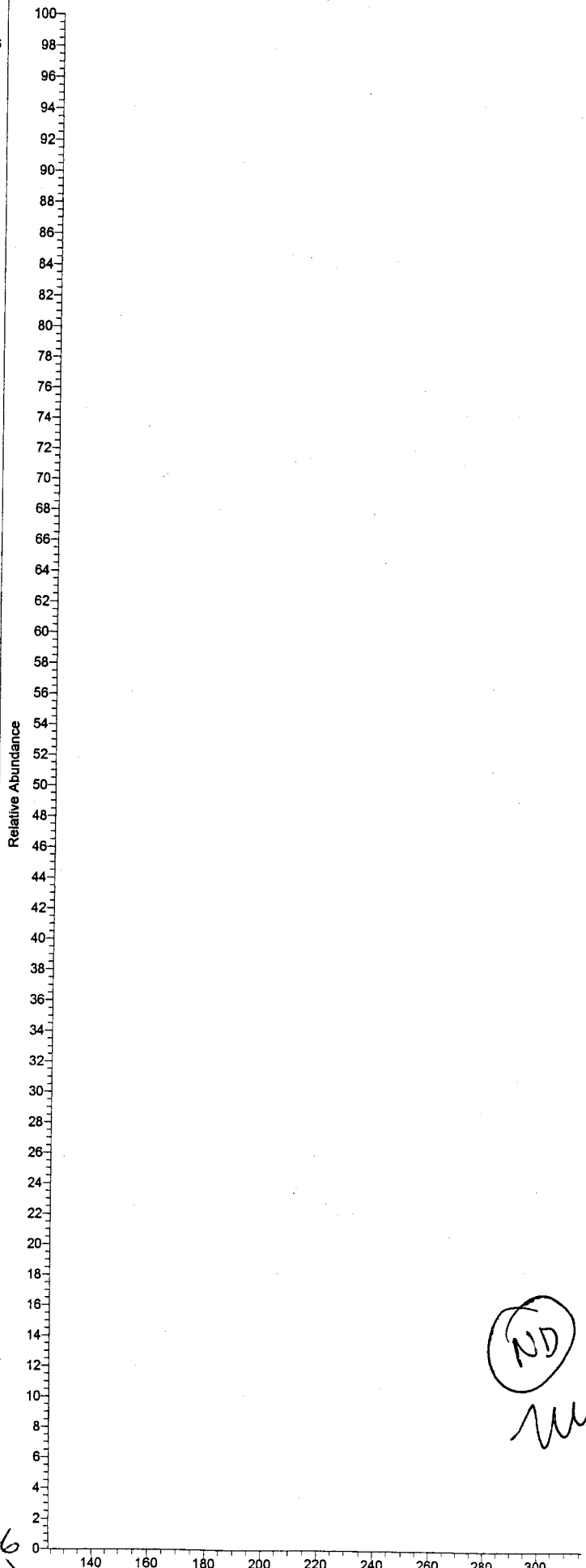
RT: 0.00 - 9.99 SM: 5G







21620#65 RT: 0.88 AV: 1 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



ND

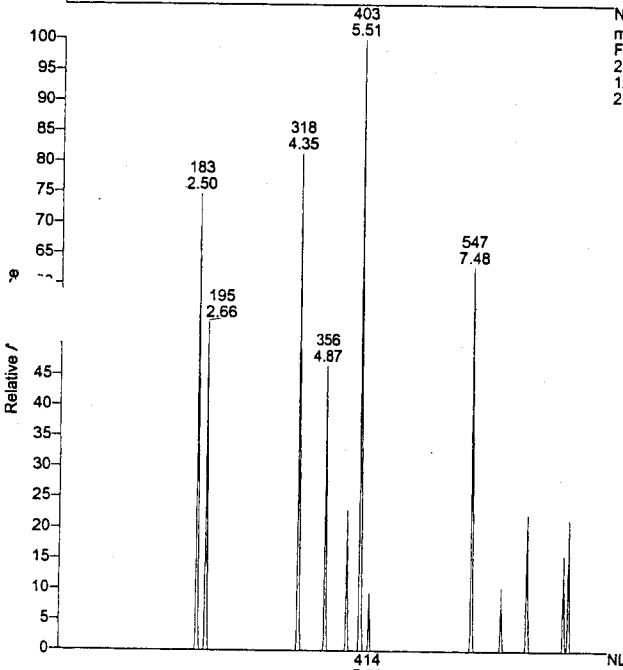
Ex 446

C:\Xcalibur\data\Brewer\070201013\21621 JD 02/16/07 03:40:49 BLANK (neg blood, DI H2O ext.)

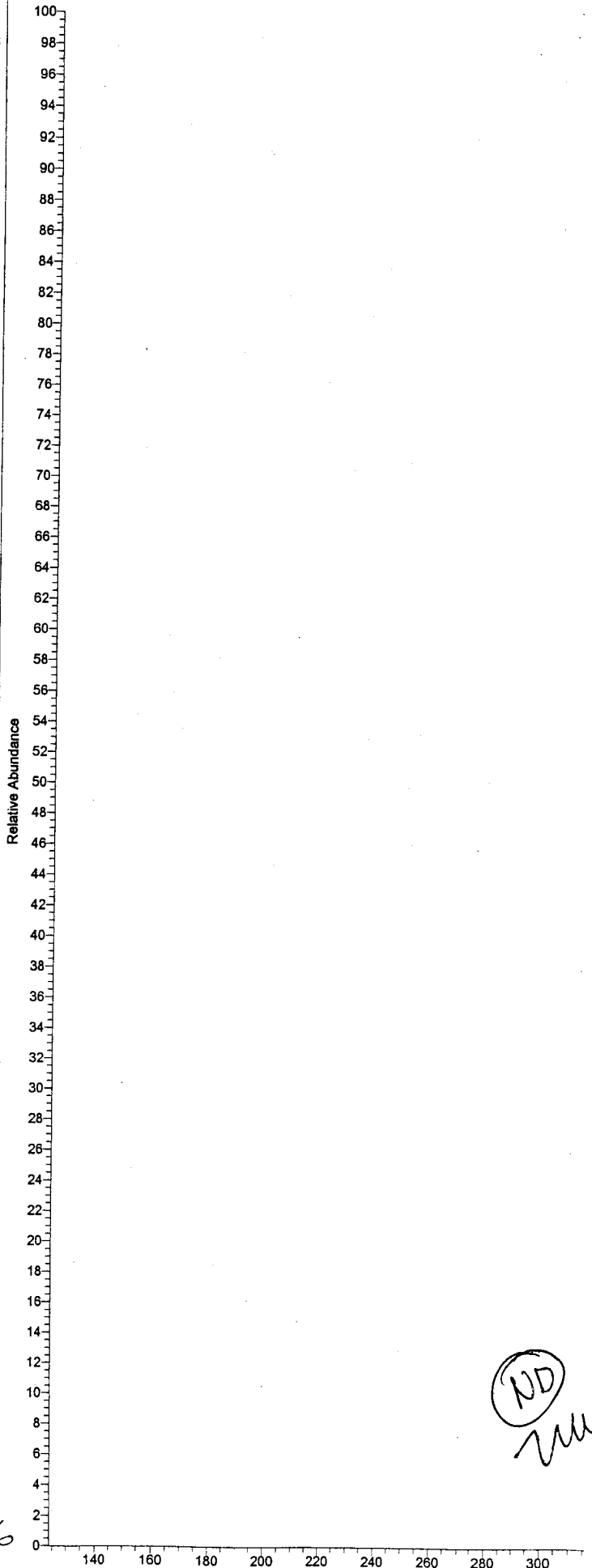
RT: 0.00 - 10.00 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21621

NL: 7.75E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21621



21621#65 RT: 0.88 AV: 1 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



ND

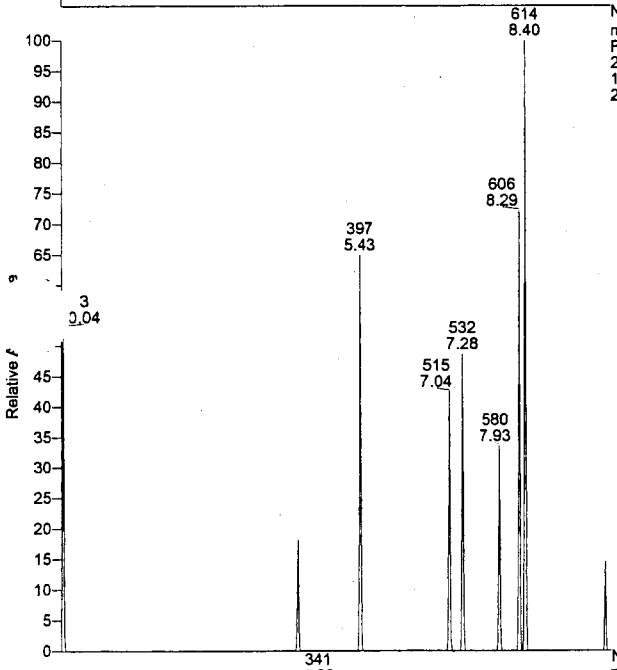
Ex 446

C:\Xcalibur\data\Brewer\070201013\21622 JMB 02/16/07 03:51:42 BLANK (neg blood, DI H2O ext.)

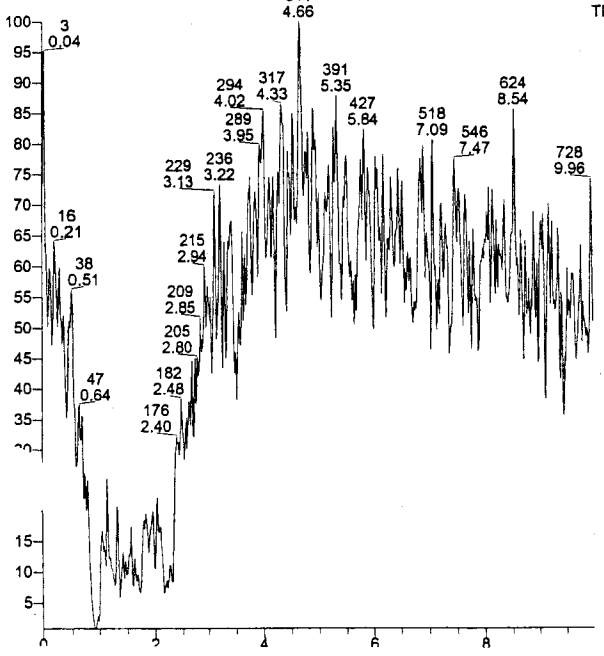
RT: 0.00 - 9.99 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21622

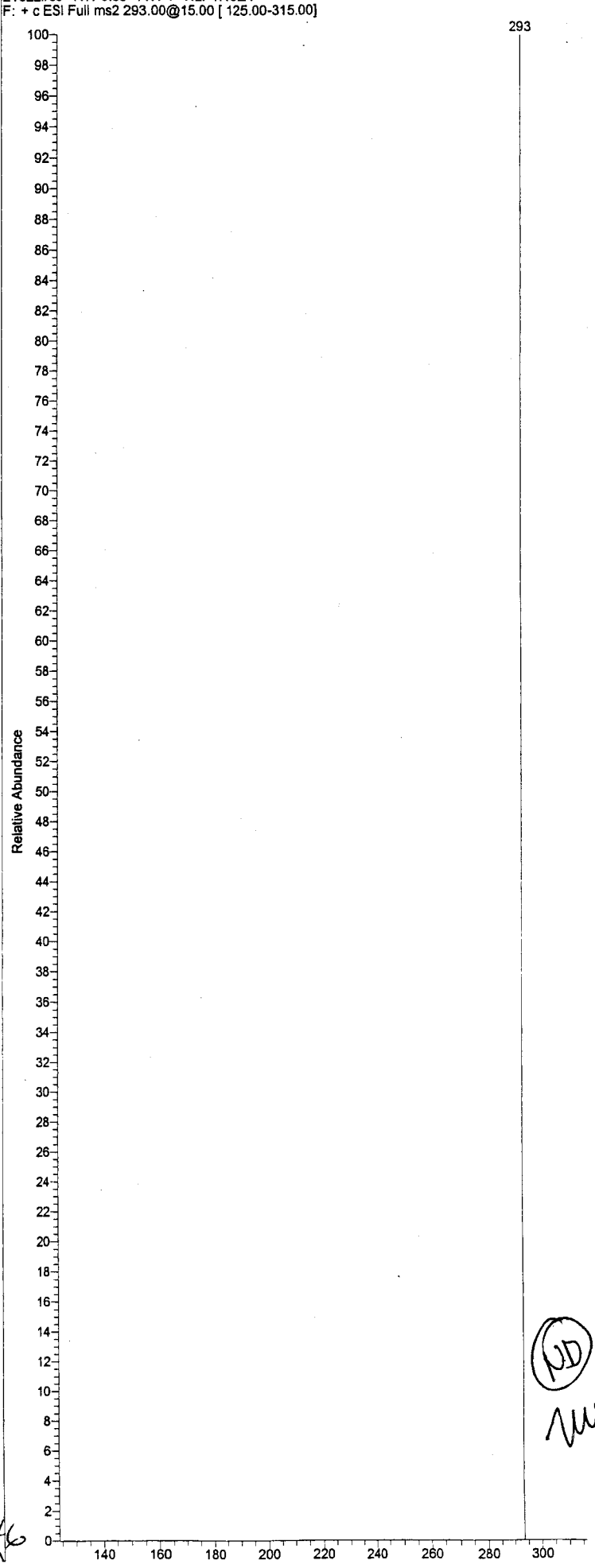
NL: 2.63E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21622



NL: 4.71E5
TIC MS 21622

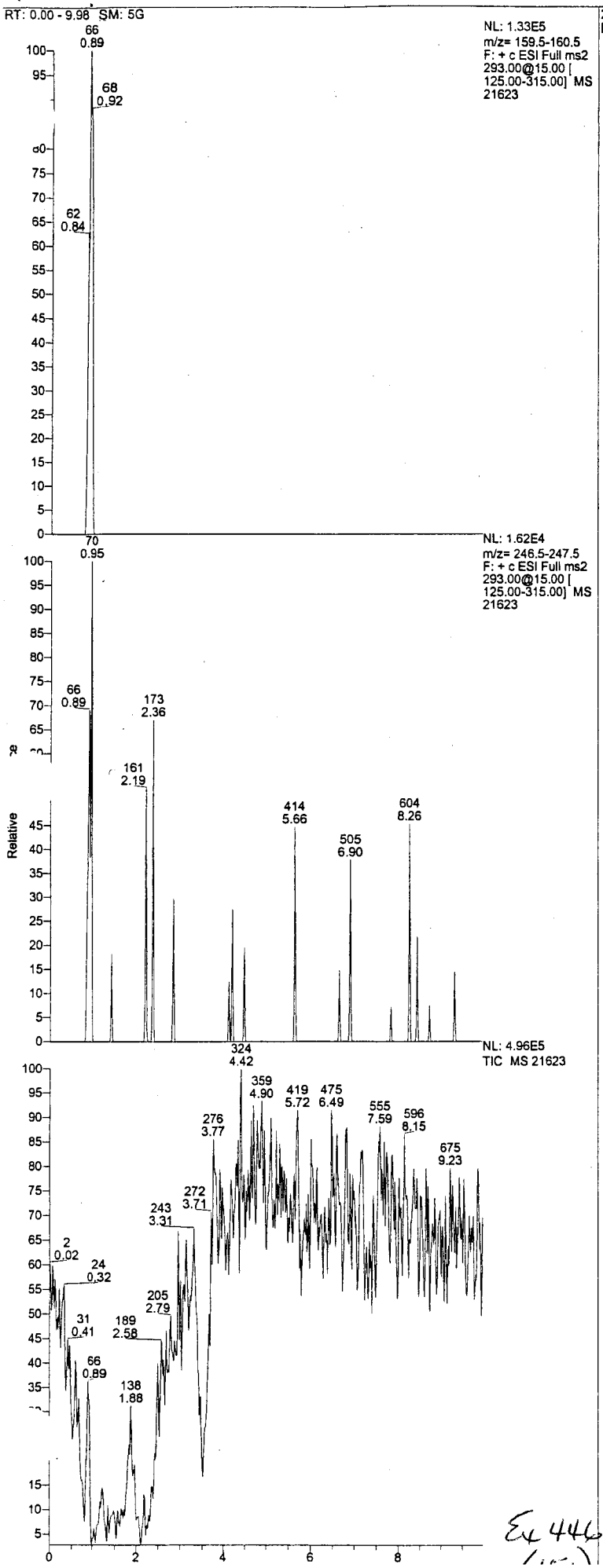


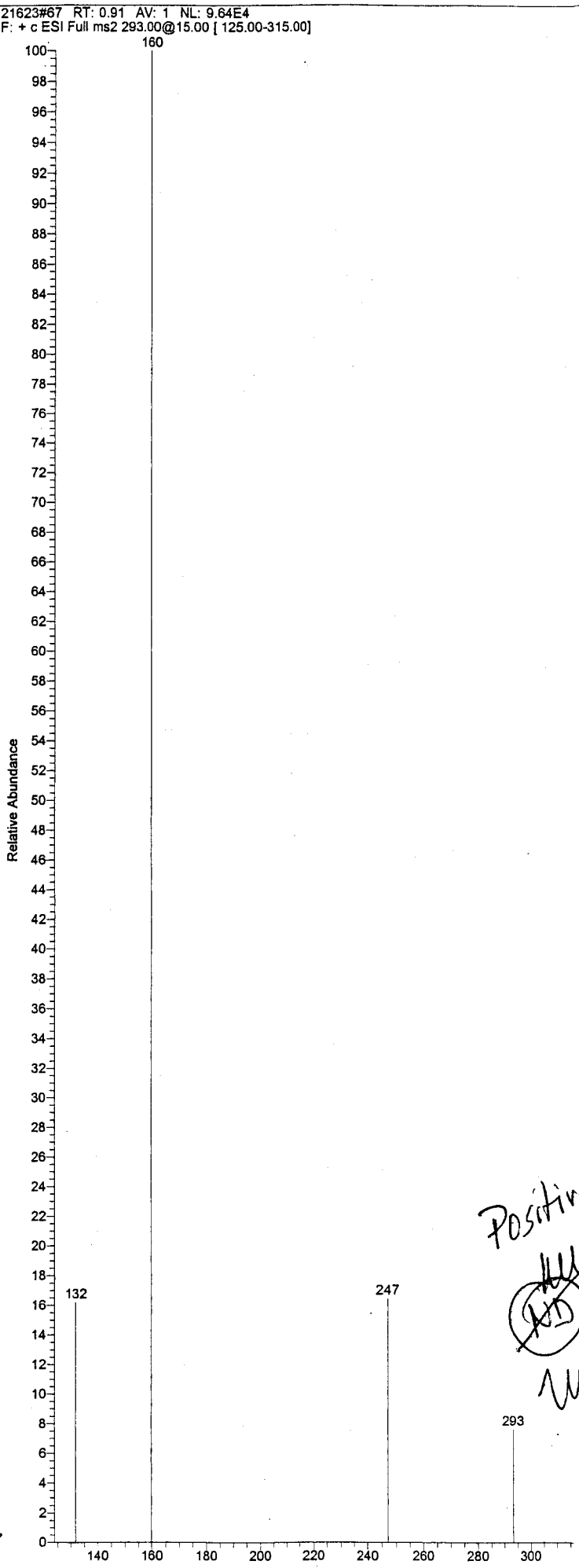
21622#65 RT: 0.88 AV: 1 NL: 1.13E4
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



Ex 446

C:\Xcalibur\data\Brewer\070201013\21623 JuB 02/16/07 04:02:32 Pos. Cont. A (MAL EDTA ext.)

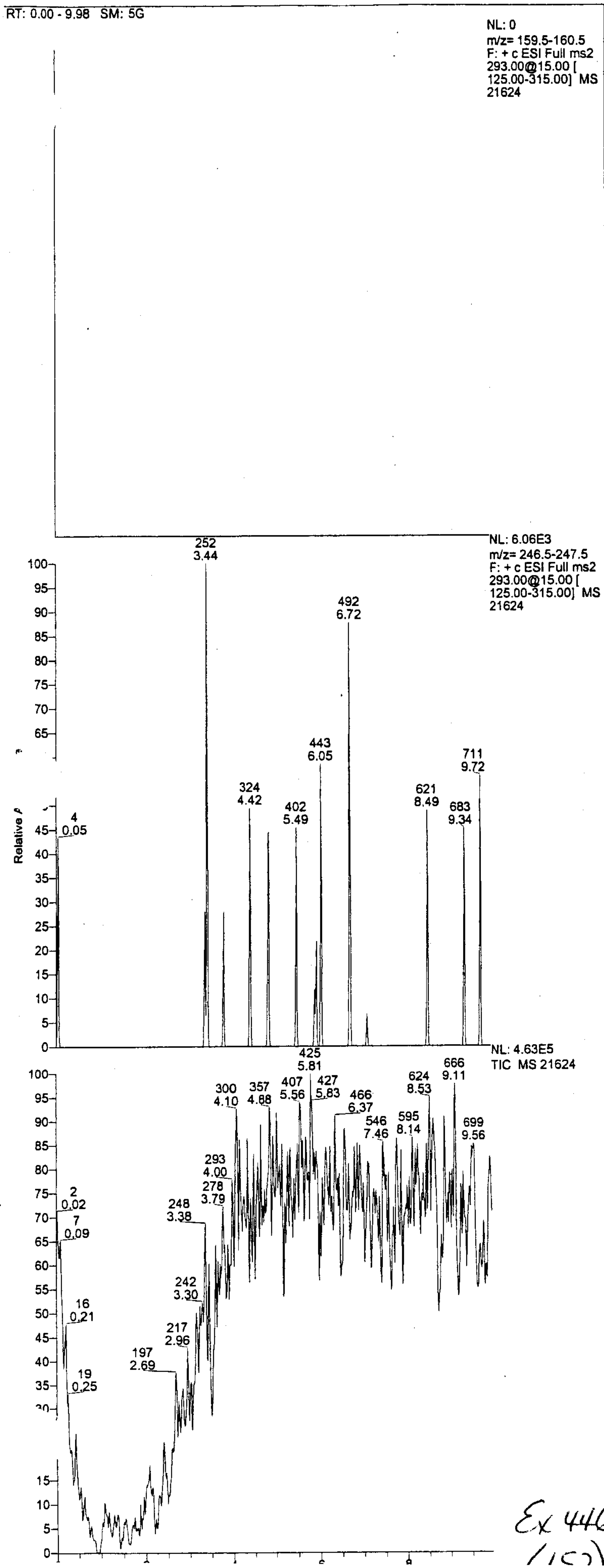


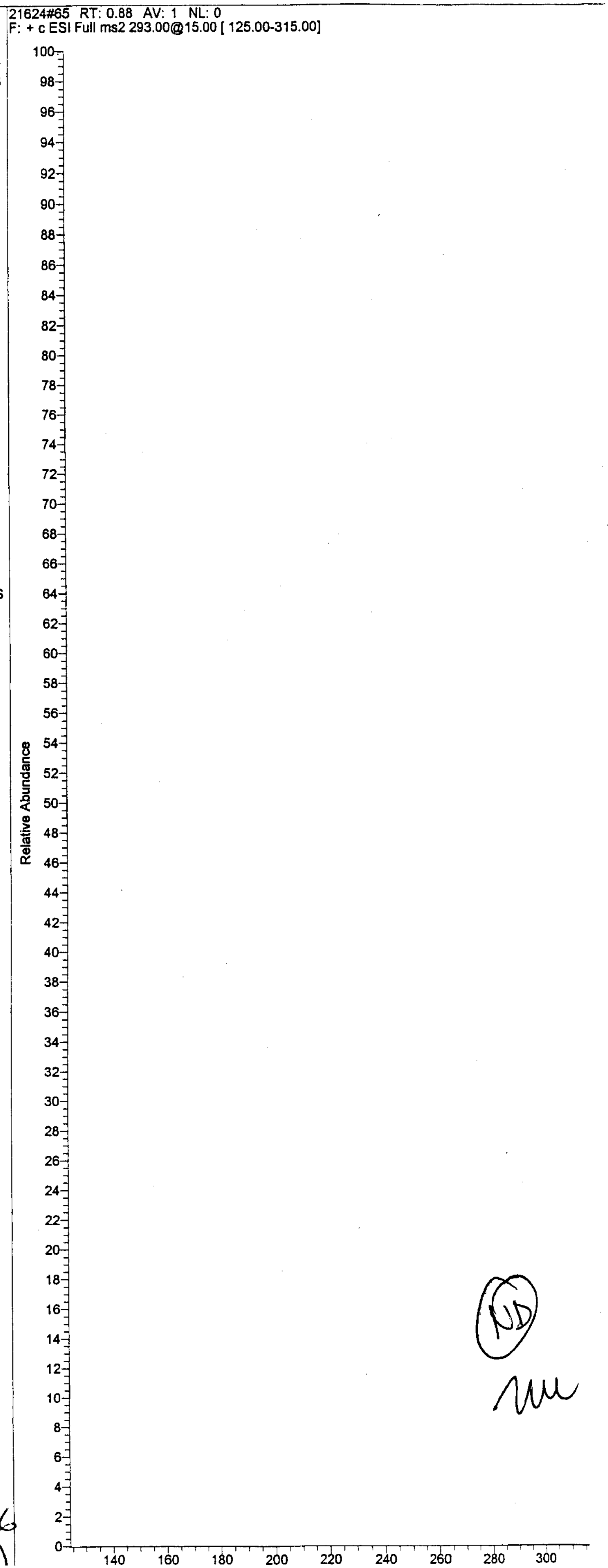


Positive

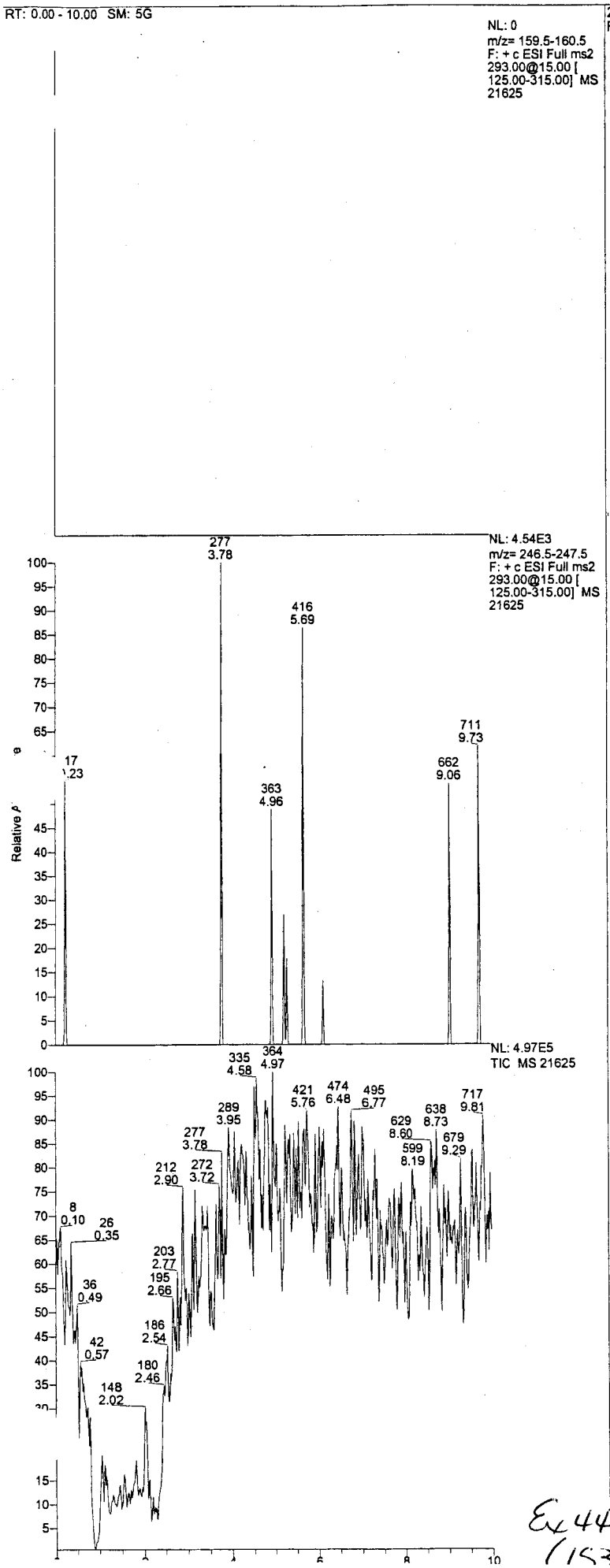
Ex 446

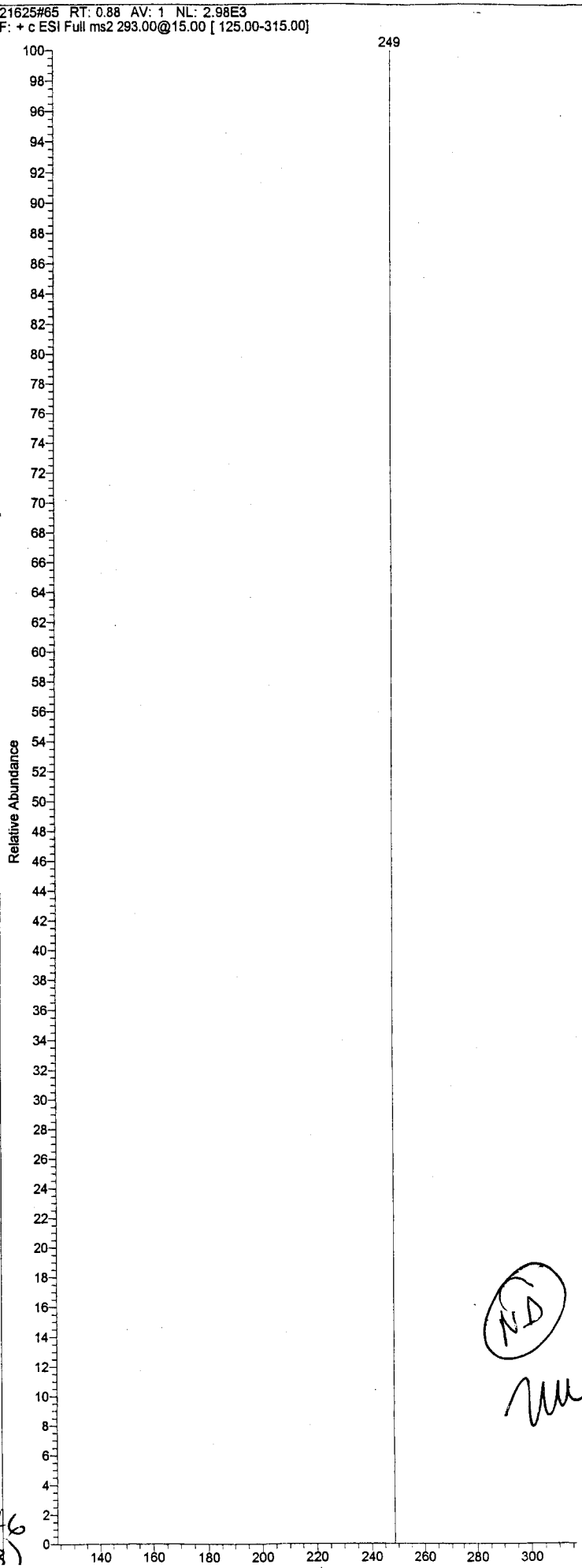
C:\Xcalibur\data\Brewer\070201013\21624 JDB 02/16/07 04:13:22 BLANK (neg blood, DI H2O ext.)





C:\Xcalibur\data\Brewer\070201013\21625 JSB 02/16/07 04:24:14 BLANK (neg blood, DI H2O ext.)

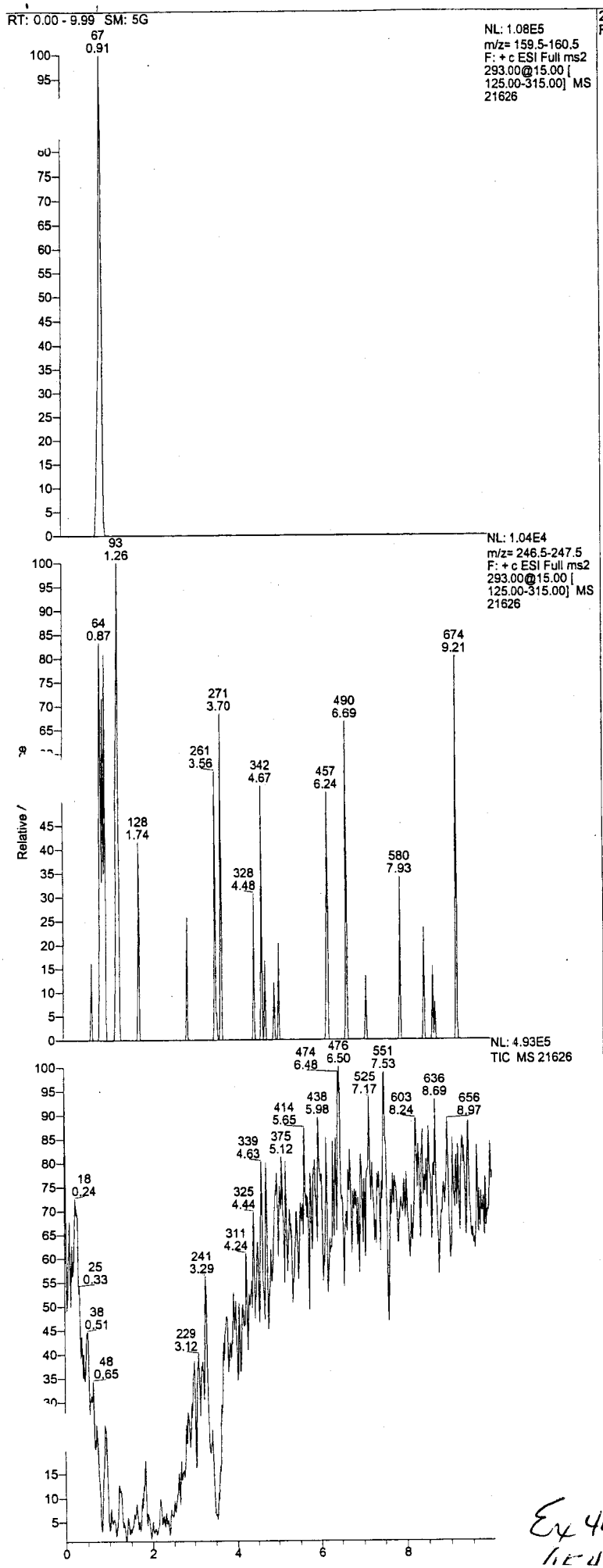


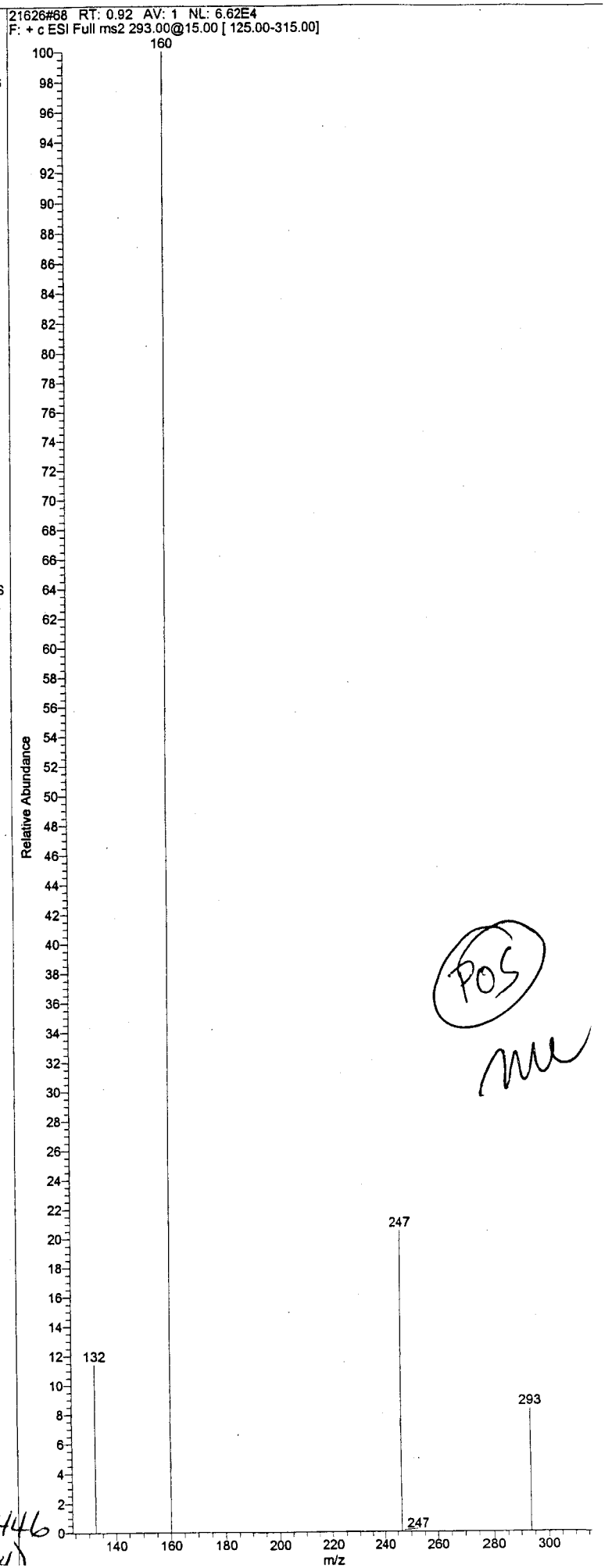


Ex 446
(153)

ND

C:\Xcalibur\data\Brewer\070201013\21626 JDB 02/16/07 04:35:06 Pos. Cont. B (Q49 ext.)

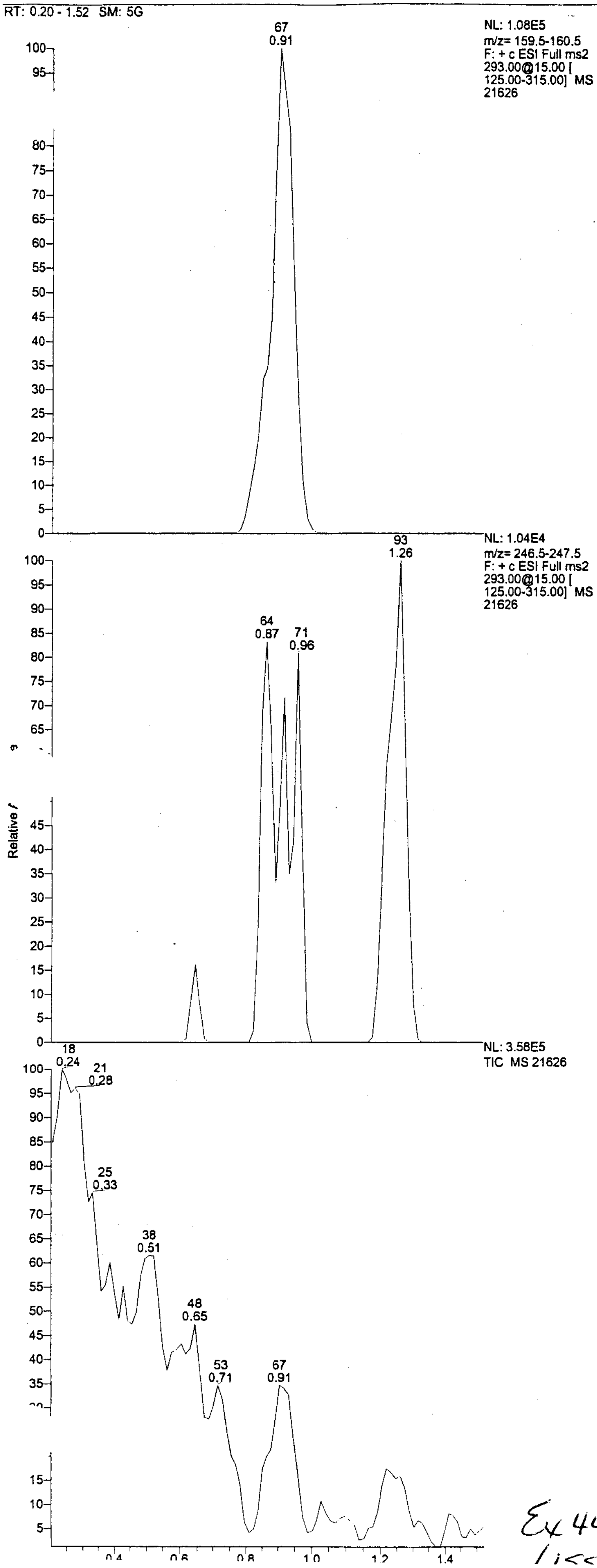


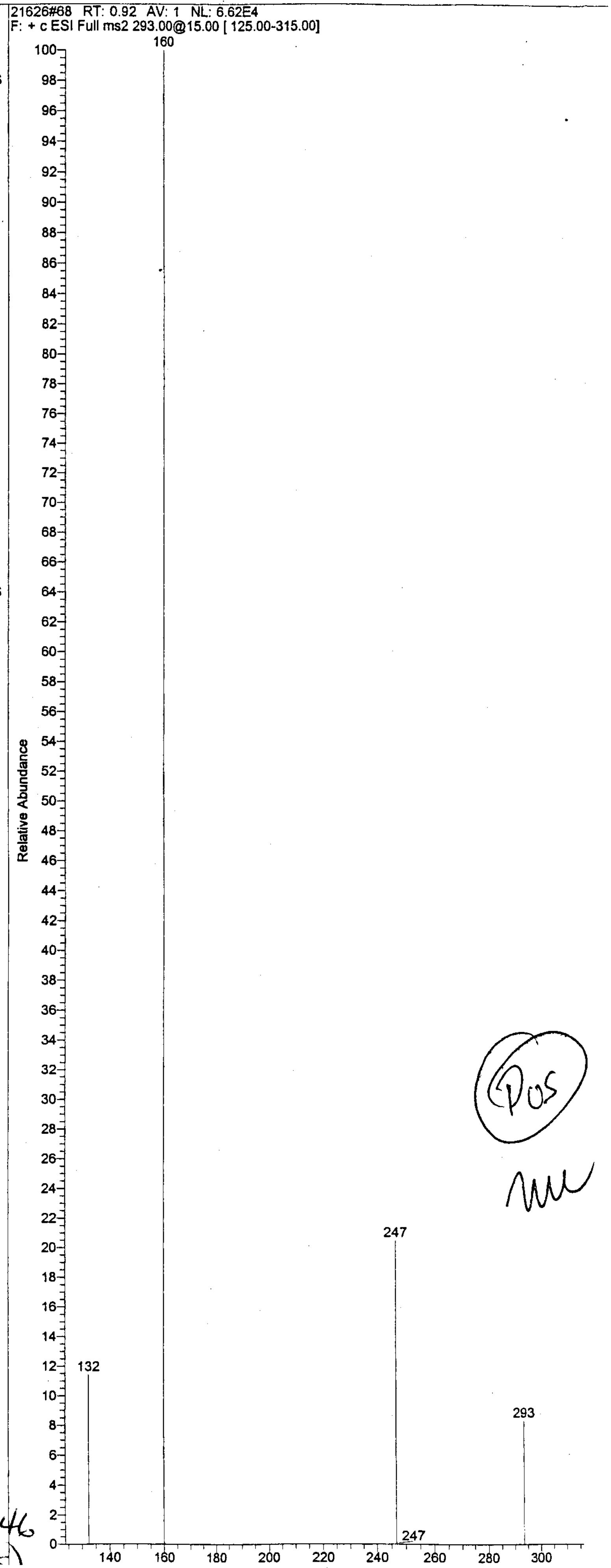


POS

Ex 446

C:\Xcalibur\data\Brewer\070201013\21626 (zoom) JDB 02/16/07 04:35:06 Pos. Cont. B (Q49 ext.)



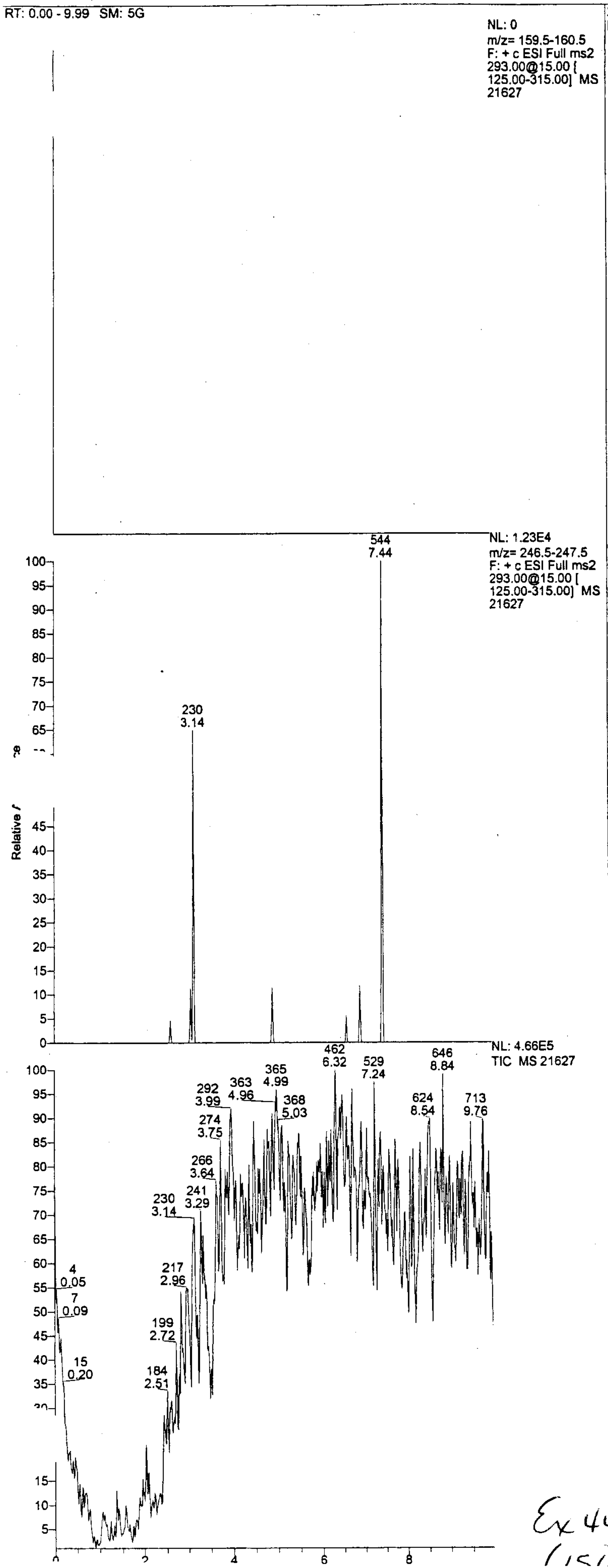


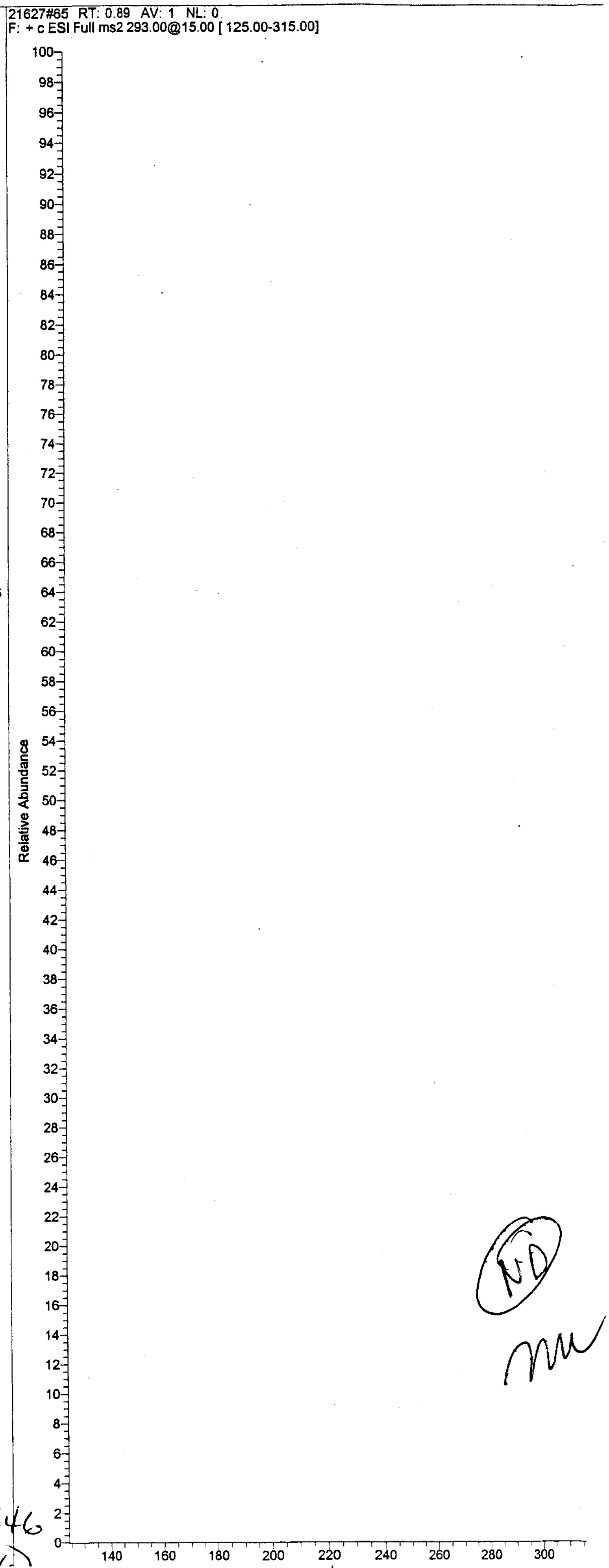
Ex 446 (iss)

Pos

MW

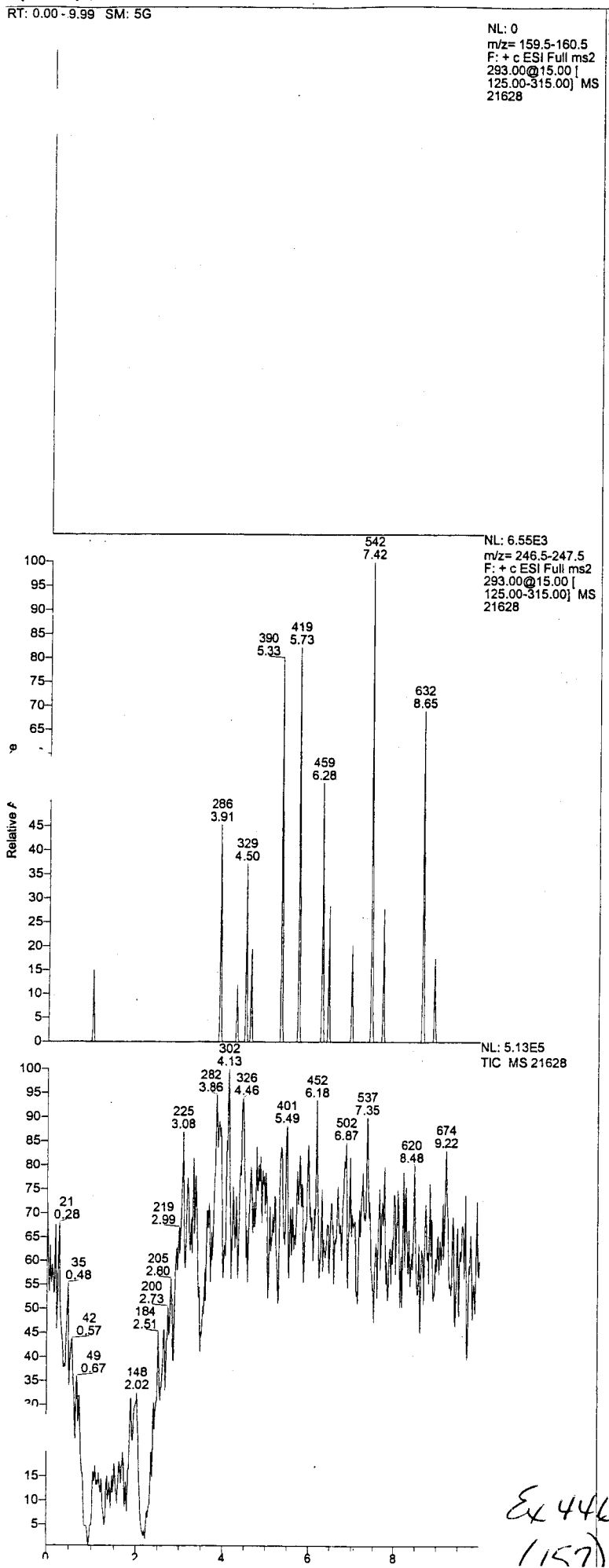
C:\Xcalibur\data\Brewer\070201013\21627 JDB 02/16/07 04:45:57 BLANK (neg blood, DI H2O ext.)

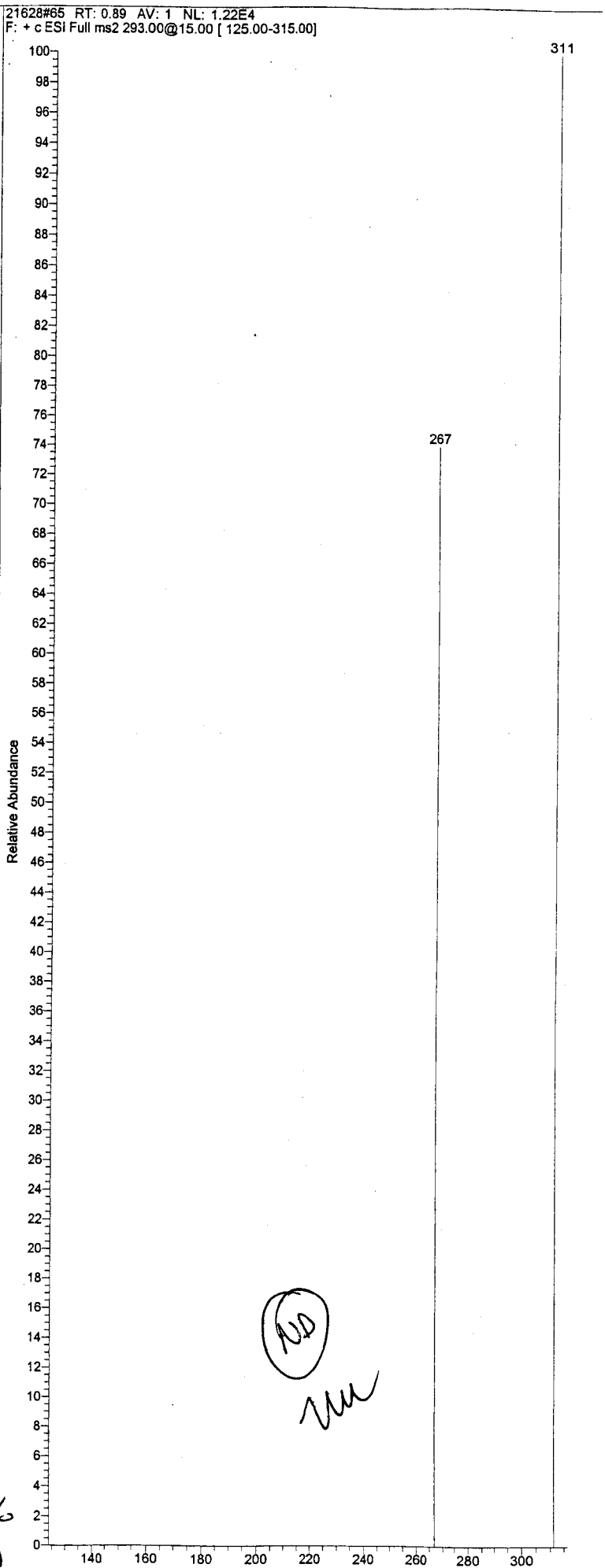




Ex 446
(156)

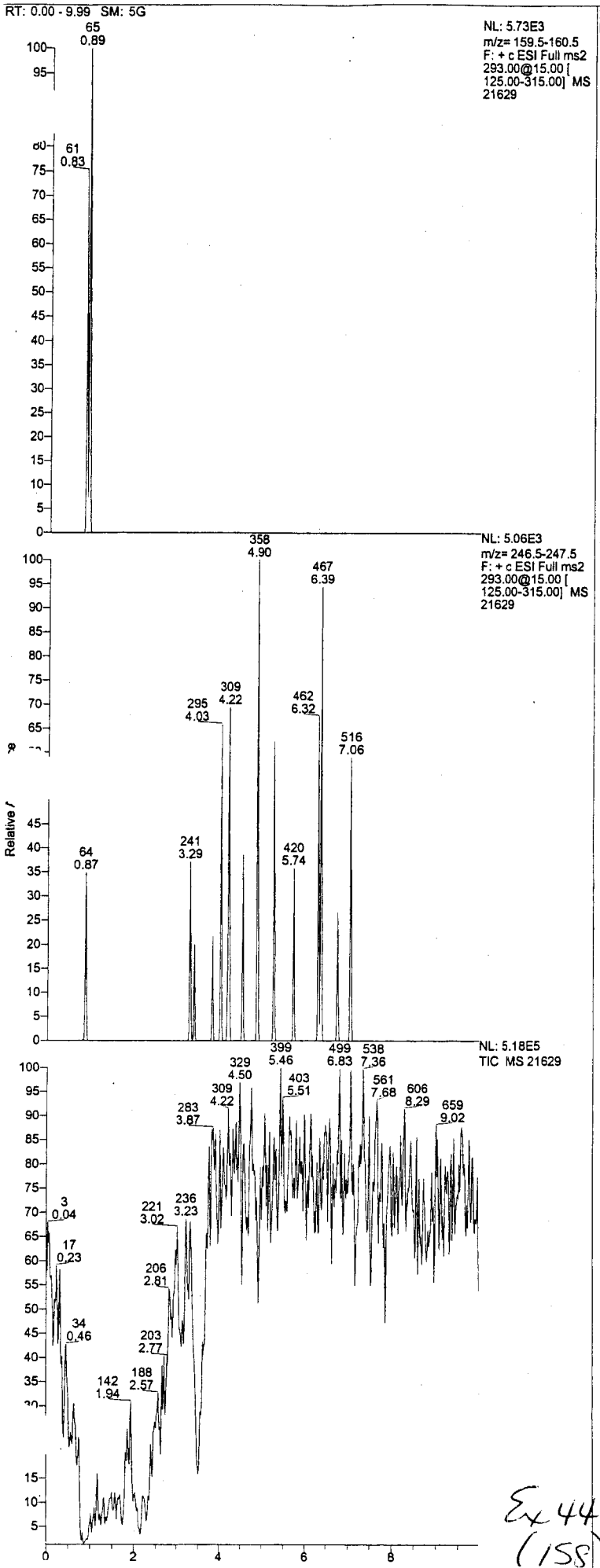
C:\Xcalibur\data\Brewer\070201013\21628 JVB 02/16/07 04:56:49 BLANK (neg blood, DI H2O ext.)

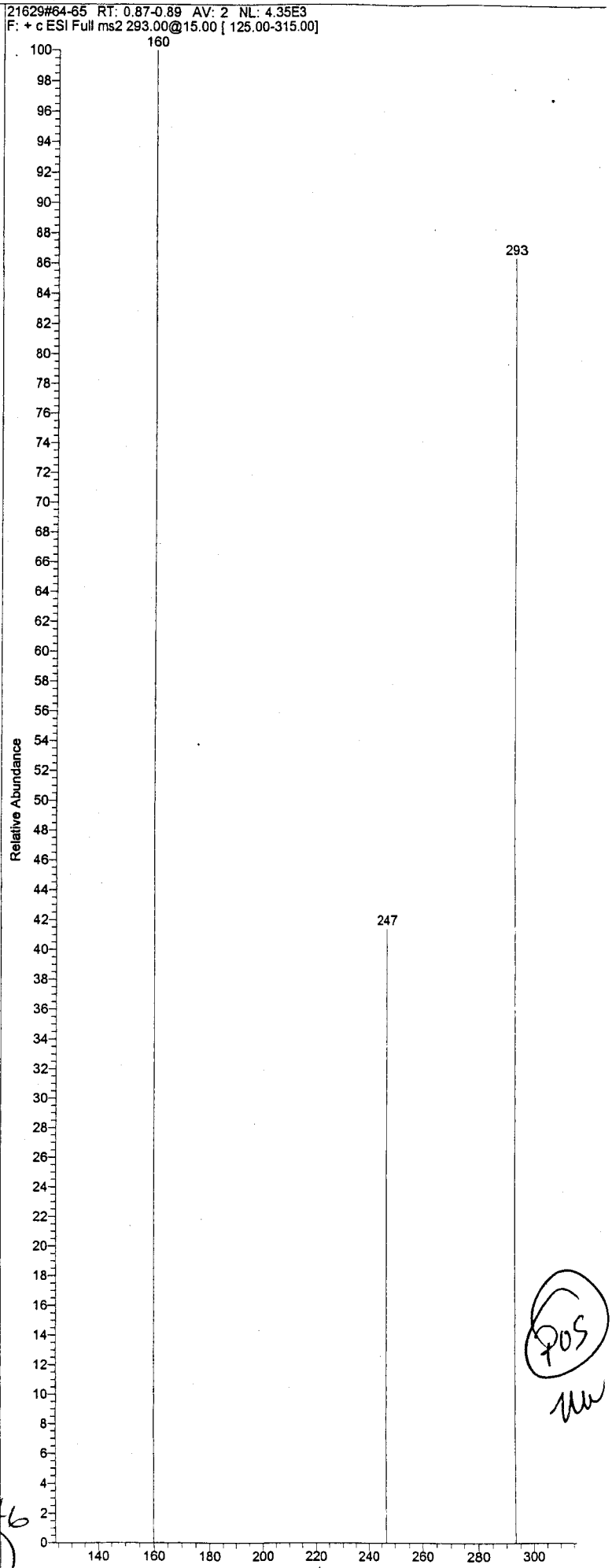




Ex 446
(157)

C:\Xcalibur\data\Brewer\070201013\21629 JDP 02/16/07 05:07:38 Spot LOD, 1uL Q49

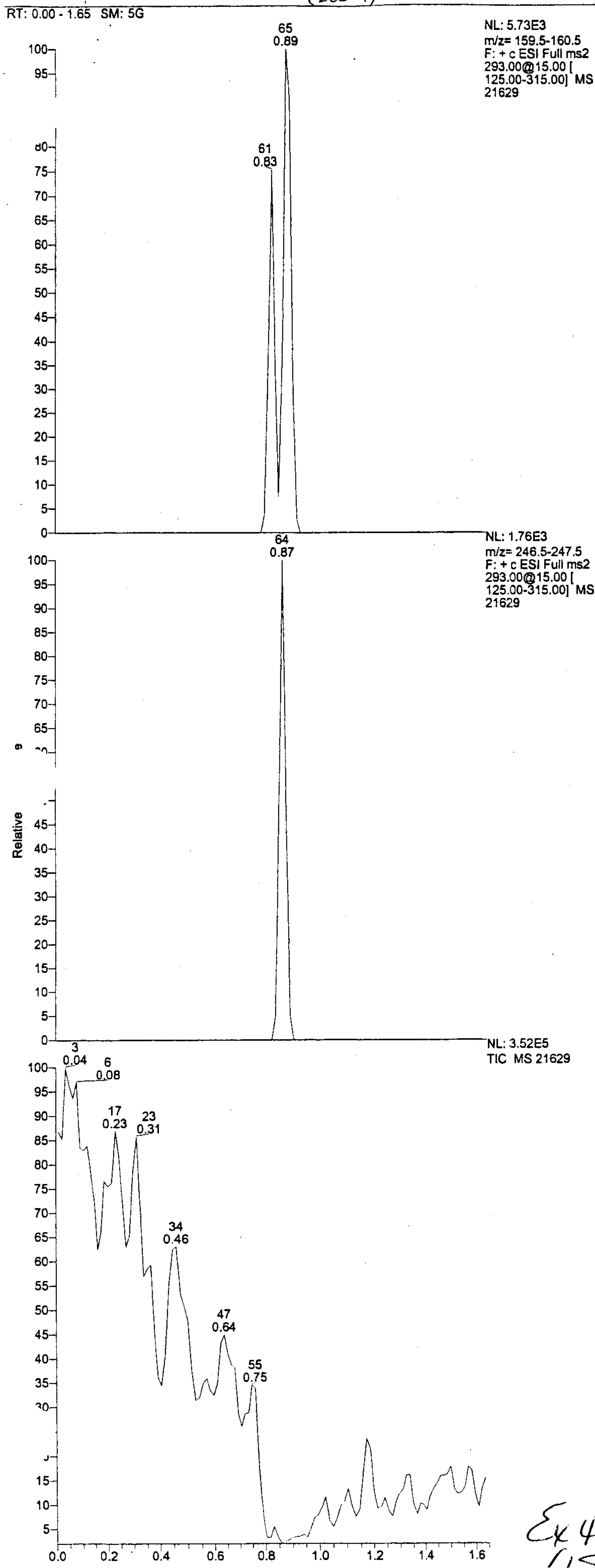


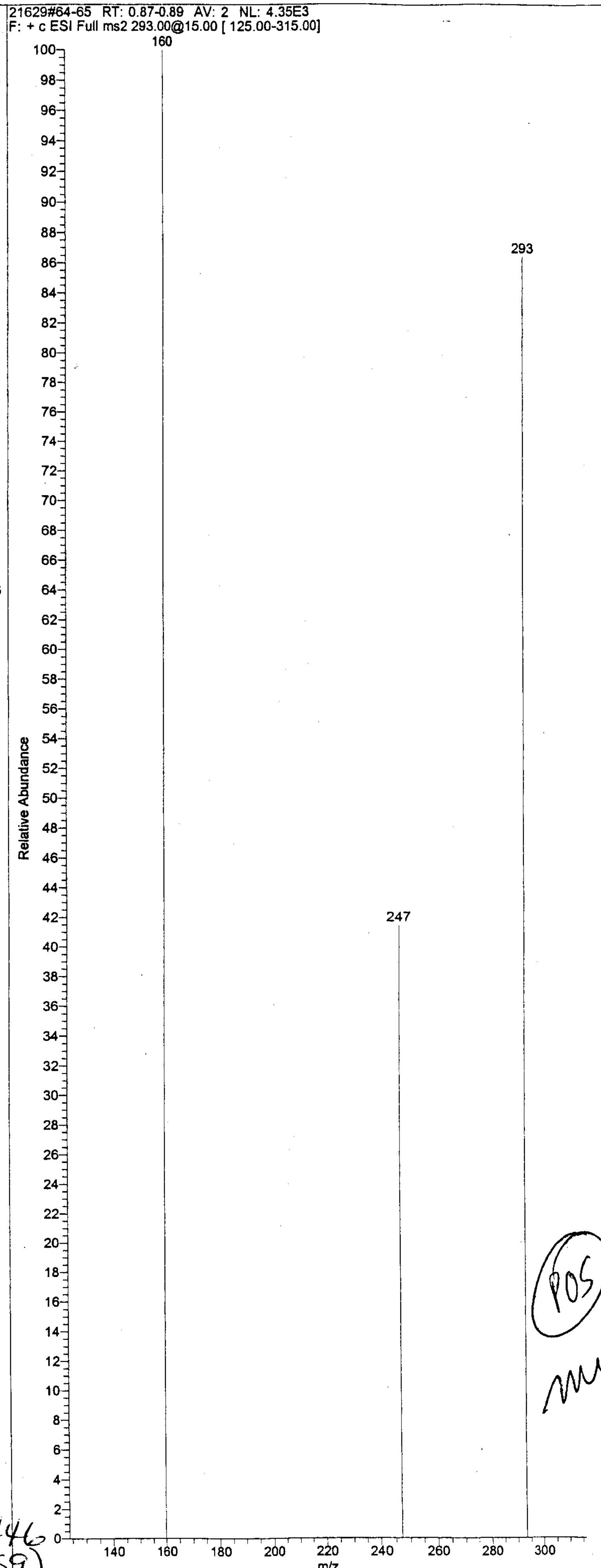


Ex 446
(158)

POS

C:\Xcalibur\data\Brewer\070201013\21629 (ZOOM) JDB 02/16/07 05:07:38 Spot LOD, 1uL Q49



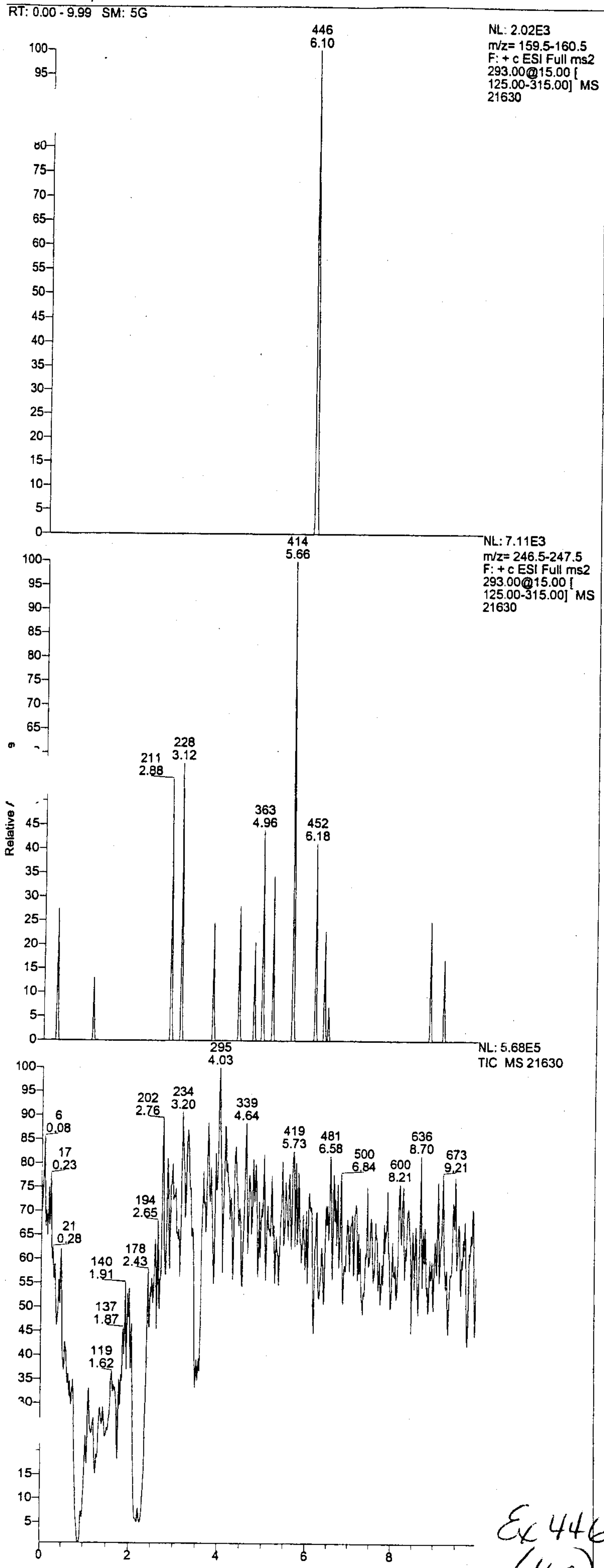


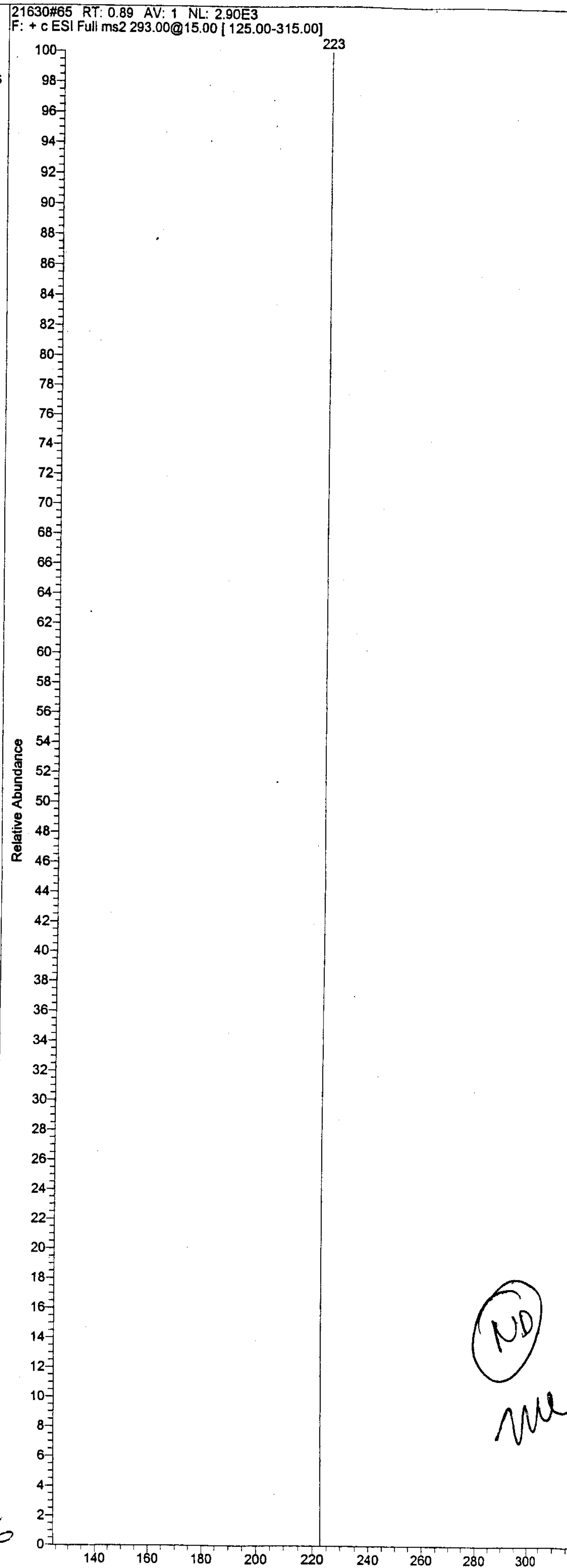
Ex 446 (159)

POS

MU

C:\Xcalibur\data\Brewer\070201013\21630 JDB 02/16/07 05:18:30 BLANK (neg blood, DI H2O ext.)



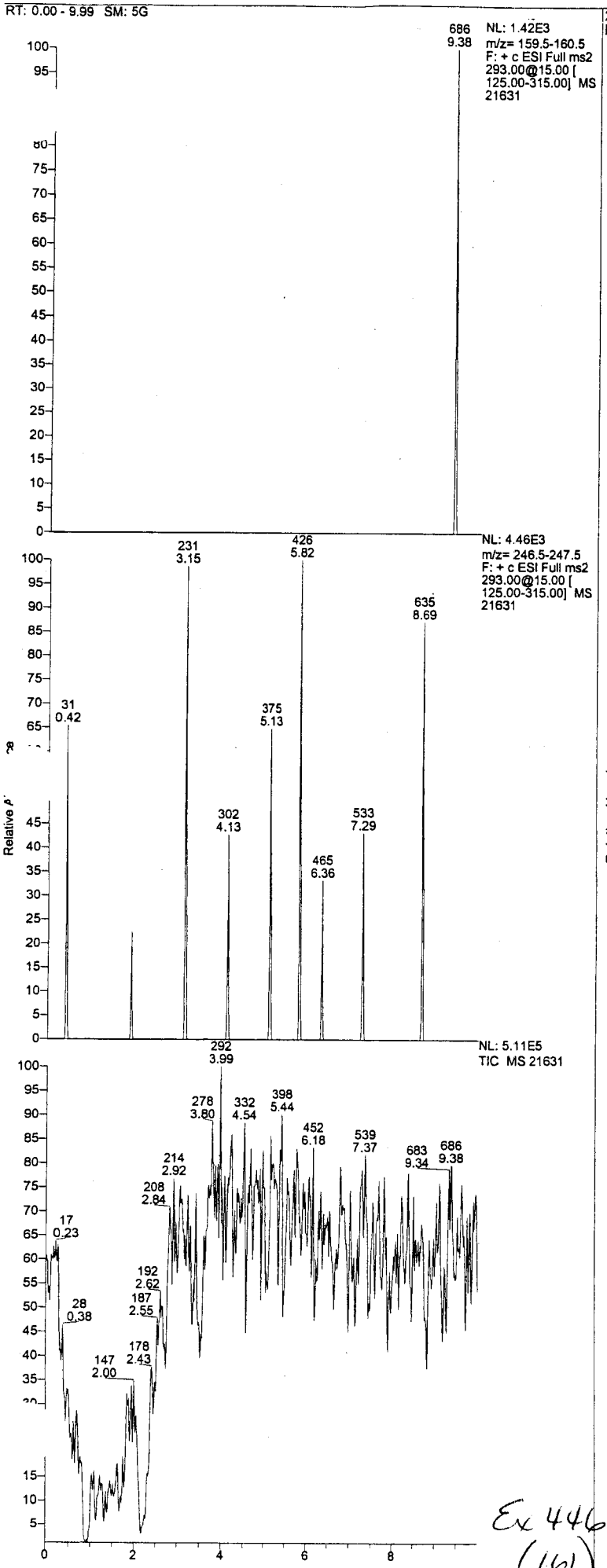


ND

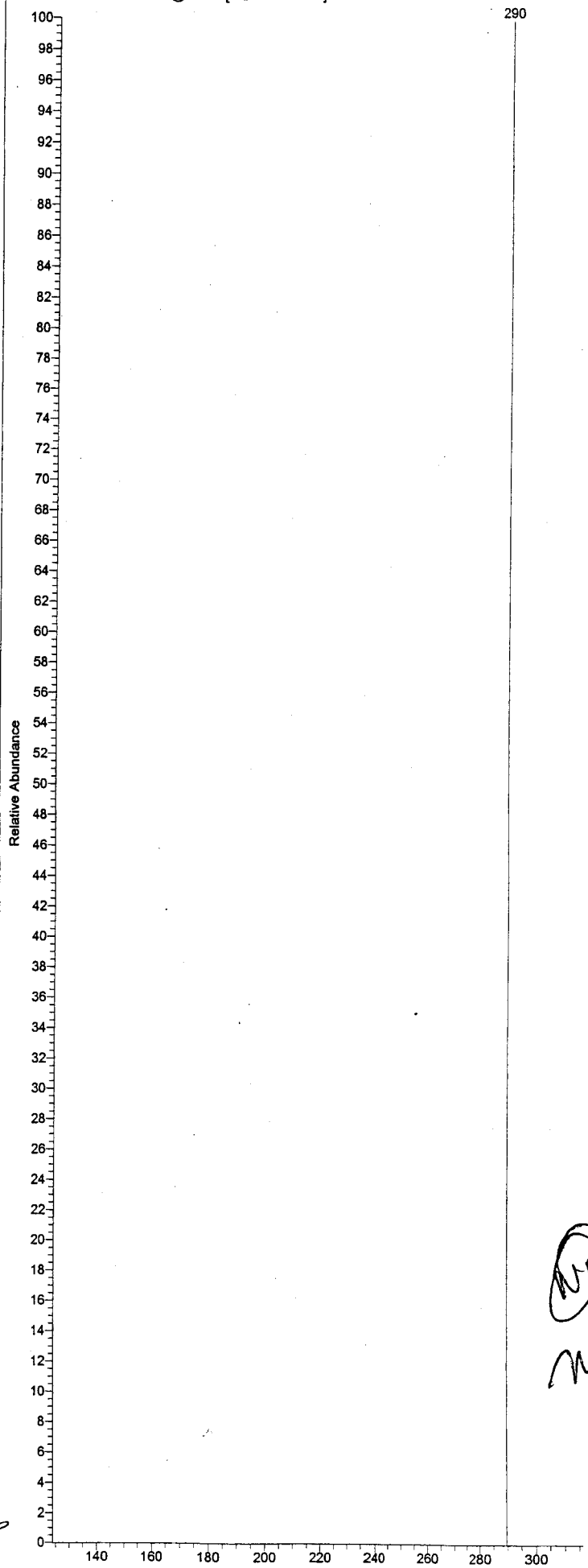
Ex 446

C:\Xcalibur\data\Brewer\070201013\21631 JDB 02/16/07 05:29:22 BLANK (neg blood, DI H2O ext.)

RT: 0.00 - 9.99 SM: 5G

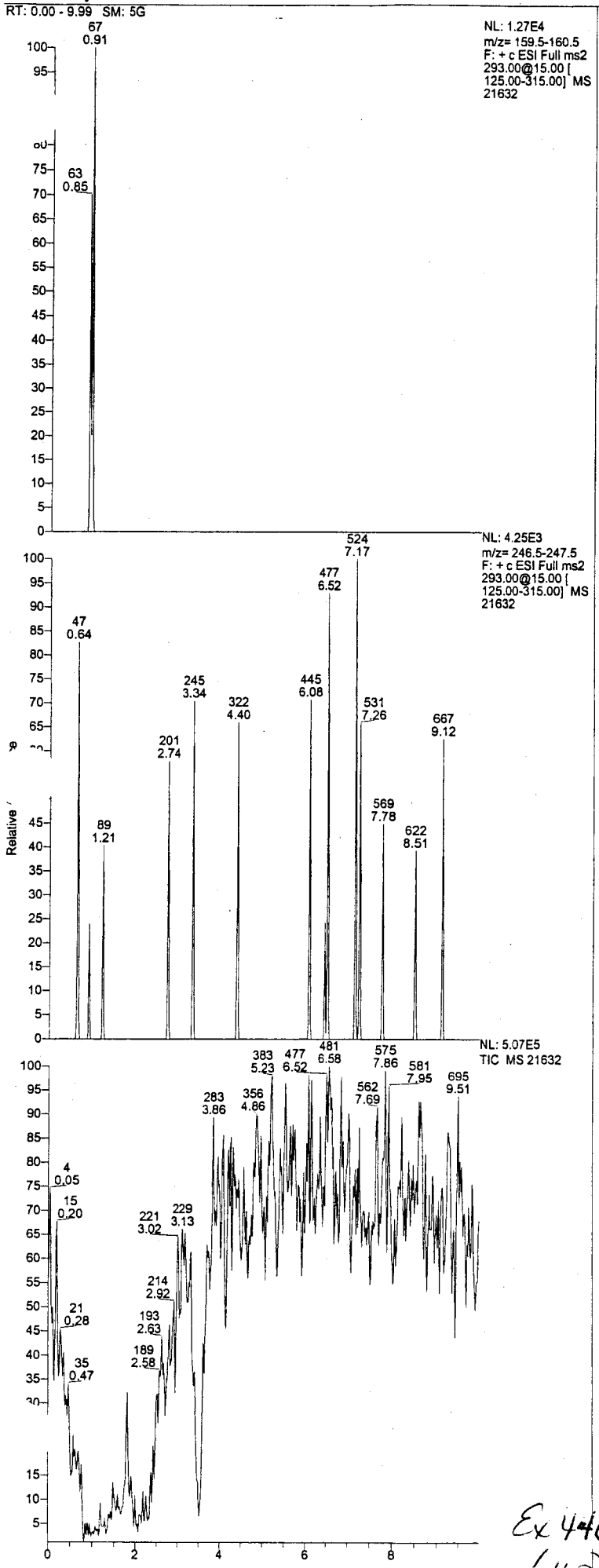


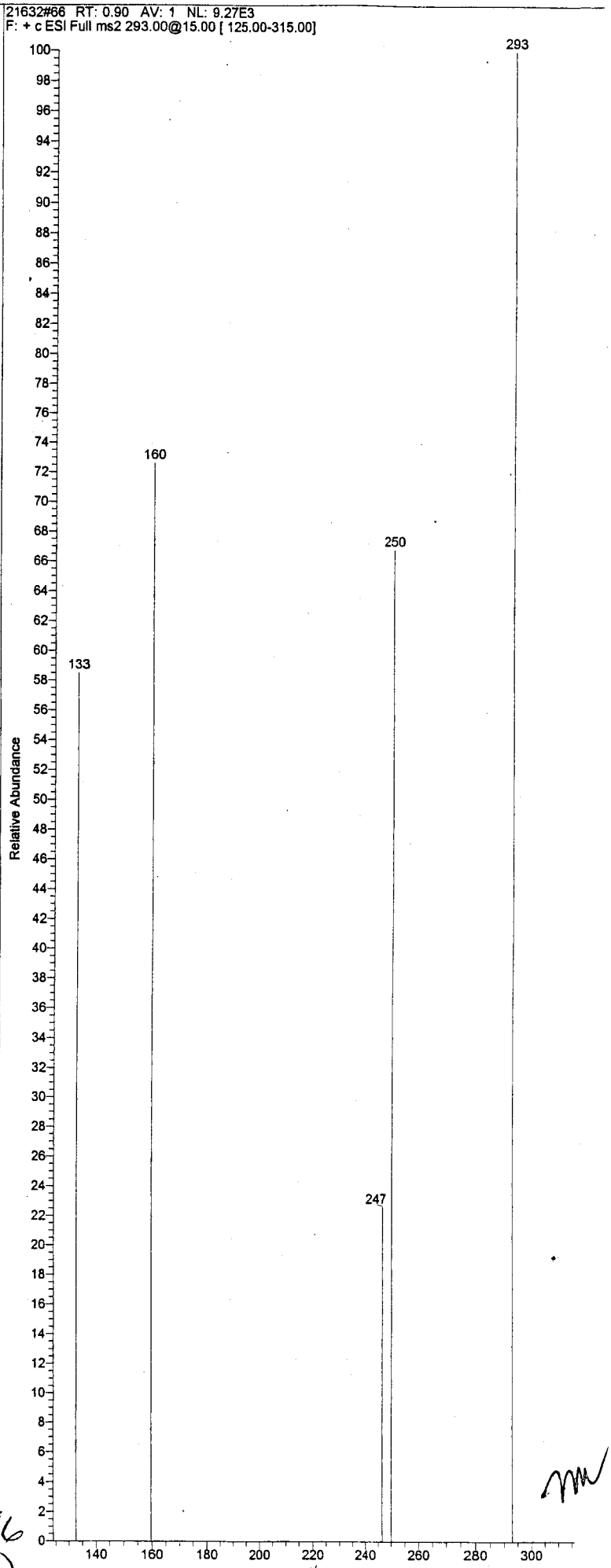
21631#65 RT: 0.88 AV: 1 NL: 1.03E4
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



Ex 446
(46)

C:\Xcalibur\data\Brewer\070201013\21632 JDP 02/16/07 05:40:13 Spot LOD, 2 uL Q49

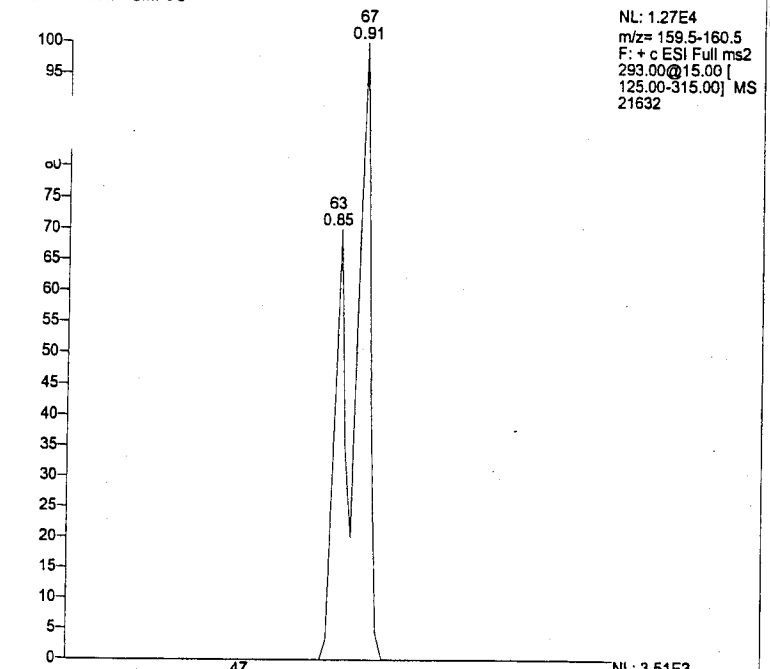


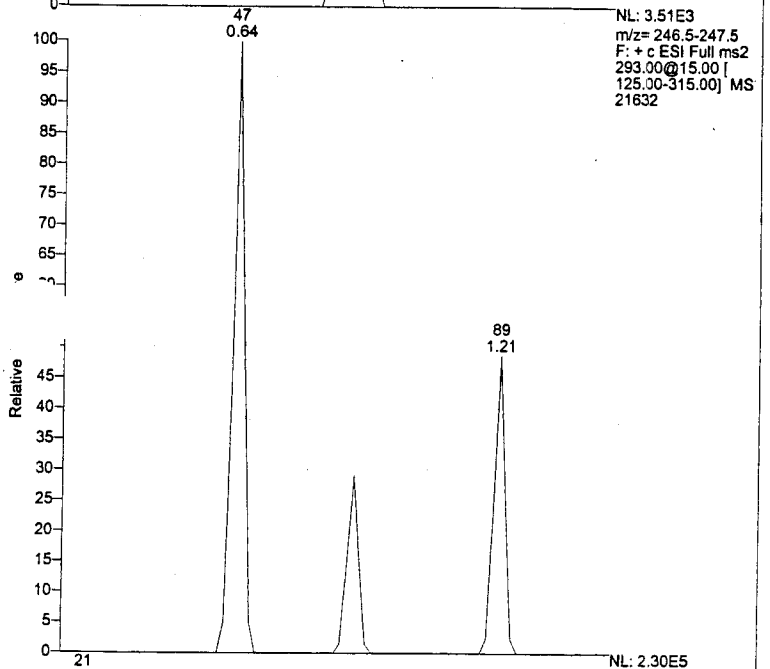


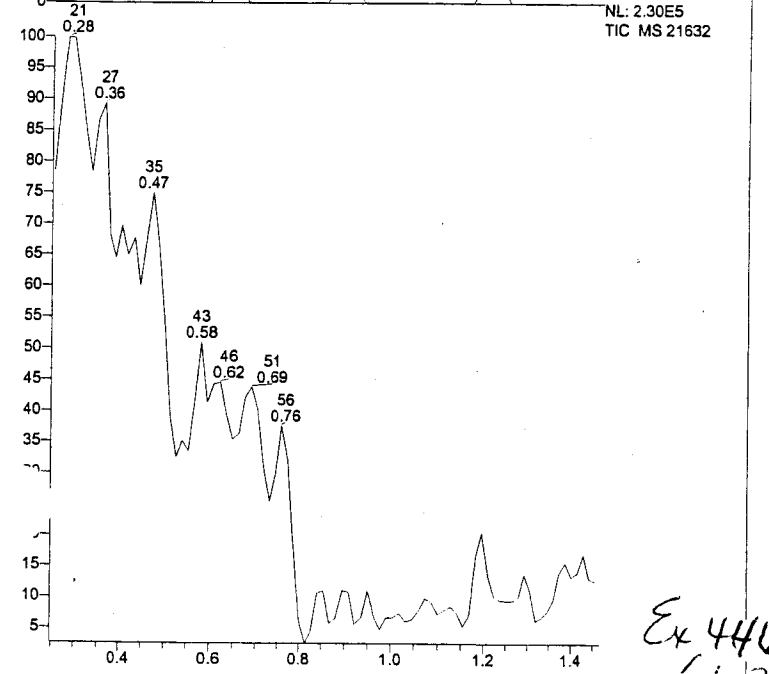
Ex 446

C:\Xcalibur\data\Brewer\070201013\21632 (zoom) JDB 02/16/07 05:40:13 Spot LOD, 2 uL Q49

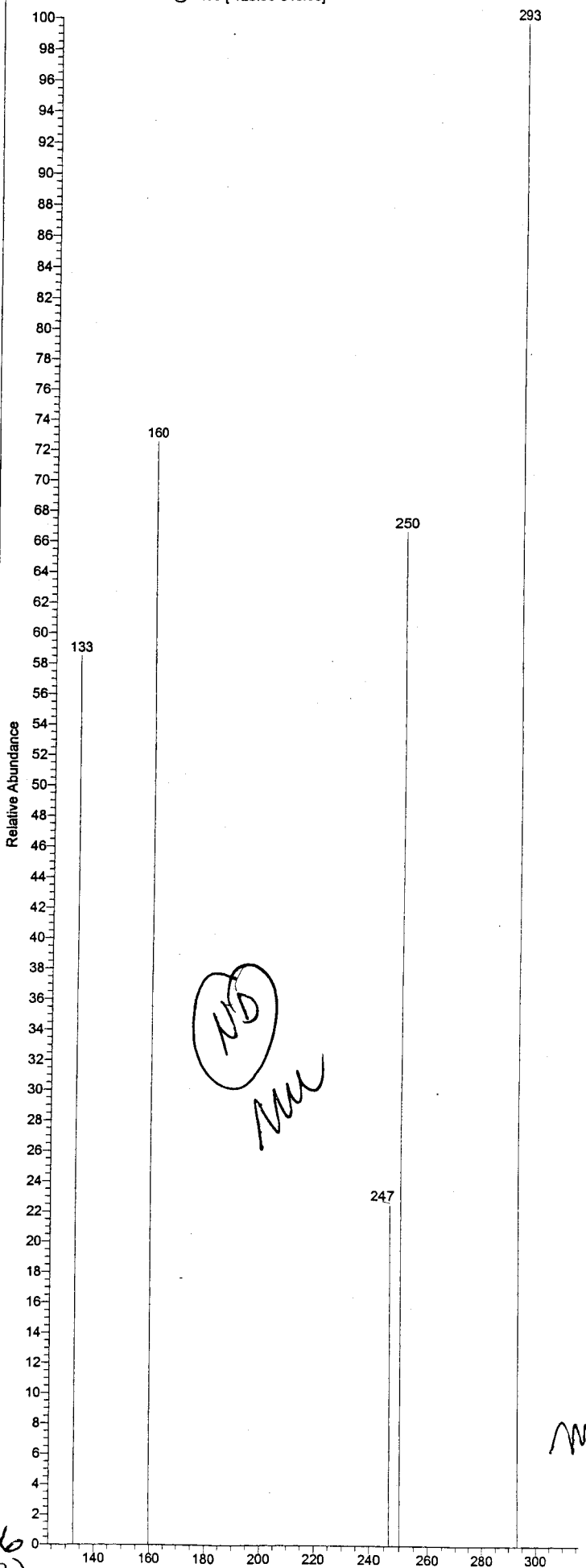
RT: 0.25 - 1.47 SM: 5G







21632#66 RT: 0.90 AV: 1 NL: 9.27E3
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

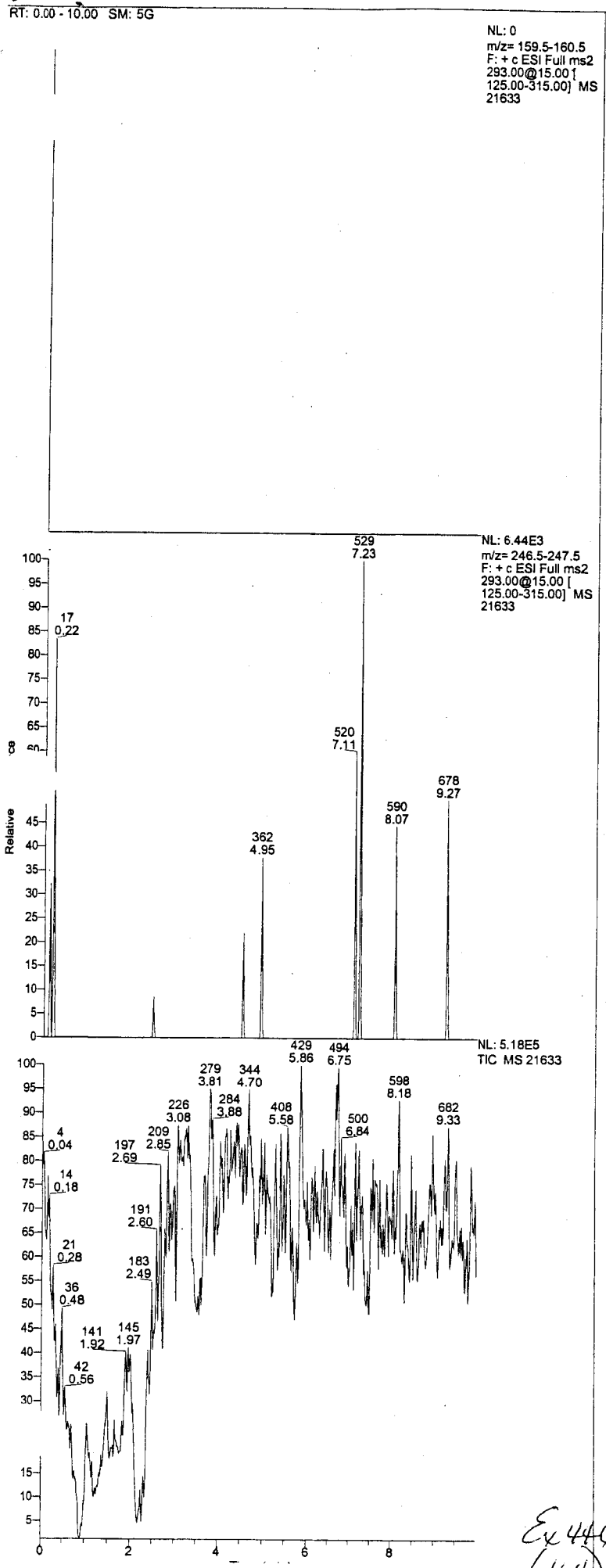


ND

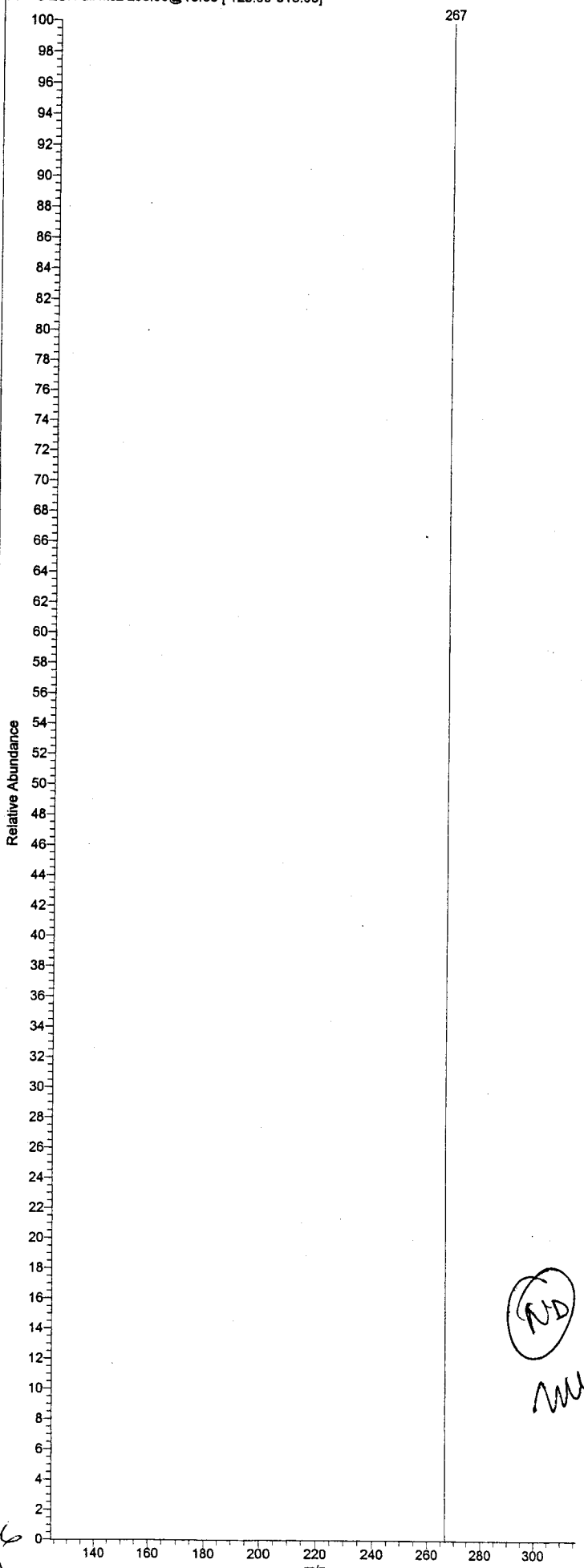
Ex 4446

C:\Xcalibur\data\Brewer\070201013\21633 JOB 02/16/07 05:51:09 BLANK (neg blood, DI H2O ext.)

RT: 0.00 - 10.00 SM: 5G



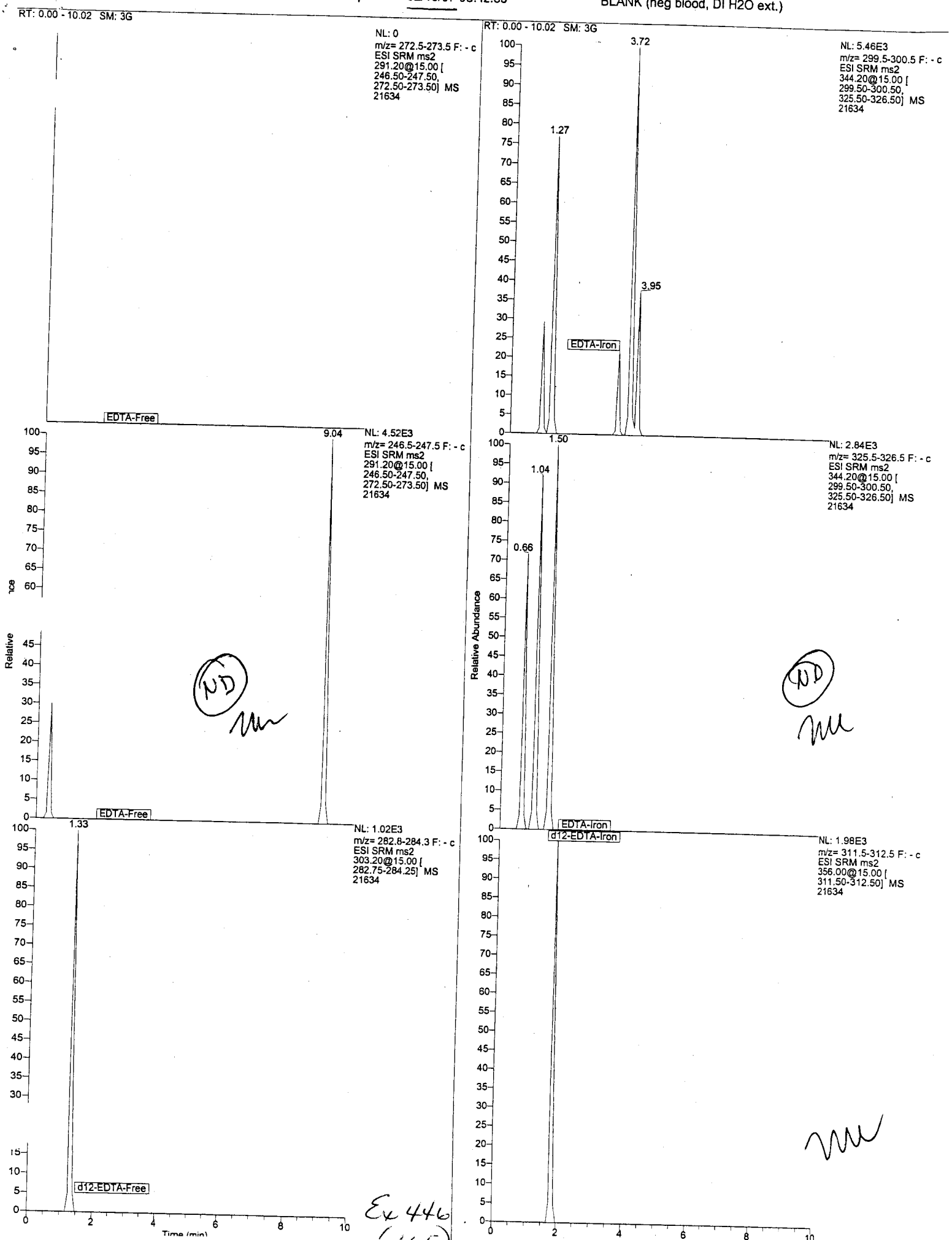
21633#65 RT: 0.88 AV: 1 NL: 8.68E3
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



ND

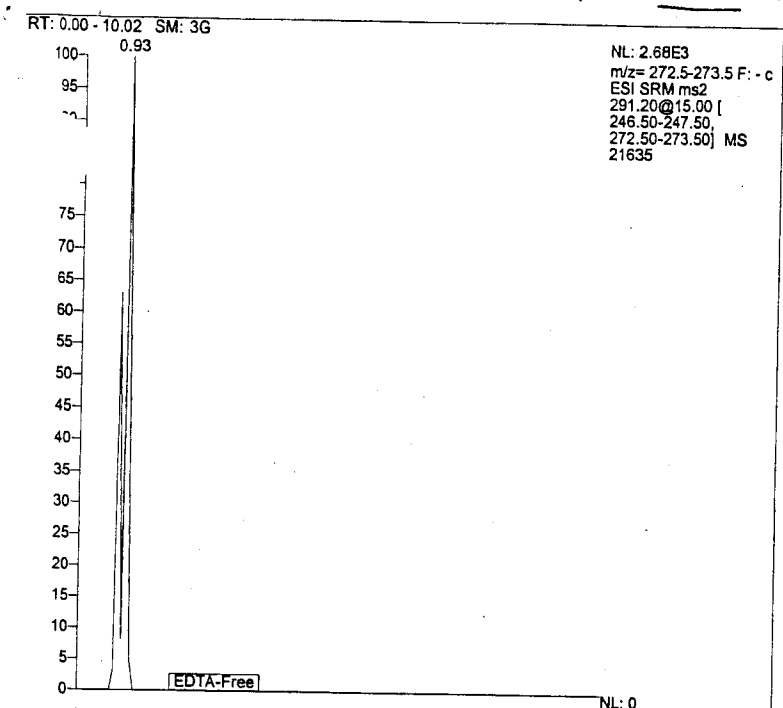
Ex 446

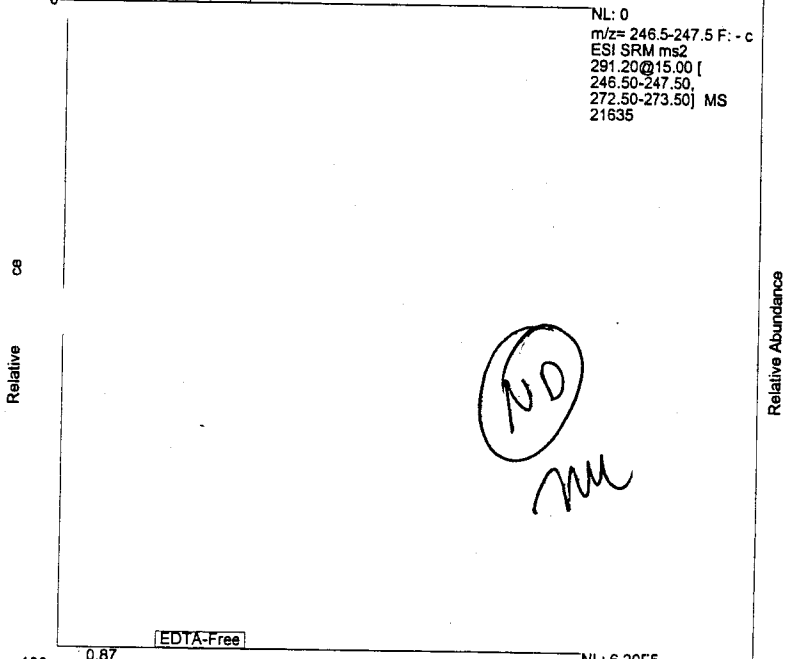
C:\Xcalibur\...\Negative Ion\21634 070201013 JDB 02/16/07 06:42:35 BLANK (neg blood, DI H2O ext.)



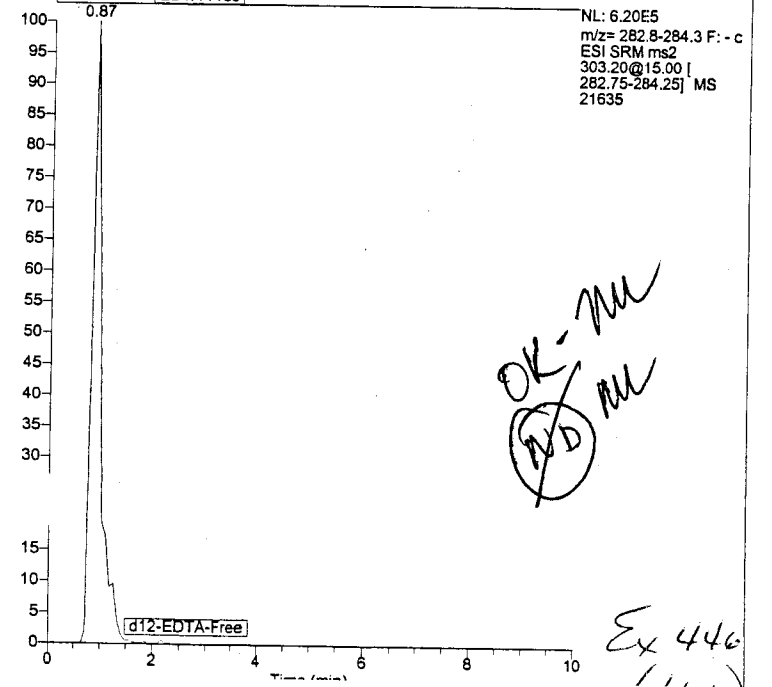
Ex 446

C:\Xcalibur\...\Negative Ion\21635 070201013 JDB 02/16/07 06:53:36 Neg. Control (-EDTA blood swab ext.)





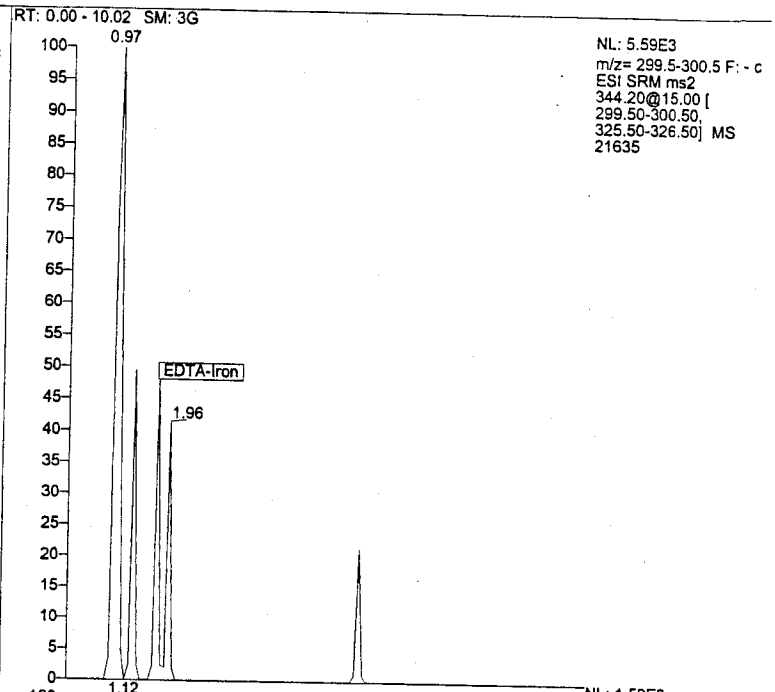
ND

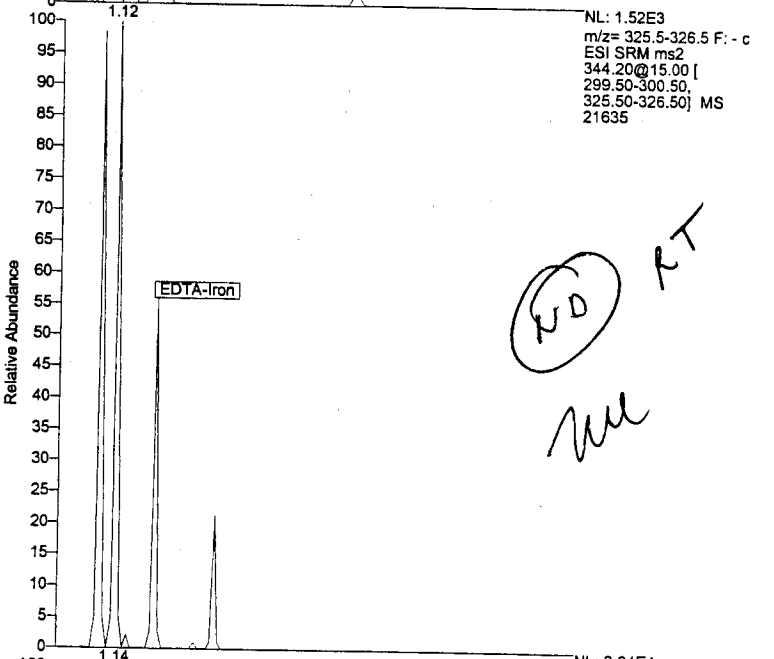


OK - MM

ND MM

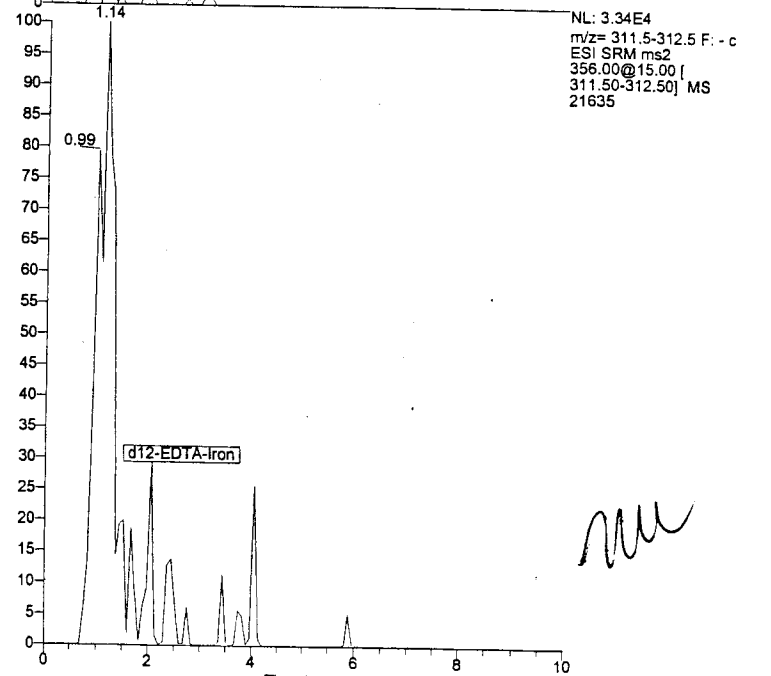
Ex 446





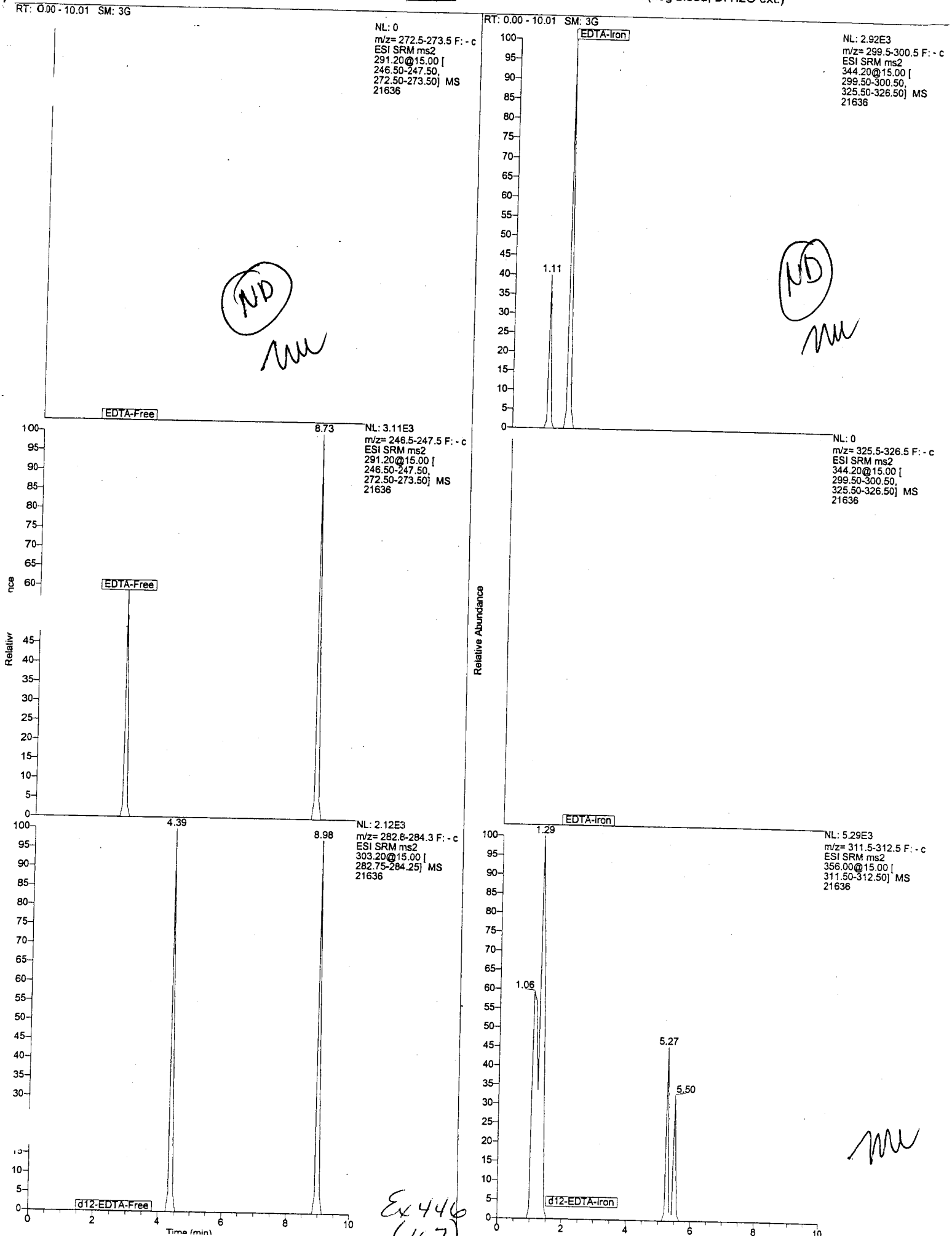
ND RT

MM



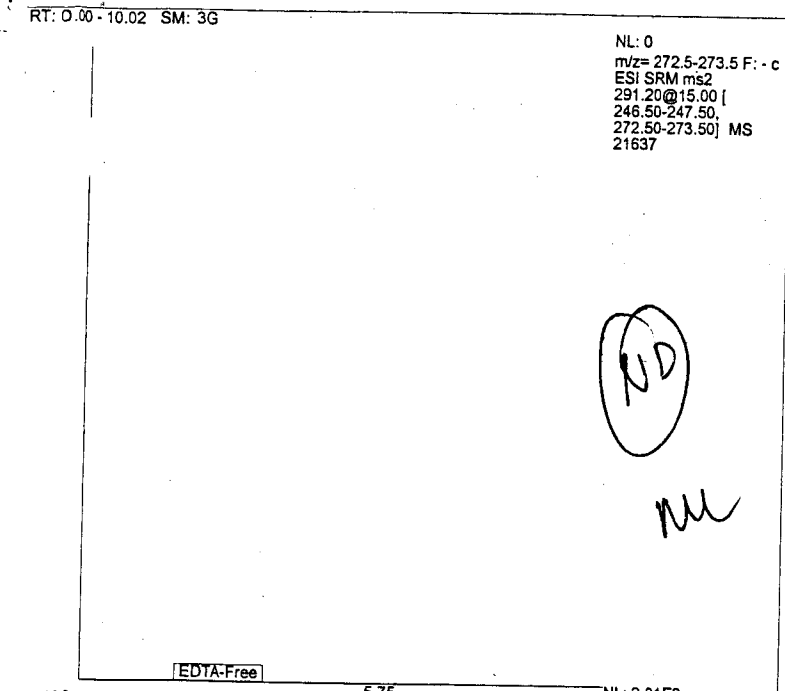
MM

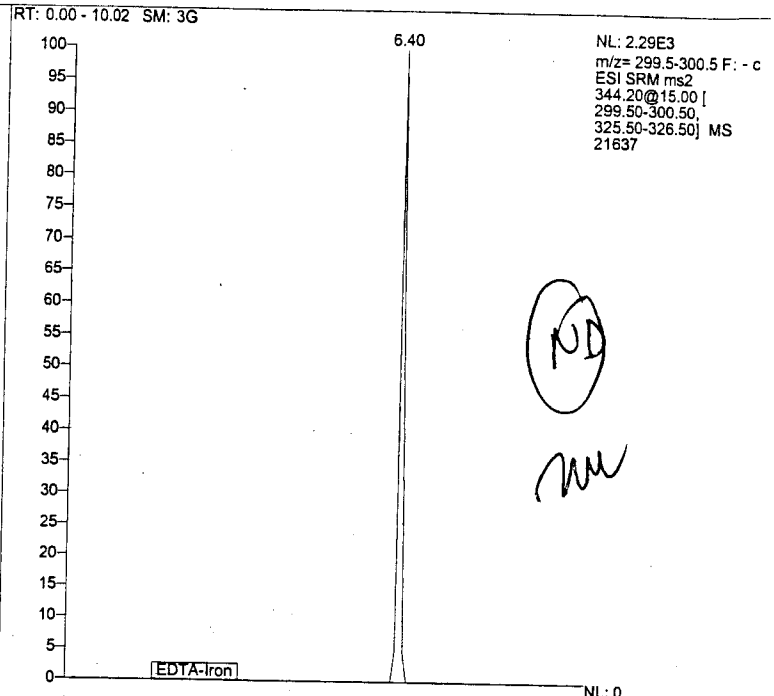
C:\Xcalibur\...\Negative Ion\21636 070201013 JDP 02/16/07 07:04:28 BLANK (neg blood, DI H2O ext.)

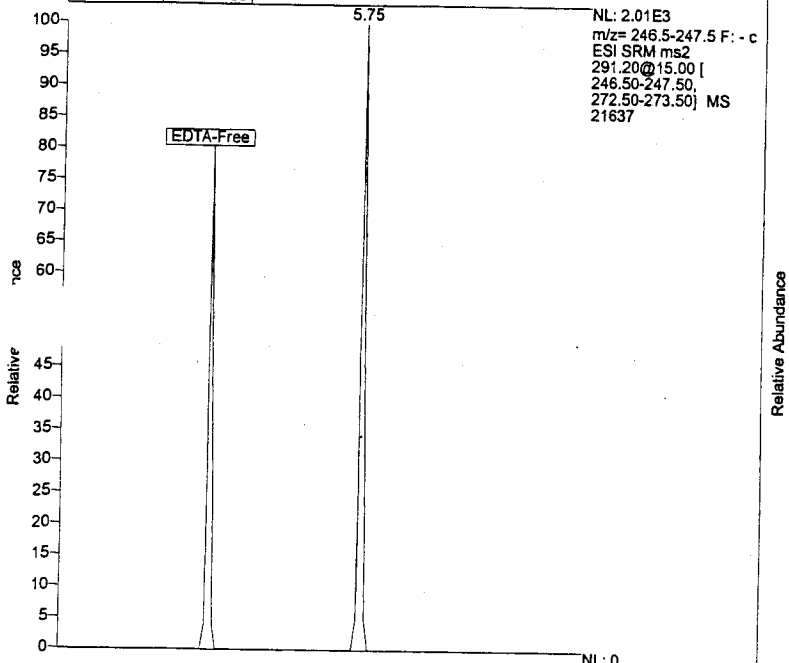


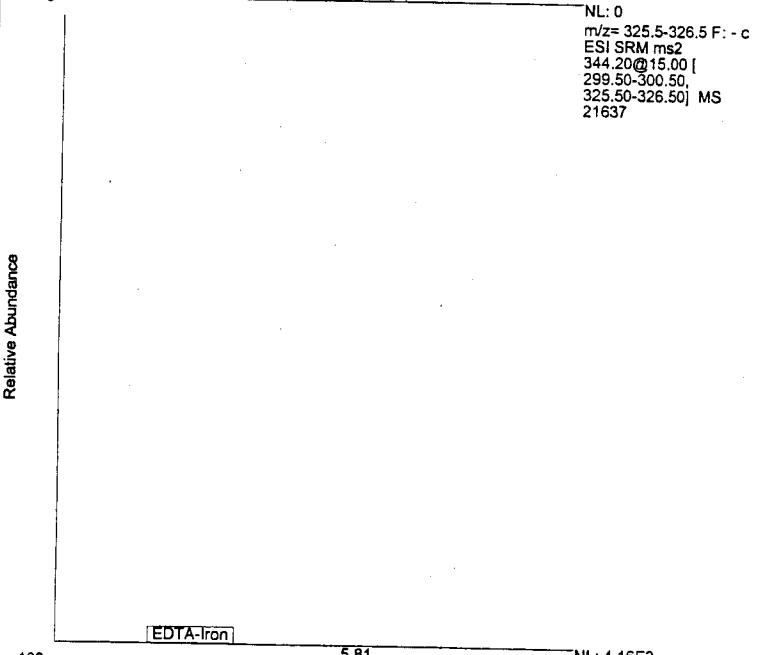
Ex 446
(4/7)

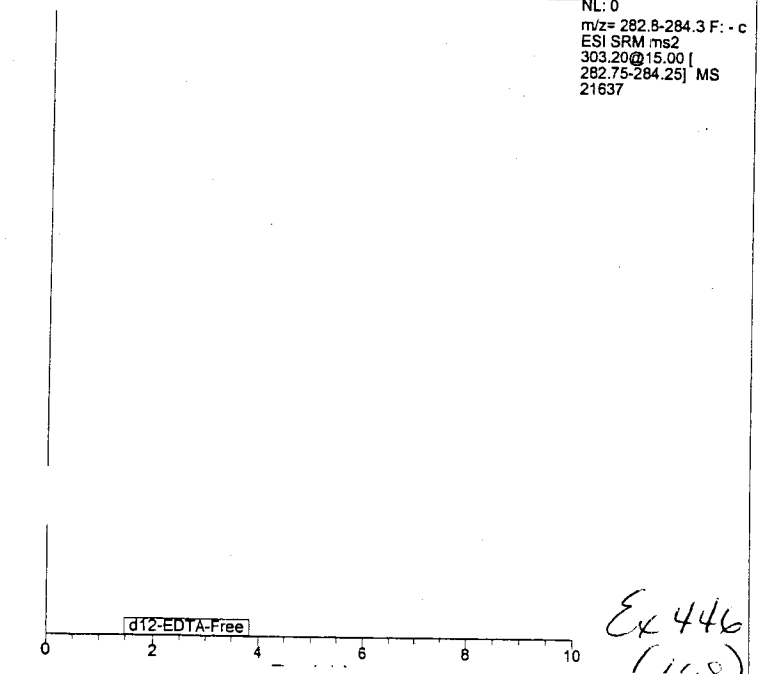
C:\Xcalibur\...\Negative Ion\21637 070201013 JDB 02/16/07 07:15:23 BLANK (neg blood, DI H2O ext.)

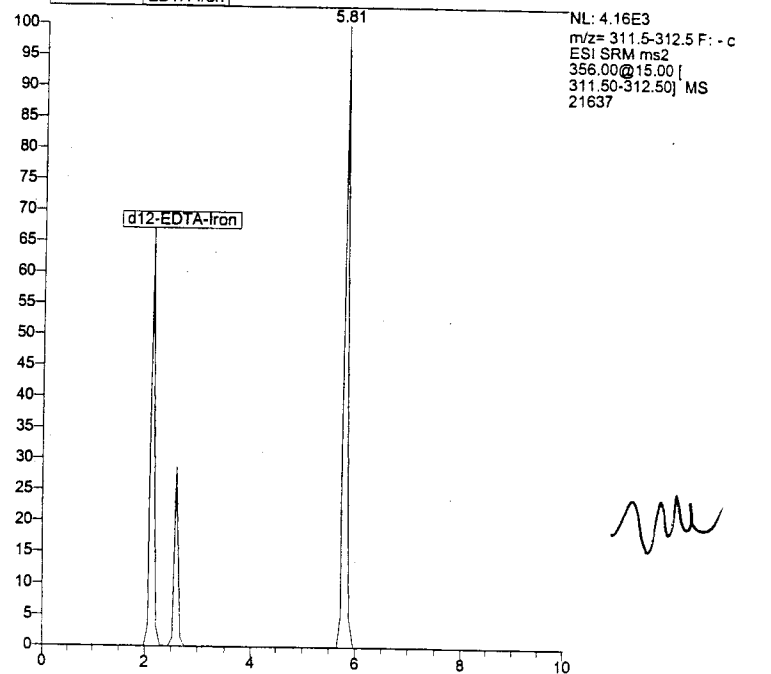




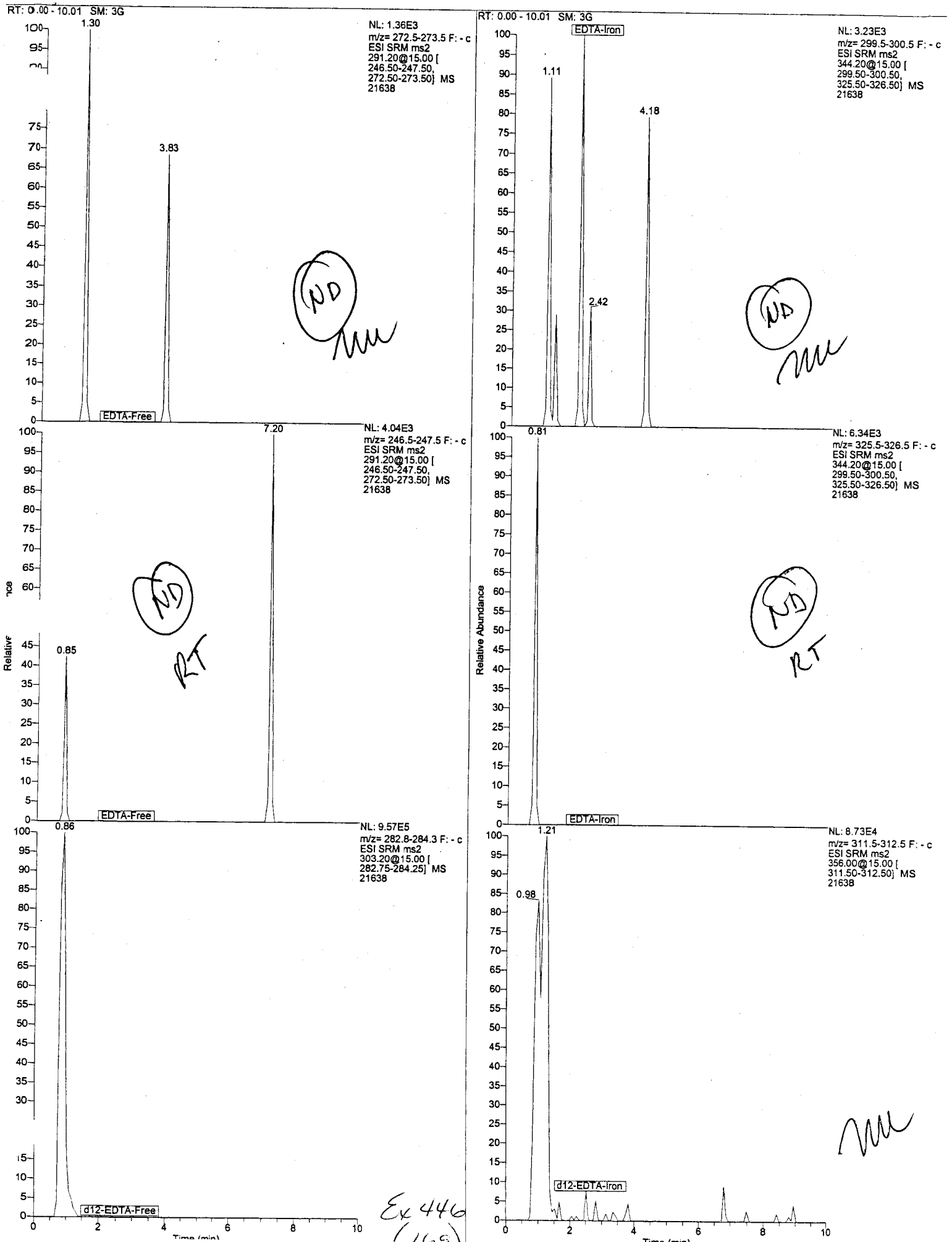






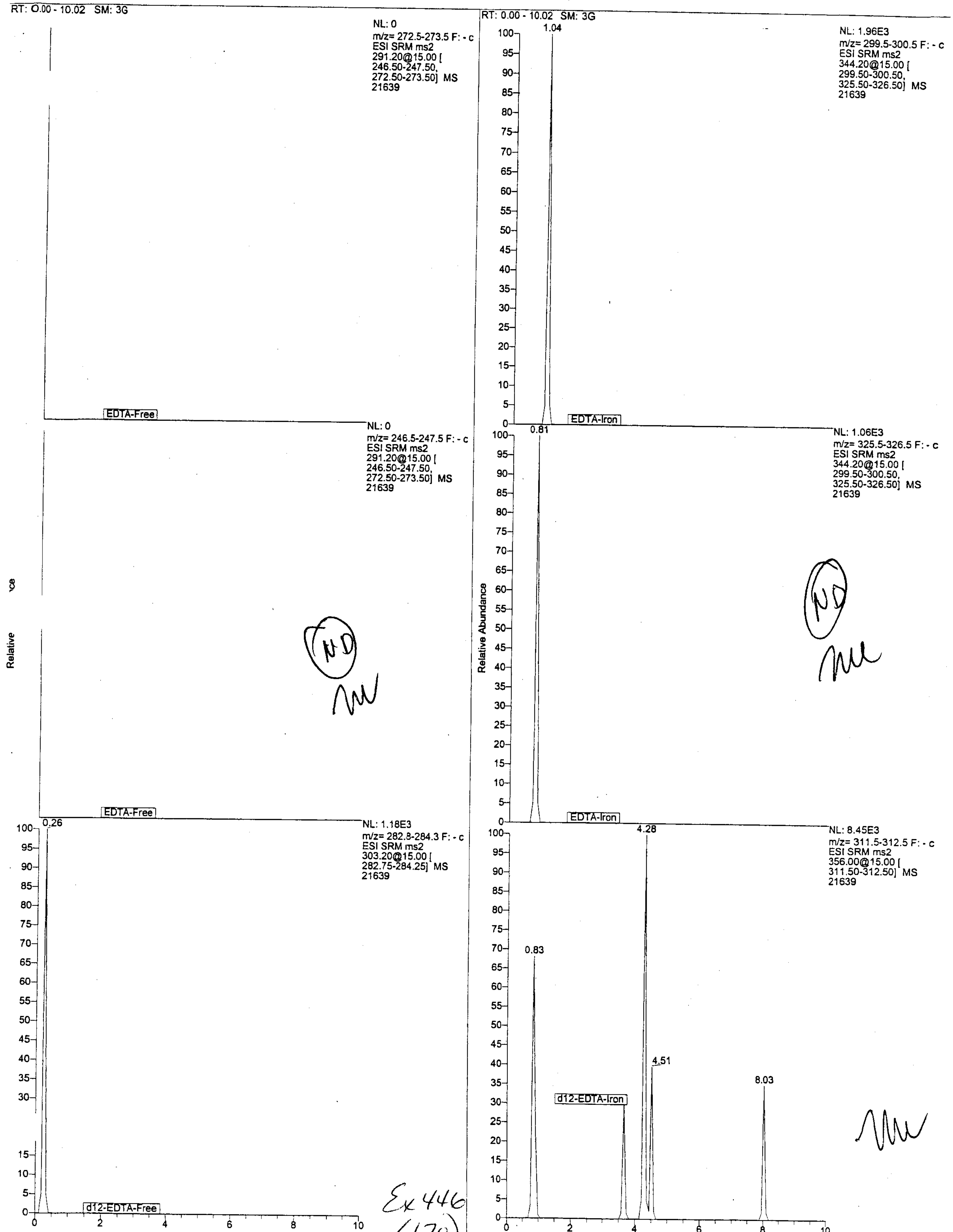


C:\Xcalibur\...\Negative Ion\21638 070201013 JDβ 02/16/07 07:26:12 K2 extract

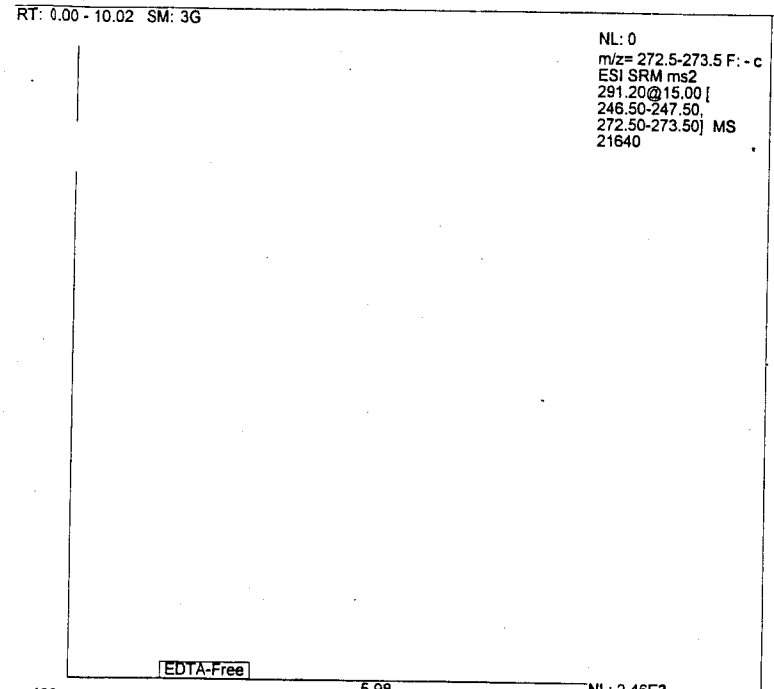


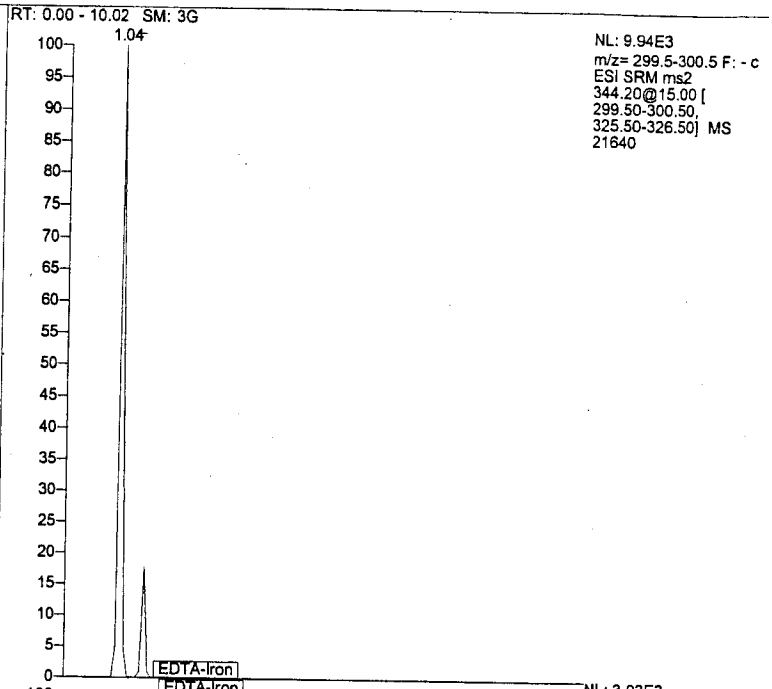
Ex 446

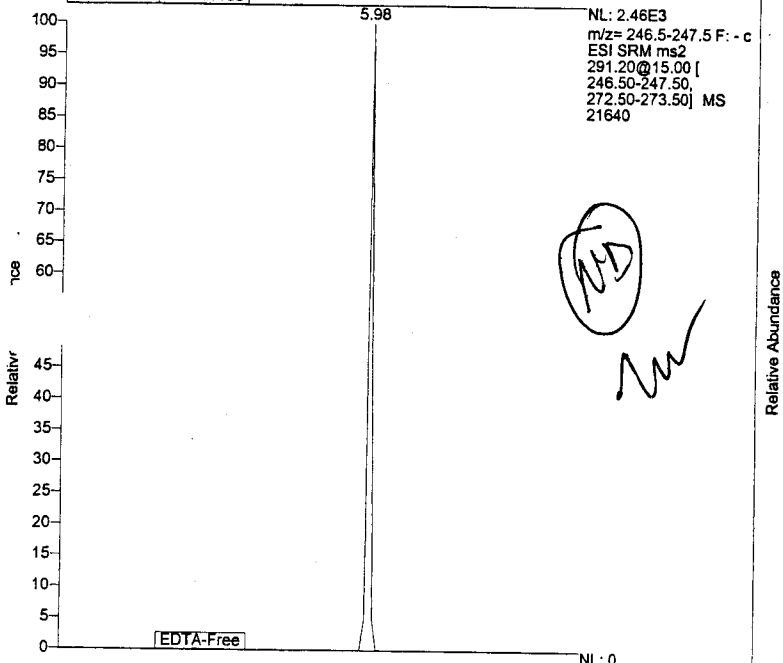
C:\Xcalibur\...\Negative Ion\21639 070201013 JDB 02/16/07 07:37:08 BLANK (neg blood, DI H2O ext.)



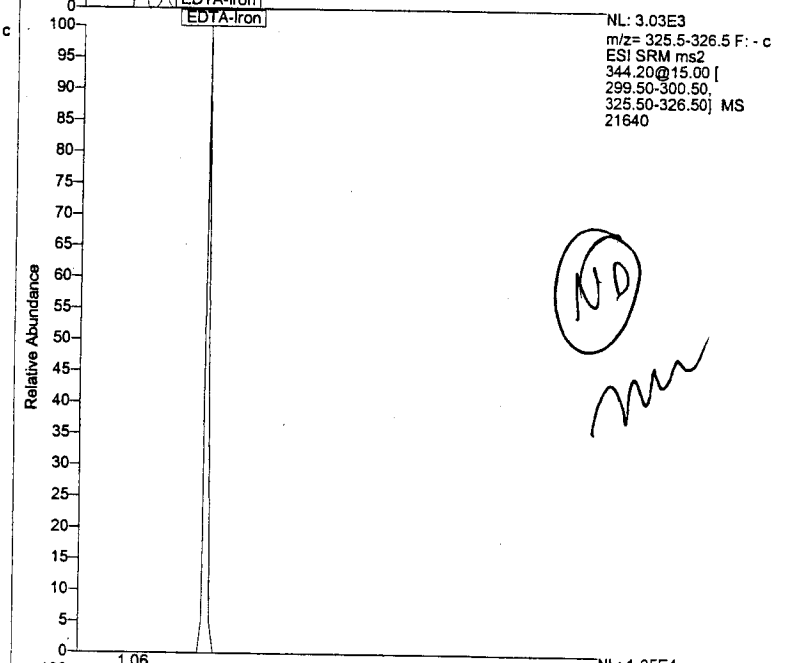
C:\Xcalibur\...\Negative Ion\21640 C7020/013 JDB 02/16/07 07:48:03 BLANK (neg blood, DI H2O ext.)



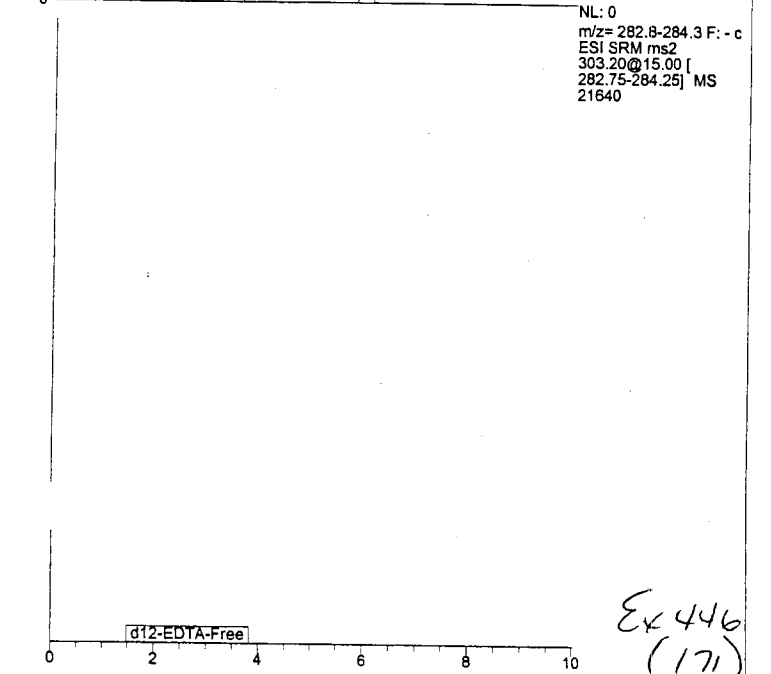




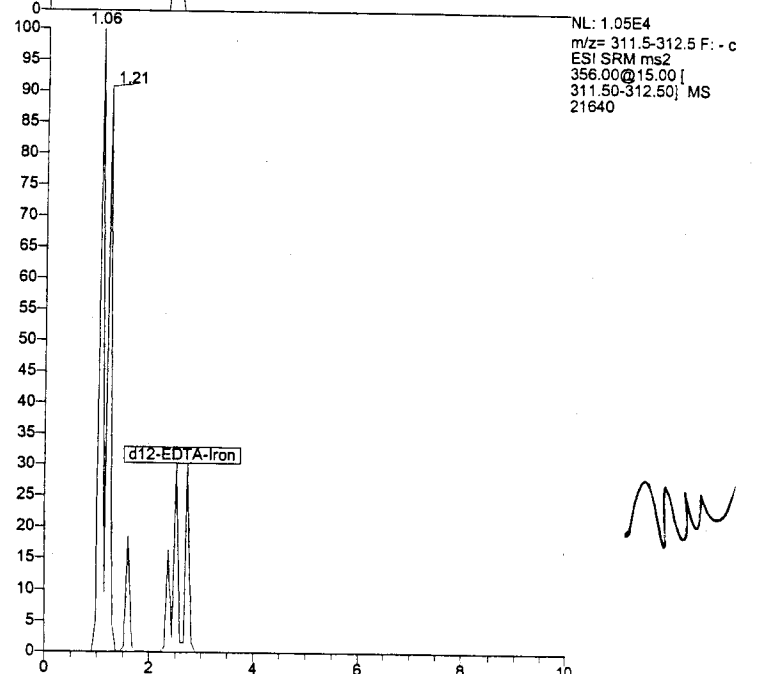
ND



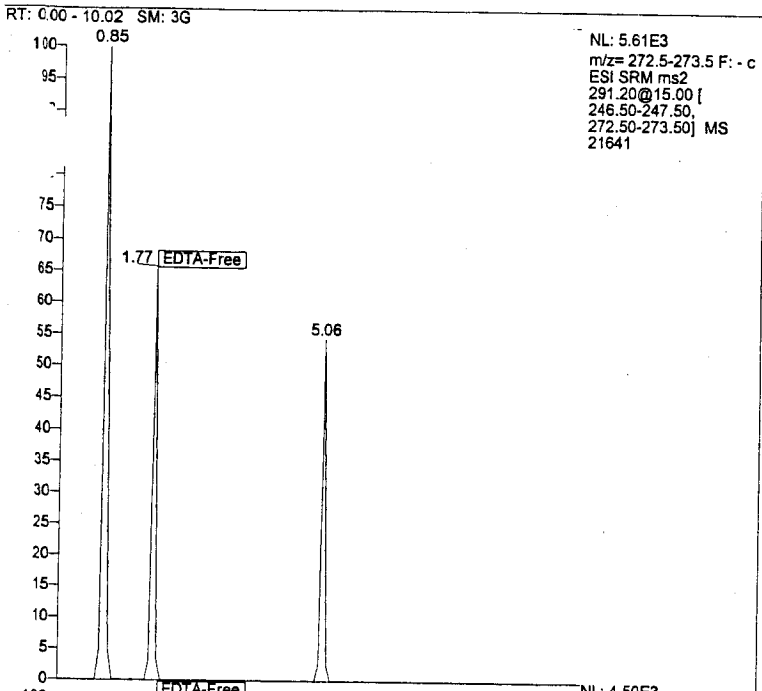
ND

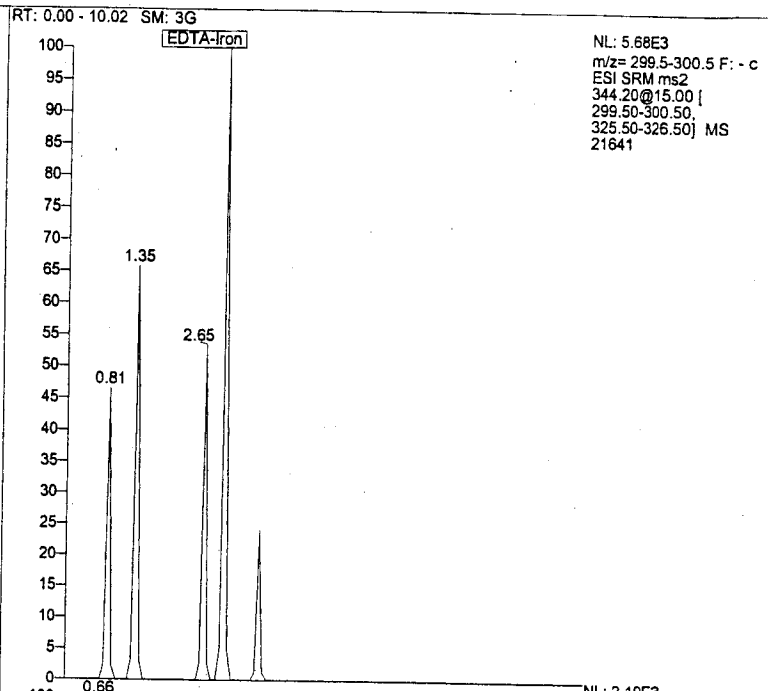


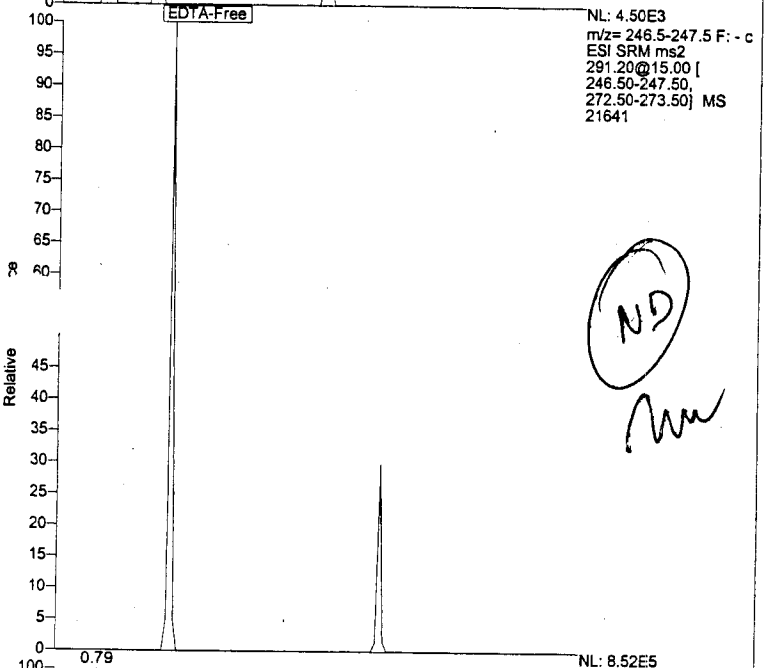
Ex 446 (171)

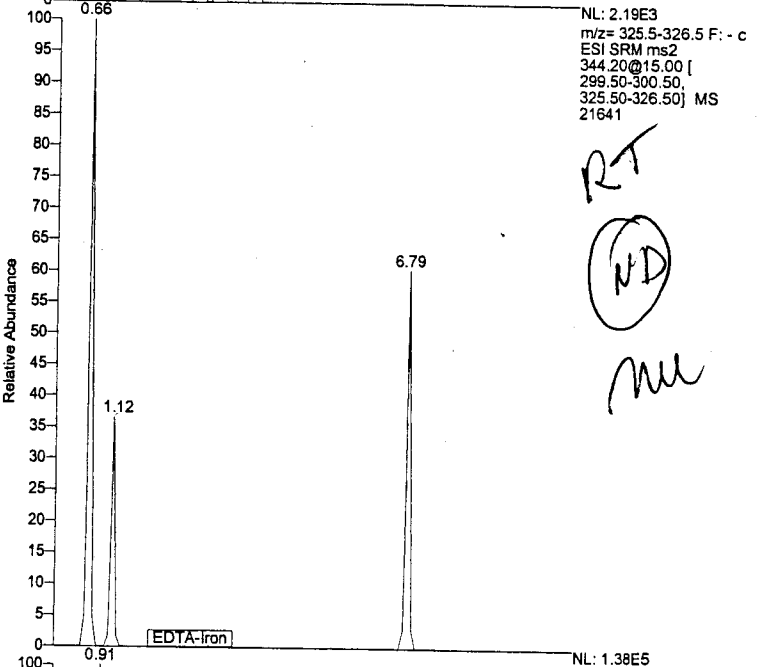


C:\Xcalibur\...\Negative Ion\21641 070201013 JDB 02/16/07 07:58:57 Q46 extract





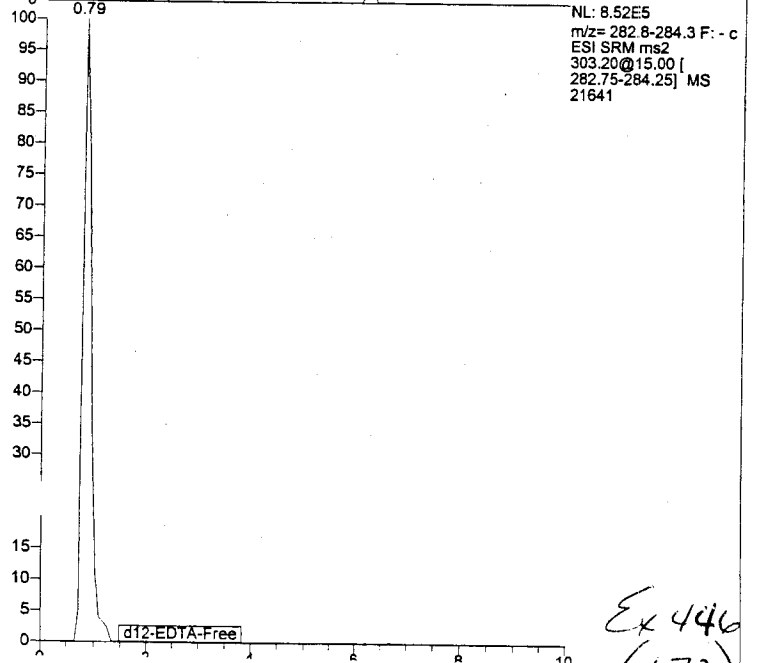


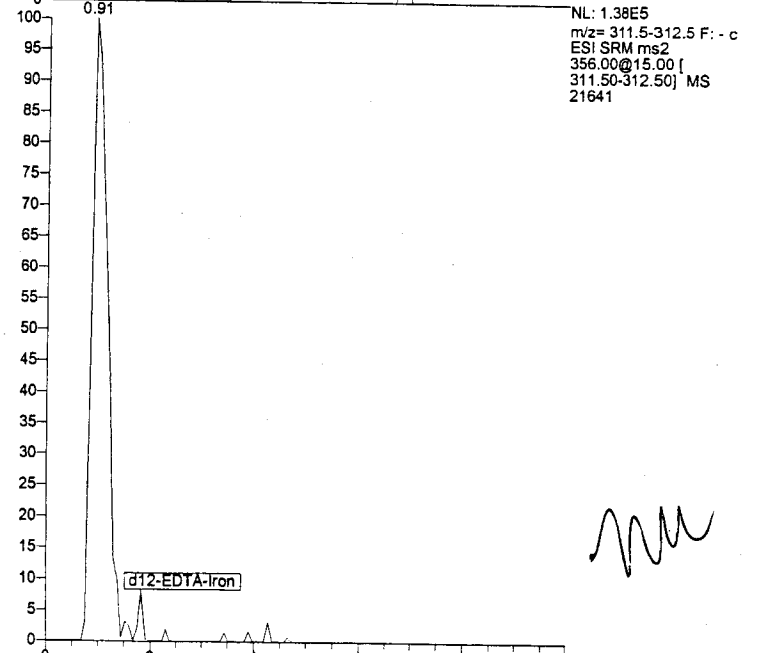


ND

RT

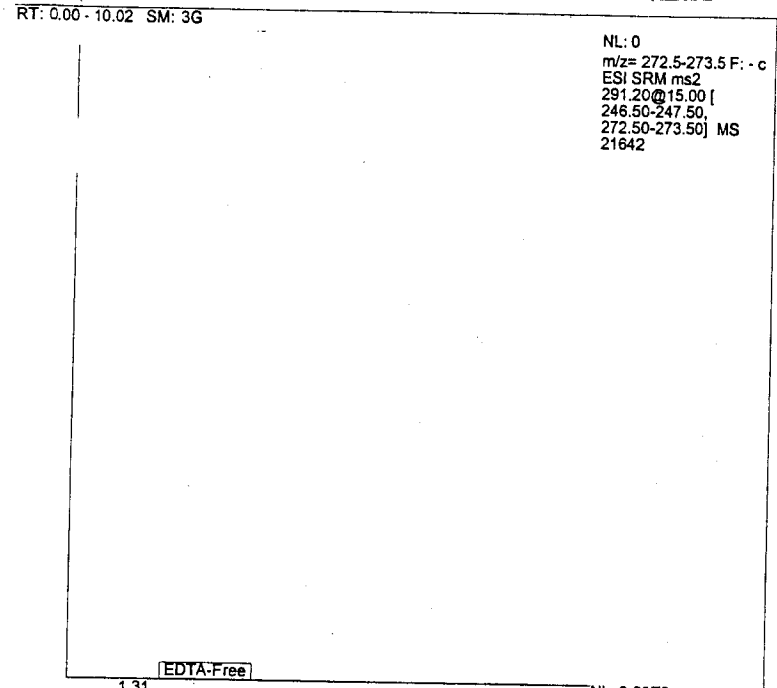
ND

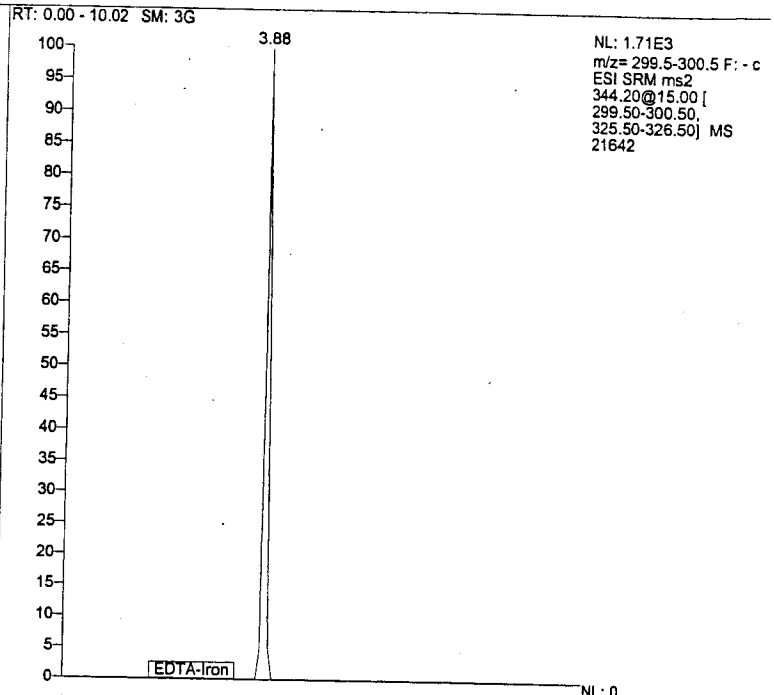


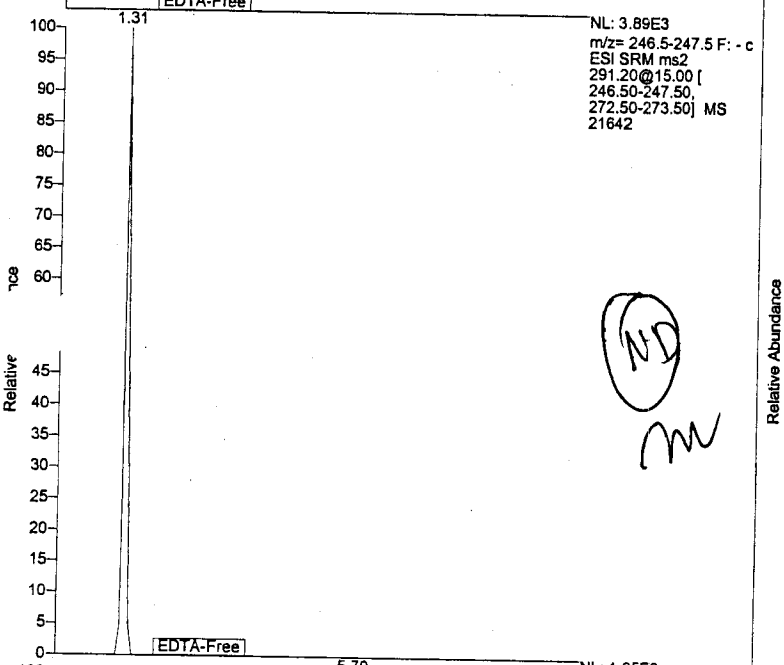


Ex 446

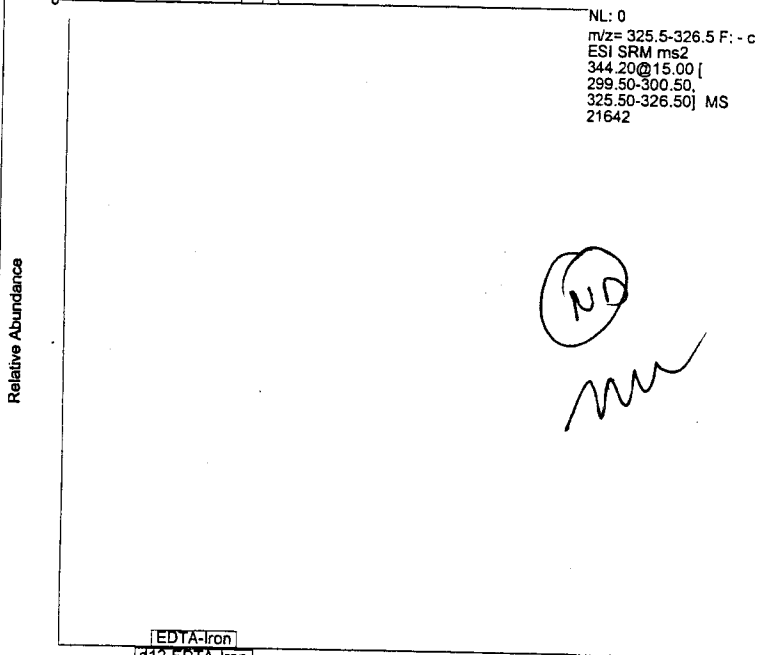
C:\Xcalibur\...\Negative Ion\21642 070201013 JDB 02/16/07 08:09:50 BLANK (neg blood, DI H2O ext.)



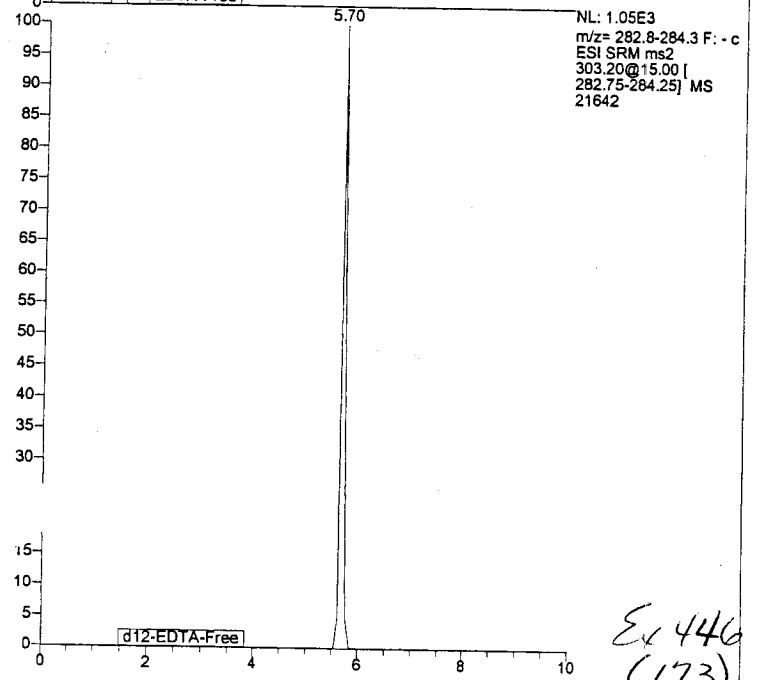




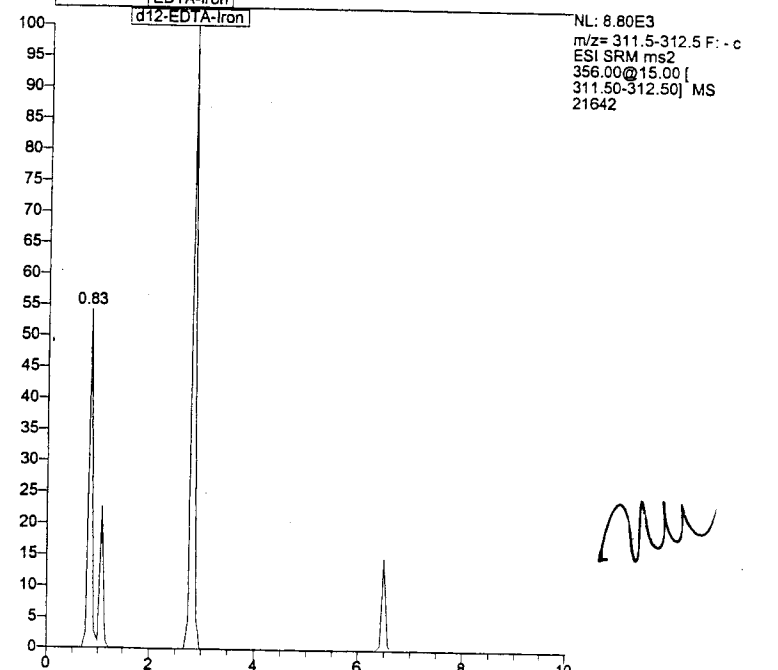
ND



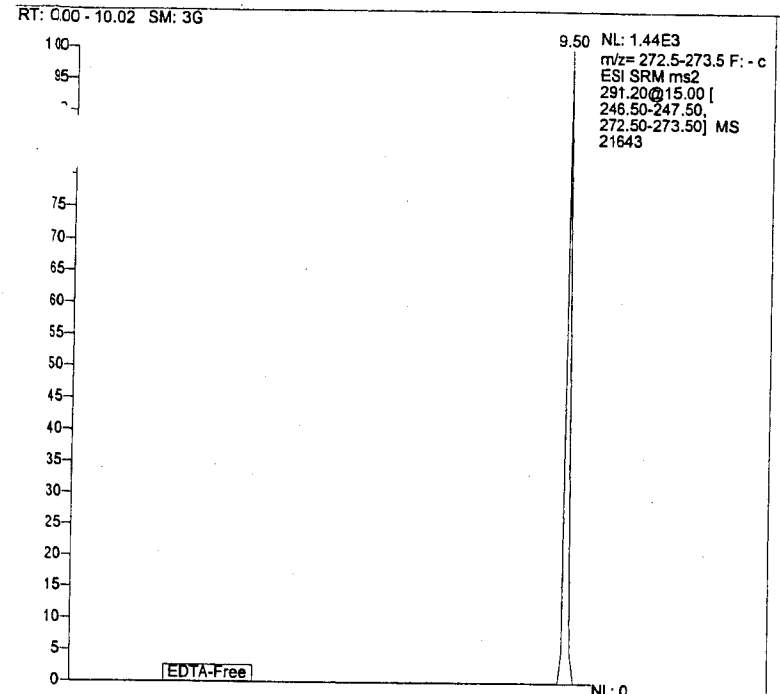
ND

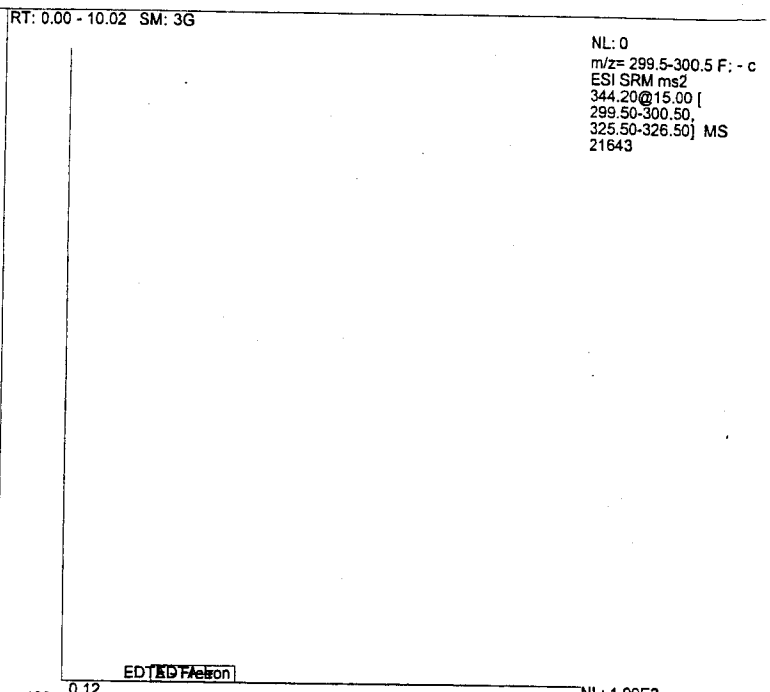


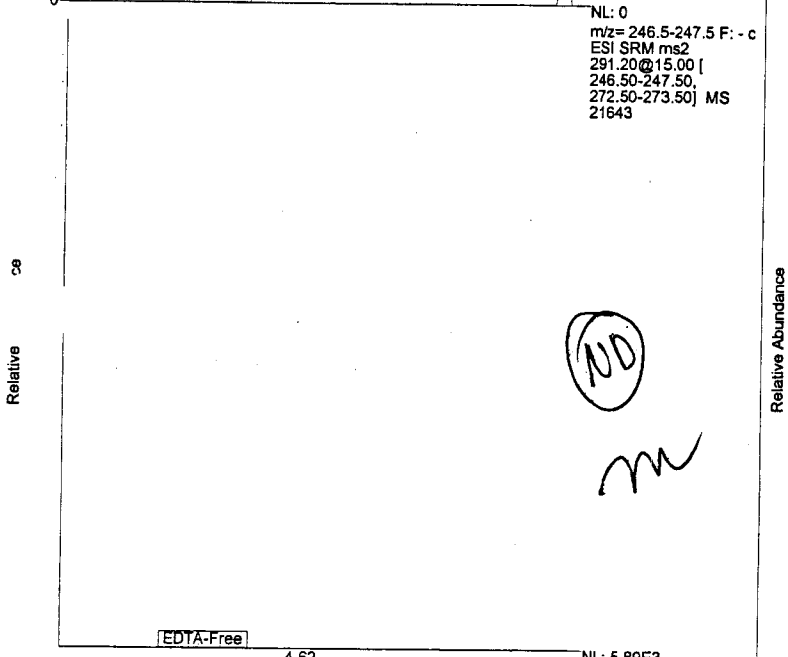
Ex 446
(173)

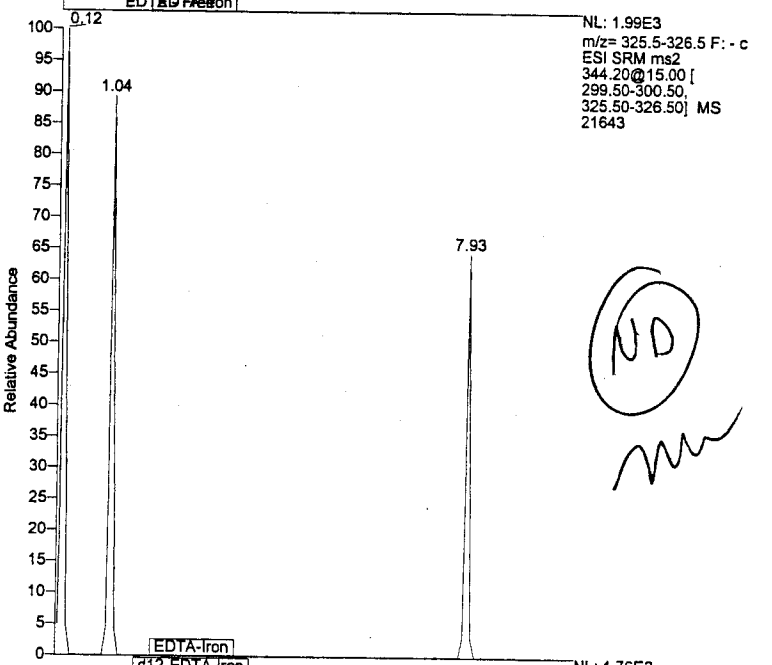


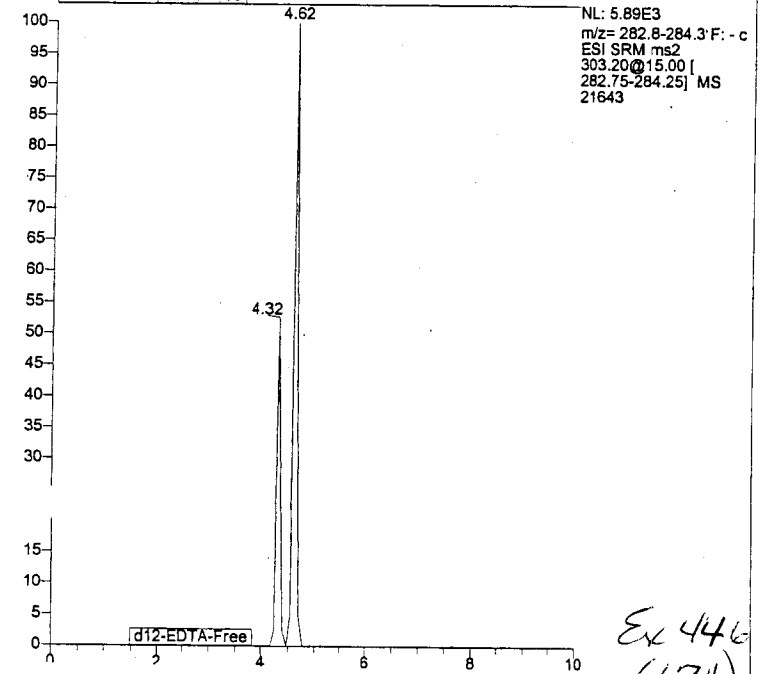
C:\Xcalibur\...\Negative Ion\21643 07020103 JDB 02/16/07 08:20:45 BLANK (neg blood, DI H2O ext.)

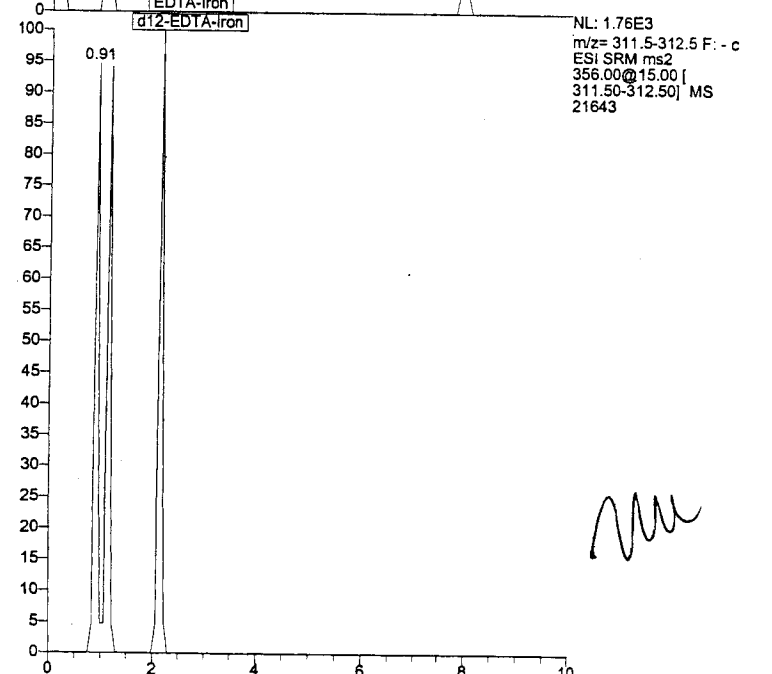




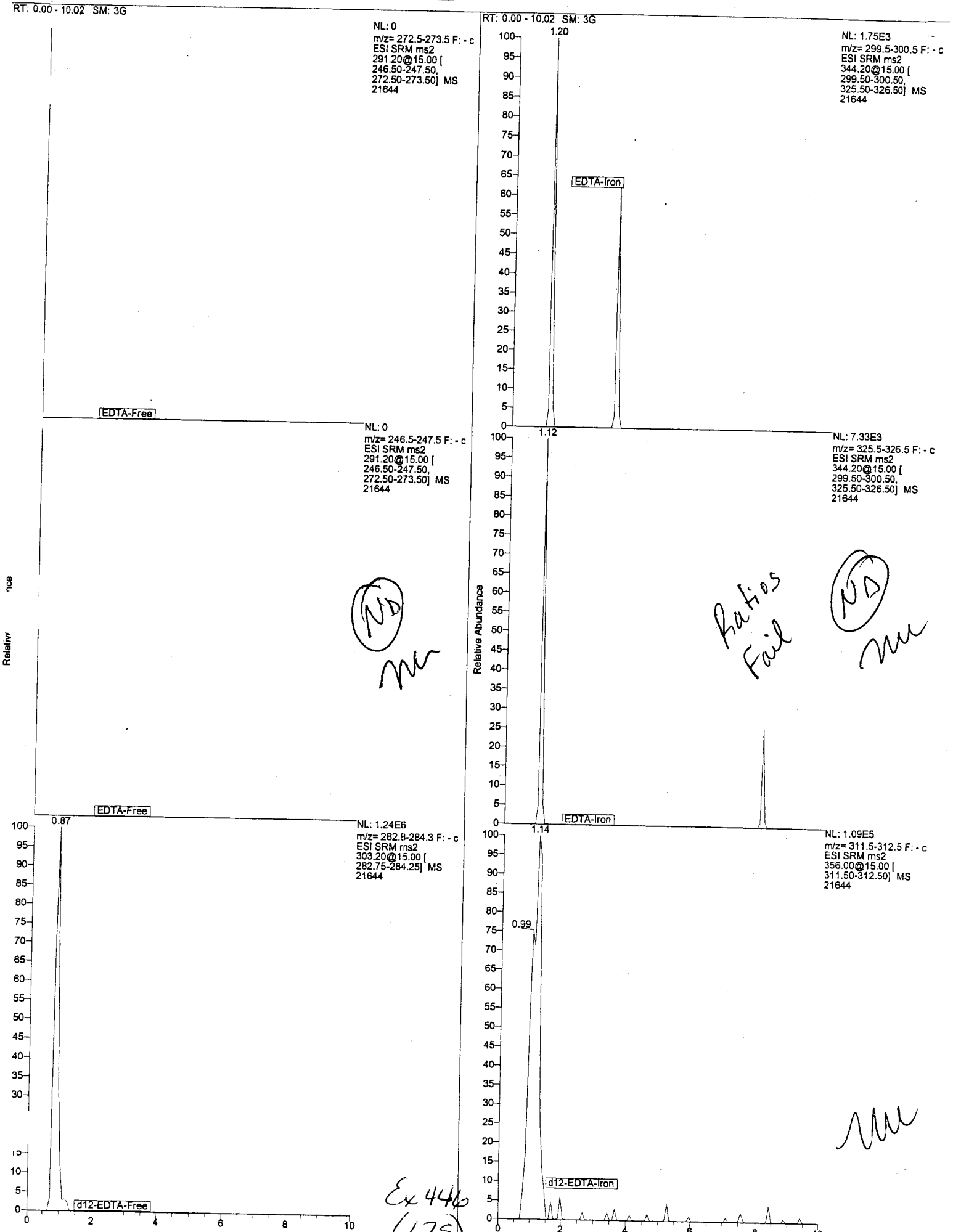




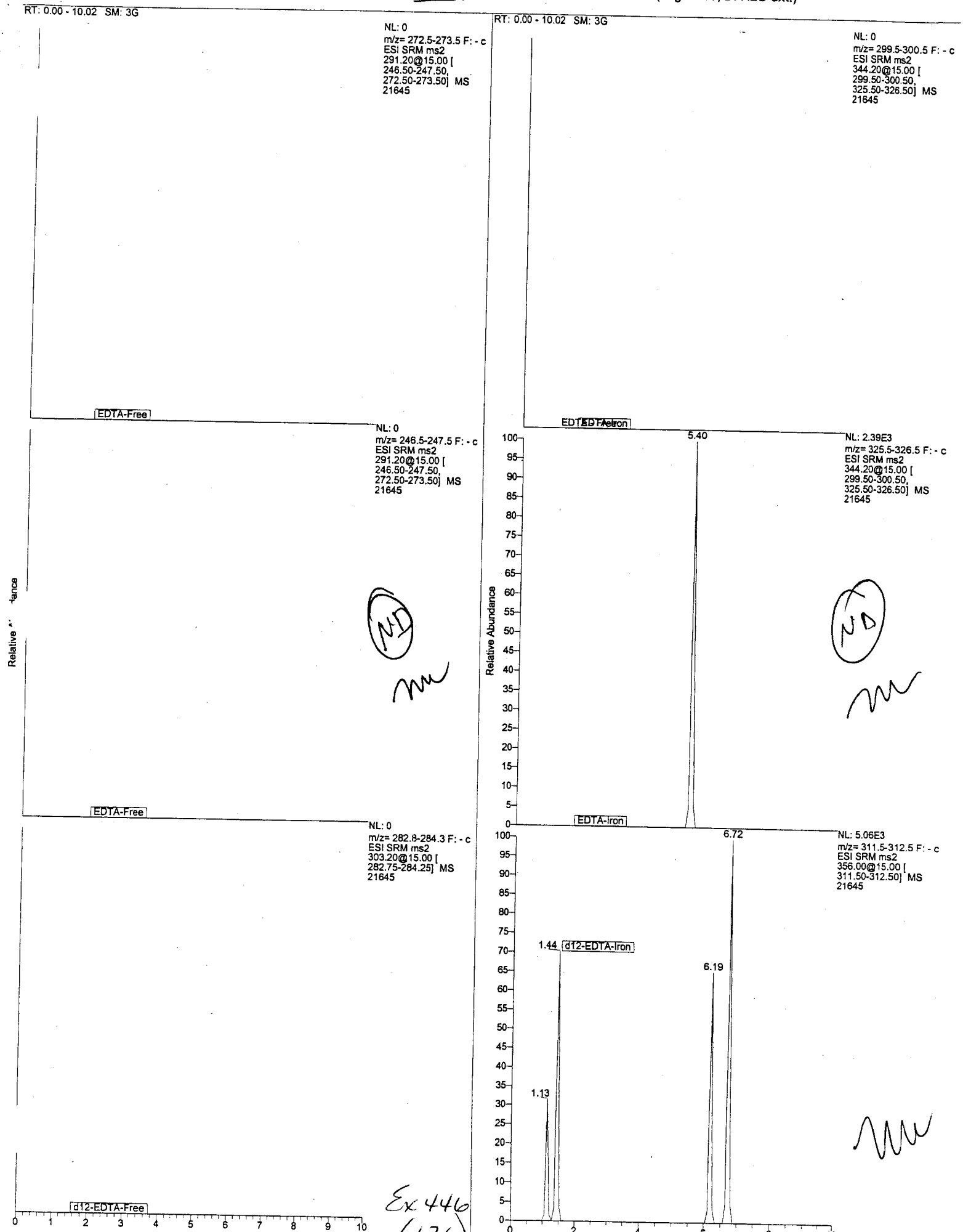




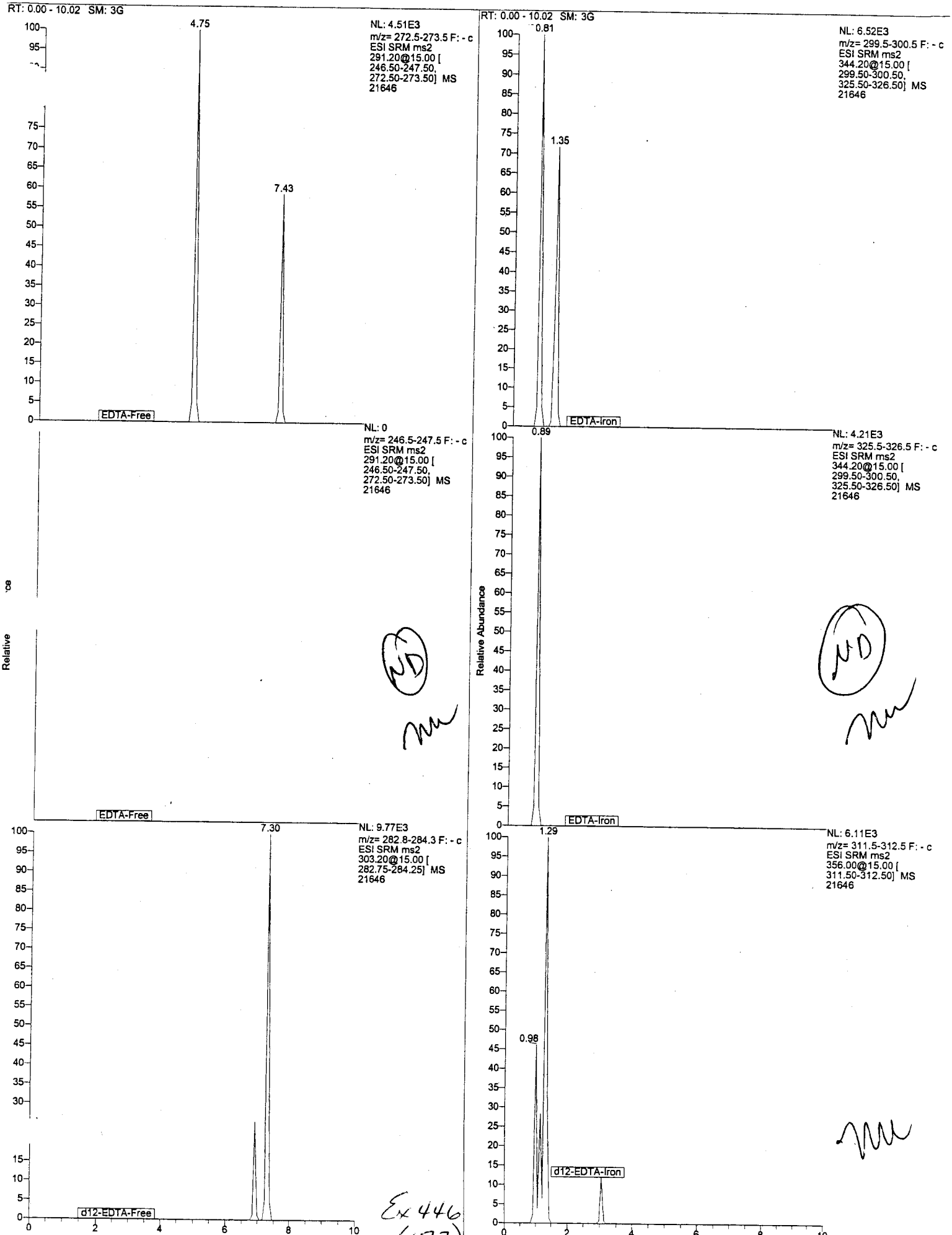
C:\Xcalibur\...\Negative Ion\21644 07020103 JDB 02/16/07 08:31:35 K3 extract



C:\Xcalibur\...\Negative Ion\21645 070201013 JDB 02/16/07 08:42:24 BLANK (neg blood, DI H2O ext.)



C:\Xcalibur\...\Negative Ion\21646 070201013 JDB 02/16/07 08:53:19 BLANK (neg blood, DI H2O ext.)

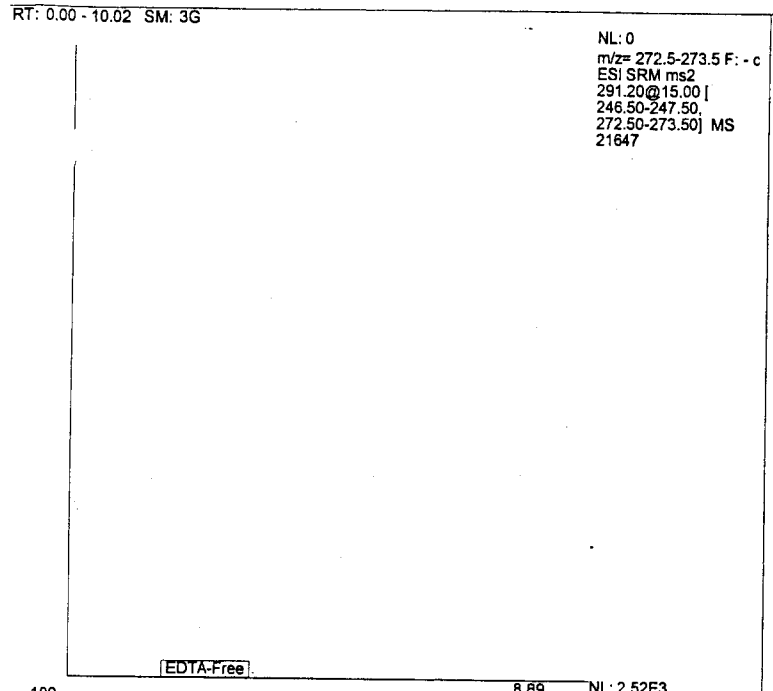


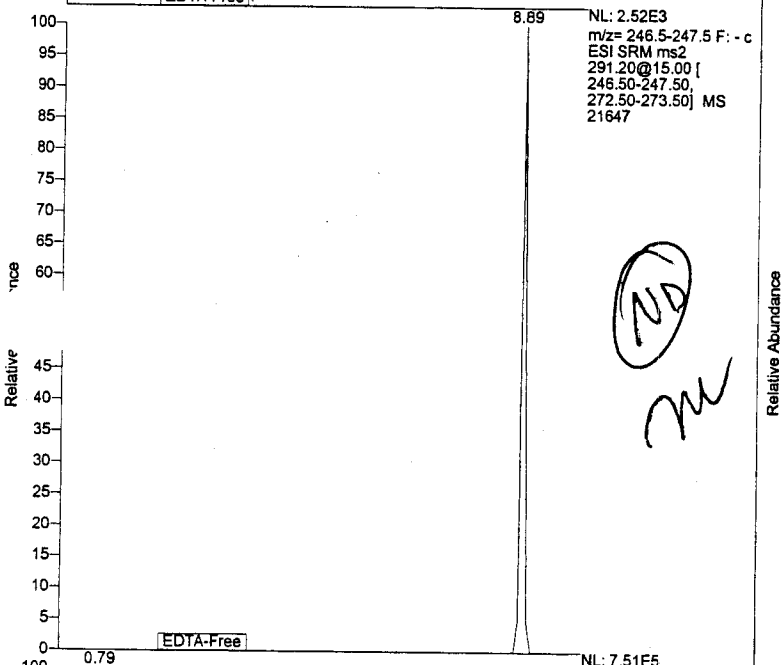
ND

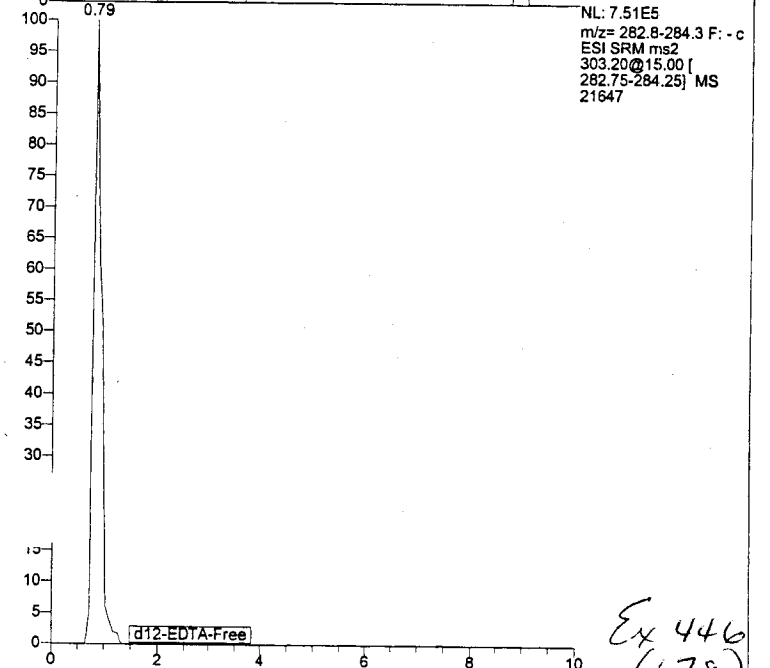
ND

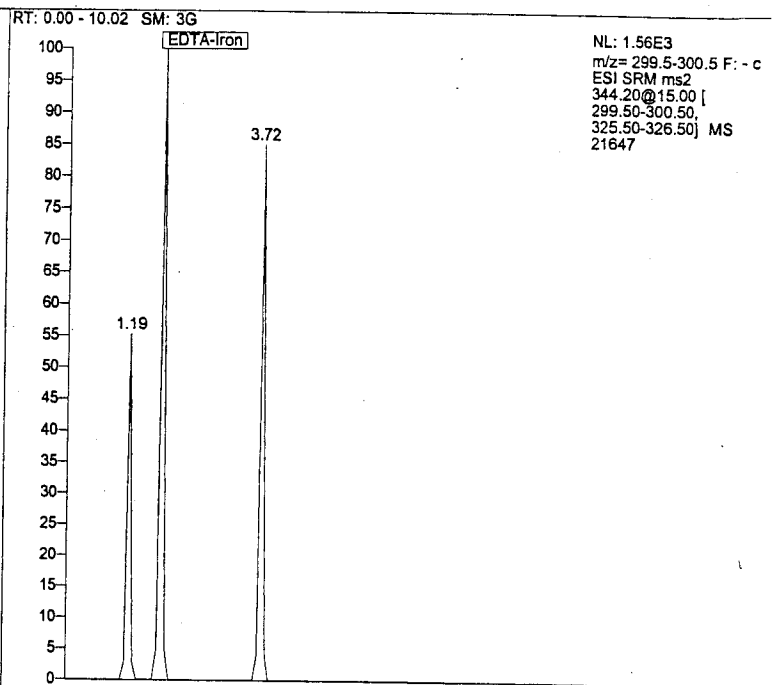
Ex 446

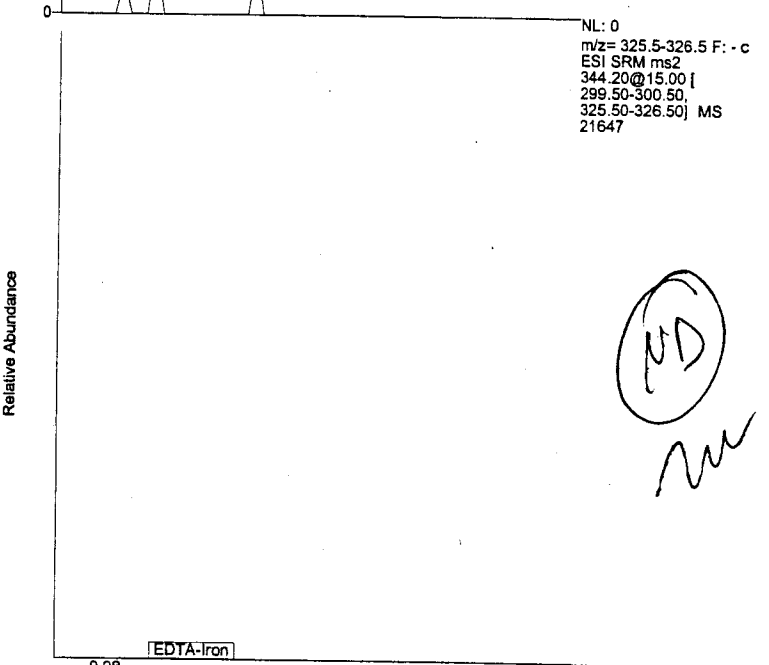
C:\Xcalibur\...\Negative Ion\21647 07020103 JDB 02/16/07 09:04:15 Q47 extract

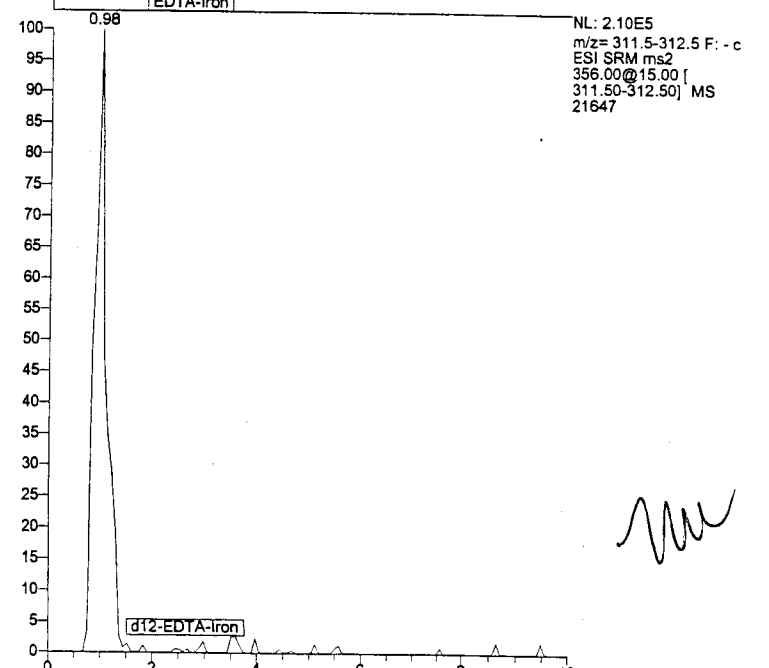






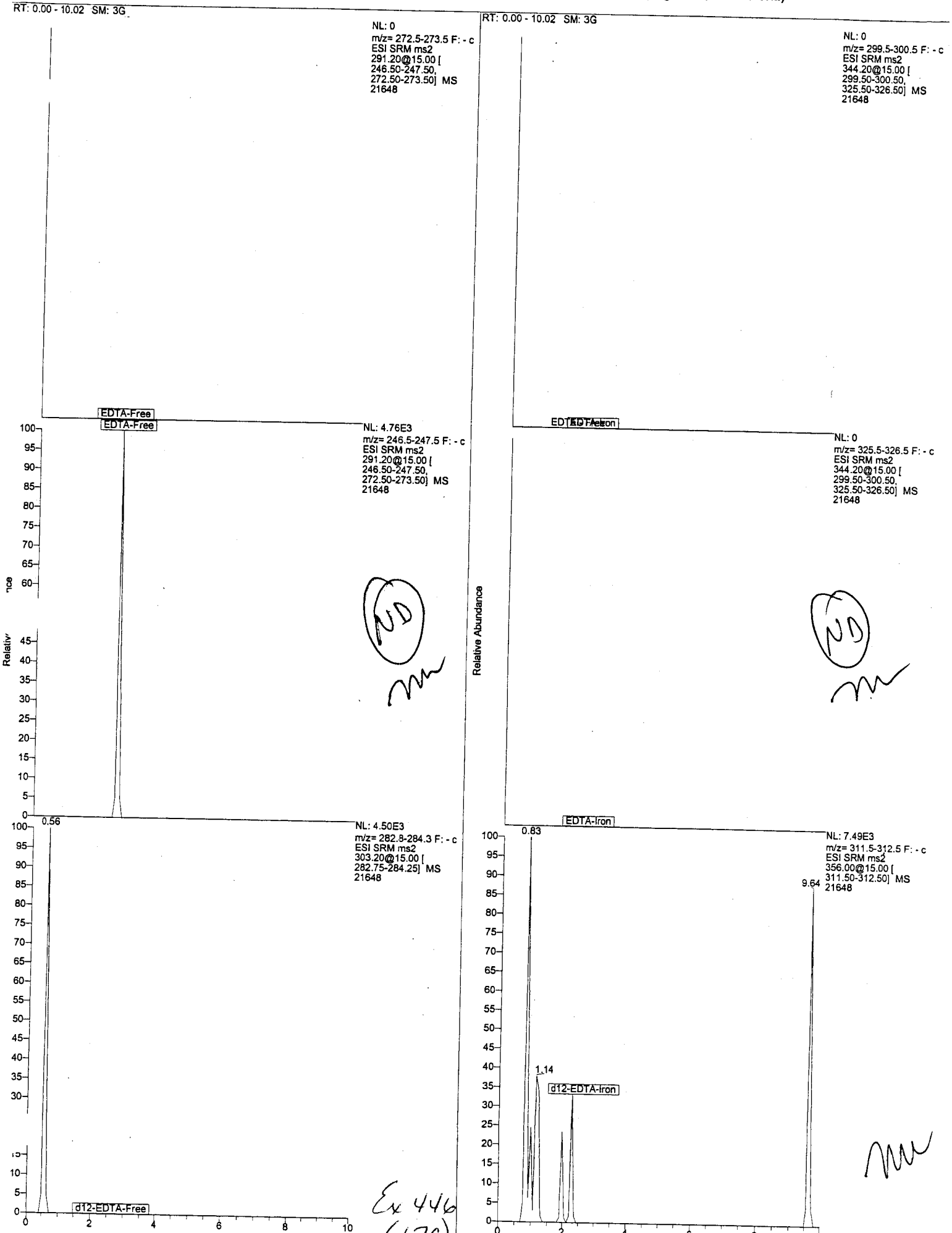




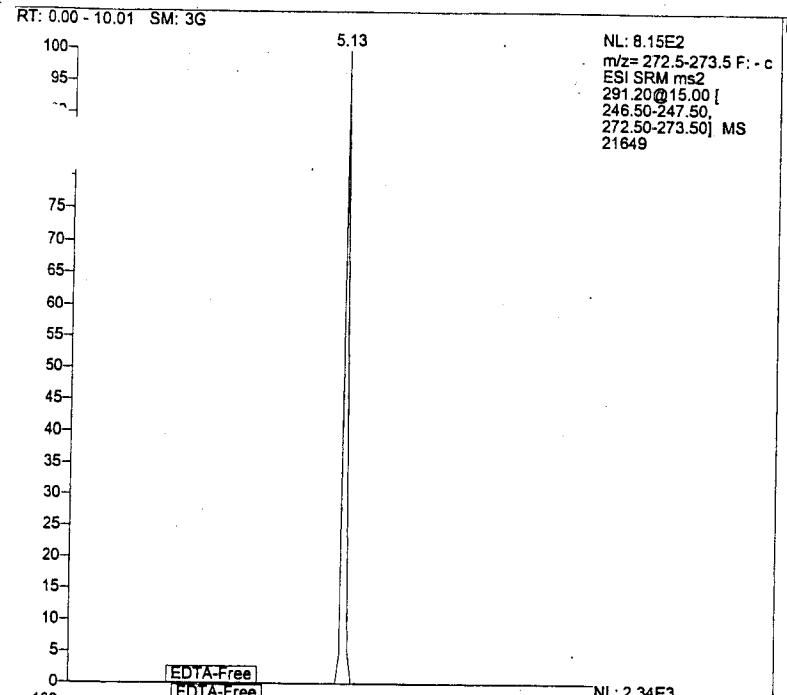


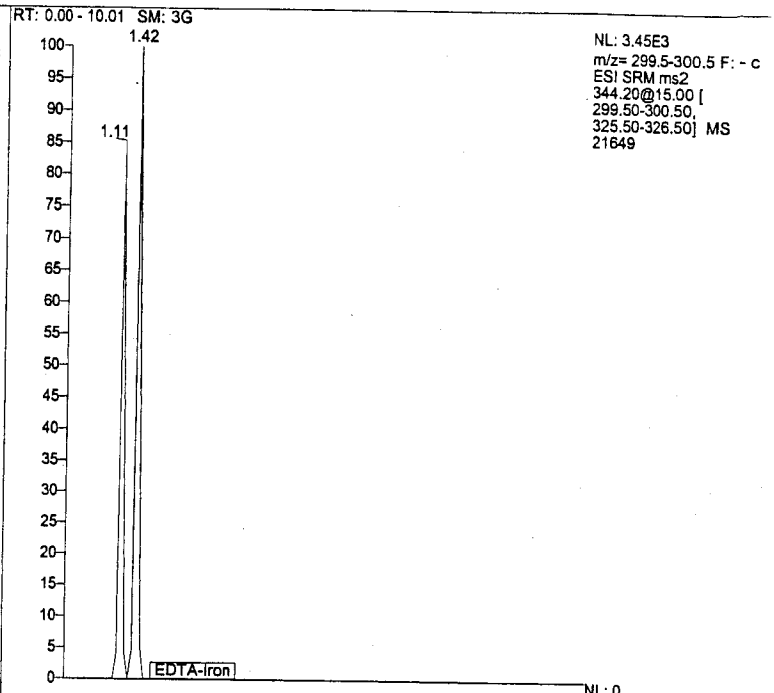
Ex 446

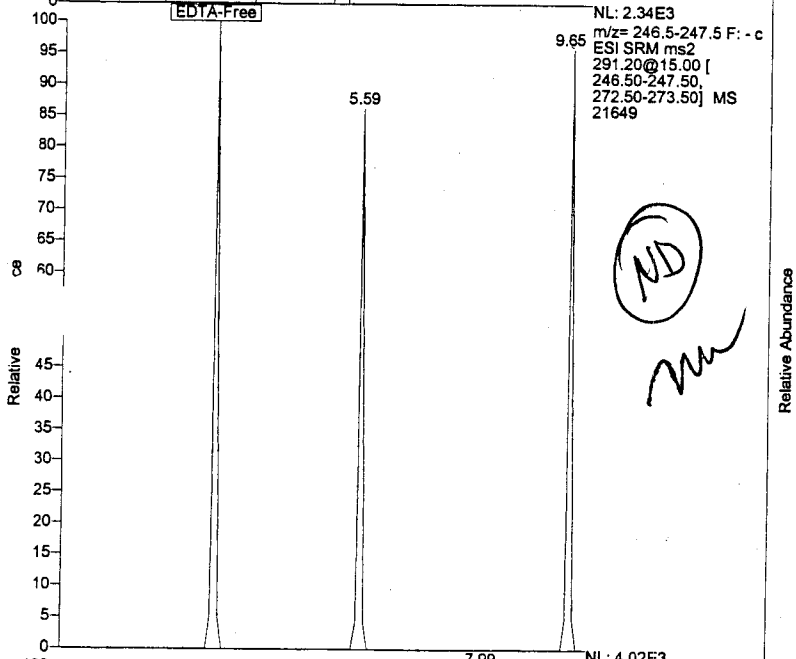
C:\Xcalibur\...\Negative Ion\21648 070201013 JDB 02/16/07 09:15:08 BLANK (neg blood, DI H2O ext.)

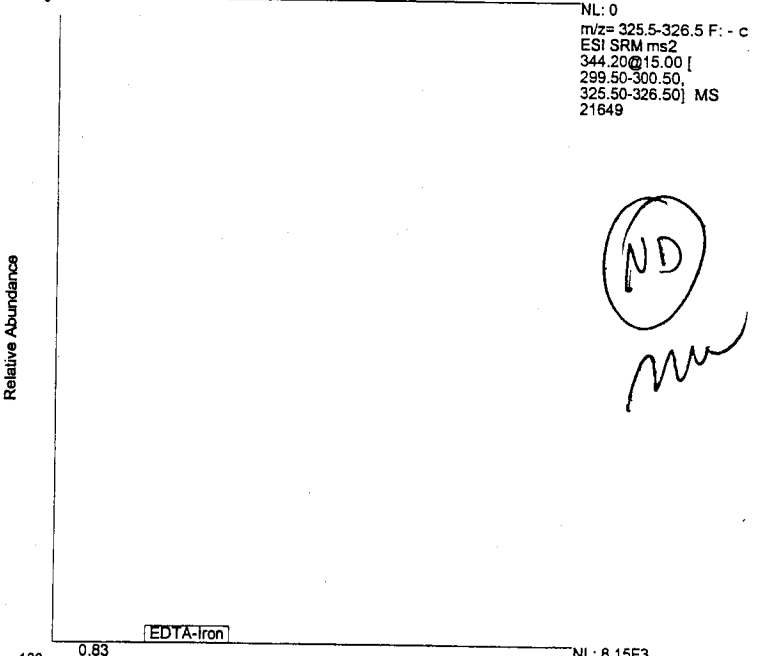


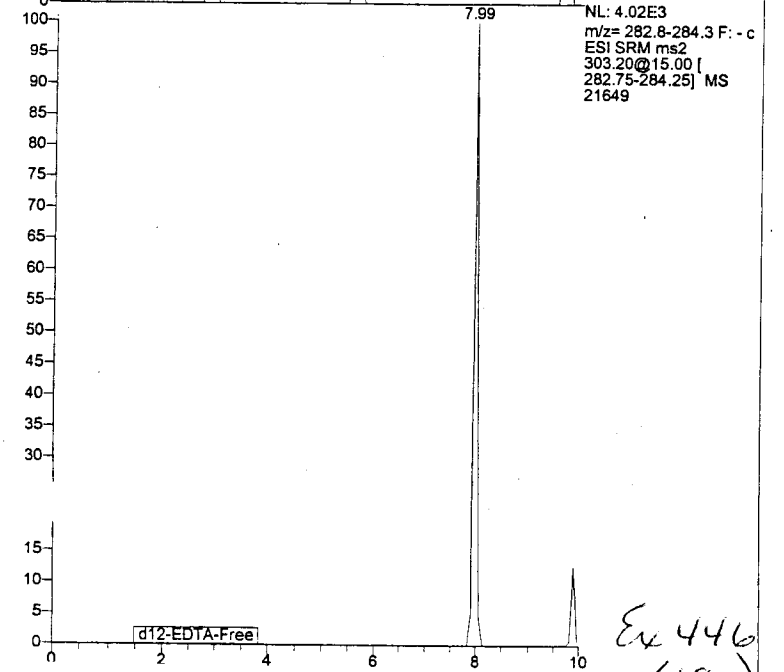
C:\Xcalibur\...\Negative Ion\21649 070201013 TDB 02/16/07 09:26:04 BLANK (neg blood, DI H2O ext.)

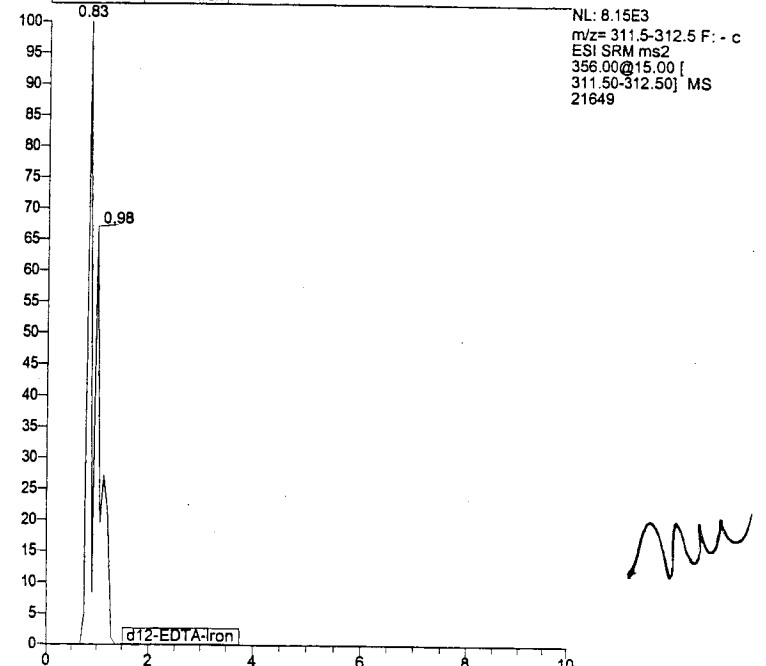




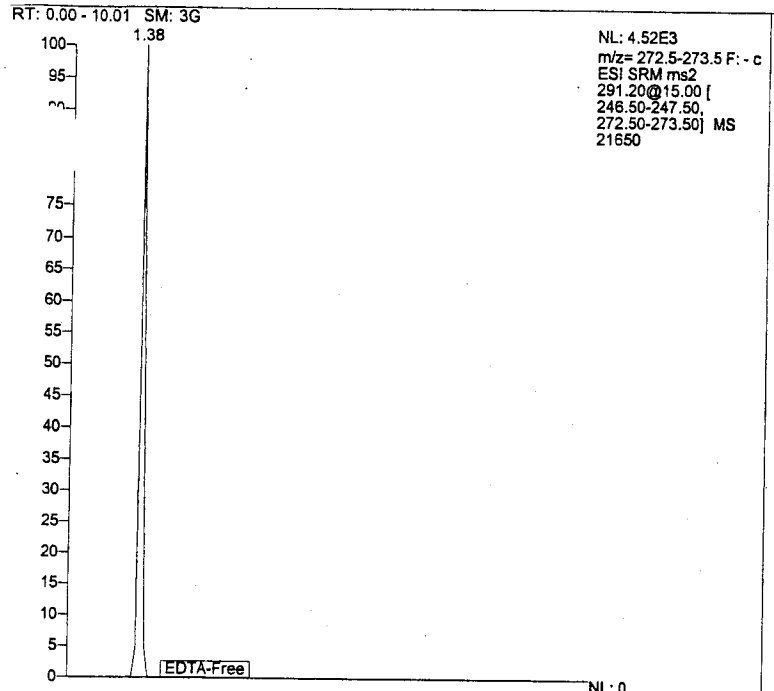


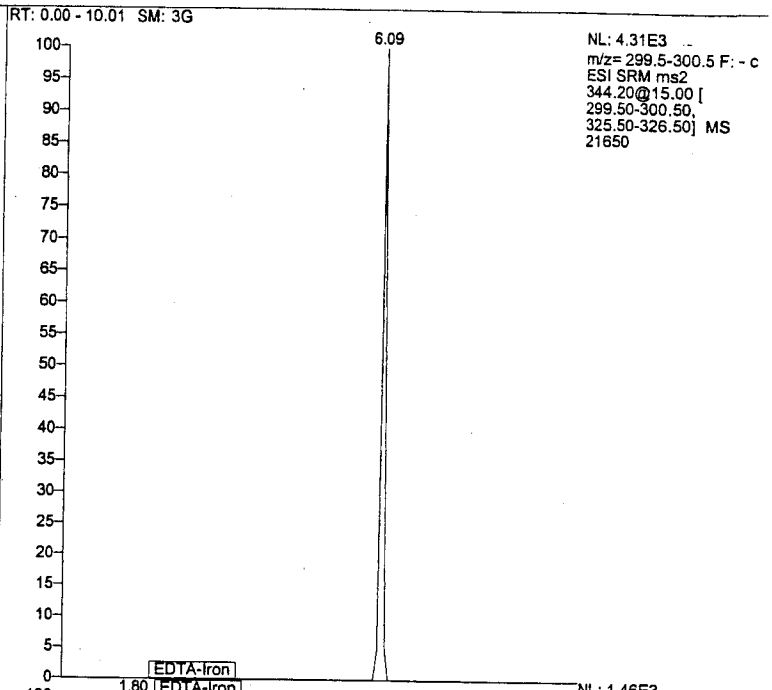


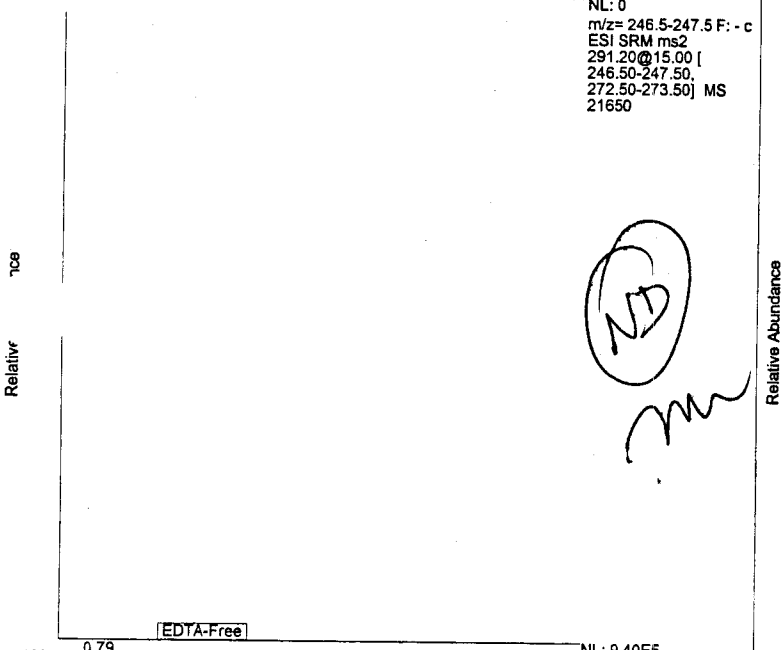


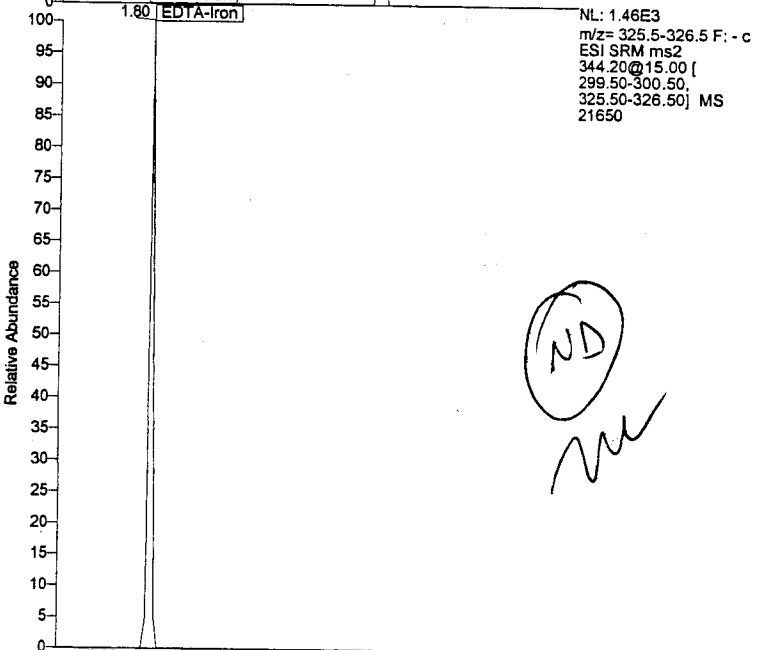


C:\Xcalibur\...\Negative Ion\21650 070201013 JDB 02/16/07 09:36:53 K4 extract



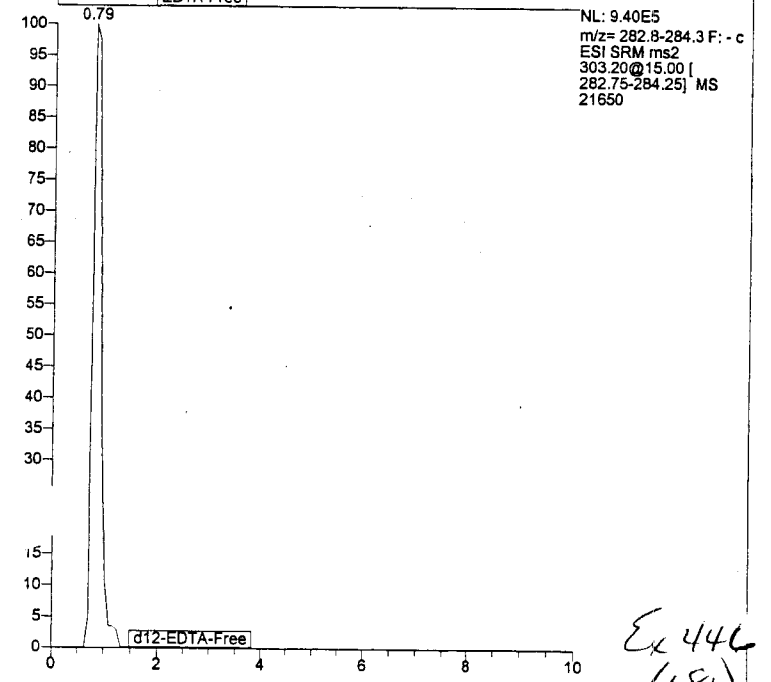


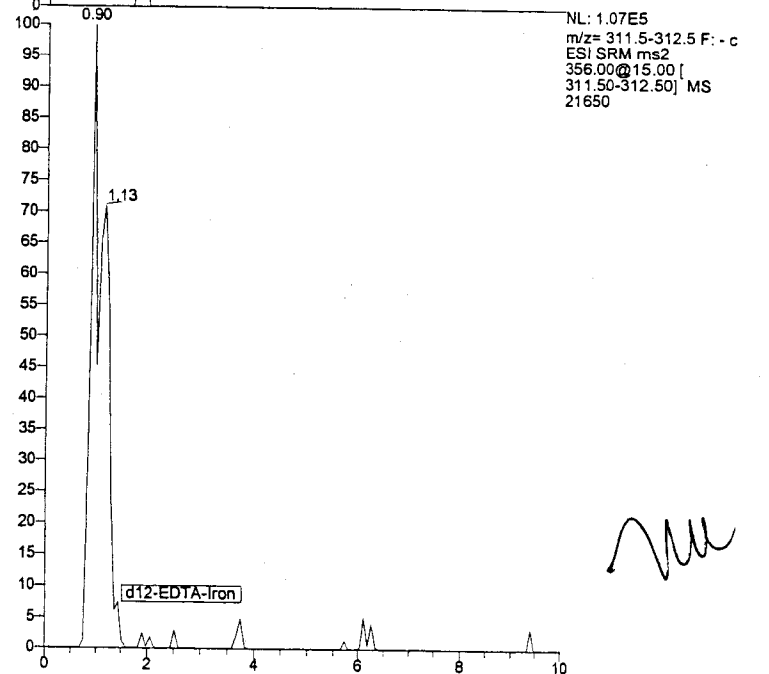




ND

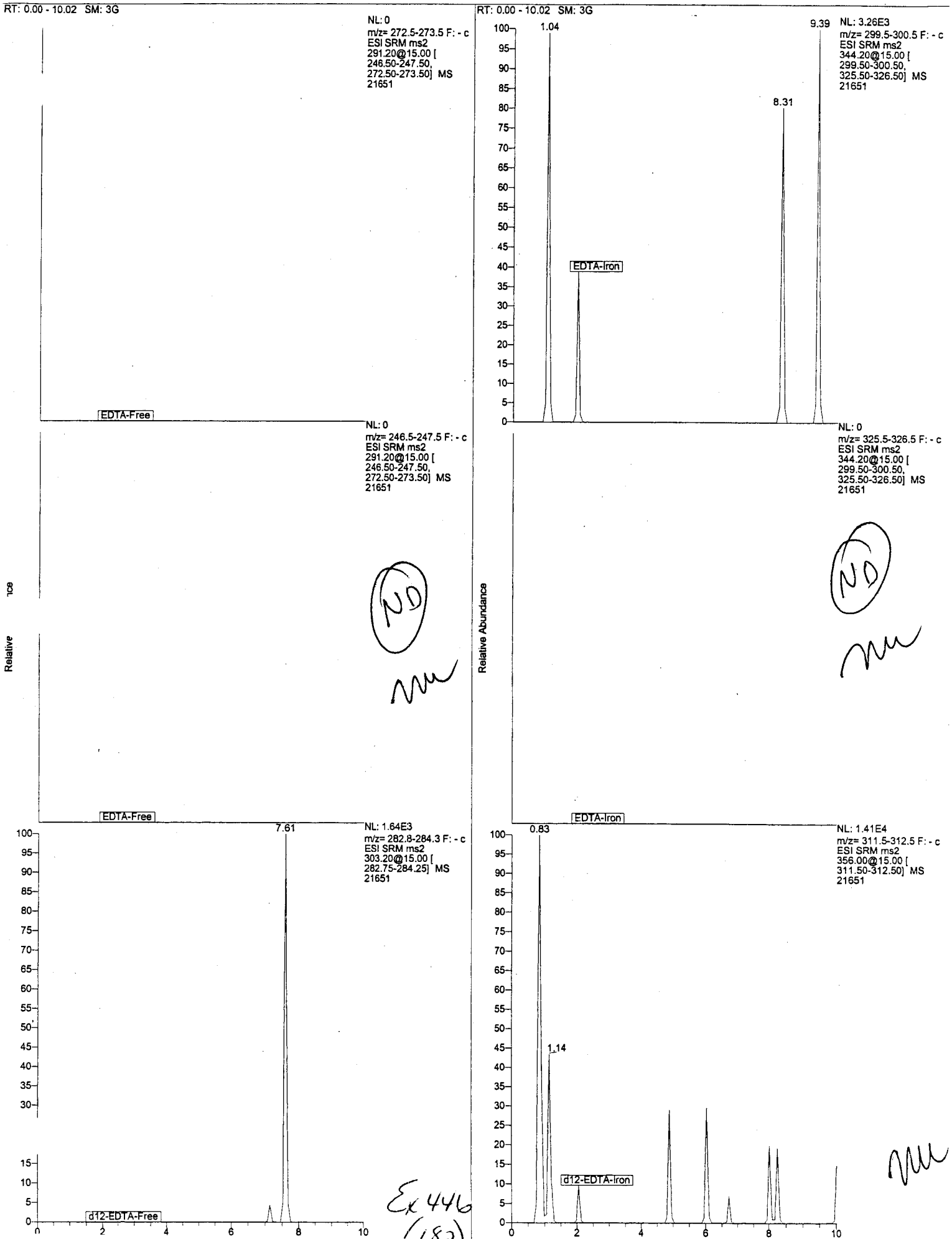
ND





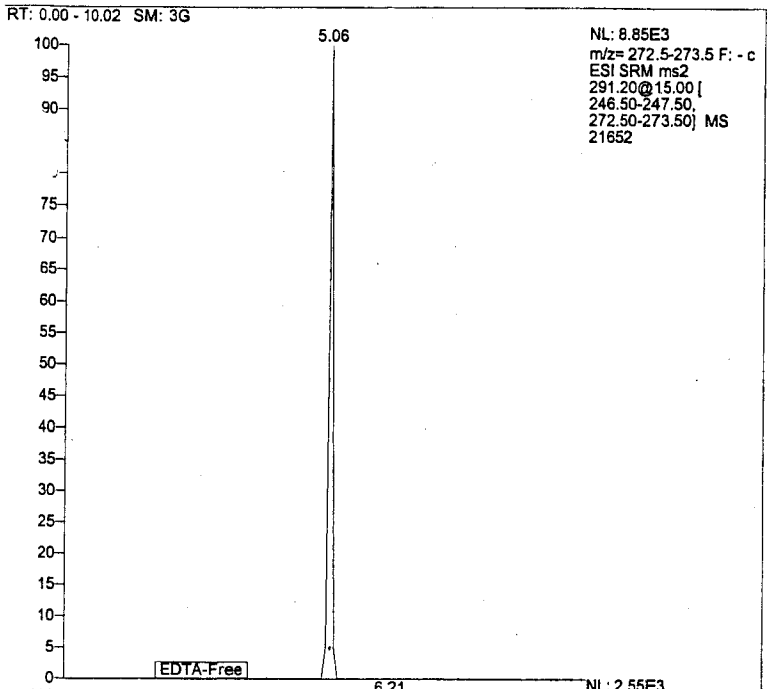
Ex 446

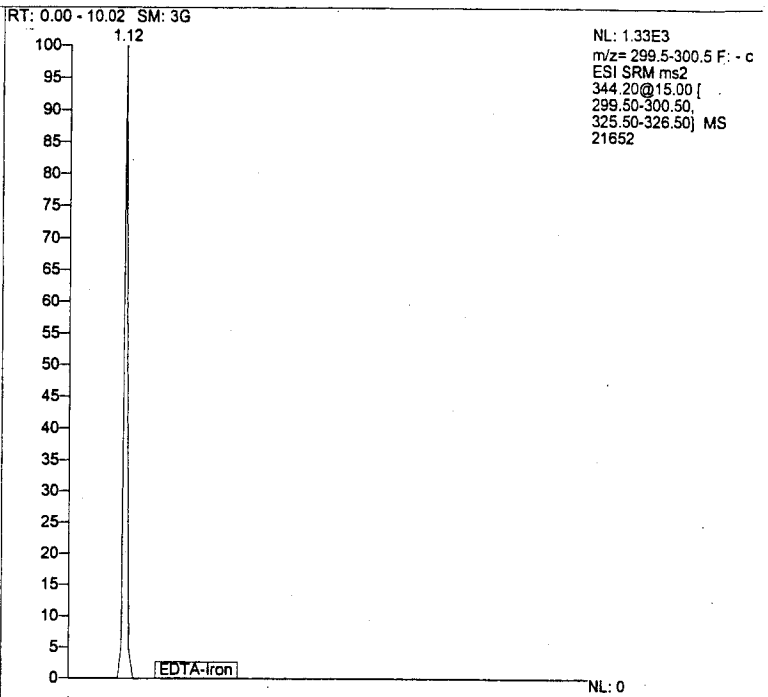
C:\Xcalibur\...\Negative Ion\21651 070201013 JBB 02/16/07 09:47:49 BLANK (neg blood, DI H2O ext.)

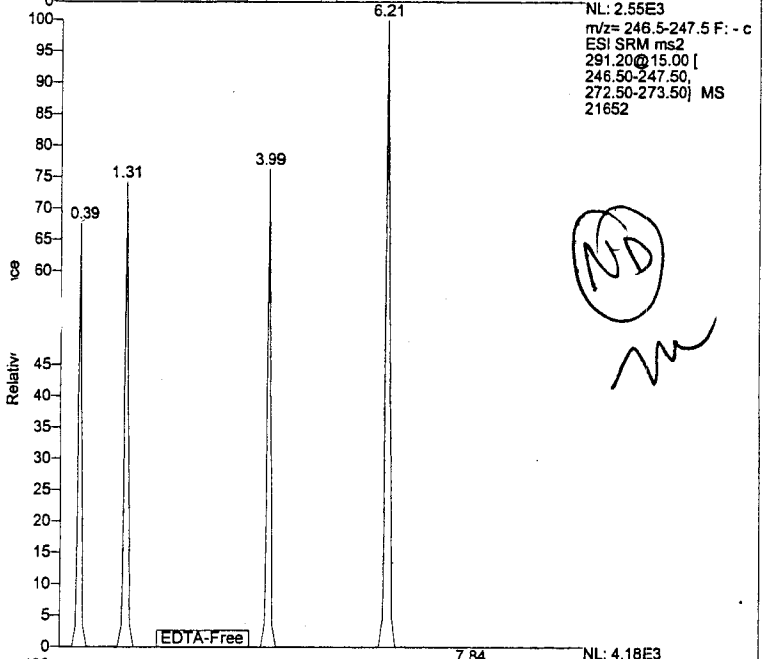


Ex 446
(182)

C:\Xcalibur\...\Negative Ion\21652 07020103 JDF 02/16/07 09:58:44 BLANK (neg blood, DI H2O ext.)







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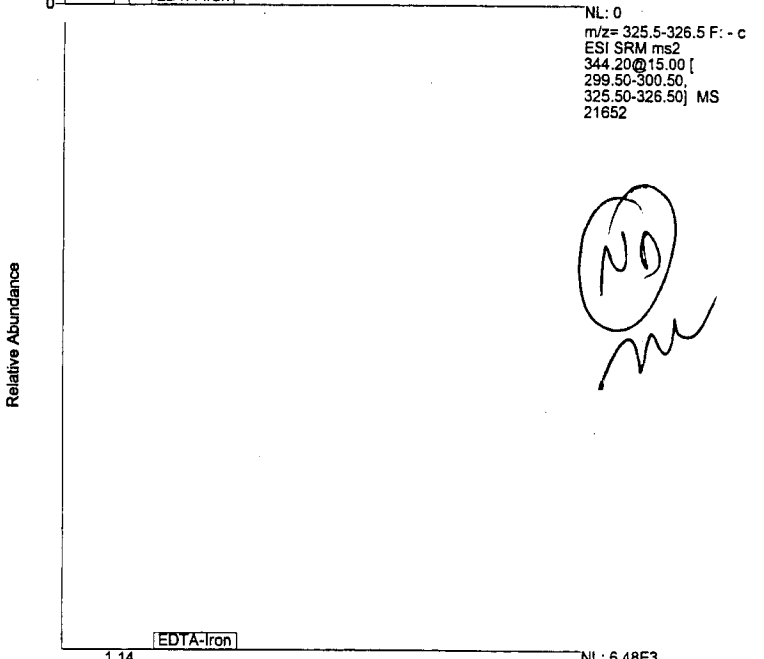
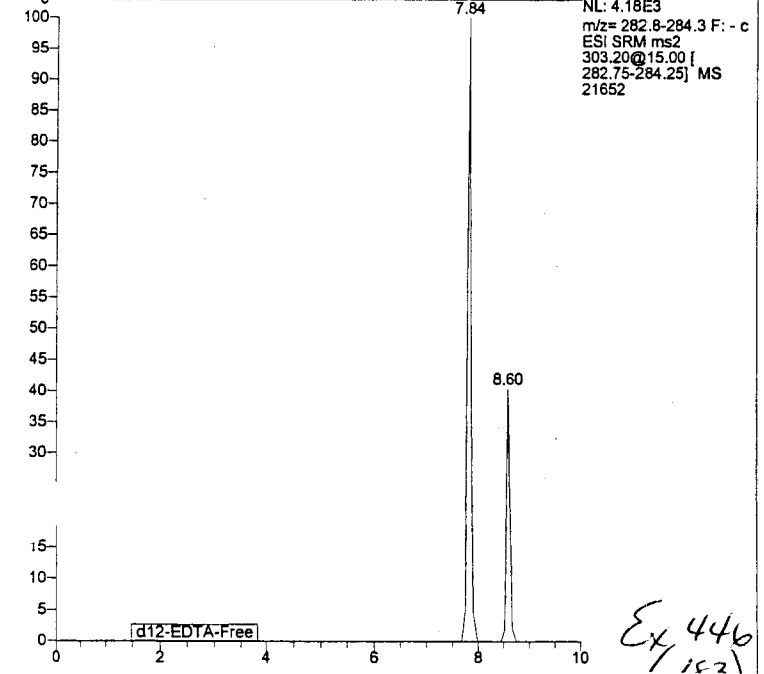
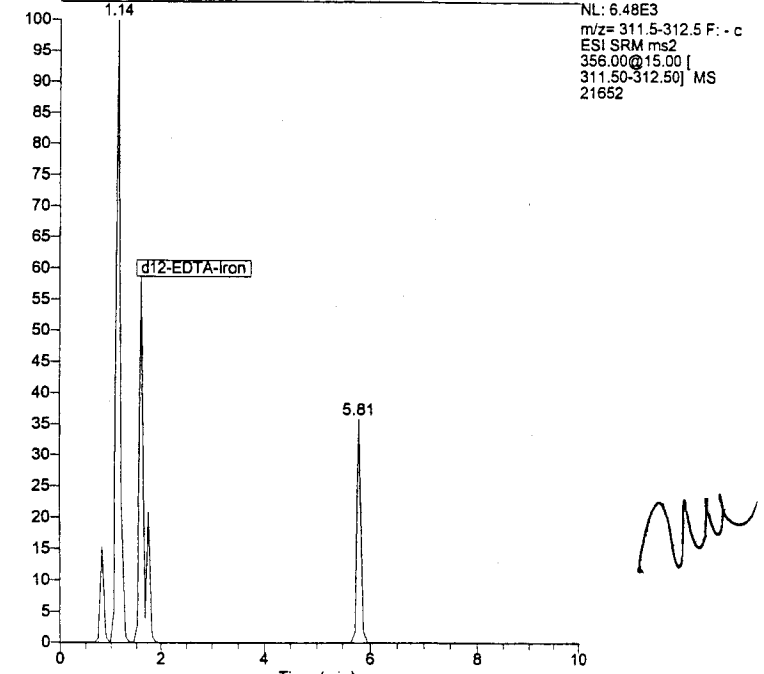


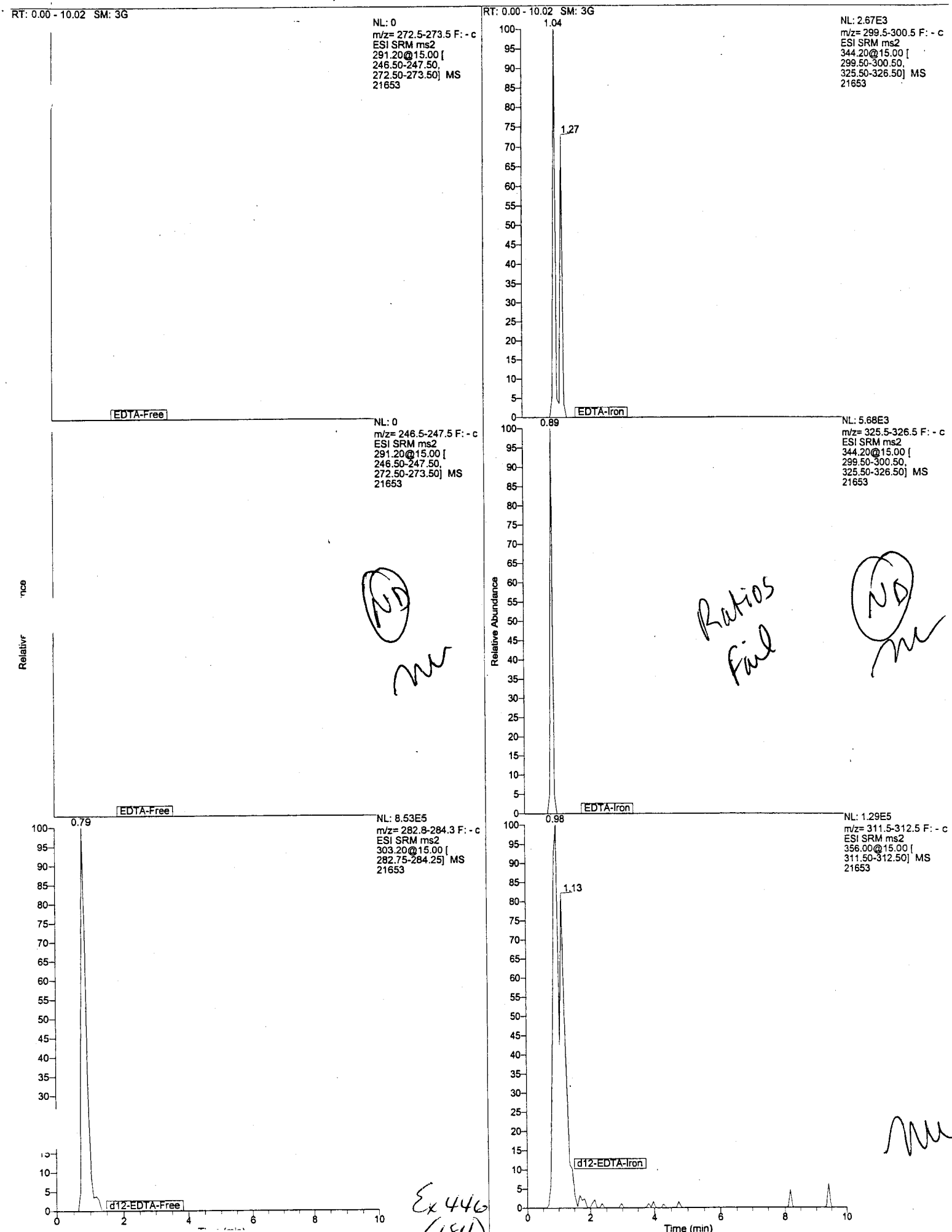
ND



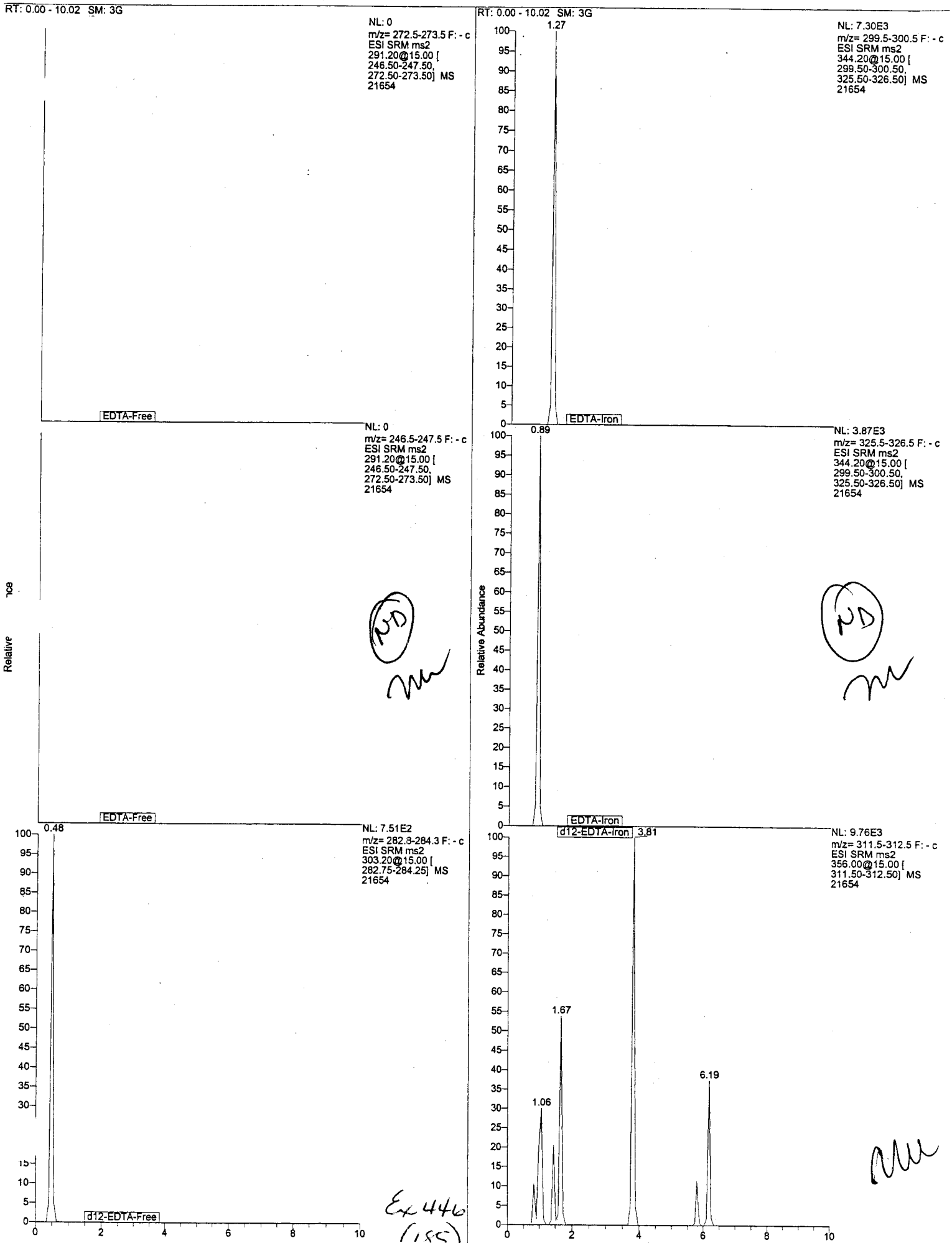
Ex 446 (153)



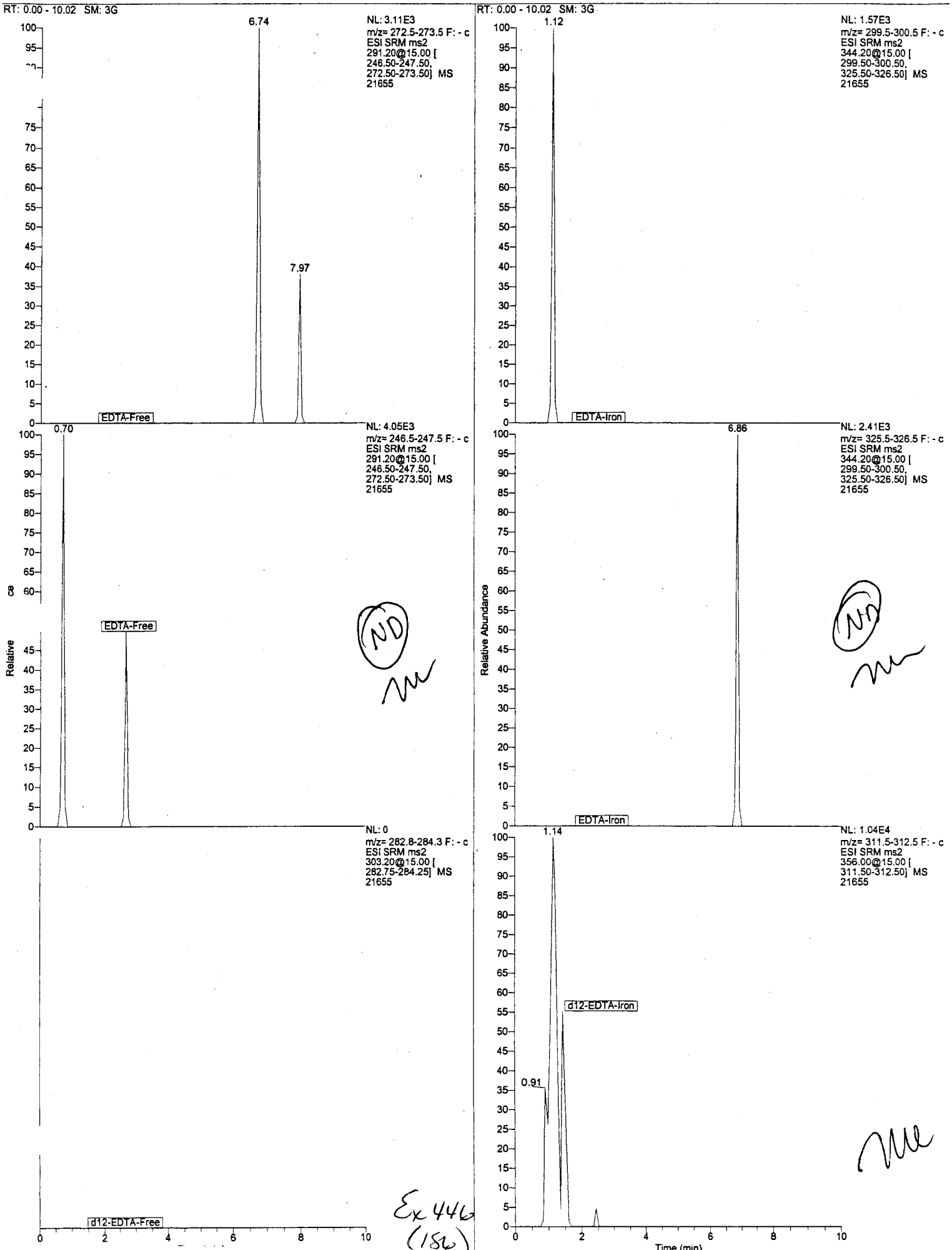
C:\Xcalibur\...\Negative Ion\21653 070201013 JDB 02/16/07 10:09:33 Q48 extract



C:\Xcalibur\...\Negative Ion\21654 07020101B JDB 02/16/07 10:20:25 BLANK (neg blood, DI H2O ext.)

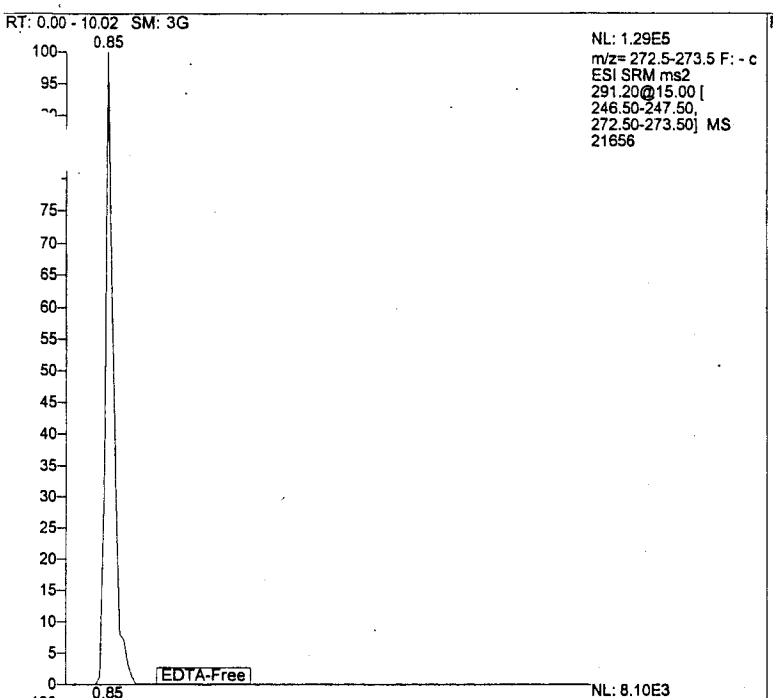


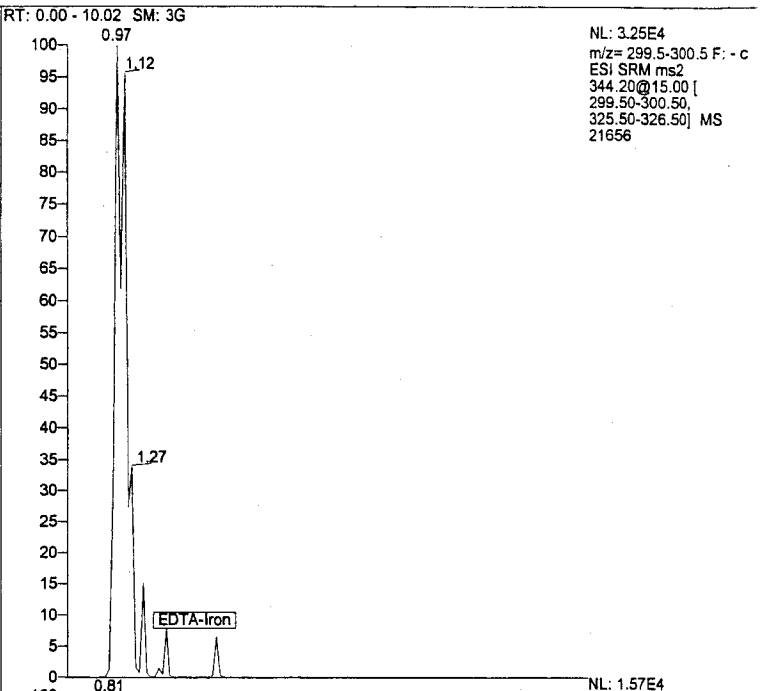
C:\Xcalibur\...\Negative Ion\21655 07020103 JDB 02/16/07 10:31:22 BLANK (neg blood, DI H2O ext.)

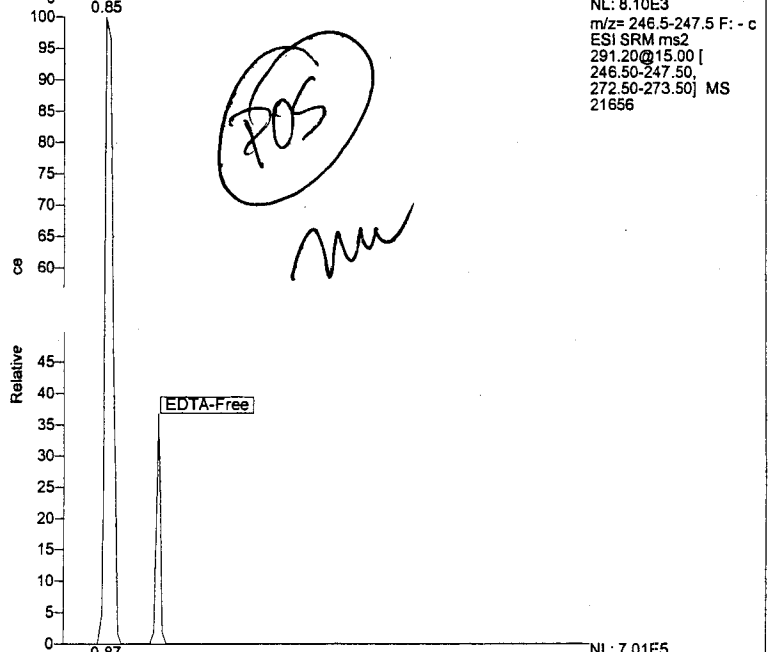


Ex 446 (182)

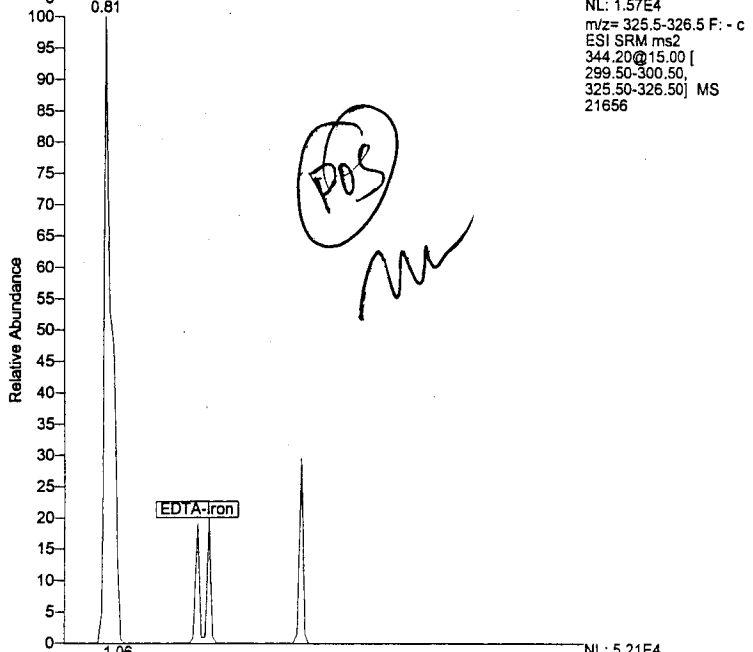
C:\Xcalibur\...\Negative Ion\21656 070201013 JDB 02/16/07 10:42:14 Pos. Cont. A (MAL EDTA ext.)



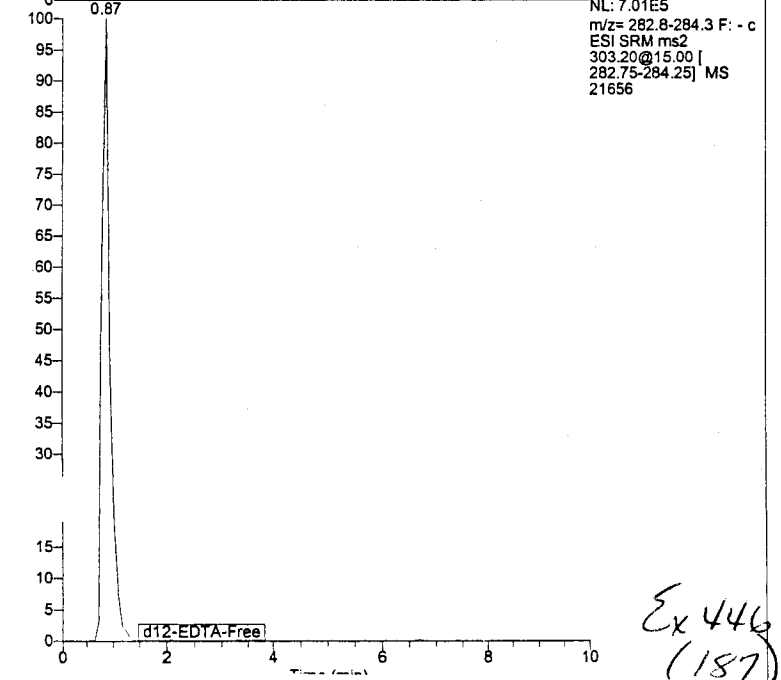


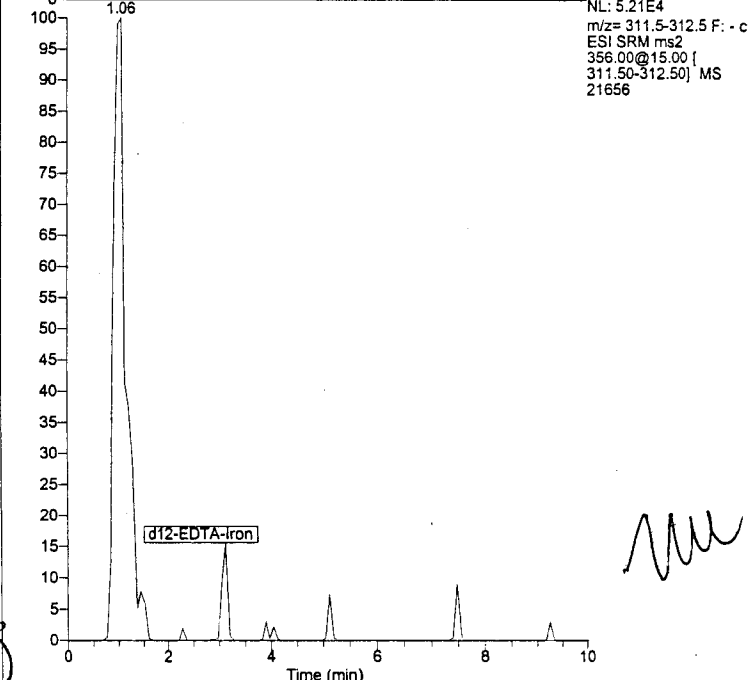


POS



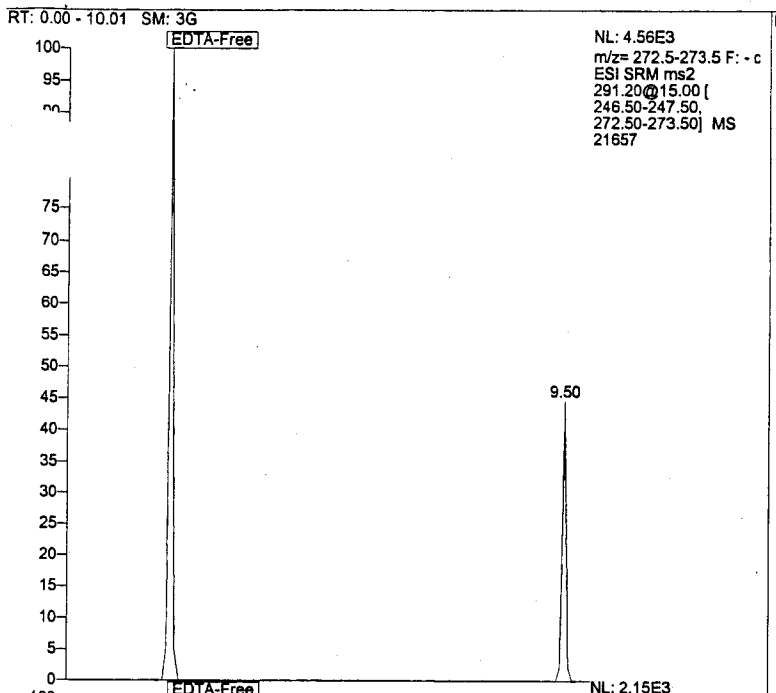
POS

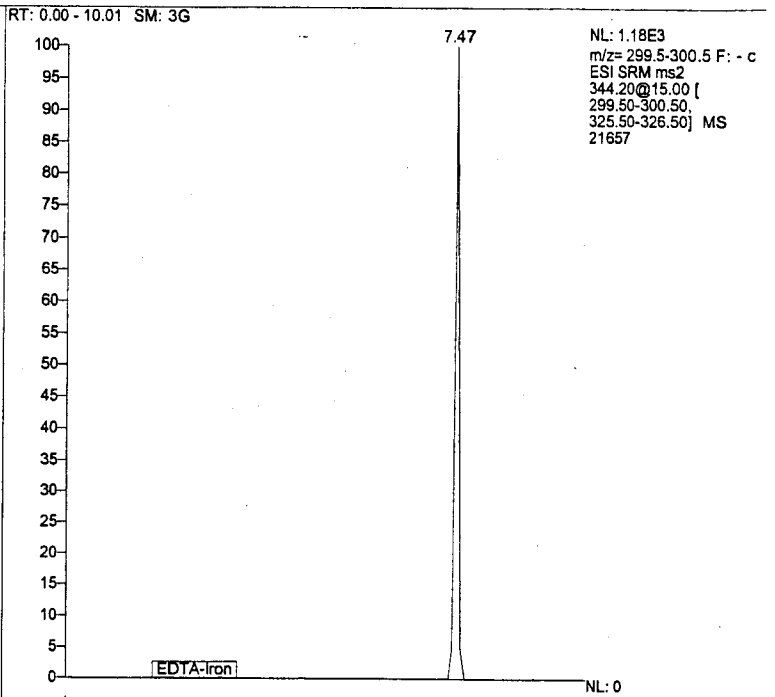


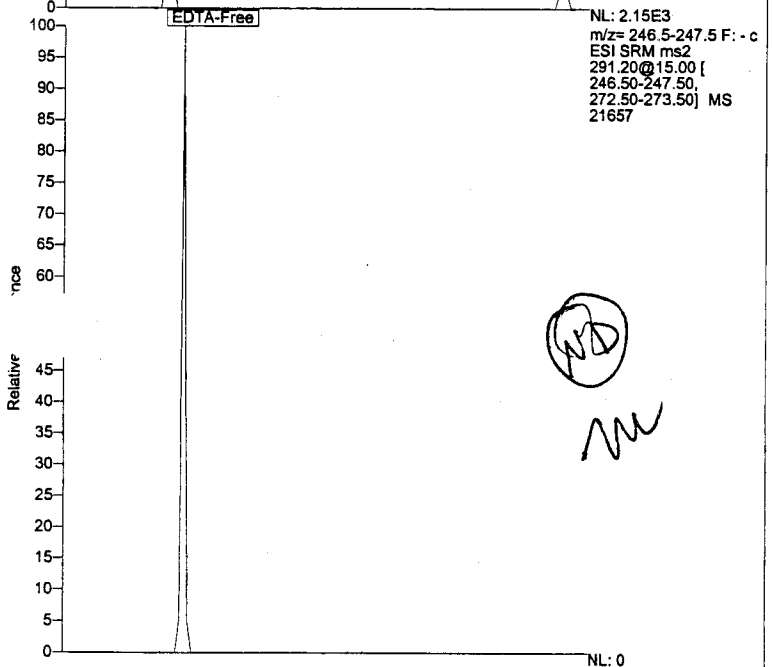


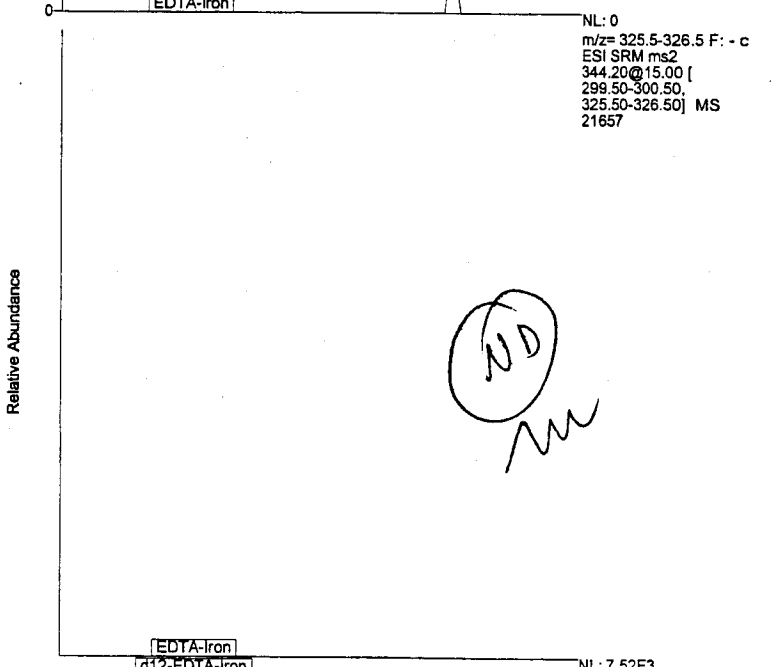
Ex 446
(187)

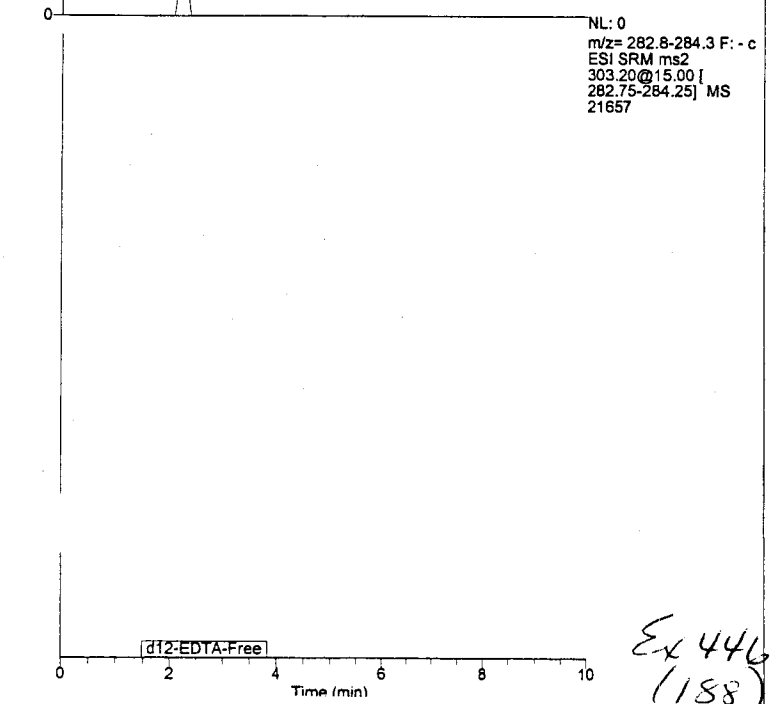
C:\Xcalibur\...\Negative Ion\21657 07020103 JOB 02/16/07 10:53:13 BLANK (neg blood, DI H2O ext.)

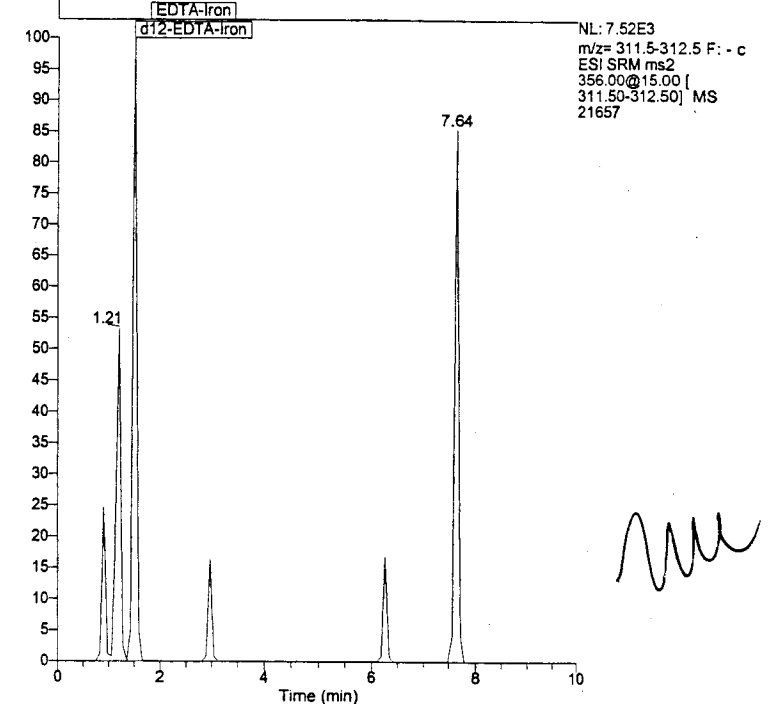




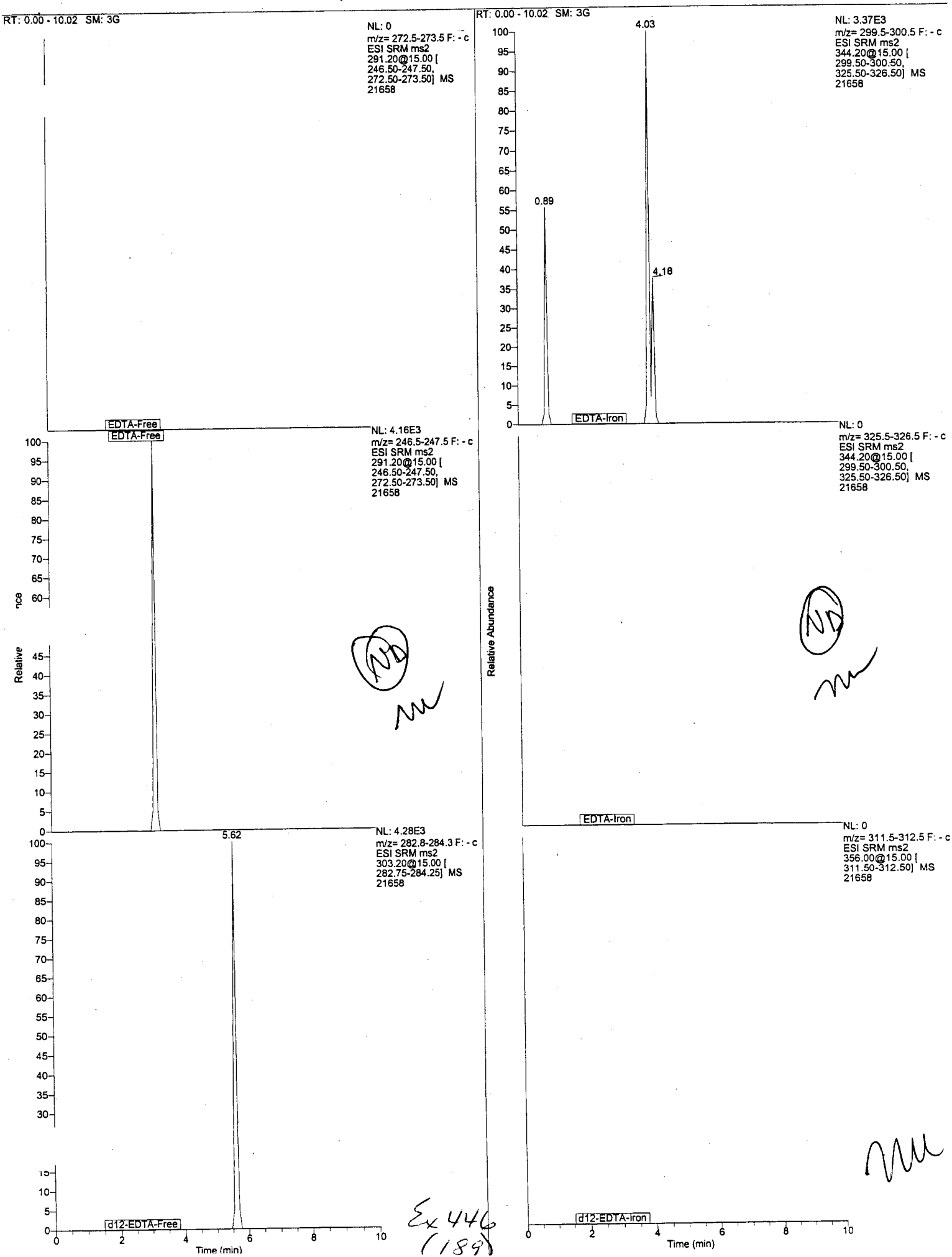






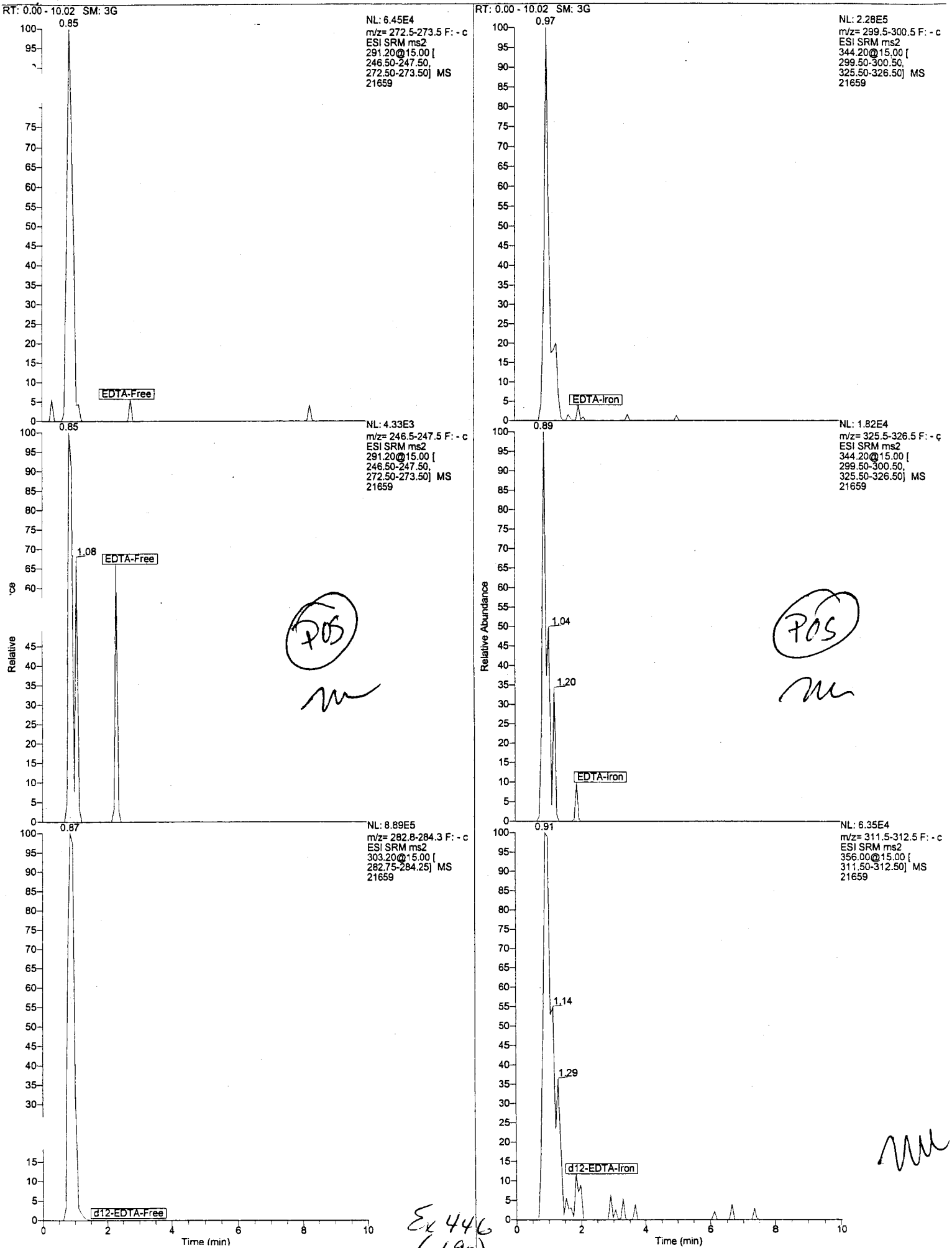


C:\Xcalibur\...\Negative Ion\21658 07020/013 JOB 02/16/07 11:04:07 BLANK (neg blood, DI H2O ext.)



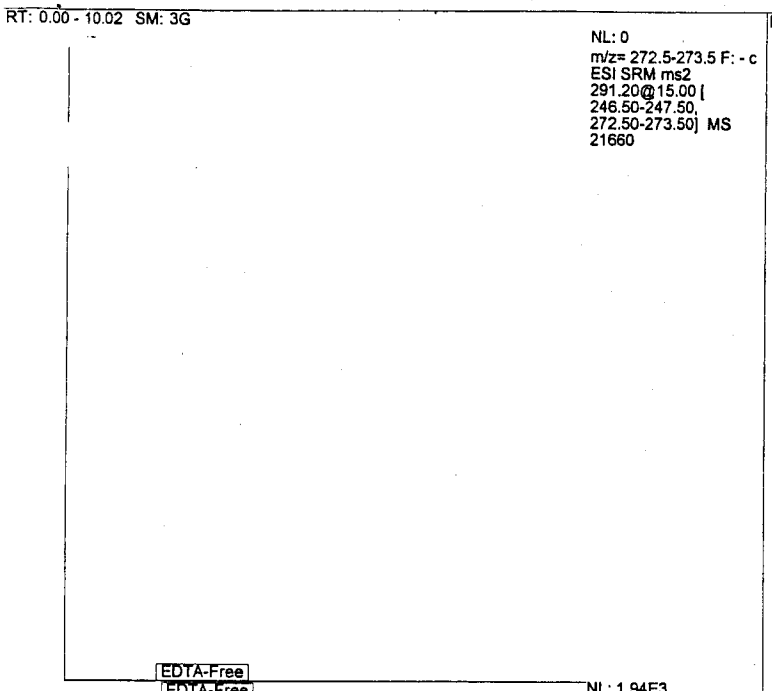
Ex 446
(189)

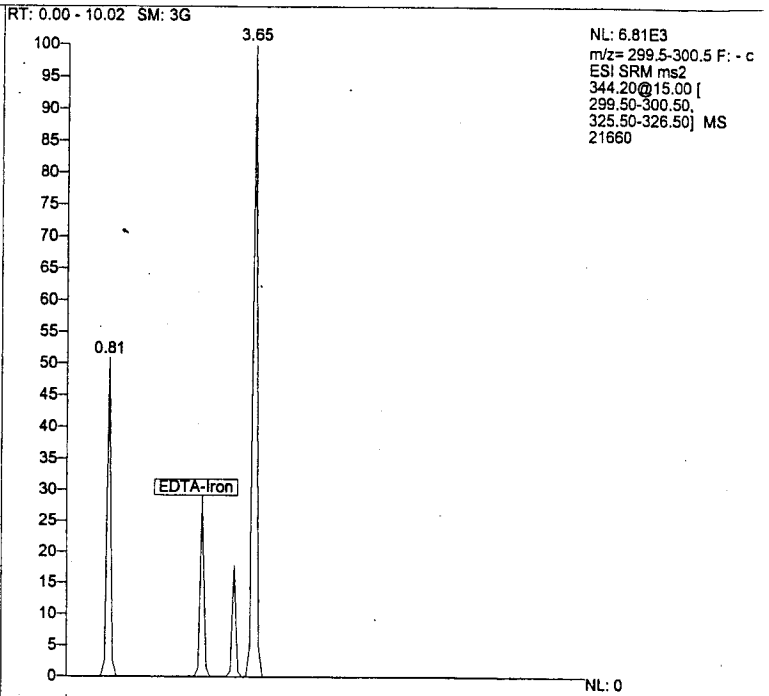
C:\Xcalibur\...\Negative Ion\21659 070201013 JDB 02/16/07 11:14:58 Pos. Cont. B (Q49 ext.)

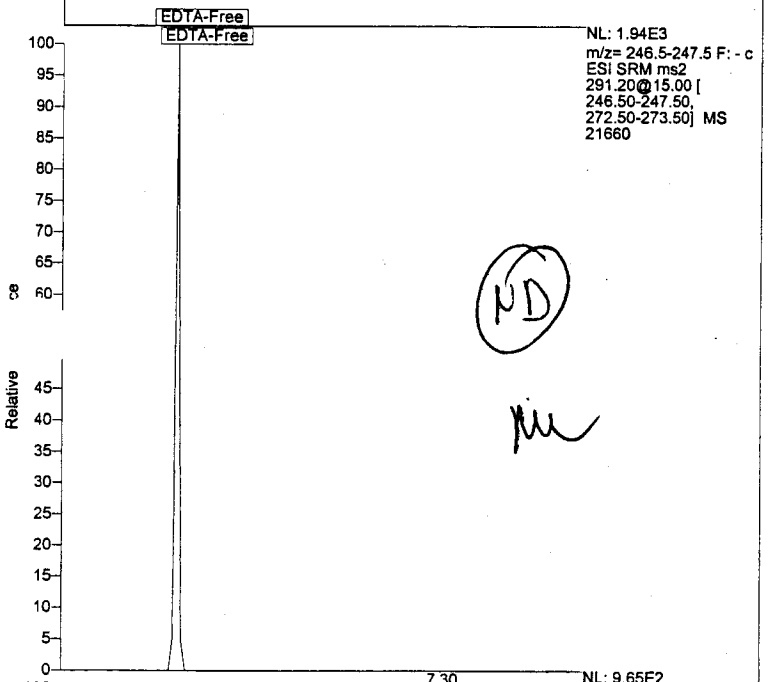


Ex 446
(49)

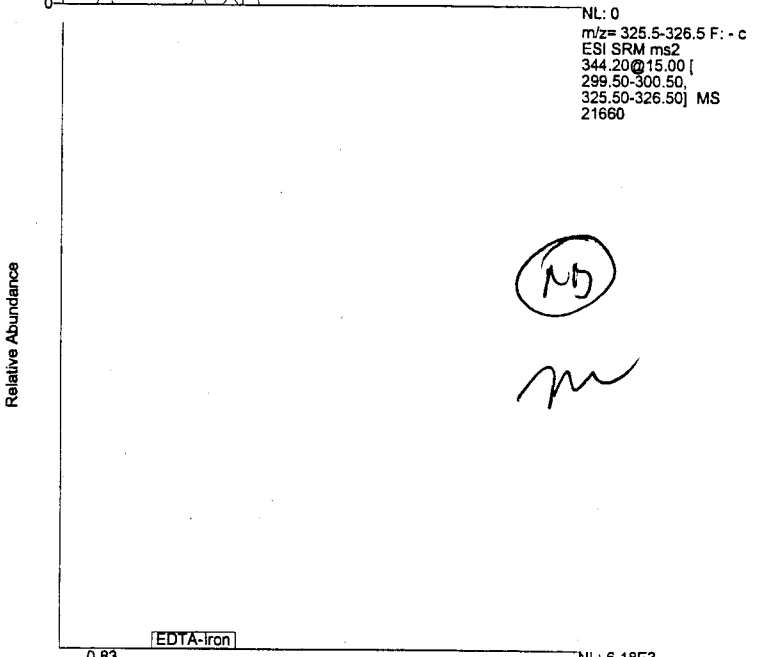
C:\Xcalibur\...\Negative Ion\21660 07020103 JDB 02/16/07 11:25:51 BLANK (neg blood, DI H2O ext.)



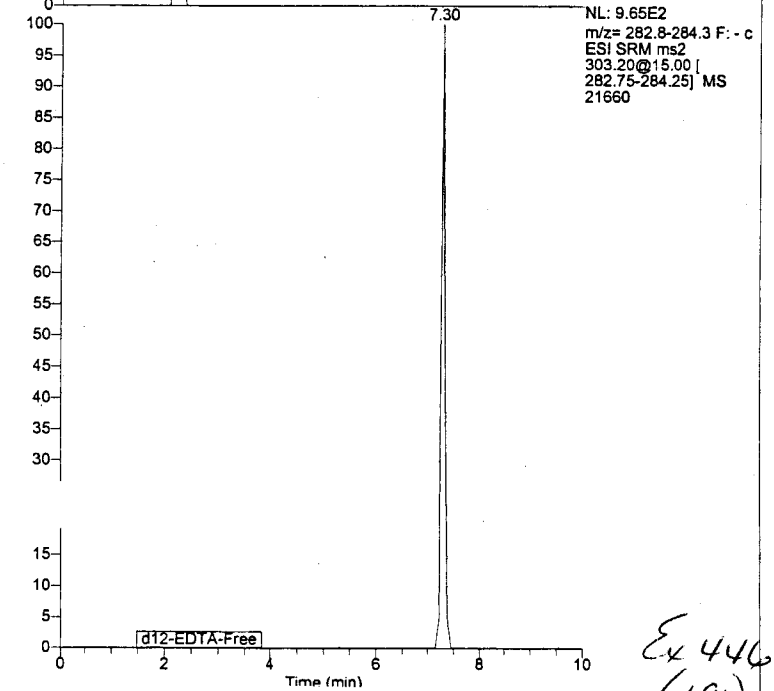




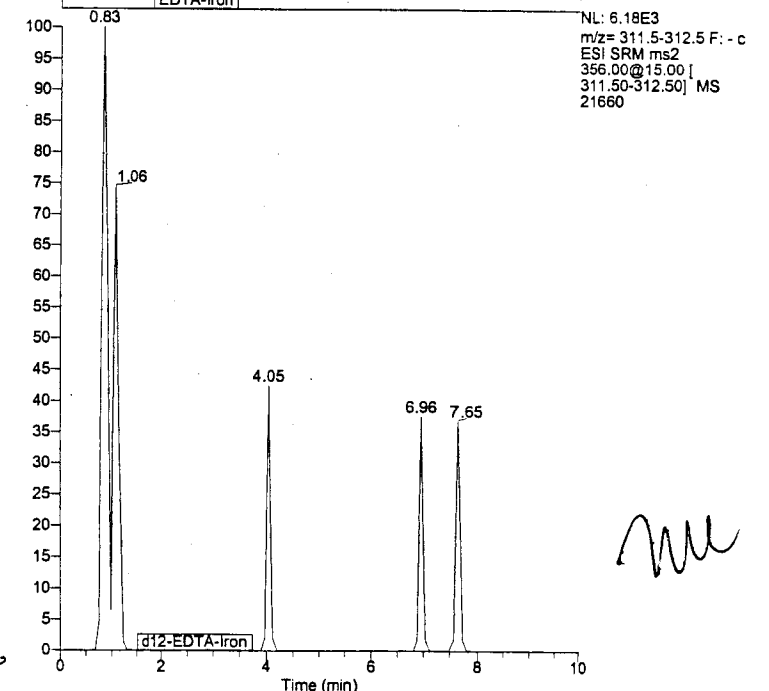
ND



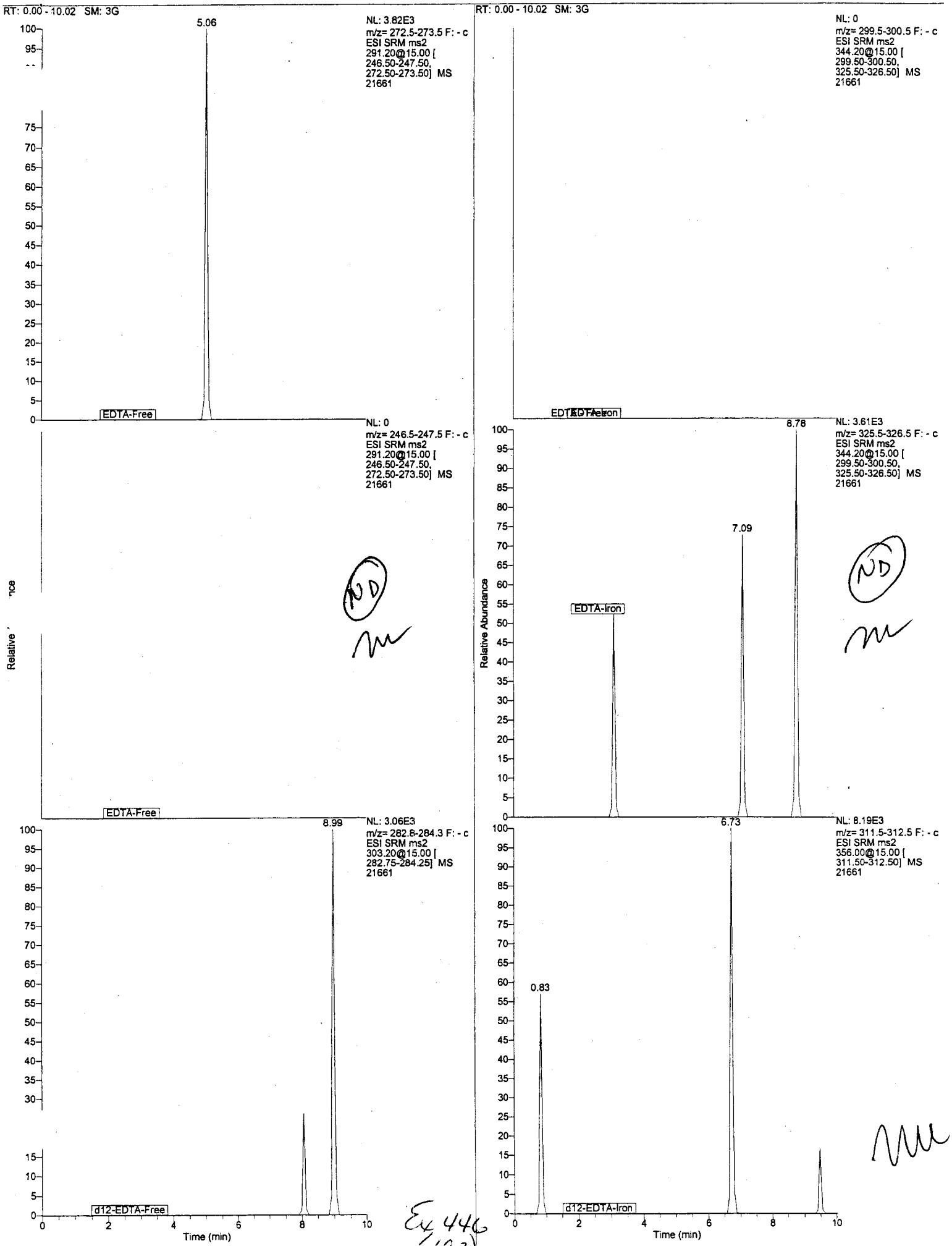
ND



Ex 446

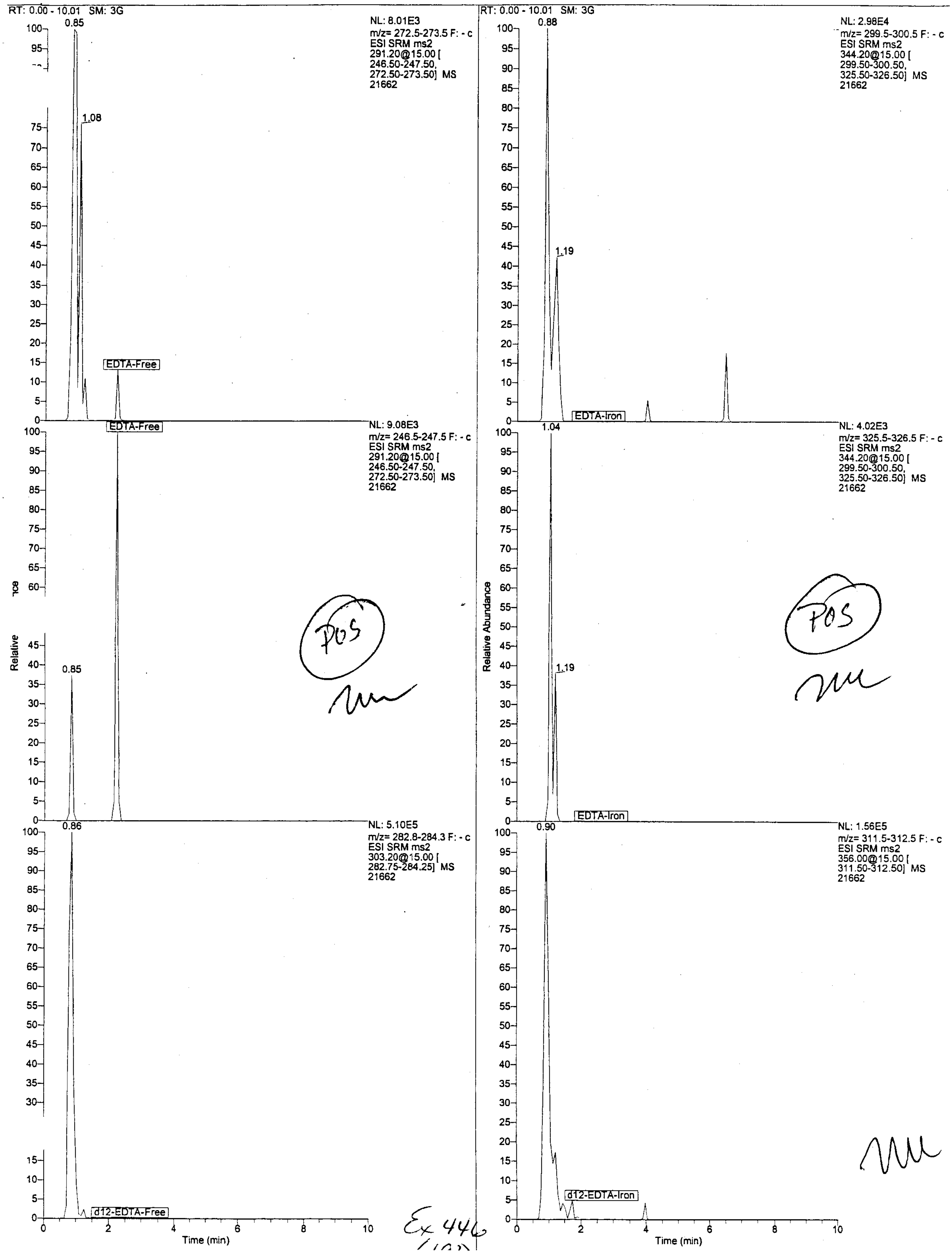


C:\Xcalibur\...\Negative Ion\21661 07020103 JDB 02/16/07 11:36:47 BLANK (neg blood, DI H2O ext.)



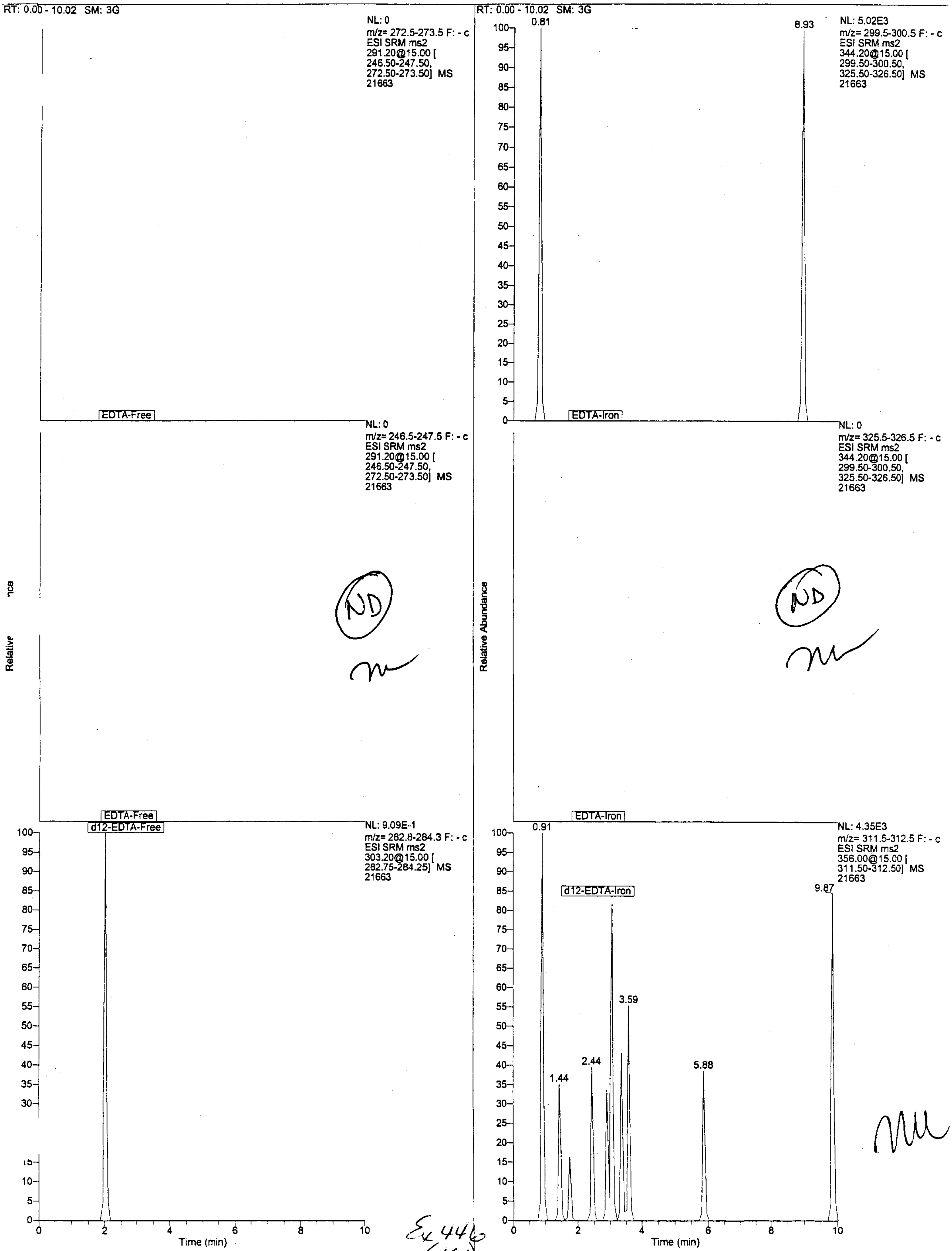
Ex 446

C:\Xcalibur\...\Negative Ion\21662 *070201013 JDB* 02/16/07 11:47:36 Spot LOD, 1uL Q49



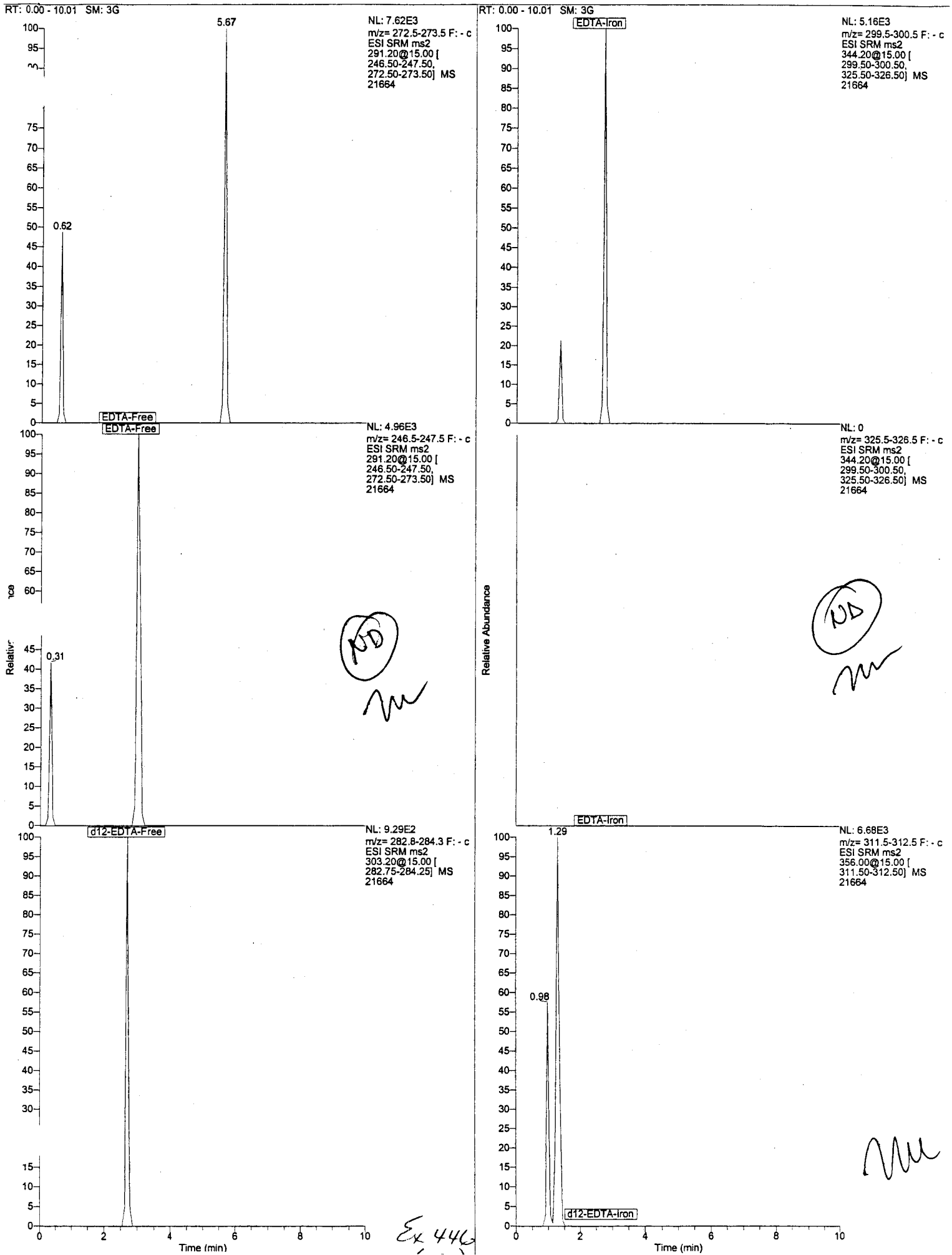
Ex 446 (193)

C:\Xcalibur\...\Negative Ion\21663 070201013 JOB 02/16/07 11:58:29 BLANK (neg blood, DI H2O ext.)



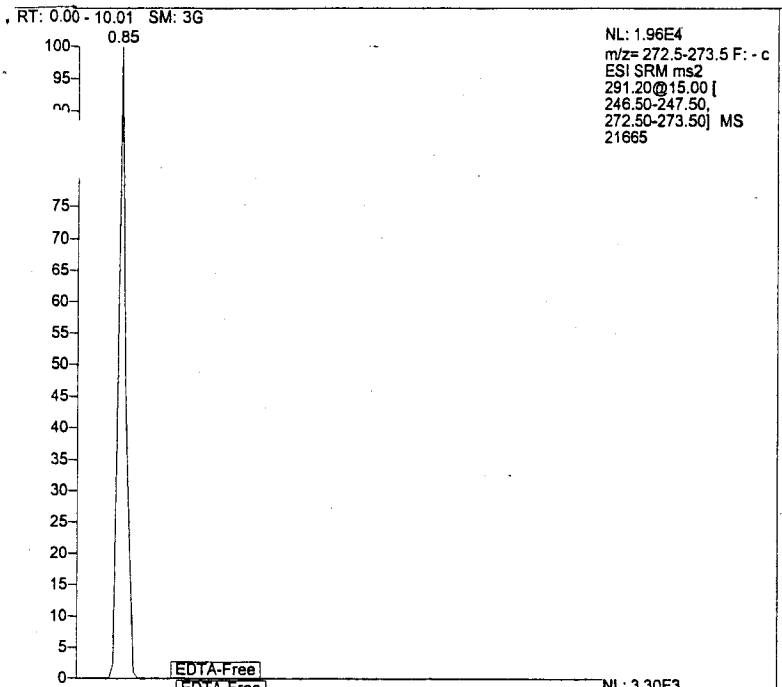
Ex 446

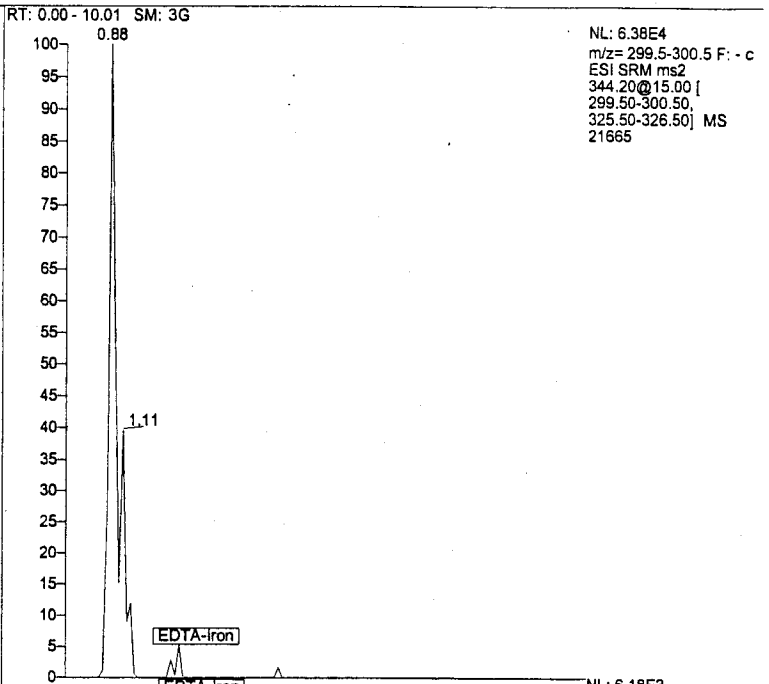
C:\Xcalibur\...\Negative Ion\21664 070201013 JDB 02/17/07 12:09:21 BLANK (neg blood, DI H2O ext.)

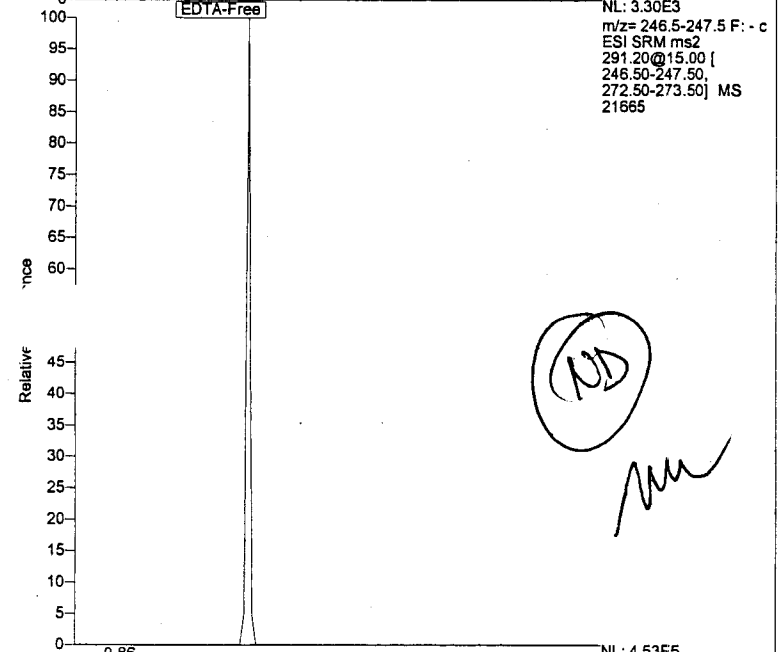


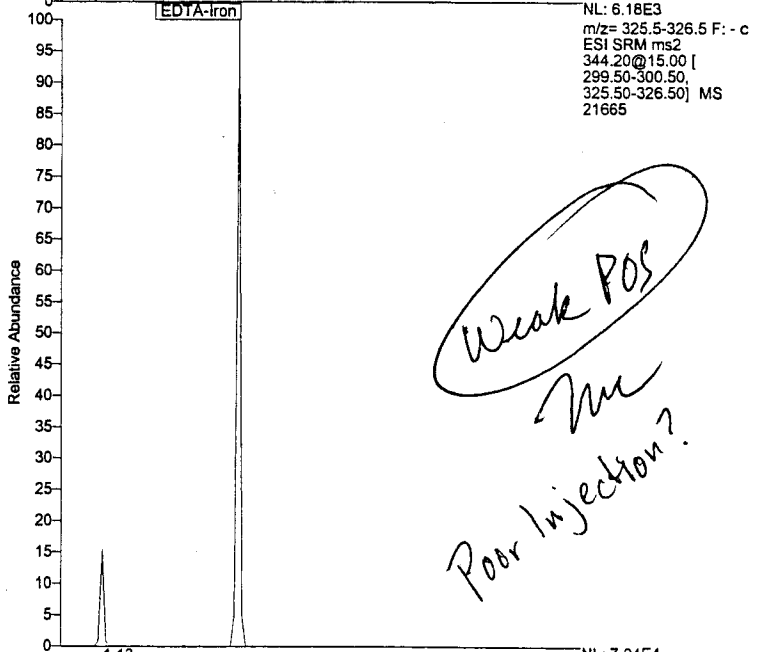
Ex 446

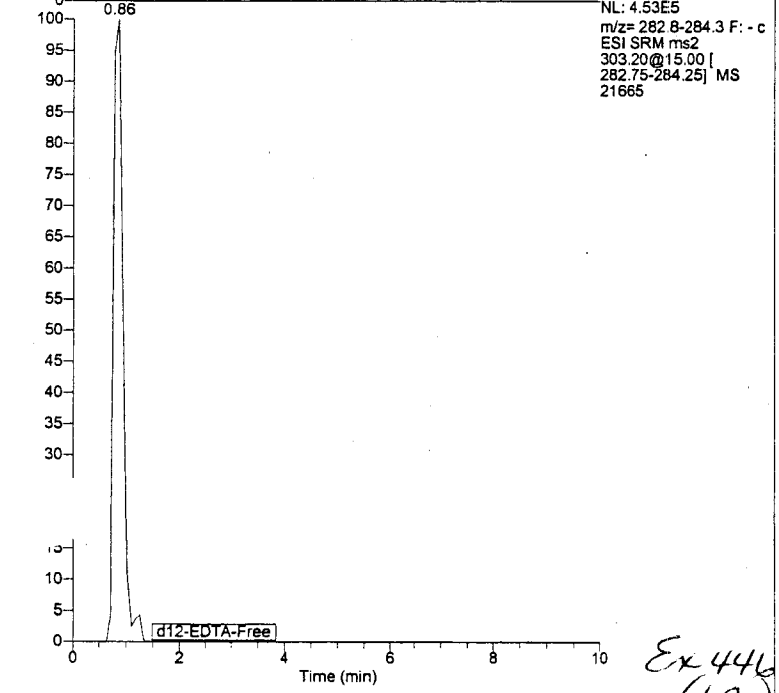
C:\Xcalibur\...\Negative Ion\21665 070201013 JDB 02/17/07 12:20:10 Spot LOD, 2 uL Q49

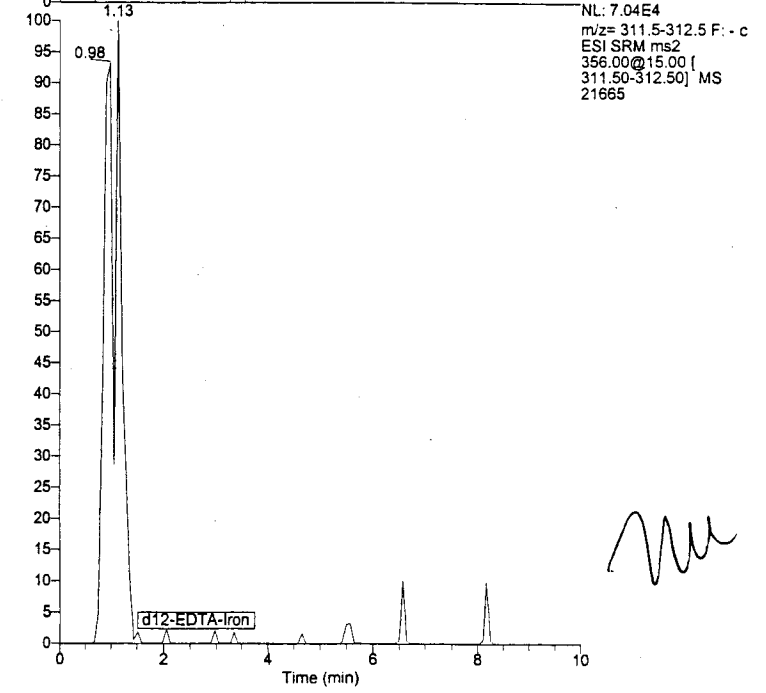




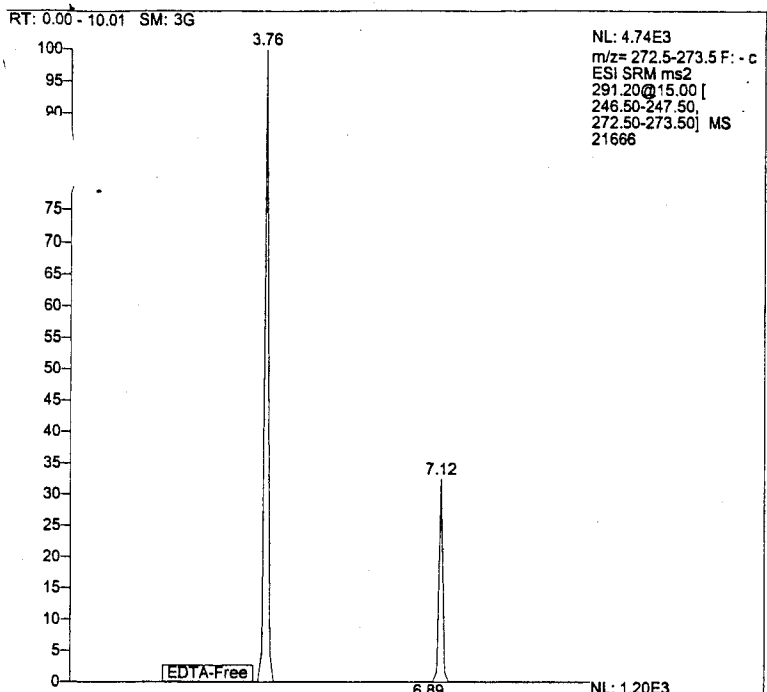


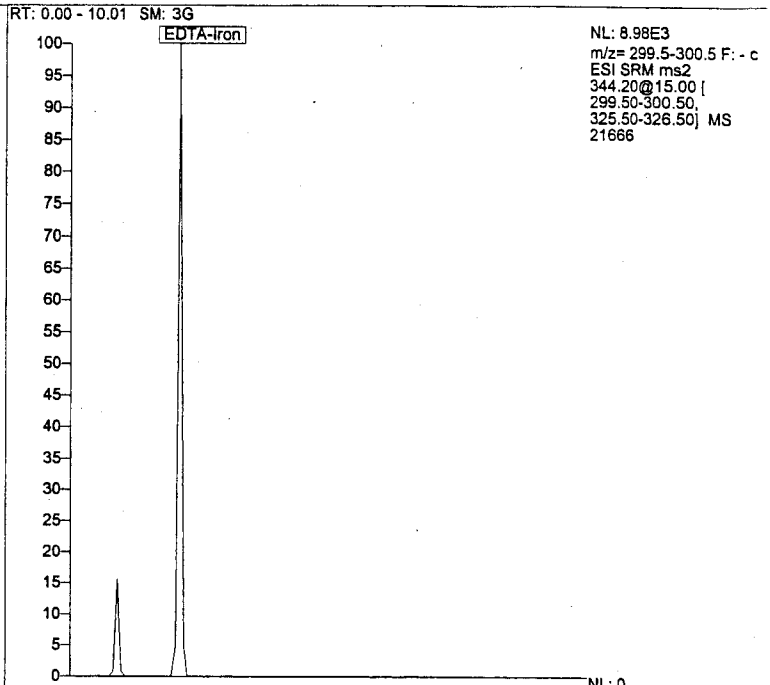


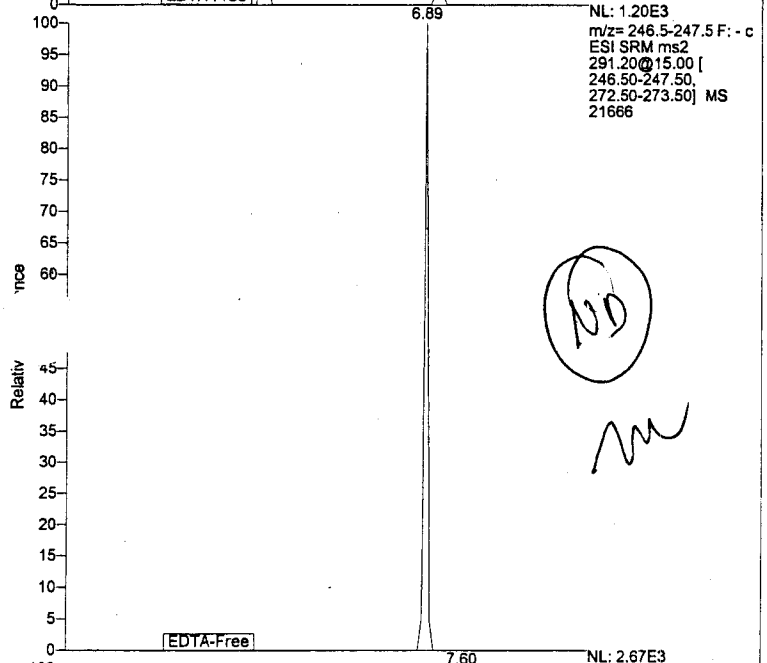


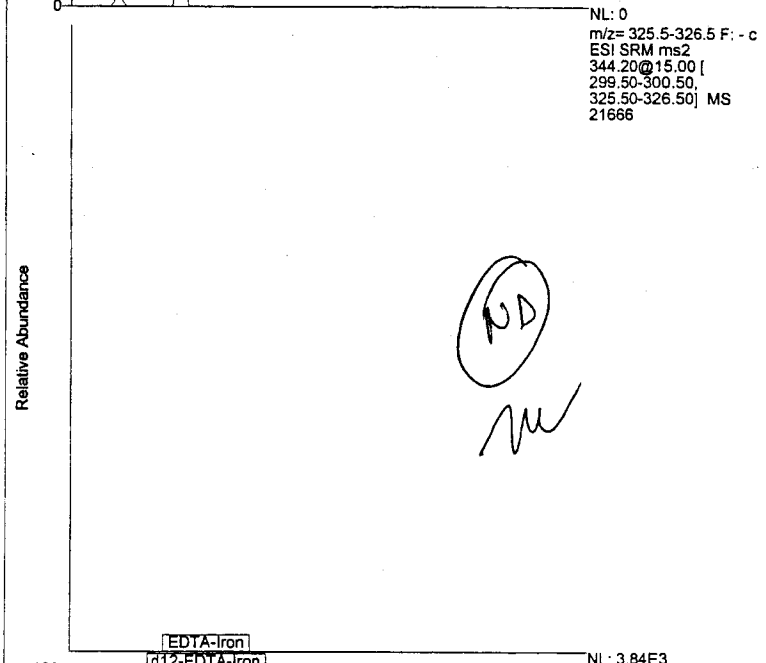


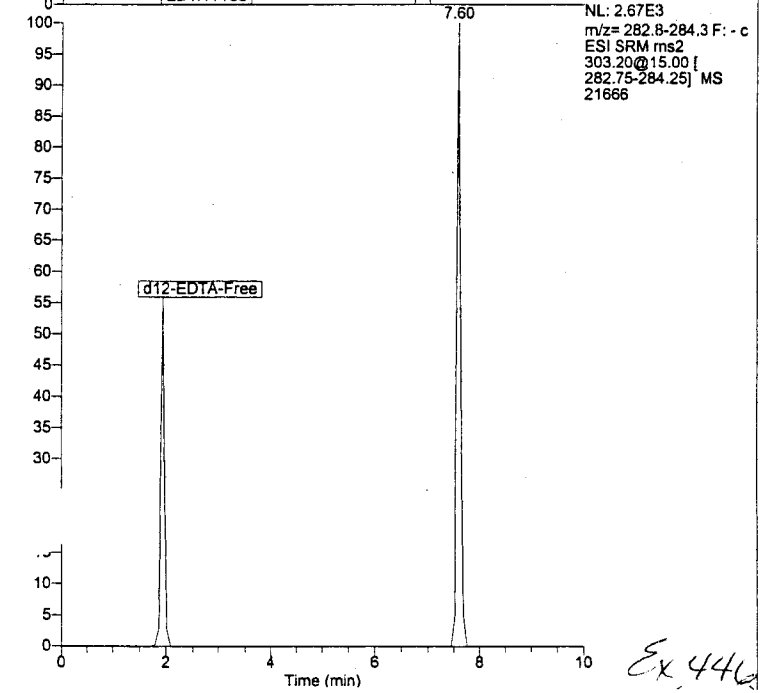
C:\Xcalibur\...\Negative Ion\21666 070201013 JDP 02/17/07 12:31:06 BLANK (neg blood, DI H2O ext.)

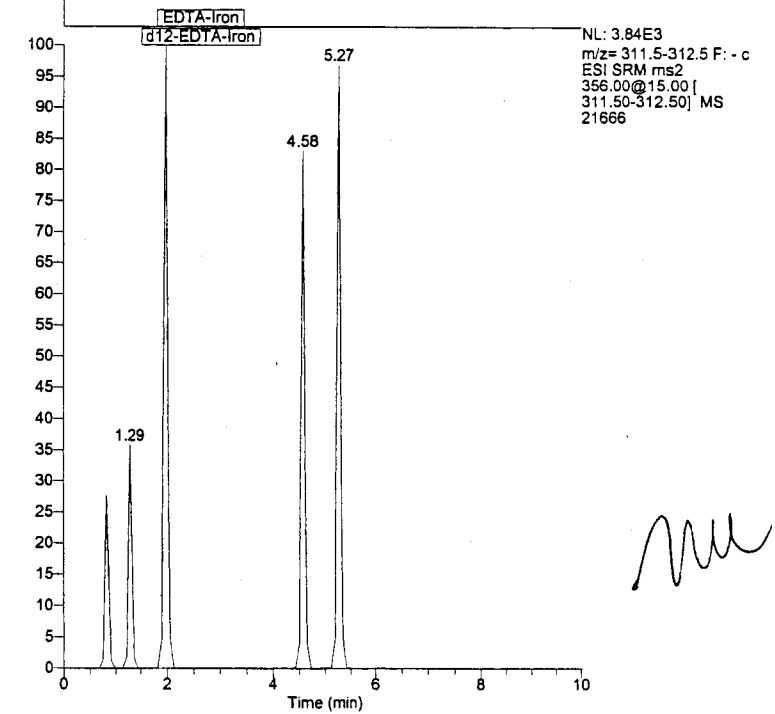












Ex 446

Instrument Method: EDTA_Neg_Swabs.meth

LCQ Instrument Method

Creator: Administrator
Last modified: 2/16/07 by Administrator

MS Run Time (min): 10.00

Divert Valve: not used during run

MS Detector Settings:

Segment 1 Information

Duration (min): 10.00
Number of Scan Events: 4
Tune Method: EDTA_NEG

Scan Event Details:
1: Neg ·(291.2)->oS(246.5-247.5 272.5-273.5)
MS/MS: CE 15.0% IsoW 1.0

2: Neg ·(303.2)->oS(282.8-284.3)
MS/MS: CE 15.0% IsoW 1.0

3: Neg ·(344.2)->oS(299.5-300.5 325.5-326.5)
MS/MS: CE 15.0% IsoW 1.0

4: Neg ·(356.0)->oS(311.5-312.5)
MS/MS: CE 15.0% IsoW 1.0

Ex 446
(195)

07020103
02/16/2007

Instrument Method: EDTA_Neg_Swabs.meth

Waters 2690 LC System

Injector parameters:
Syringe draw rate (µl/sec):2.50
Injection volume (µl):5

Pump settings:
Solvent A:80:20 ACN:Water + 0.03% NH4OH
Solvent B:B
Solvent C:C
Solvent D:D
Min pressure (PSI):0
Max pressure (PSI):5000
Chart output:Pressure
Chart output:Normal

Gradient program:

Time(min)	Flow(ml/min)	A(%)	B(%)	C(%)	D(%)	Curve
0.00	0.30	100.0	0.0	0.0	0.0	Linear - 6
3.00	0.30	100.0	0.0	0.0	0.0	Linear - 6

Timed events:
Initial states:
Switch 1:No change
Switch 2:No change
Switch 3:No change
Switch 4:No change

Time(min)	Event	Action	Parameter
0.00	Switch1	No change	

Ex 446
(199)

070201013
02/16/2007
TWP

Sequence---070201013_Neg.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21634	1	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Neg. Control (-EDTA blood swab ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21635	02	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			2	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(200)

070201013
02/16/2007
JOB

Sequence---070201013_Neg.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21636	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21637	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

070201013
02/16/2007
JDB

Ex 446
(201)

Sequence---070201013_Neg.sld [Open]

Sample Name: K2 extract
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21638	03	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			3	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21639	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

070201013
02/16/2007
JDB

Ex 446
(202)

Sequence---070201013_Neg.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21640	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Q46 extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21641	04	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			4	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(203)

070201013
02/16/2007
JDB

Sequence---070201013_Neg.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21642	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21643	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(204)

070201013
02/16/2007
JMB

Sequence---070201013_Neg.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21646	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Q47 extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21647	06	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			6	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(205)

070201013
02/16/2007
JDB

Sequence---070201013_Neg.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21648	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21649	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(206)

070201013
02/16/2007
JDB

Sequence---070201013_Neg.sld [Open]

Sample Name: K4 extract
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21650	07	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			7	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21651	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(207)

070201013
02/16/2007
JDB

Sequence---070201013_Neg.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21652	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Q48 extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21653	08	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			8	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(208)

070201013
02/16/2007
JVB

Sequence---070201013_Neg.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21654	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21655	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(209)

070201013
02/16/2007
JDB

Sequence---070201013_Neg.sld [Open]

Sample Name: Pos. Cont. A (MAL EDTA ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21656	09	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			9	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21657	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(210)

070201017
02/16/2007
JMB

Sequence---070201013_Neg.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21658	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Pos. Cont. B (Q49 ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21659	10	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			10	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

070201013
02/16/2007
JDB

Ex 446
(211)

Sequence---070201013_Neg.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21660	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21661	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(212)

070201013
02/16/2007
JDB

Sequence---070201013_Neg.sld [Open]

Sample Name: Spot LOD, 1uL Q49
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21662	11	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21663	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

070201013
02/16/2007
JMB

Ex 446
(213)

Sequence---070201013_Neg.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21664	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Spot LOD, 2 uL Q49
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21665	12	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

070201013
02/16/2007
JDB

Ex 446
(214)

Sequence---070201013_Neg.sld [Open]

Sample Name: BLANK (neg blood, DI H2O ext.)

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	21666	01	C:\Xcalibur\Data\Brewer\070201013\Negative Ion

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(215)

070201013
02/16/2007
JDB

Vial List---C:\Xcalibur\methods\brewer\070201013\070201013_Neg.sld

Position	Sample Type	Level	Sample ID	Sample Name
1	Unknown		1	BLANK (neg blood, DI H2O ext.)
	Unknown		02	Neg. Control (-EDTA blood swab ext.)
3	Unknown		03	K2 extract
4	Unknown		04	Q46 extract
5	Unknown		05	K3 extract
6	Unknown		06	Q47 extract
7	Unknown		07	K4 extract
8	Unknown		08	Q48 extract
9	Unknown		09	Pos. Cont. A (MAL EDTA ext.)
10	Unknown		10	Pos. Cont. B (Q49 ext.)
11	Unknown		11	Spot LOD, 1uL Q49
12	Unknown		12	Spot LOD, 2 uL Q49

Ex 446
(216)

070201013
02/16/2007
JDB

SOPs

Ex 446
(217)

Analysis of EDTA in Dried Bloodstains

1 Introduction

The collection of blood at crime scenes and for legal proceedings is a common practice that may be used to inculpate or exculpate individuals suspected of being involved in the crime. Occasionally, there are allegations that blood evidence collected from crime scenes was "planted". This issue may be resolved by the determination of exogenous components in the bloodstains (e.g. preservatives) that should not be present in authentic crime scene evidence.

Ethylenediaminetetraacetic acid (EDTA) is an anti-coagulant and enzyme inhibitor that is commonly used in blood specimen collection tubes. Blood specimen collection tubes containing EDTA have lavender-colored tops and are the most common collection tube used to collect reference specimens for DNA testing. Therefore, most allegations of blood evidence "planting" focus on EDTA-preserved blood samples.

EDTA-preserved blood tubes use either the disodium, dipotassium, or tripotassium salt forms of EDTA. The concentration of EDTA in its free acid form in a drawn blood tube is typically 1000-2000 mg/L, depending on the volume of blood and the capacity of the tube. At this concentration, the free acid and salt forms of EDTA are soluble in the blood. EDTA readily forms water-soluble chelates with nearly all heavy metals, so aqueous extractions of dried bloodstains should isolate EDTA.

2 Scope

This procedure allows for the screening and confirmation of EDTA in suspected blood stains.

3 Principle

This method takes advantage of the water solubility of EDTA and EDTA-complexes. A dried bloodstain is first extracted with deionized water and then subjected to ultrafiltration. Following ultrafiltration, the filtrate is analyzed by liquid chromatography/mass spectrometry/mass spectrometry (LC/MS/MS) in both the positive and negative electrospray ionization (ESI) modes.

4 Specimens

This procedure can be used for assaying bloodstains from a cotton swab or other cotton-based matrices.

Ex 446
(215)

5 Equipment/Materials/Reagents

Guidance for the preparation of reagents may be found in the *Preparation of Chemical Reagents* standard operating procedure (Tox 103).

a. Liquid chromatograph/mass spectrometer equipped with a Hamilton PRP-1 polymeric column (2.1 mm x 150 mm x 5µm) or equivalent

b. Laboratory scissors

c. Millipore Amicon Ultra-4 10,000 Molecular Weight Cutoff Centrifugal Filter Device

d. Wheaton (or similar) pipette – 200 µL

e. Centrifuge

f. EDTA-preserved whole blood

g. EDTA-free whole blood

h. Deionized water

i. Acetonitrile (HPLC grade)

j. Ammonium Hydroxide (HPLC grade)

k. Deionized Water (95%) / Acetonitrile (5%) / Ammonium Hydroxide (0.06%) – Mobile Phase for Positive Electrospray Ionization

l. Acetonitrile (80%) / Deionized Water (20%) / Ammonium Hydroxide (0.03%) – Mobile Phase for Negative Electrospray Ionization

m. Common laboratory supplies such as glassware, Pasteur pipettes, etc.

6 Standards and Controls

a. EDTA LC/MS/MS(ESI) Performance Mix – 100 µg/mL:
Accurately weigh 12.7 mg of the disodium salt of EDTA (reagent grade, Aldrich) and dilute with deionized water to a final volume of 100 mL. Store at room temperature in a clear glass container. Stable for at least 6 months.

Ex 446
(219)

b. d_{12}-EDTA Working Internal Standard Solution – 500 µg/mL:
Accurately weigh 5 mg of the free acid of d_{12}-EDTA (reagent grade, Cambridge Isotope Laboratories) and dilute with deionized water to a final volume of 10 mL. Store frozen in a brown glass container. Stable for at least 6 months.

c. Negative Bloodstain Control:
Add 50 µL of EDTA-free whole blood to a cotton-tip applicator. Dry for at least 30 minutes at room temperature before use. Store at room temperature in a glass test tube or paper envelope. Stable for at least 2 years.

d. Positive Bloodstain Control:
Add 50 µL of EDTA-preserved whole blood to a cotton-tip applicator. Dry for at least 30 minutes at room temperature before use. Store at room temperature in a glass test tube or paper envelope. Stable for at least 2 years.

7 Calibration

This procedure has not been validated for quantitative analysis.

8 Sampling

Not applicable.

9 Procedure

a. Carefully cut the tip from a cotton swab (negative control, positive control, or questioned swab) using clean laboratory scissors.

b. Place the cotton swab tip into a separately labeled molecular weight cutoff filter device.

c. Add 200 µL of the d_{12}-EDTA Working Internal Standard Solution directly to each cotton swab tip in the molecular weight cutoff filter device. Allow to sit at room temperature for 45 minutes.

d. Centrifuge the molecular weight cutoff filter device for 10 minutes at 2500 RPM.

e. Transfer the filtrate to an autosampler vial and inject 5 µL into the LC/MS/MS system that is in negative ion mode and monitor for both the free acid of EDTA and the EDTA-iron complex.[1] Samples that screen positive are confirmed by injection of 5 µL of the filtrate into the LC/MS/MS system in the positive ion mode, in which the free acid of EDTA can be confirmed.

10 Instrumental Conditions

10.1 Liquid Chromatograph Parameters

10.1.1 Positive Electrospray Ionization Mode

Mobile Phase Composition: Deionized Water (95%) / Acetonitrile (5%) / Ammonium Hydroxide (0.06%)

Column Parameters: Hamilton PRP-1 (2.1 mm x 150 mm x 5µm) at ambient temperature

Isocratic Flow Rate: 0.3 mL/minute

10.1.2 Negative Electrospray Ionization Mode

Mobile Phase Composition: Acetonitrile (80%) / Deionized Water (20%) / Ammonium Hydroxide (0.03%)

Column Parameters: Hamilton PRP-1 (2.1 mm x 150 mm x 5µm) at ambient temperature

Isocratic Flow Rate: 0.3 mL/minute

10.2 Mass Spectrometer Parameters

10.2.1 Positive Electrospray Ionization Mode

Spray Voltage: 4.5 kV

Capillary Temperature: 230°C

Capillary Voltage: +30V

Collision Induced Dissociation: 100%

---

[1] See note on carryover in the Limitations section of this procedure (Section 14).

MS/MS Mode: Products of *m/z* 293.0 (EDTA Free Acid)→ (*m/z* 125.0 – 315.0); Collision Energy = 15.0%[2]

Acquisition Time: 10 minutes

10.2.2 Negative Electrospray Ionization Mode

Spray Voltage: 4.5 kV

Capillary Temperature: 230°C

Capillary Voltage: -10V

Collision Induced Dissociation: 0% (Off)

SRM Mode: All Collision Energies Set to 15%

Segment 1 (EDTA Free Acid): *m/z* 291.2 → (*m/z* 246.5-247.5; *m/z* 272.5-273.5)

Segment 2 (d_{12}-EDTA Free Acid): *m/z* 303.2 → (*m/z* 282.8-284.3)

Segment 3 (EDTA Iron Complex): *m/z* 344.2 → (*m/z* 299.5-300.5; *m/z* 325.5-236.5)

Segment 4 (d_{12}-EDTA Iron Complex): *m/z* 356.0 → (*m/z* 311.5-312.5)

Acquisition Time: 10 minutes

11 Decision Criteria

11.1 Performance Mix Suitability

Proper calibration and sensitivity of the LC/MS/MS (ESI) are demonstrated each day samples are analyzed. The EDTA LC/MS/MS (ESI) Performance Mix effectively evaluates system suitability. Proper mass assignments, elution times, and signal-to-noise responses can be assessed by analyzing 5 µL of the Performance Mix. In all instances, the elution time should be ±2% of the retention time (relative or absolute) obtained from the previous run's injection of the Performance Mix. A stacked Gaussian-shaped peak must be present for the EDTA Free Acid analyte with a signal-to-noise ratio exceeding 50:1 for all extracted ions.

[2] *m/z* 160 and 247 are monitored for EDTA confirmation.

Ex 446
(222)

11.2 Analyte Suitability

The following criteria are used as guidelines in determining the acceptability of the data produced in this assay. In general, compound identification should be based on a comparison of the chromatography and mass spectrometry for the analyte peak of interest with data from a contemporaneously analyzed reference standard or extracted Positive Control. In most cases, all of the following should be met in order to identify EDTA within a bloodstain:

11.2.1 Chromatography

The peak of interest should show good chromatographic fidelity, with reasonable peak shape, width, and resolution. Additionally, the following two criteria should be met:

11.2.1.1 Retention Time

The retention time of the peak should be within ±2% of the retention time (relative or absolute) obtained from injection of the EDTA LC/MS (ESI) Performance Mix, extracted Positive Control, or the d_{12}-EDTA internal standard.

11.2.1.2 Signal-to-Noise

To justify the existence of a peak, its signal-to-noise ratio must exceed 3. Further, the baseline signal for the peak from the sample of interest must be at least 10 fold greater than that for any observed peak at a similar retention time in a Negative Control or blank sample injected just prior to that sample.

11.2.2 Mass Spectrometry

The mass spectral results (whether run in SRM mode or full scan products mode) for the analyte of interest should match that of the appropriate reference standard or an extracted Positive Control within a reasonable degree of scientific certainty. See the *Guidelines for Comparison of Mass Spectra* standard operating procedure (Tox 104) for further guidance.

12 Calculations

Not applicable.

Ex 446
(223)

13 Uncertainty of Measurement

Not applicable.

14 Limitations

a. Limit of Detection (LOD): The LOD for EDTA was determined to be 13 µg/mL in both the positive and negative electrospray ionization modes. These detection limits were determined by triplicate analysis of serial dilutions of an EDTA solution. The lowest concentration that reproducibly met the decision criteria listed in Section 11 above was determined to be the LOD.

A separate LOD study was conducted to determine the minimum volume of EDTA-preserved blood that was detectable using this analytical method. Three milliliters of whole blood were placed into a 4-mL lavender-topped blood collection tube containing 7.5 mg of EDTA and thoroughly mixed. The EDTA-preserved blood was placed on a clean, non-porous surface in triplicate at the following volumes: 1 µL, 5 µL, and 10 µL. Following a 1-hour drying period, the blood stains were swabbed using deionized water and cotton-tipped applicators. Each swab was extracted and analyzed to determine the minimum sized EDTA-preserved blood stain required in order to detect EDTA on the swab using the decision criteria requirements of Section 11. The 1 µL drop was readily detectable using this technique.

b. Selectivity: Selectivity was determined by extraction and analysis of 10 matrix blanks (swabs dipped into different blood samples with a variety of non-EDTA preservatives added to them). None of the 10 matrix blanks exhibited peaks of EDTA that met the decision criteria requirements of Section 11. Additionally, d_{12}-EDTA was evaluated for interferences in the analysis and none was observed.

c. Matrix Effects: Five extracted matrix blanks were spiked with an equal amount of an EDTA standard at both low and high concentrations. Following analysis of these samples, the results were compared with equal concentrations of neat EDTA to determine the amount of ion suppression caused by the blood matrix. While ion suppression was not noted in the positive electrospray ionization mode, suppression of 3% and 34% were noted in the negative electrospray ionization mode at EDTA concentrations of 50 µg/mL and 500 µg/mL, respectively.

d. Carryover: It has been reported in the literature that trace amounts of EDTA may be absorbed in the chromatographic system and released in a subsequent analysis of a sample. This carryover was assessed during validation, but was determined to be minor in nature appearing mainly after the injection of a high concentration of EDTA. To demonstrate the

lack of carryover within an analytical run, a minimum of two matrix-matched negative samples (i.e. whole blood extracts) must be analyzed between case samples.

15 Safety

Take standard precautions for the handling of chemicals and biological materials. Refer to the *FBI Laboratory Safety Manual* for guidance.

16 References

Miller, M.L., McCord, B.R., Martz, R., and Budowle, B. "The Analysis of EDTA in Dried Bloodstains by Electrospray LC-MS-MS and Ion Chromatography", *Journal of Analytical Toxicology*, Vol. 21, 1997, 521-528.

Sheppard, R.L. and Henion, J. "Determining EDTA in Blood", *Analytical Chemistry*, Vol. 69, 1997, 477A-480A.

Preparation of Chemical Reagents (Tox 103); FBI Laboratory Chemistry Unit – Toxicology Subunit SOP Manual.

Guidelines for Comparison of Mass Spectra (Tox 104); FBI Laboratory Chemistry Unit – Toxicology Subunit SOP Manual.

FBI Laboratory Chemistry Unit - Instrument Operation and Support Subunit SOP Manual.

FBI Laboratory Safety Manual.

Rev. #	Issue Date	History
0	02/15/07	New document.

Approval

Chemistry Unit Chief: Marc A. LeBeau Date: 2/15/07

QA Approval

Quality Manager: Robert B. Stacey Date: 2/15/2007

Issuance

Tox Subunit Manager: Marc A. LeBeau for M. Montgomery Madeline A. Montgomery Date: 2/15/07

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Ex 446
(336)

Performance Monitoring Protocol (QA/QC) for the Finnigan LCQ LC/MS (ESI)

1 Scope

This document addresses the performance monitoring (QA/QC) of the Finnigan LCQ LC/MS system consisting of a Finnigan LCQ MS and a Waters LC. It is an analytical instrument used to analyze a wide variety of evidence and must be maintained in such a way as to verify its reproducibility from analysis to analysis and its reliability in court.

2 Principle

The LCQ system is comprised of a Waters Liquid Chromatograph (LC) and a Finnigan ion trap LCQ Mass Spectrometer (MS). The instrument is configured with an API source that is capable of both electrospray (ESI) and atmospheric pressure chemical (APCI) ionization. The instrument is primarily used in ESI mode. However, this protocol can also be used for APCI provided the method of ionization is clearly labeled in the resulting data and documentation. Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Policy."

3 Equipment/Materials/Reagents

a. Instrumentation - Finnigan LCQ MS, API Source, Waters Alliance 2690/2695 LC, and Data System with XCalibur software (or equivalent)

b. API Gas - Nitrogen, 99.99% (high purity or equivalent)

c. Ion Trap Gas - Helium, 99.99% (high purity or equivalent)

d. Methanol, HPLC grade

e. Deionized Water, 18 MΩ Milli-Q or equivalent

f. Acetonitrile, HPLC grade

g. Acetic Acid, reagent grade

Ex 446
(227)

h. Ultramark 1621 (Finnigan or equivalent)

i. Caffeine (Sigma or equivalent)

j. MRFA (L-methionyl-arginyl-phenylalanyl-alanine acetate) (Finnigan or equivalent)

k. Ammonium Hydroxide (NH_4OH), reagent grade

l. Codeine (Sigma or equivalent)

m. Brucine (Sigma or equivalent)

n. Reserpine (Sigma or equivalent)

o. Volumetric glassware

p. Infusion Syringe - 10 to 500 µL LC syringe (Hamilton or equivalent)

4 Standards and Controls

4.1 Testmix

The Testmix is used to assess daily operating performance, mass assignment, and continued integrity of the system. To prepare, weigh 5.0 mg caffeine, 1.0 mg codeine, 1.0 mg brucine, and 1.0 mg reserpine into a 100-mL volumetric flask. Bring to the mark with methanol and mix well. Store the solution in the refrigerator. It has a shelf-life of three years. This preparation may be appropriately scaled up.

4.2 Calibration Solution

The calibration solution is used for coarse tuning and calibrating the mass spectrometer over the entire mass range. This procedure only needs to be performed when the instrument has been moved, down for a long period of time, undergone a major repair, or warranted based on system performance.

The calibration solution is a solution of caffeine, MRFA, and Ultramark 1621 in acetonitrile:methanol:water containing 1% acetic acid. To prepare this solution, follow the procedure in the LCQ 'Getting Started' manual. Store the solution in the refrigerator. It has a shelf-life of three years. This preparation may be appropriately scaled up.

Ex 446
(228)

5 Calibration

The calibration procedure should be performed as needed, when the instrument has been moved, down for a long period of time, undergone a major repair, or warranted based on system performance.

a. Load a 250 µL syringe with the calibration solution.

b. Using capillary tubing, connect the infusion syringe to the ESI probe assembly, and place in the syringe pump.

c. Set the syringe pump to the correct syringe type and set the pump rate to10 µL/minute.

d. Load the tune file "ESI_TUNE" (or equivalent).

e. Check that instrument is in POSITIVE ION mode and collecting CENTROID data.

f. Set the detector using the parameters listed in the 'Instrumental Conditions' section of this protocol.

g. Turn on the syringe pump and verify that the solution is flowing out the ESI needle.

h. Engage the ESI probe and turn on the MS.

i. In Tune Plus, open the Calibrate dialog box, choose the 'Automatic' tab and check the individual tests or 'Select All' and then 'Start.'

j. When the calibration is complete, it will display whether or not the calibration was successful.

- If the procedure fails, repeat the calibration.
- When the procedure passes, print the report and evaluate the calibration solution spectrum using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the spectrum of the calibration solution.

k. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Policy." If any requirements fail, the IOSS

Ex 446
(229)

Manager will determine the corrective action to be taken.

6 Sampling

Not applicable.

7 Procedures

7.1 Daily Checks

The following steps are to be performed daily. Enter the appropriate information in the QA/QC log for tracking purposes.

a. Record the remaining disk space on the hard drive. Use Windows Explorer program (WindowsNT) to verify that the hard disk has at least 100 MB of free disk space. Do not use if less than 100 MB remain.

b. Record the line pressure of the building nitrogen supply (API gas). The regulator should read between 60 and 100 p.s.i. If it cannot maintain this pressure, contact IOSS. If the nitrogen is supplied by a gas cylinder, record the tank pressure. Change the tank if less than 100 p.s.i. remaining.

c. Record the line pressure of the building helium supply (ion trap gas). The regulator should read between 30 and 60 p.s.i. If it cannot maintain this pressure, contact IOSS. If the helium is supplied by a gas cylinder, record the tank pressure. Change the tank if less than 100 p.s.i. remaining.

d. Check the Ion Gauge to ensure that no significant leaks are present in the system. Do not use if the pressure is higher than 1×10^{-4} torr.

e. Prepare the instrument for analysis of Testmix. Verify that the instrument has the correct source probe installed (ESI), the correct tune file loaded (esi_tune or equivalent), positive ion mode selected, and centroid data being collected. If a column is installed, remove it from that system and replace it with the infusion capillary tube.

f. Perform an analysis of the Testmix prior to the analysis of evidence using parameters listed in the 'Instrumental Conditions' section of this protocol. Start the HPLC pump. Engage the ESI probe and turn on the MS. Start an acquisition using a filename such

as 'testmix' (or equivalent). Make three 5 µL injections of the Testmix solution approximately 10 seconds apart by using the manual loop injector, and then stop the data collection. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the TIC and spectra for all components in the Testmix.

g. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Policy." If any requirements fail, contact IOSS.

7.2 As Needed Checks

a. Re-cut or replace the sample capillary as needed.

b. Clean or replace the heated capillary as needed.

8 Instrumental Conditions

Refer to the "General Instrument Maintenance Policy" for procedures on minor deviations.

8.1 Testmix

Liquid Chromatograph	
Mobile Phase:	95:5 methanol:water + 0.03% ammonium hydroxide
Flow Rate:	0.2 mL/min
Column:	None
Inj Volume:	5 µL
Number of Inj:	3

Mass Spectrometer	
Ionization:	ESI
Tune File:	esi_tune
Scan Mode:	Full Scan
Scan Range:	100-650 m/z

Ex 446
(231)

8.2 Calibration

Mass Spectrometer

Ionization:	ESI
Tune File:	esi_tune
Scan Mode:	Full Scan
Scan Range:	100-2000 m/z

9 Decision Criteria

9.1 Testmix

Verify the results of the Testmix. The following ions should be observed in the three Testmix injections:

- Caffeine 195 m/z
- Codeine 300 m/z
- Brucine 395 m/z
- Reserpine 609 m/z

9.2 Calibration

Verify the results of the calibration. The calibration will indicate if the procedure was successful. The individual ions for the calibration solution are:

• Caffeine	195 m/z	greater than 5%
• MRFA	524 m/z	greater than 50%
• Ultramark	1022 m/z	greater than 5%
	1122 m/z	greater than 20%
	1222 m/z	greater than 50%
	1322 m/z	greater than 50%
	1422 m/z	greater than 80%
	1522 m/z	greater than 50%
	1622 m/z	greater than 50%
	1722 m/z	greater than 20%
	1822 m/z	greater than 10%
	1922 m/z	present

Ex 446
(232)

10 Calculations

Not applicable.

11 Uncertainty of Measurement

Not applicable.

12 Limitations

Only properly trained personnel shall perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

13 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis. Many instrument components are held at temperatures of 250°C and higher. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

14 References

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Policy" (Inst 001) *Instrument Operation and Support Subunit SOP Manual.*

"Liquid Chromatograph General Maintenance Protocol" (Inst 003) *Instrument Operation and Support Subunit SOP Manual.*

"Mass Spectrometer General Maintenance Protocol" (Inst 004) *Instrument Operation and Support Subunit SOP Manual.*

FBI Laboratory Safety Manual.

Ex 446
(233)

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0	06/21/06	New document which replaces original titled "Performance Monitoring Protocol (QA/QC) for the Finnigan LCQ LC/MS (ESI)."

Approval

Chemistry Unit Chief: _____ Date: 6/12/06
Marc A. LeBeau

QA Approval

Quality Manager: _____ Date: 6/15/2006
Robert B. Stacey

Issuance

IOSS Manager: _____ Date: 6/21/06
Jeffrey N. Leibowitz

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Ex 446
(234)

Preparation of Chemical Reagents

1 Scope

This procedure provides instructions for the preparation and storage of all reagents used in the various standard operating procedures of the Chemistry Unit's Toxicology Subunit. This document does not provide information for materials used directly as obtained from the manufacturer. Neither does it provide instructions for the preparation of calibrators, controls, or analytical standards. Prepared reagents are listed, in alphabetical order, in section 6, "Procedure" and materials needed for preparation of these reagents are listed in section 2, "Equipment/ Materials/Reagents." Refer to the *Chemistry Unit Procedure for Verification of Reagents, Kits, Solvents and Standards* (CUQA 9) for guidance in labeling and testing the reliability of reagents.

2 Equipment/Materials/Reagents

a. Electronic balance

b. pH paper in various ranges

c. Ultrasonicator

d. Vacuum filtration apparatus (1 liter size) with 0.5 µm PTFE filter membranes

e. Miscellaneous routine laboratory glassware and supplies

f. Acetic acid, glacial (17 M) (ACS grade)

g. Acetonitrile (HPLC grade)

h. Ammonium acetate (reagent grade)

i. Ammonium hydroxide, concentrated (15 M) (ACS grade)

j. Calcium chloride (reagent grade)

k. Chloramine T (reagent grade)

l. Chloroform (GC^2 grade and HPLC grade)

m. o-Cresol (reagent grade)

n. Cupric sulfate pentahydrate (reagent grade, $CuSO_4 \cdot 5H_2O$)

o. Curcumin (reagent grade)

p. Diethyl ether (high purity grade)

q. Dimethylsulfoxide (ACS grade)

r. Diphenylamine (reagent grade)

s. Ethyl acetate (HPLC grade)

t. Ethyl alcohol (200 proof and pharmaceutical grade)

u. Ferric chloride hexahydrate (reagent grade, $FeCl_3 \cdot 6H_2O$)

v. Ferric nitrate nonahydrate (reagent grade, $Fe(NO_3)_3 \cdot 9H_2O$)

w. Gold(III) chloride hydrochloride trihydrate (reagent grade, $HAuCl_4 \cdot 3H_2O$)

x. Heptafluorobutyric acid (aka HFBA) (reagent grade)

y. Hexamethonium hydroxide solution, 0.1 M (obtained from Fluka Chemical Company)

z. Hexane (UV grade)

aa. Hydrochloric acid, concentrated (12 M) (ACS grade)

bb. Indigo Carmine (reagent grade)

cc. Iodine (reagent grade)

dd. Isopropanol (2-propanol) (HPLC grade)

ee. Magnesium nitrate (high purity grade I)

ff. Mercuric chloride (reagent grade, $HgCl_2$)

gg. Methanol (GC^2 grade and HPLC grade)

hh. Methylene Chloride (dichloromethane) (HPLC grade)

ii. Nitric acid, concentrated (16 M) (ACS grade and Optima grade)

Ex 446
(23)

jj. Palladium matrix modifier solution (0.1% obtained from High-Purity Standards, Inc.)

kk. PIC reagent (methanesulfonic acid) (reagent grade)

ll. PICB-8 reagent (octanesulfonic acid) (low UV grade obtained from Waters Corp.)

mm. Potassium cyanide (reagent grade)

nn. Potassium ferricyanide (reagent grade)

oo. Potassium hydroxide (reagent grade)

pp. Potassium iodide (reagent grade)

qq. Potassium phosphate, monobasic (ACS grade, KH_2PO_4)

rr. Pyromellitic acid (reagent grade)

ss. Saponin (reagent grade)

tt. Silver nitrate (reagent grade)

uu. Sodium acetate trihydrate (reagent grade)

vv. Sodium bicarbonate (reagent grade)

ww. Sodium borate (sodium tetraborate decahydrate) (ACS grade, $Na_2B_4O_7 \cdot 10H_2O$)

xx. Sodium chloride (reagent grade)

yy. Sodium dithionite (reagent grade)

zz. Sodium hydroxide (ACS grade)

aaa. Sodium phosphate, dibasic heptahydrate(ACS grade, $Na_2HPO_4 \cdot 7H_2O$)

bbb. Sodium phosphate, monobasic monohydrate (ACS grade, $NaH_2PO_4 \cdot H_2O$)

ccc. Sulfuric acid, concentrated (18 M) (ACS grade)

ddd. Tetramethylammonium hydroxide (ACS grade)

Ex 446
(237)

eee. Toluene (HPLC grade)

fff. Triethanolamine (reagent grade)

ggg. Trifluoroacetic acid (98+% purity)

hhh. Water, Deionized (18+MΩ grade)

3 Standards and Controls

Not applicable.

4 Calibration

Not applicable.

5 Sampling

Not applicable.

6 Procedure

Unless specifically noted otherwise, all reagents can be prepared in larger or smaller total volumes, as needed, by appropriate scaling of component volumes and masses. Listed grades or qualities of chemicals are the minimum acceptable levels. Unless specifically noted otherwise, a higher quality of the same chemical may be substituted.

a. 50 mM Acetic Acid:
To a 100-mL graduated cylinder, add 80 mL deionized water and 0.25 mL glacial acetic acid. Mix well and bring to 85 mL with deionized water. Store in glass at room temperature. Stable 3 months.

b. 0.1 M Acetic Acid:
To a 100-mL graduated cylinder, add 80 mL deionized water and 0.5 mL glacial acetic acid. Mix well and bring to 85 mL with deionized water. Store in glass at room temperature. Stable 3 months.

c. Dilute (~ 1.5 M) Acetic Acid:
Mix 2 mL glacial acetic acid with 20 mL deionized water and shake to combine. Store in glass at room temperature. Stable 3 months.

d. 1:1 Acetonitrile:Water:
Combine 100-mL HPLC grade acetonitrile with 100 mL deionized water and mix well. Store in glass at room temperature. Stable 6 months.

e. 0.1 M Ammonium Acetate:
Add 3.85 g ammonium acetate to a 500-mL volumetric flask containing 300 mL deionized water. Mix well to dissolve, and bring to volume with deionized water. Store in refrigerated in glass. Stable 2 months.

f. 0.24 M Ammonium Hydroxide:
Add 1.6 mL concentrated ammonium hydroxide to 50 mL deionized water in a 100-mL graduated cylinder. Fill to the 100-mL mark with deionized water and mix well. Store in glass at room temperature. Stable 1 month.

g. 2 M Ammonium Hydroxide:
Add 10 mL concentrated ammonium hydroxide to 50 mL deionized water in a 100-mL graduated cylinder. Fill to the 75-mL mark with deionized water and mix well. Store in glass at room temperature. Stable 1 month.

h. 5% (w/v) Calcium Chloride Solution:
Dissolve 1 g calcium chloride in 20 mL deionized water. Store in glass at room temperature. Stable 1 year.

i. CE (Capillary Electrophoresis) Run Buffer – Anions:
To a 1000-mL volumetric flask, add 500 mL deionized water, 612 mg pyromellitic acid, 280 mg sodium hydroxide, 238 mg triethanolamine, and 7.5 mL 0.1M hexamethonium hydroxide solution. Mix well to dissolve, and bring to volume with deionized water. Store refrigerated in plastic. Stable 1 week.

j. 0.5% (w/v) Chloramine T:
To a 100-mL volumetric flask, add 80 mL deionized water and 0.5 g chloramine T. Mix well to dissolve and bring to volume with deionized water. Store refrigerated in glass. Stable 6 months.

k. 4:1 Chloroform:Methanol:
Combine 40 mL GC^2 grade chloroform with 10 mL GC^2 methanol. Mix well. Store in brown glass at room temperature. Stable 1 month.

Ex 446
(239)

l. 1% (by volume) o-Cresol:
Place 1 mL o-cresol in a 100-mL volumetric flask and fill to the mark with deionized water. Mix well and allow to stand for at least 24 hours before use. Store refrigerated in brown glass. Stable 6 months.

m. 5% (w:v) Cupric Sulfate Solution:
Dissolve 1.56 g cupric sulfate pentahydrate in 20 mL deionized water. Store in glass at room temperature. Stable 1 year.

n. Curcumin Solution (saturated in ethanol):
Add curcumin to 10 mL 200 proof ethanol in a test tube with mixing until no more will dissolve. Centrifuge at low speed for 5 min and transfer the supernatant. Store in glass at room temperature. Stable 1 year.

o. Cyanmethemoglobin Reagent (Drabkin's Solution):
To a 1000-mL volumetric flask containing 500 mL deionized water, add 200 mg potassium ferricyanide, 50 mg potassium cyanide, and 1 g sodium bicarbonate. Mix well to dissolve and bring to volume with deionized water. Store refrigerated in brown glass. Stable 4 months.

p. Diphenylamine Reagent (0.5% w:v in sulfuric acid):
Dissolve 0.5 g diphenylamine in 100 mL concentrated sulfuric acid. Store in glass with a PTFE-lined cap at room temperature. Stable 1 year.

q. 80% (by volume) Ethanol:
Measure 80 mL pharmaceutical grade ethanol into a 100-mL volumetric flask. Bring to volume with deionized water and mix well. Store in glass at room temperature. Stable for 6 months.

r. 1:1 Ether:Toluene:
Combine 50 mL HPLC grade toluene with 50 mL diethyl ether. Mix well. Store in glass at room temperature. Stable 1 month.

s. 5% (w/v) Ferric Chloride Solution:
Dissolve 1.67 g ferric chloride hexahydrate in 20 mL deionized water. Store in glass at room temperature. Stable 1 year.

t. 0.5% (w/v) Gold Chloride Solution:
Dissolve 130 mg gold(III) chloride hydrochloride trihydrate in 20 mL deionized water. Store in brown glass at room temperature. Stable 1 year.

Ex 446
(240)

u. 0.1% (w/v) Heptafluorobutyric Acid:
Add 0.5 g HFBA to 400 mL deionized water in a 500-mL volumetric flask and mix well. Bring to volume with deionized water. Store in glass at room temperature. Stable 3 months.

v. 2 mM Hydrochloric Acid:
In a 100-mL volumetric flask, combine 80 mL deionized water with 16 µl concentrated hydrochloric acid and mix well. Bring to volume with deionized water. Store in glass at room temperature. Stable 6 months.

w. 0.1 M Hydrochloric Acid:
To a 100-mL graduated cylinder, add 80 mL deionized water and 0.8 mL concentrated hydrochloric acid. Bring to 96 mL with deionized water and mix well. Store in glass at room temperature. Stable 6 months.

x. 0.96 M Hydrochloric Acid:
To a 100-mL volumetric flask, add 80 mL deionized water. Add 8 mL concentrated hydrochloric acid and mix well. Bring to volume with deionized water. Store in glass at room temperature. Stable 6 months.

y. 1 M Hydrochloric Acid:
To a 100-mL graduated cylinder, add 80 mL deionized water. Add 8 mL concentrated hydrochloric acid and mix well. Bring to 96 mL with deionized water. Store in glass at room temperature. Stable 6 months.

z. 6 M Hydrochloric Acid (~ 50% v:v):
To a 25-mL graduated cylinder containing 10 mL deionized water, add 12 mL concentrated hydrochloric acid and mix well. Bring to 24 mL with deionized water. Store in glass at room temperature. Stable 6 months.

aa. 0.01% (w/v) Indigo Carmine Reagent:
Dissolve 10 mg indigo carmine in 100 mL deionized water. Store in glass at room temperature. Stable 1 year.

bb. Iodine Test Solution:
Dissolve 0.4 g iodine and 0.6 g potassium iodide in 20 mL deionized water. Store in glass at room temperature. Stable 6 months.

cc. LC (Liquid Chromatography) Mobile Phase – Alkaline#1 / Cocaine (95:5:0.03 methanol:water:ammonia):
Combine 950 mL HPLC grade methanol and 50 mL deionized water. Mix well and vacuum filter through a 0.5 µm PTFE membrane. Add 0.3 mL concentrated ammonium

Ex 446
(241)

hydroxide and mix gently. Verify pH>8. Store in glass at room temperature. Stable 1 month.

dd. LC Mobile Phase – Alkaline#2 (5:95:0.03 methanol:water:ammonia):
Combine 25 mL HPLC grade methanol and 475 mL deionized water. Mix well and vacuum filter through a 0.5 µm PTFE membrane. Add 0.15 mL concentrated ammonium hydroxide and mix gently. Verify pH>8. Store in glass at room temperature. Stable 1 month.

ee. LC Mobile Phase – Benzodiazepines (60:40:0.03 methanol:water:ammonia):
Combine 300 mL HPLC grade methanol and 200 mL deionized water. Mix well and vacuum filter through a 0.5 µm PTFE membrane. Add 0.15 mL concentrated ammonium hydroxide and mix gently. Verify pH>8. Store in glass at room temperature. Stable 1 month.

ff. LC Mobile Phase – Rodenticide #1 (0.1% acetic acid in water):
Vacuum filter 500 mL deionized water through a 0.5 µm PTFE membrane. Add 0.5 mL ACS grade glacial acetic acid. Store in glass at room temperature. Stable 1 month.

gg. LC Mobile Phase – Rodenticide #2 (0.1% acetic acid in methanol):
Vacuum filter 500 mL Optima grade methanol. Add 0.5 mL ACS grade glacial acetic acid. Vacuum filter through a 0.5 µm PTFE membrane. Store in glass at room temperature. Stable 1 month.

hh. LC Mobile Phase – Mivacurium #1 (acetonitrile):
Measure out 1000 mL HPLC grade acetonitrile and vacuum filter through a 0.5 µm PTFE membrane. Store in glass at room temperature. Stable 2 months.

ii. LC Mobile Phase – Mivacurium #2 (5 mM octanesulfonic acid):
Quantitatively transfer the contents of one vial of PICB-8 reagent into a 1000-mL volumetric flask and bring to the mark with deionized water. Vacuum filter through a 0.5 µm PTFE membrane. Store in glass at room temperature. Stable 1 month.

jj. LC Mobile Phase – Mivacurium MSMS (40:60:0.015 acetonitrile:water:methanesulfonic acid):
Combine 200 mL HPLC grade acetonitrile, 300 mL deionized water, and 75 µl PIC reagent. Vacuum filter through a 0.5 µm PTFE membrane, and verify 2<pH<3.5. Store at room temperature in brown glass. Stable 1 month.

kk. LC Mobile Phase – Succinylmonocholine #1 (92:8:0.1 water:methanol:PIC reagent):
Combine 460 mL deionized water, 40 mL HPLC methanol, and 0.5 mL PIC reagent. Mix well and vacuum filter through a 0.5 µm PTFE membrane. Store at room temperature in brown glass. Stable for 1 month.

Ex 446
(342)

ll. LC Mobile Phase – Succinylmonocholine #2 (80:15:4.75:0.25 0.1% HFBA:0.1 M ammonium acetate:acetonitrile:isopropanol):
Combine 400 mL 0.1% heptafluorobutyric acid, 75 mL 0.1 M ammonium acetate, 23.75 mL HPLC grade acetonitrile, and 1.25 mL HPLC grade isopropanol. Mix well and vacuum filter through a 0.5 µm PTFE membrane. Store in glass at room temperature. Stable 1 month.

mm. 1:1 Methanol:Dilute Hydrochloric Acid:
Combine 2 mL GC^2 grade methanol with 2 mL 1 M hydrochloric acid, and mix well. Store in glass at room temperature. To be prepared fresh.

nn. 95:5 Methanol:Water:
Combine 95 mL HPLC grade methanol with 5 mL deionized water and mix well. Store in glass at room temperature. Stable 6 months.

oo. 1:1 Methanol:Water:
Combine 50 mL HPLC methanol with 50 mL deionized water and mix well. Store in glass at room temperature. Stable 6 months.

pp. Nitric Acid, Dilute (33% by volume):
Mix 5 mL concentrated nitric acid with 10 mL deionized water and shake to combine. Store in glass at room temperature. Stable 1 year.

qq. 0.2% (by volume) Nitric Acid:
To a 1000-mL Nalgene volumetric flask containing 600 mL deionized water, add 2 mL Optima grade concentrated nitric acid. Bring to volume with deionized water and mix well. Store in plastic at room temperature. Stable 1 year.

rr. Opiates Extraction Solvent (90:10 chloroform:isopropanol):
Combine 50 mL HPLC grade isopropanol and 450 mL HPLC grade chloroform and mix well. Store at room temperature in brown glass. Stable 1 month.

ss. Palladium / Magnesium Nitrate Matrix Modifier for AAS (Atomic Absorption Spectroscopy):
To a 100-mL Nalgene volumetric flask containing 50 mL deionized water, add 15 mg magnesium nitrate and 25 mL palladium matrix modifier solution. Bring to volume with deionized water and mix well. Store at room temperature in Nalgene container. Stable 5 years.

Ex 446
(243)

tt. 11.8 M Potassium Hydroxide:
To a 100-mL Nalgene volumetric flask add 66 g potassium hydroxide and 50 mL deionized water. Mix well to dissolve and bring to volume with deionized water. Store at room temperature in Nalgene container. Stable 1 year.

uu. 5% (w/v) Potassium Phosphate Buffer (pH 4.5):
To a 100-mL volumetric flask, add 80 mL deionized water. Add 5 g monobasic potassium phosphate and mix well to dissolve. Bring to volume with deionized water, and verify 4.0<pH<5.0. Store refrigerated in glass. Stable 1 month.

vv. 10% (w/v) Silver Nitrate Solution:
Dissolve 2 g silver nitrate in 20 mL deionized water. Store at room temperature in an opaque container. Stable 1 year.

ww. 0.1 M Sodium Acetate Buffer (pH 7):
To a 250-mL volumetric flask, add 3.4 g sodium acetate trihydrate and 200 mL deionized water. Mix well and adjust to 6.5<pH<7.5 by slow addition of 1 N hydrochloric acid. Bring to volume with deionized water. Store refrigerated in glass. Stable 2 months.

xx. Sodium Acetate Buffer with 5% Methanol:
Add 5 mL HPLC grade methanol to a 100-mL volumetric flask and bring to volume with 0.1 M sodium acetate buffer (pH 7). Store refrigerated in glass. Stable 2 months.

yy. 1.1 M Sodium Acetate Buffer (pH 5.5):
To a 100-mL volumetric flask, add 14.95 g sodium acetate trihydrate, 60 mL deionized water, and 2.2 mL glacial acetic acid. Mix well to dissolve, and bring to volume with deionized water. Verify 5<pH<6. Store refrigerated in glass. Stable 2 months.

zz. 0.1 M Sodium Borate Buffer (pH 9):
To a 100-mL volumetric flask add 3.8 g sodium borate and bring to volume with deionized water. Sonicate for 15 minutes to assist dissolution, and verify 8.5<pH<9.5. Store refrigerated in glass. Stable 3 months.

aaa. Saturated Sodium Chloride Solution (~ 35% w/v):
To a 500-mL volumetric flask, add 450 mL deionized water and 175 g sodium chloride. Gently heat with continuous stirring for at least one hour. Remove the stirbar, fill to volume with deionized water, and mix by inversion. A small amount of undissolved solid should remain in the bottom of the flask. Store in glass at room temperature. Stable for one year.

Ex 446
(244)

bbb. 0.287 M Sodium Dithionite Reducing Agent:
To a 50-mL volumetric flask, add 40 mL deionized water and 2.96 g sodium dithionite. Mix well to dissolve and bring to volume with deionized water. Store in glass at room temperature. Stable 1 month.

ccc. 0.1 M Sodium Hydroxide:
To a 100-mL Nalgene volumetric flask, add 60 mL water and 0.4 g sodium hydroxide. Mix well to dissolve and bring to volume with deionized water. Store in Nalgene containers at room temperature. Stable 1 year.

ddd. 5 M (20% w/v) Sodium Hydroxide:
To a 100-mL Nalgene volumetric flask, add 60 mL water and 20 g sodium hydroxide. Mix well to dissolve and bring to volume with deionized water. Store in Nalgene containers at room temperature. Stable 1 year.

eee. 0.1 M Sodium Phosphate Buffer (pH 6.0):
To a 500-mL volumetric flask, add 400 mL deionized water, 6.1 g sodium phosphate monobasic monohydrate, and 1.6 g sodium phosphate dibasic heptahydrate. Mix well to dissolve. Verify 5.8<pH<6.1, adjusting pH by addition of 0.1 M dibasic sodium phosphate (increases pH) or 0.1 M monobasic sodium phosphate (decreases pH) as necessary. Bring to volume with deionized water. Store refrigerated in glass. Stable 1 month.

fff. 0.1 M Sodium Phosphate, Dibasic:
To a 100-mL volumetric flask, add 2.7 g sodium phosphate dibasic heptahydrate and 80 mL deionized water. Mix well to dissolve and bring to volume with deionized water. Store refrigerated in glass. Stable 1 month.

ggg. 0.1 M Sodium Phosphate, Monobasic:
To a 100-mL volumetric flask, add 1.4 g sodium phosphate monobasic monohydrate and 80 mL deionized water. Mix well to dissolve and bring to volume with deionized water. Store refrigerated in glass. Stable 1 month.

hhh. SPE (Solid Phase Extraction) Alkaline / Cocaine Elution Solvent (78:20:2 methylene chloride:isopropanol:ammonia):
Combine 20 mL HPLC grade isopropanol with 2 mL concentrated ammonium hydroxide and mix well. Add 78 mL HPLC grade methylene chloride and mix well. Store in glass at room temperature. To be prepared fresh.

iii. SPE Benzodiazepines Elution Solvent (49:1 ethyl acetate:ammonia):
Combine 49 mL ethyl acetate with 1 mL concentrated ammonium hydroxide and mix well. Store in glass at room temperature. To be prepared fresh.

Ex 446
(245)

jjj. SPE Benzodiazepines Wash Solvent (20% acetonitrile in 0.1 M phosphate buffer):
Combine 80 mL 0.1 M phosphate buffer (pH 6) with 20 mL HPLC grade acetonitrile and mix well. Store in glass at room temperature. Stable 1 month.

kkk. SPE THC Elution Solvent aka Rodenticide Wash Solvent (95:5 hexane:ethyl acetate):
Combine 95 mL hexane with 5 mL ethyl acetate and mix well. Store in glass at room temperature. Stable 3 months.

lll. SPE THC-COOH Elution Solvent aka Rodenticide Elution Solvent (75:25:1 hexane:ethyl acetate:acetic acid):
Combine 75 mL hexane with 25 mL ethyl acetate and 1 mL glacial acetic acid. Mix well. Store in glass at room temperature. Stable 1 month.

mmm. 5 N Sulfuric Acid:
To a 100-mL graduated cylinder containing 70 mL deionized water, slowly add 12.5 mL concentrated sulfuric acid. Mix well and bring to 90 mL with deionized water. Store in glass at room temperature. Stable 1 year.

nnn. 5% (by volume) Sulfuric Acid:
To a 100-mL volumetric flask containing 80 mL deionized water, slowly add 5 mL concentrated sulfuric acid. Mix well and bring to volume with deionized water. Store in glass at room temperature. Stable 1 year.

ooo. 1 M Sulfuric Acid with 1.5% (w/v) Saponin:
Add 80 mL deionized water and 1.35 g saponin to a 100-mL graduated cylinder and mix well to dissolve. Slowly add 5 mL concentrated sulfuric acid. Bring to the 90-mL mark with deionized water and mix well. Store in glass at room temperature. Stable 1 year.

ppp. THC-COOH Extraction Solvent (7:1 hexane:ethyl acetate):
Combine 70 mL hexane with 10 mL ethyl acetate and mix well. Store in glass at room temperature. Stable 3 months.

qqq. TMAH Reagent:
Dissolve 0.25 g tetramethylammoniumhydroxide in 1 mL deionized water. Add 20 mL dimethylsulfoxide and mix well. Store refrigerated in brown glass. Stable 1 year.

rrr. 0.04% (by volume) Trifluoroacetic Acid (TFA):
To a 100-mL volumetric flask, add 90 mL deionized water and 40 µl trifluoroacetic acid. Mix well and bring to volume with deionized water. Store in glass at room temperature. Stable 3 months.

Ex 446
(246)

sss. Trinder's Reagent:
Add 400 mg mercuric chloride, 400 mg ferric nitrate nonahydrate, and 0.1 mL concentrated hydrochloric acid to 5 mL deionized water in a 10-mL volumetric flask. Mix well to dissolve and bring to volume with deionized water. Store refrigerated in glass. Stable 6 months.

7 Instrumental Conditions

Not applicable.

8 Decision Criteria

Not applicable.

9 Calculations

Not applicable.

10 Uncertainty of Measurement

Not applicable.

11 Limitations

Not applicable.

12 Safety

Follow standard precautions for the handling of chemicals and biological materials. Refer to the *FBI Laboratory Safety Manual* for guidance.

Ex 446
(247)

13 References

Shugar, G. J.; Ballinger, J.T. *Chemical Technicians' Ready Reference Handbook, 3rd Ed.*; McGraw-Hill: New York, NY, 1990.

FBI Laboratory Safety Manual.

Toxicology Subunit SOP Manual.

Chemistry Unit Procedure for Verification of Reagents, Kits, Solvents and Standards (CUQA 9); FBI Laboratory Chemistry Unit Quality Assurance Manual.

Ex 446
(24K)

Rev. #	Issue Date	History
0	1/30/06	New document.
1	6/21/06	Added new reagent (saturated sodium chloride), and removed several (insulin ELISA reagents).
2	10/27/06	Added new reagents (mobile phases for rodenticides).

Approval

Chemistry Unit Chief: _____ Date: 10/10/06
Marc A. LeBeau

QA Approval

Quality Manager: _____ Date: 10/25/2006
Robert B. Stacey

Issuance

Tox Subunit Manager: _____ Date: 10/27/06
Madeline A. Montgomery

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Ex 446
(249)

Guidelines for Comparison of Mass Spectra

1 Introduction

Many of the analytical procedures used in the Toxicology Subunit rely on mass spectrometry to help establish identification of individual chemical entities within a sample. In order to ensure consistency and reproducibility in compound identification, it is desirable to have guidelines for the comparison of known and unknown mass spectra.

2 Scope

This document provides guidelines to help determine what constitutes a match between known and unknown mass spectra. Various critical characteristics of a mass spectrum are defined, and procedures for using these characteristics to evaluate matching between spectra are laid out. Note that this document provides guidelines for the matching of mass spectra, and does not directly address compound identification. A good quality mass spectral match will normally be only one element in establishing the identity of an unknown substance. These protocols are intended for application to full scan, tandem, and selected ion monitoring (SIM) mass spectra acquired in electron impact (EI), chemical (CI), electrospray (ESI), and atmospheric pressure chemical ionization (APCI) modes. Other mass spectral techniques are beyond the scope of this document.

3 Principle

The spectrum of a given unknown of interest is compared to the known spectrum of a target analyte. The unknown spectrum should contain all the significant ions present in the known spectrum, and should not contain any unexplained significant ions not seen in the known spectrum. The relative intensities (hereafter referred to as "ion ratios") of several selected characteristic ions should match in both spectra, to within defined tolerances. These guidelines draw heavily on technical document 2003IDCR from the World Anti-Doping Agency, the 2003 recommendations of the American Society for Mass Spectrometry's Measurements and Standards Committee, and the 2002 National Committee for Clinical Laboratory Standards Approved Guideline for GC/MS (gas chromatography/mass spectrometry) Confirmation of Drugs.

4 Specimens

Not applicable.

5 Equipment/Materials/Reagents

Ex 446
(250)

Not applicable.

6 Standards and Controls

Not applicable.

7 Calibration

The calibration of all mass spectrometers should be verified regularly per the appropriate instrument protocols in the *Instrument Operations and Support Subunit SOP Manual*.

8 Sampling

Not applicable.

9 Procedure

Provided below are procedures for defining and determining critical characteristics of a mass spectrum to be used in establishing whether or not two spectra match. An abbreviated list of the key points is provided in Appendix 1.

9.1 Averaging and Background Subtraction of Mass Spectra

In many real-world samples, it may be necessary to correct mass spectra of interest for the presence of ions resulting from sample background, instrument background, or partially coeluting sample components. The necessary background-subtracted spectrum will usually be generated by averaging not more than five spectra across the peak of interest and then subtracting the average of a number of background spectra equal to not more than twice the number of sample spectra. The background spectra may come before and/or after the sample spectra, and should all be selected from outside the region integrated for determination of ion ratios. This background-subtracted spectrum will be used to establish the list of significant ions and the base peak for that spectrum.

9.2 Determination of "Significant Ions" in a Mass Spectrum

Any ion signal greater than 15% of the most intense ion signal in a background-subtracted mass spectrum will normally be considered a ***significant ion***. An ion that would otherwise be considered significant may be excluded if it can be demonstrated that the ion arises from, or is significantly disturbed by, an uncorrectable chemical interference. Such interferences will

normally be demonstrated by showing that a reconstructed ion trace for the ion in question is not coincident with the traces for other ions associated with the component of interest.

9.3 Determination of "Diagnostic Ions" in a Mass Spectrum

Diagnostic ions are those ions in a mass spectrum that are characteristic of the chemical compound under investigation. Determination of diagnostic ions depends upon knowledge of the chemical structure of a component under investigation, and may therefore only be determined from mass spectra of known standards. The definition of what makes any given ion "characteristic" of a particular chemical structure is somewhat nebulous, and there does not appear to be any universally accepted standard in the field. This means that good and consistent judgment by the examiner is essential. There are, however, recommendations as to what renders some ions nonspecific and not diagnostic, and an examiner should abide by these practices when eliminating ions from consideration as diagnostic.

Adduct ions will normally be excluded, except that one pseudomolecular adduct ion may be considered diagnostic. Isotopomers will be excluded unless they are characteristic of a specific chemical composition. Normally this will be limited to chlorine and bromine isotopomers, but other possibilities may arise. Ions resulting purely from a derivatizing or complexing reagent will normally be excluded from the list of diagnostic ions. For example, the m/z 73 ion of a trimethylsilyl derivative may not be chosen as a diagnostic ion. Normally the (pseudo)molecular ion for a compound will be considered diagnostic, unless the intensity for that ion is less than 5% of the intensity for the base peak in the background-subtracted spectrum of the component in question.

9.4 Determination of the "Base Peak" in a Mass Spectrum

The ***base peak*** for the mass spectrum of a known standard is the most intense signal for a diagnostic ion in the background-subtracted spectrum. For the purpose of determining relative ion intensities the base peak in an unknown mass spectrum will be taken as the base peak of the standard spectrum to which it is being compared, even if a different diagnostic ion shows higher intensity in that spectrum.

In instances where it can be demonstrated that the nominal base peak signal is significantly disturbed by an uncorrectable chemical interference, the second most intense diagnostic ion present in the spectrum may be used as the base peak. Such interference will normally be demonstrated by showing that a reconstructed ion trace for the ion in question is not coincident with the traces for other ions associated with the component of interest.

9.5 Method for Calculating Ion Ratios

Ion ratios will normally be determined by integrating reconstructed ion traces for each diagnostic ion present in a given component. All integrations of reconstructed ion traces from a given

component should have comparable stop and start points. Ion ratios are then calculated by dividing the area for each ion trace by the area for the trace of the base peak ion, and expressing the result as a percentage. In instances where the reconstructed ion traces produce non-integratable data, it is acceptable to substitute ion abundances from the background subtracted spectrum of the compound of interest for the integrated areas from reconstructed ion traces. This will normally happen in situations where multiple sorts of mass spectral data are simultaneously acquired in a single analytical run, resulting in discontinuous data streams for the various individual mass spectral experiments.

10 Instrument Conditions

Not applicable.

11 Decision Criteria

Provided below are guidelines for establishing a match between a known mass spectrum and that of an unknown spectrum. Note that some analytical standard operating procedures (SOP's) include detailed criteria for the evaluation of mass spectra of individual target analytes. Such specific instructions will supercede the guidelines provided below.

In almost all cases, unknown spectra should be matched against known spectra obtained from contemporaneously analyzed reference material. Exceptions are discussed in section 12.5 of these guidelines. When assessing spectra for a targeted analyte from multiple unknown samples in a single analytical run, it is acceptable to compare each unknown spectrum to the known spectrum resulting from a different contemporaneously analyzed reference sample. The mass spectra of many chemical entities are known to vary with analyte load. It is acceptable to dilute and reanalyze a sample containing a high level of a suspected target compound in order to be able to more appropriately match its spectrum to a lower concentration standard, control, or calibrator.

11.1 For Full Scan Mass Spectra

In order to establish a match between known and unknown mass spectra in the full scan mode, both of the following criteria should be met:

a. Every significant ion present in the known spectrum should be present in the unknown spectrum, and vice-versa.

b. All relative intensities for diagnostic ions in the unknown spectrum should match those observed in the known spectrum within the tolerances shown in Table 1 or Table 2. If these limits would produce an acceptable lower bound of less than 1% for a given ion ratio,

the lower limit will be set at 1%. Ion ratios for specific diagnostic ions may be excluded from consideration if they meet any of the following criteria:

1. The ion ratio for that ion in the known spectrum is less than 5% (less than 10% for CI, ESI, or APCI spectra).
2. The signal-to-noise ratio of the reconstructed ion trace for that ion in the unknown spectrum is less than 3.
3. It can be shown that the signal for that ion in either the known or the unknown spectrum is significantly disturbed by an uncorrectable chemical interference. Such interference will normally be demonstrated by showing that a reconstructed ion trace for the ion in question is not coincident with the traces for other ions associated with the component of interest.

If there are more than four diagnostic ions in the known spectrum, then the examiner need only evaluate the ratios for four diagnostic ions (three ratios) in order to establish a scientifically valid match between the spectra. For compounds with a molecular weight less than 80 AMU, only three diagnostic ions (2 ratios) need be evaluated to establish a scientifically valid match. The selected ions will normally include the base peak and the (pseudo)molecular ion, unless those ions meet one of the three exclusion criteria given above. If fewer than three diagnostic ions are available for evaluation, the spectra may still be matched, but the examiner should be aware that the information content derived from such a match is limited, and should be regarded with caution. Examiners should also take care to ensure that the chosen scan range provides adequate "buffer space" around the diagnostic and significant ions of the substance in question.

Table 1: Ion Ratio Matching Tolerances for EI Mass Spectra

If the ion ratio in the known spectrum is:	>50%	≥25% and ≤50%	<25%
Then the ion ratio in the unknown spectrum should be within:	10% absolute	20% relative	5% absolute

Table 2: Ion Ratio Matching Tolerances for CI, ESI, and APCI Mass Spectra

If the ion ratio in the known spectrum is:	>60%	≥40% and ≤60%	<40%
Then the ion ratio in the unknown spectrum should be within:	15% absolute	25% relative	10% absolute

11.2 For SIM Mass Spectra

Selected ion monitoring experiments can allow for the detection of very low levels of analyte in complex sample matrices, at the cost of reducing the information content of that experiment. Examiners should take great care in selecting monitored ions for a SIM experiment. Ions for a SIM experiment must be based upon a known full scan spectrum of the species of interest collected

Ex 446
(254)

on the instrument to be used for the SIM experiment. Four diagnostic ions will normally be selected (three ions for compounds with a molecular weight less than 80 AMU; see 12.4 for another exception), and, if possible, all should be significant as well as diagnostic. The base peak will normally be one of the chosen ions, and the (pseudo)molecular ion should be included if it has a relative intensity greater than 5% in the known full scan spectrum. In order to establish a match between a known SIM spectrum and an unknown SIM spectrum, all resulting ion ratios should meet the tolerances specified in Table 1 or Table 2, as appropriate.

11.3 For Tandem Mass Spectrometry (MS/MS)

Tandem mass spectrometry can lend a great deal of additional specificity to mass spectral experiments by greatly increasing the confidence that the ions in a given spectrum are all associated with a single substance. Due to the nature of most collision-induced dissociation processes, however, ion ratios in tandem mass spectra tend to be much less stable, and much more dependent on analyte load, than is true for classic electron impact mass spectra.

Tandem mass spectra tend to be much "cleaner" than full scan mass spectra, with fewer extraneous ions. Therefore, the limit for determination of significant ions in a tandem mass spectrum is lowered to 10% (from 15%) of the most intense observed ion in the background subtracted spectrum. The high probability of ion association in tandem mass spectrometry means that nearly all ions of reasonable intensity observed in an MS/MS experiment should be considered diagnostic, with the exception of ions resulting purely from the loss of an adduct.

Due to vagaries of the physical processes involved in the precursor ion isolation and fragmentation events in an ion trap mass spectrometer, tandem mass spectra acquired on such an instrument will occasionally show an "ion-splitting" artifact for a precursor ion returned in a product ion mass spectrum. This is evidenced by the presence of two ions, separated by a fraction of an AMU, at the nominal mass of the precursor ion in the product ion spectrum. In instances where this phenomenon is observed, the response for the affected ion should be taken as the total of the response for both components of the "split" ion signal.

11.3.1 Product Ion Experiments

When conducting product ion experiments, the selection of a precursor ion is critical to obtaining useful and reliable information. In most cases, the (pseudo)molecular ion of the species under consideration should be selected, if available. It is also acceptable to use a diagnostic isotopomer of the (pseudo)molecular ion, if one is available. If the (pseudo)molecular ion is not available, or is not suitable for some reason, then the selected precursor ion should be both significant and diagnostic in the full scan mass spectrum of the substance under consideration. With product ion spectra, it is also important to ensure that the observed fragment spectrum is, in fact, emerging from the selected precursor ion. For this reason, one of the two following criteria should normally be met for a product ion spectrum:

a. The precursor ion should be observed in the product ion spectrum with an ion ratio of at least 5%.
b. If full scan mass spectral data are collected concurrently with the product ion spectra, the full scan spectrum of the component of interest should show no ions within 1.5 AMU of the precursor ion with greater than three times the intensity of the precursor ion.

In order to establish a match between a known product ion spectrum and the product ion spectrum of an unknown, both of the following criteria should be met:

a. Every significant ion present in the known spectrum should be present in the unknown spectrum, and vice-versa.
b. All relative intensities for diagnostic ions in the unknown spectrum should match those observed in the known spectrum to within the tolerances shown in Table 3. If these limits would produce an acceptable lower bound of less than 1% for a given ion ratio, the lower limit will be set at 0.5%. Ion ratios for specific diagnostic ions may be excluded from consideration if they meet any of the following criteria:

    1. The ion ratio for that ion in the known spectrum is less than 5%.
    2. The signal-to-noise ratio of the reconstructed ion trace for that ion in the unknown spectrum is less than 3.
    3. It can be shown that the signal for that ion in either the known or the unknown spectrum is significantly disturbed by an uncorrectable chemical interference. Such interference will normally be demonstrated by showing that a reconstructed ion trace for the ion in question is not coincident with the traces for other ions associated with the component of interest.

If there are more than three diagnostic ions in the known spectrum, then the examiner need only evaluate the ratios for three diagnostic ions (two ratios) in order to establish a scientifically valid match between the spectra. The selected three ions should include the base peak and the precursor ion (if present), unless those ions meet one of the three exclusion criteria given above. If only a single diagnostic ion is observed in the product ion spectrum, spectra may still be matched, but the examiner should be aware that the information content derived from such a match is limited, and should be regarded with caution.

Table 3: Ion Ratio Matching Tolerances for MS/MS Product Ion Spectra

If the ion ratio in the known spectrum is:	>40%	≤40%
Then the ion ratio in the unknown spectrum should be within:	25% relative	10% absolute

Ex 446
(256)

11.3.2 Precursor Ion and Neutral Loss Experiments

The practical information content for precursor ion and neutral loss MS/MS experiments is generally low, but circumstances may still arise in which one of these techniques can provide critical additional information about a given substance. For precursor ion experiments, a match between a known and an unknown spectrum may be established if all significant ions present in the known spectrum are present in the unknown spectrum, and vice-versa. For neutral loss experiments, a match between a known and an unknown spectrum may be established if all significant transition pairs present in the known spectrum are present in the unknown spectrum and vice-versa.

11.3.3 Selected Reaction Monitoring (SRM) Experiments

SRM analysis shares many features, advantages, and limitations with SIM analysis, but benefits from the added specificity afforded by tandem mass spectrometry. Three diagnostic ion transitions should be chosen for an SRM experiment. Generally, all three transitions should share a common precursor ion, although it is appropriate to use multiple precursor ions if all are part of a diagnostic isotope cluster in the full scan spectrum of the substance in question. It is desirable that the chosen precursor ion be the (pseudo)molecular ion of the substance in question. If this is not possible, or not practical, then the chosen precursor ion should be both significant and diagnostic in the full scan spectrum of the substance in question. In order to establish a match between a known SRM spectrum and an unknown SRM spectrum, both resulting ion ratios should meet the tolerances specified in Table 3.

11.3.4 Higher Order (MS^n) Tandem Mass Spectrometry

Tandem mass spectra of order higher than 2 are beyond the scope of this document. There is little to no discussion of this subject in the various published technical guidelines, and the technique is rarely practiced in forensic and regulatory settings. When used, higher order tandem mass spectra will be addressed on a case-by-case basis, usually as a part of method validation. Examiners should consider using the criteria for product ion MS/MS in section 12.3.1 as a starting point for such evaluation.

11.4 Exact (Precise) Mass Measurement Techniques

Exact mass measurement can provide a significant additional level of information content in a mass spectrum, giving an examiner more confidence in any conclusions based upon that spectrum. The use of exact mass measurement techniques does not, however, allow an examiner to disregard other aspects of the mass spectrum under consideration. As such, mass spectra obtained using exact mass techniques should still meet all of the matching criteria for the appropriate mass spectral techniques given above, but different standards may be used in selecting diagnostic ions, and more confidence can be placed in matches based upon a limited set of diagnostic ions.

Ions in an unknown spectrum will be considered to be an exact mass match to those in a known spectrum if the measured masses agree to within 0.005 AMU. For a SIM experiment, only three ions, instead of four, need be monitored, and show appropriate ion ratio agreement, if all three ions meet this exact mass match criterion. When determining diagnostic ions, any isotopomer of a (pseudo)molecular ion may be considered diagnostic if it meets this exact mass match criterion. One additional adduct ion, beyond the pseudomolecular ion, may also be considered diagnostic if it meets this exact mass match criterion.

11.5 Matching to Library Spectra

While mass spectral libraries (either commercial compendia or collections generated in-house) can be invaluable tools in helping to direct examinations and suggest possible targets for further investigation, an examiner should remain aware of the limitations of these libraries. Most commercial libraries do not clearly indicate the instrumentation the spectra were acquired on, or at what level of sample loading. In-house library data may have been acquired on the same instrumentation used to obtain a given unknown spectrum, but it is very difficult to ensure that long-term drift in instrument performance has not compromised the reproducibility of those library spectra.

Despite these limitations, there may arise rare instances in which it is necessary to compare an unknown spectrum to a library entry, for example if a standard of the substance in question cannot be readily obtained, or for purposes of screening to direct further investigation. In cases where such matching is attempted, all criteria for the appropriate type of mass spectrometry, given above, should still be observed, with one significant change. In these instances, ion abundances for the determination of ion ratios will be measured as the intensity of the ion in the spectrum, rather than as the integrated area of a reconstructed ion trace. For the unknown spectrum, all criteria regarding averaging and background subtraction from section 10.1 should still be observed.

12 Calculations

$IR_x = (A_x/A_b)x100\%$, where:

IR_x = the ion ratio for ion x
A_x = the integrated area of the reconstructed ion trace for ion x
A_b = the integrated area of the reconstructed ion trace for the base peak ion
(Ion abundances from background subtracted mass spectra may be substituted for integrated areas under certain circumstances detailed in section 9.5.)

13 Uncertainty of Measurement

Not applicable.

14 Limitations

This procedure, while extensive, is not intended to be exhaustive, and situations will arise in which their blind application could lead to inappropriate conclusions. No set of rules can ever replace the good judgment of a trained and experienced examiner. The mere fact that an unknown mass spectrum matches well to the spectrum of a known standard will rarely, *by itself*, be sufficient grounds to claim the presence of that compound in the questioned sample. All of the analytical data for the samples in question should be considered when drawing such final conclusions. Similarly, the fact that an unknown mass spectrum fails to match that of a known standard generally will not, *by itself*, constitute grounds for concluding that the compound is not present in the questioned specimen.

This protocol does not excuse an examiner from exercising care in the acquisition of mass spectral data. It should be remembered that poor practices in the acquisition of mass spectra will yield data that is useless at best, and misleading at worst. Samples that show evidence of severe chromatographic overload should generally be diluted and reinjected in order to obtain reliable mass spectra. Instrument calibration is also critical for obtaining useful mass spectral data, and it is incumbent upon any examiner to run appropriate test samples to demonstrate proper instrument function.

15 Safety

Not applicable.

16 References

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Instrument Operations and Support Subunit SOP Manual.

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Approval

Chemistry Unit Chief: Marc A. LeBeau Date: 6/9/06

QA Approval

Quality Manager: Robert B. Stacey Date: 6/15/2006

Issuance

Tox Subunit Manager : Madeline A. Montgomery Date: 6/16/06

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Ex 446
(261)

Appendix 1: Mass Spectra Key Points

1. Averaging/Background Subtraction of Mass Spectra: (* = subtracted spectrum)
   a. <6 scans averaged for sample (N)
   b. <2N+1 scans for background
   c. background outside area integrated for ion ratios

2. Significant Ions: Ion signal > 15% (10% for MS/MS) of most intense ion signal (*)

3. Diagnostic Ions (determine from mass spectra of known standard):
   a. Characteristic of chemical compounds chemical structure
   b. Diagnostic criteria (not well defined):
      (Pseudo)molecular ion is diagnostic if intensity > 5% base peak (*)
   c. Non-diagnostic ions (generally exclude):
      Adduct ions except pseudomolecular ion
      Isotopomers except chlorine and bromine isotopomers
      Ions 2^{ndry} to a derivatizing reagent (e.g. m/z 73 TMS deriv)

4. Base Peak (determined from mass spectra of known standard):
   a. Most intense signal for diagnostic ion (*)
   b. Base peak standard is base peak unknown (Exception: can use second most intense diagnostic ion if base peak has an uncorrectable chemical interference)

5. Method for calculating relative ion intensities:
   a. Determine ion abundances = integrate RIC's / EIC's for each diagnostic ion
   b. Integration - comparable stop & start points
   c. Ion ratios (expressed as %) = each ion trace area/base peak ion area
   d. Spectral peak height may be substituted for integrated area if the RIC data in non-integrable.

6. Decision Criteria: Unknown spectra matched against known spectra.

6.1 Full Scan Mass Spectra
   a. Every significant ion in known spectra present in unknown and vice-versa
   b. Relative ion intensities (EI)
      Known Ion ratio > 50% - unknown 10% absolute
      Known Ion ratio 25-50% - unknown 20% relative
      Known Ion ratio < 25% - unknown 5% absolute
   c. Relative ion intensities (CI, ESI, APCI)
      Known Ion ratio > 60% - unknown 15% absolute
      Known Ion ratio 40-60% - unknown 25% relative
      Known Ion ratio < 40% - unknown 10% absolute

Ex 446
(262)

6.2 SIM Mass Spectra
- a. Four diagnostic ions (three for compounds <80 AMU), all should be significant
- b. Base peak one of the chosen ions
- c. (Pseudo)molecular ion RI > 5%
- d. Meet relative ion intensities

6.3 Tandem MS (ion ratio dependant on analyte load, less stable)
- a. All ions of reasonable intensity are diagnostic (Exception 2^{ndry} loss of adduct)

6.3.1 Product Ion (Daughter Ion) Experiment
- a. Select precursor ion (parent ion):
  (Pseudo)molecular ion or one of its isotopomers
- b. Product spectra:
  Precursor ion observed with ion ratio >5% OR full scan show no ions within 1.5 AMU of precursor ion with > 3x intensity of the precursor ion
- c. Comparing product spectra (Standard to unknown = std to unk)
  Significant ions present both std and unk
  If > 3 diag ions –need only ratio 3 diag (2 ratios)
  Ion ratios:
    Known > 40% - unknown 25% relative
    Known < 40% - unknown 10% absolute
  Exclude ion ratios if:
    Ion ratio known < 5%
    Signal-to-noise RIC unknown < 3
    Chemical interference in ion

6.3.2 Precursor ion and Neutral loss – must match stds with unknowns

6.3.3 Selected Reaction Monitoring
- a. 3 diagnostic ion transitions
- b. Share common precursor ion, except isotopomer clusters
- c. Ion ratios:
  Known > 40% - unknown 25% relative
  Known < 40% - unknown 10% absolute

6.4 Exact Mass (Accurate Mass)
- a. m/z < 600: Std to Unk measured masses within 3 mAMU
- b. m/z > 600: Std to Unk measured masses within 5ppm
- c. SIM: only 3 ions need monitoring, in correct ratios
- d. If above criteria is met:
  Can use (pseudo)molecular ion if < 5% RI
  Can use any isotopomer of (pseudo)molecular ion

Ex 446
(263)

Can use up to one additional adduct ion, beyond pseudomolecular ion

Appendix 2: Glossary of Terms

Adduct Ion – Any ion to which another chemical entity has been attached by a means other than covalent bonding.

Base Peak – The most intense or abundant diagnostic ion in the mass spectrum of a substance.

Diagnostic Ion – Any ion observed in the mass spectrum of a substance that is characteristic of the chemical structure of that substance.

Ion Ratio – The relative abundance or intensity of two ion signals in a mass spectrum.

Isotopomers – Two or more chemical species that differ only in isotopic composition. For example, CH_3OH and CD_3OH are two isotopomers of methanol.

Molecular Ion – A charged intact molecular species, with charge acquired solely through the gain or loss of electrons. Normally denoted as M^+ or M^- for singly charged species.

Precursor Ion – In tandem mass spectrometry, the ion selected for fragmentation. Often referred to as a "parent ion".

Product Ion – In tandem mass spectrometry, an ion resulting from the fragmentation of another ion. Often referred to as a "daughter ion".

Pseudomolecular Ion – A charged molecule in which charge has been acquired through adduction of an ion or through loss of a moiety able to dissociate in solution. Examples include $(M+H)^+$, $(M-H)^-$, $(M+Na)^+$, and $(M+NH_4)^+$.

Reconstructed Ion Trace – A display of the abundance or intensity of a single ion signal as a function of time during an analysis. Often also called an "extracted ion chromatogram" (EIC) or "reconstructed ion chromatogram" (RIC).

Significant Ion – Any ion in the mass spectrum of a substance present above a specified intensity or abundance.

VALIDATION

Ex 446
(265)

Chemistry Unit
Method Validation Review Form

Procedure Name: ANALYSIS OF EDTA IN DRIED BLOODSTAINS

Scope: ALLOWS FOR SCREENING AND CONFIRMATION OF EDTA IN SUSPECTED BLOOD STAINS.

Intended Use (✓ as appropriate):

- ☐ Measurement of Physical/Chemical Property
- ☑ Establishment of Presence/Absence of Specific Analyte(s)/Class(es)
- ☐ Quantitation of Specified Analyte(s)

✓	Parameters Evaluated:	Date(s):	Researcher(s):	Reviewer:
☑	*Selectivity:* Analysis of at least 10 sources of blank matrix + 2 zero samples (blank matrix spiked with internal standard). Analysis of samples spiked with other compounds expected to be in real samples.	2/9/07, 2/12/07	BREWER, LE BEAU	M.M
☐	*Calibration Model (Linearity):* Analysis of at least 5 non-zero calibrators at a minimum of 5 replicates per level.			
☐	*Bias, Repeatability, and Intermediate Precision:* QC samples at low and high conc in calibration range. Analysis of duplicates of each conc in at least 5 runs.			
☑	*Limit of Detection:* See Procedure for details.	2/12/07	BREWER, LE BEAU	M.M
☐	*Lower Limit of Quantitation:* See Procedure for details.			
☑	*Matrix Effects:* For LC-MS(-MS) procedures only. Analysis of 5 neat standards and 5 blank extracts spiked with analyte after extraction.	2/12/07, 2/13/07	BREWER, LE BEAU	MM

Additional Comments:

This method has been appropriately validated for its intended use (as specified above). The method is considered fit for the above use.

Unit Chief Approval: _____ Date: 2/16/07

Ex 446
(266)

METHOD

Ex 446
(267)

Instrument Method: EDTA_Pos_Swabs.meth

LCQ Instrument Method

Creator: Administrator
Last modified: 2/12/07 by Administrator

MS Run Time (min): 10.00

Divert Valve: not used during run

MS Detector Settings:

Segment 1 Information

Duration (min): 10.00
Number of Scan Events: 1
Tune Method: EDTA_Pos_CID

Scan Event Details:
1: Pos ·(293.0)->o(125.0-315.0)
MS/MS: CE 15.0% IsoW 1.0

Ex 446
(268)

Instrument Method: EDTA_Pos_Swabs.meth

Waters 2690 LC System

Injector parameters:
Syringe draw rate (µl/sec):2.50
Injection volume (µl):5

Pump settings:
Solvent A:5:95 ACN:Water + 0.06% NH4OH
Solvent B:B
Solvent C:C
Solvent D:D
Min pressure (PSI):0
Max pressure (PSI):5000
Chart output:Pressure
Chart output:Normal

Gradient program:

Time(min)	Flow(ml/min)	A(%)	B(%)	C(%)	D(%)	Curve
0.00	0.30	100.0	0.0	0.0	0.0	Linear - 6
3.00	0.30	100.0	0.0	0.0	0.0	Linear - 6

Timed events:
Initial states:
Switch 1:No change
Switch 2:No change
Switch 3:No change
Switch 4:No change

Time(min)	Event	Action	Parameter
0.00	Switch1	No change	

Ex 446
(269)

Instrument Method: EDTA_Pos_Swabs.meth

LCQ Instrument Method

Creator: Administrator
Last modified: 2/9/07 by Administrator

MS Run Time (min): 10.00

Divert Valve: not used during run

MS Detector Settings:

Segment 1 Information

Duration (min): 10.00
Number of Scan Events: 2
Tune Method: EDTA_Pos_CID

Scan Event Details:
1: Pos ·(293.0)->o(125.0-315.0)
MS/MS: CE 15.0% IsoW 1.0

2: Pos ·(305.0)->o(125.0-315.0)
MS/MS: CE 15.0% IsoW 1.0

(OLD METHOD) JDB

Ex 446
(270)

Instrument Method: EDTA_Pos_Swabs.meth

Waters 2690 LC System

Injector parameters:
Syringe draw rate (µl/sec):2.50
Injection volume (µl):5

Pump settings:
Solvent A:5:95 ACN:Water + 0.06% NH4OH
Solvent B:B
Solvent C:C
Solvent D:D
Min pressure (PSI):0
Max pressure (PSI):5000
Chart output:Pressure
Chart output:Normal

Gradient program:

Time(min)	Flow(ml/min)	A(%)	B(%)	C(%)	D(%)	Curve
0.00	0.30	100.0	0.0	0.0	0.0	Linear - 6
3.00	0.30	100.0	0.0	0.0	0.0	Linear - 6

Timed events:
Initial states:
Switch 1:No change
Switch 2:No change
Switch 3:No change
Switch 4:No change

Time(min)	Event	Action	Parameter
0.00	Switch1	No change	

Ex 446
(271)

METHOD

Ex 446
(272)

Instrument Method: EDTA_Neg_Swabs.meth

LCQ Instrument Method

Creator: Administrator
Last modified: 2/9/07 by Administrator

MS Run Time (min): 10.00

Divert Valve: not used during run

MS Detector Settings:

Segment 1 Information

Duration (min): 10.00
Number of Scan Events: 4
Tune Method: EDTA_NEG

Scan Event Details:
1: Neg ·(291.2)->oS(246.5-247.5 272.5-273.5)
MS/MS: CE 15.0% IsoW 1.0

2: Neg ·(303.2)->oS(282.8-284.3)
MS/MS: CE 15.0% IsoW 1.0

3: Neg ·(344.2)->oS(299.5-300.5 325.5-326.5)
MS/MS: CE 15.0% IsoW 1.0

4: Neg ·(356.0)->oS(311.5-312.5)
MS/MS: CE 15.0% IsoW 1.0

Ex 446
(273)

Instrument Method: EDTA_Neg_Swabs.meth

Waters 2690 LC System

Injector parameters:
Syringe draw rate (µl/sec):2.50
Injection volume (µl):5

Pump settings:
Solvent A:80:20 ACN:Water + 0.03% NH4OH
Solvent B:B
Solvent C:C
Solvent D:D
Min pressure (PSI):0
Max pressure (PSI):5000
Chart output:Pressure
Chart output:Normal

Gradient program:

Time(min)	Flow(ml/min)	A(%)	B(%)	C(%)	D(%)	Curve
0.00	0.30	100.0	0.0	0.0	0.0	Linear - 6
3.00	0.30	100.0	0.0	0.0	0.0	Linear - 6

Timed events:
Initial states:
Switch 1:No change
Switch 2:No change
Switch 3:No change
Switch 4:No change

Time(min)	Event	Action	Parameter
0.00	Switch1	No change	

Ex 446
(274)

SELECTIVITY

Ex 446
(275)

Sequence---select_pos.sld [Open]

Sample Name: Swab A, Case 1, Grey

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	SEL01	1	C:\Xcalibur\Data\EDTA\Brewer\SEL_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Swab A, Case 2, Red

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	SEL02	02	C:\Xcalibur\Data\EDTA\Brewer\SEL_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			2	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JDB
2/9/07

Ex 446
(276)

Sequence---select_pos.sld [Open]

Sample Name: Swab B, Case 2, Grey

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	SEL03	03	C:\Xcalibur\Data\EDTA\Brewer\SEL_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			3	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Swab C, Case 3-1, Grey

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	SEL04	04	C:\Xcalibur\Data\EDTA\Brewer\SEL_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			4	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(277)

JOB
2/9/07

Sequence---select_pos.sld [Open]

Sample Name: Swab C, Case 3-2, Red
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	SEL05	05	C:\Xcalibur\Data\EDTA\Brewer\SEL_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			5	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Swab C, Case 3-3, Grey
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	SEL06	06	C:\Xcalibur\Data\EDTA\Brewer\SEL_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			6	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(278)

JOP
2/9/07

Sequence---select_pos.sld [Open]

Sample Name: Swab D, Case 3-4, Grey

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	SEL07	07	C:\Xcalibur\Data\EDTA\Brewer\SEL_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			7	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Swab D, Case 4-1, Red

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	SEL08	08	C:\Xcalibur\Data\EDTA\Brewer\SEL_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			8	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JDP
2/9/07

Ex 446
(279)

Sequence---select_pos.sld [Open]

Sample Name: Swab D, Case 5, Red

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	SEL09	09	C:\Xcalibur\Data\EDTA\Brewer\SEL_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			9	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Swab A, MAL, Yellow

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	SEL10	10	C:\Xcalibur\Data\EDTA\Brewer\SEL_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			10	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(280)

JDB
2/9/07

Sequence---select_pos.sld [Open]

Sample Name: Swab A, MAL, Yellow, d12 spiked

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	SEL11	11	C:\Xcalibur\Data\EDTA\Brewer\SEL_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Swab D, MAL, Yellow, d12 spiked

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	SEL12	12	C:\Xcalibur\Data\EDTA\Brewer\SEL_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			12	5.0		0.000

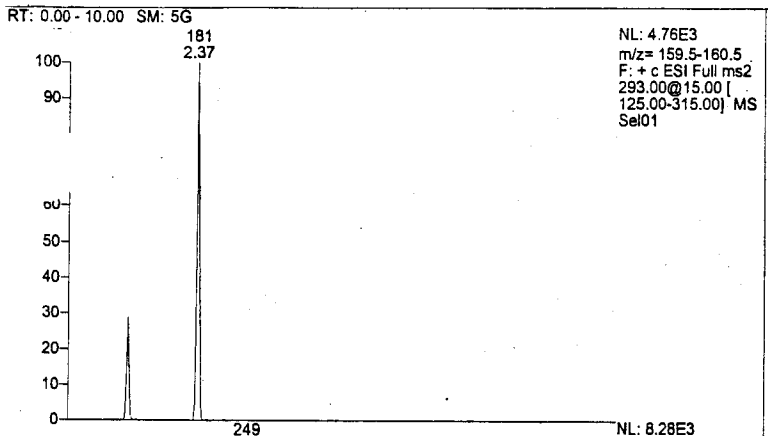
Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

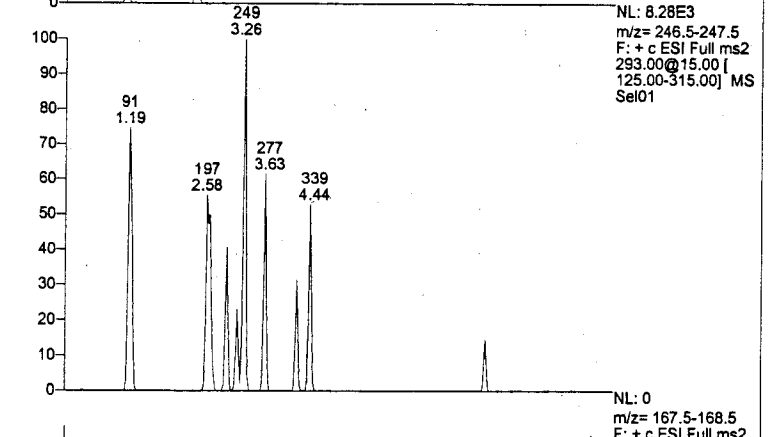
****

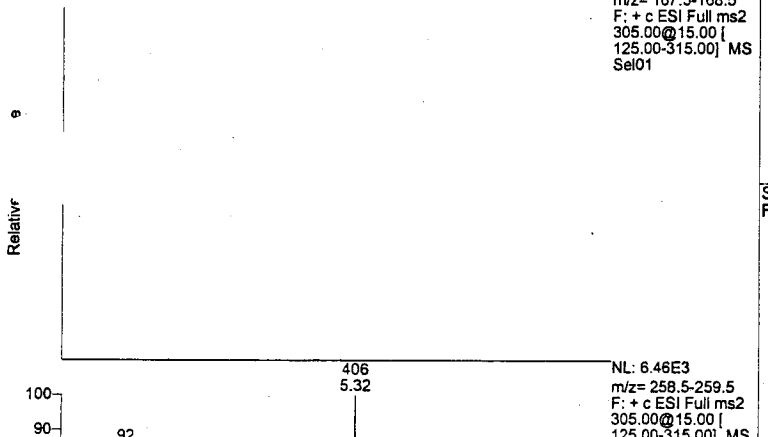
Ex 446
(281)

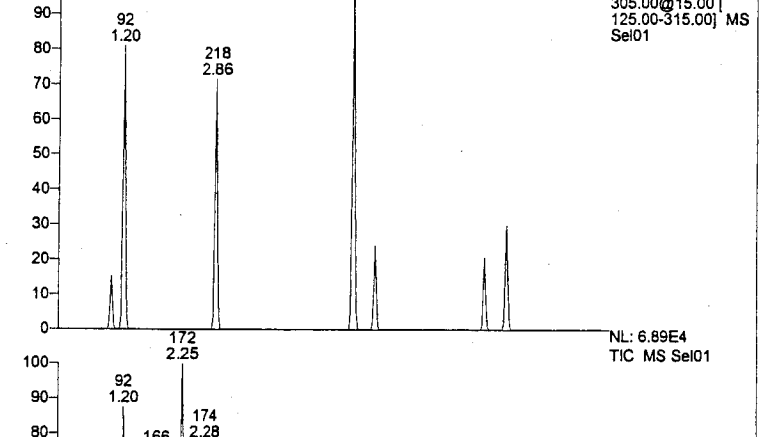
JQB
2/9/07

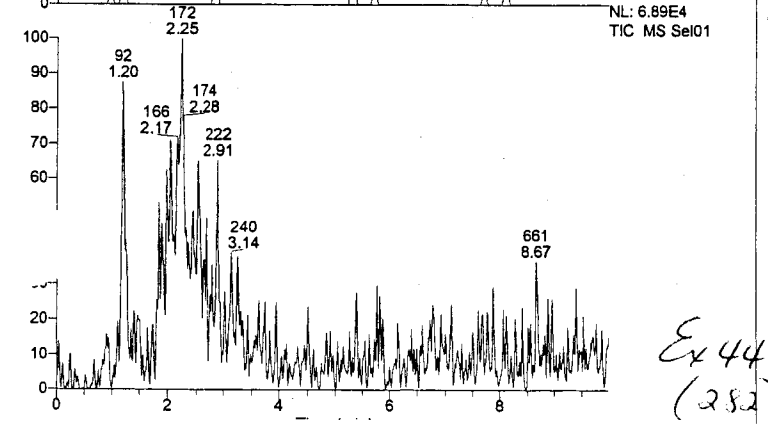
C:\Xcalibur\...\SEL_Positive\Sel01 02/09/07 10:57:05 Swab A, Case 1, Grey JMf

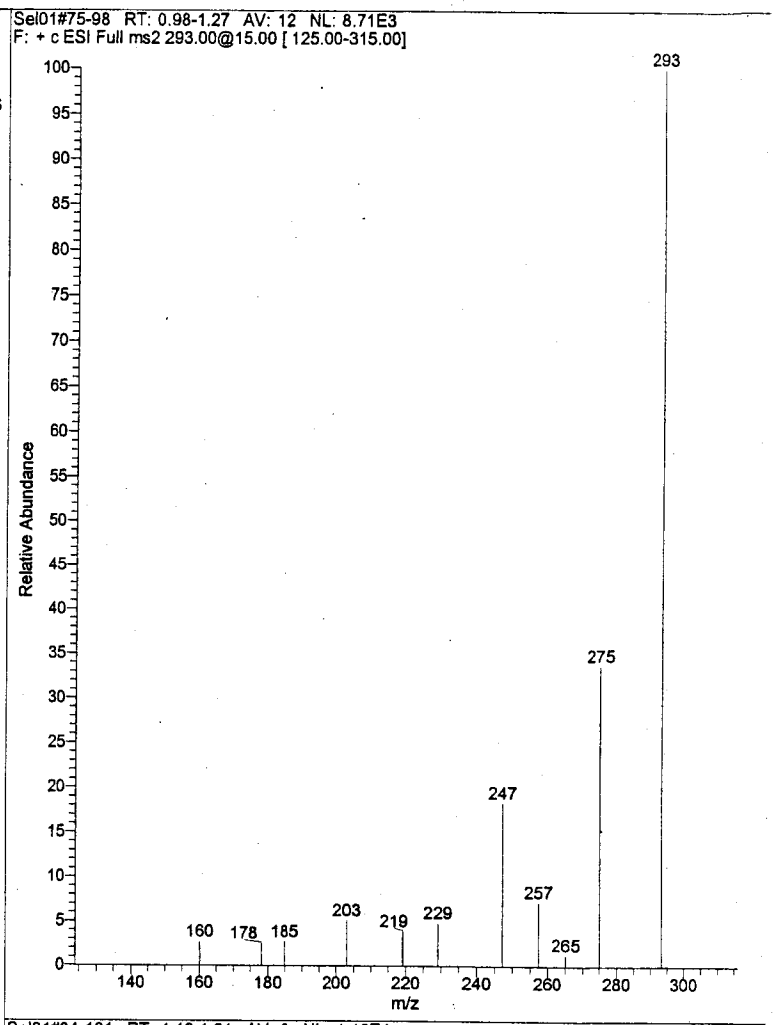


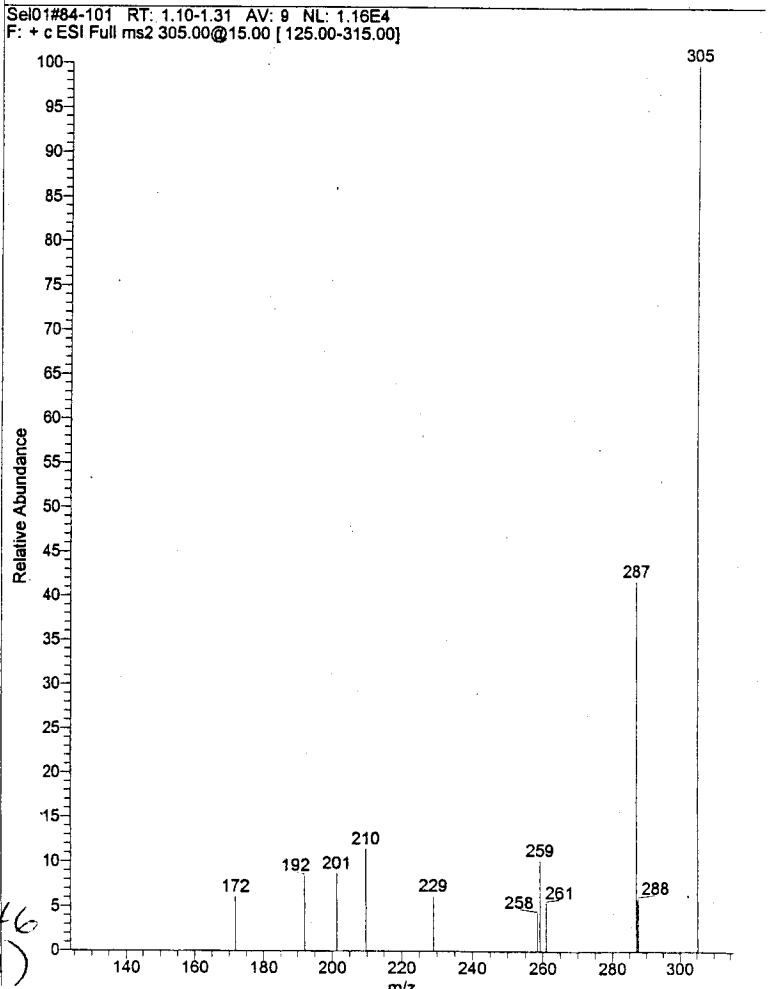








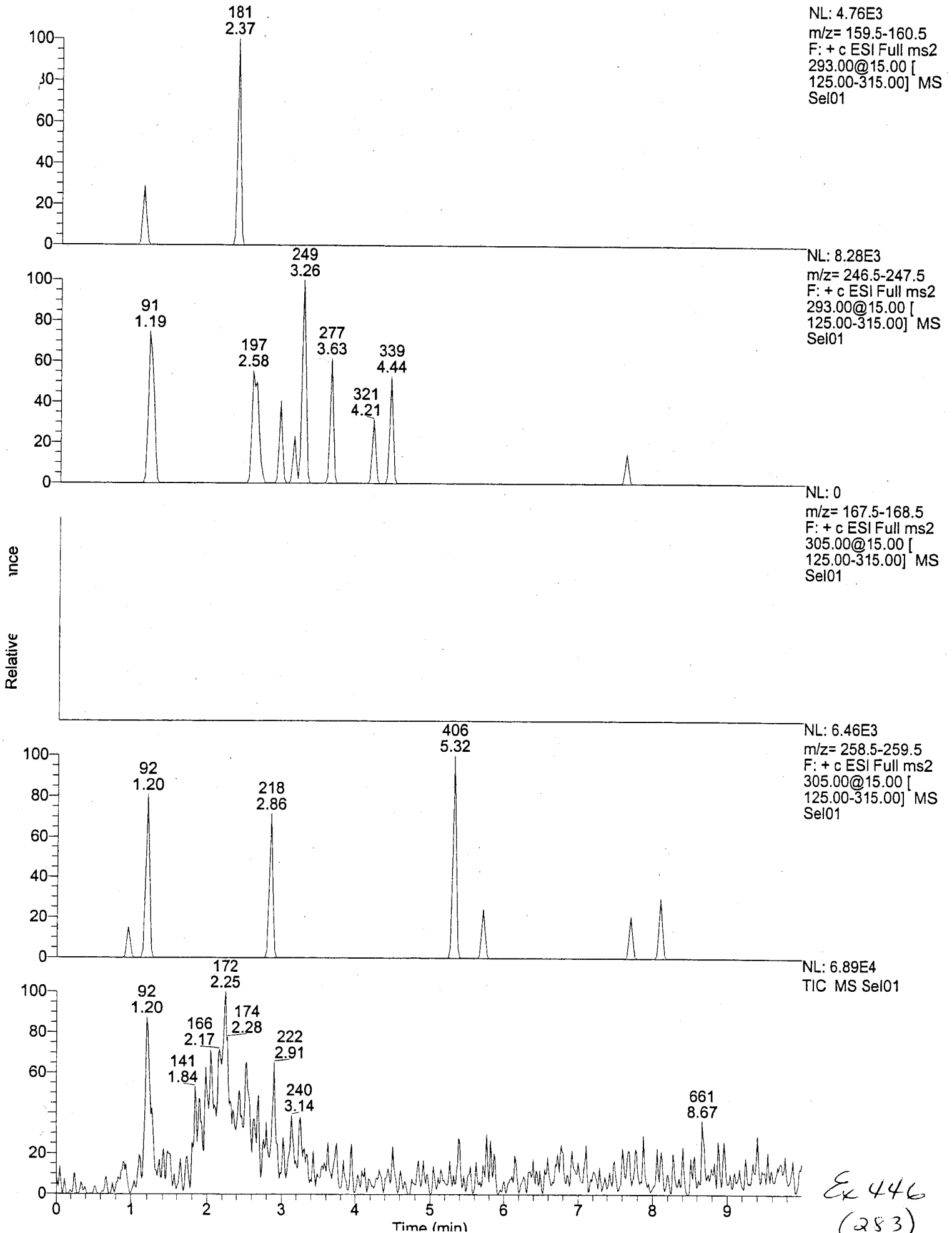




Ex 446
(282)

C:\Xcalibur\...\SEL_Positive Sel01 02/09/07 10:57:05 Swab A, Case 1, Grey JOB

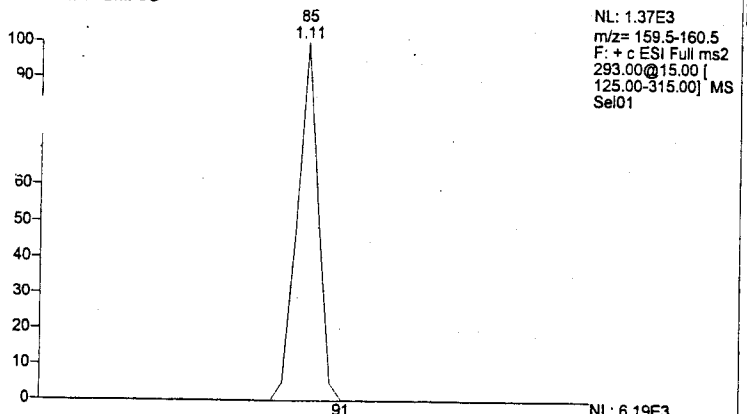
RT: 0.00 - 10.00 SM: 5G

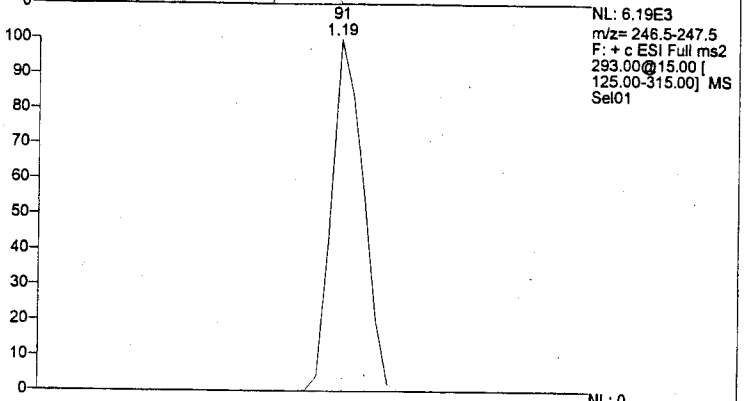


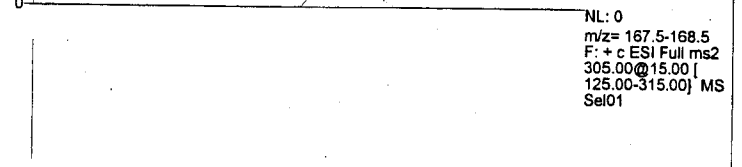
Ex 446
(283)

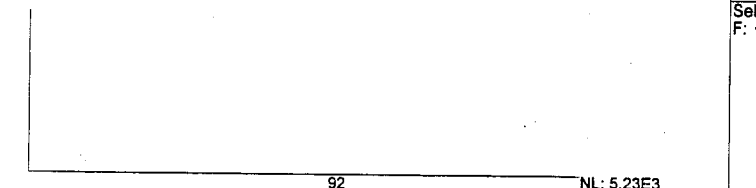
C:\Xcalibur\...\SEL_Positive\Sel01 02/09/07 10:57:05 Swab A, Case 1, Grey JOB

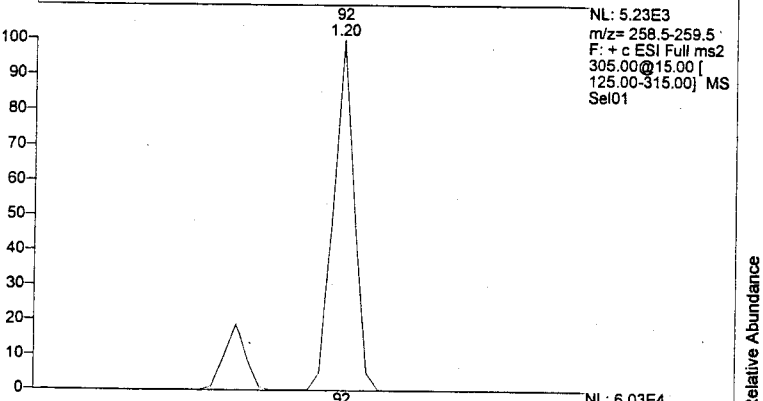
RT: 0.53 - 1.74 SM: 5G

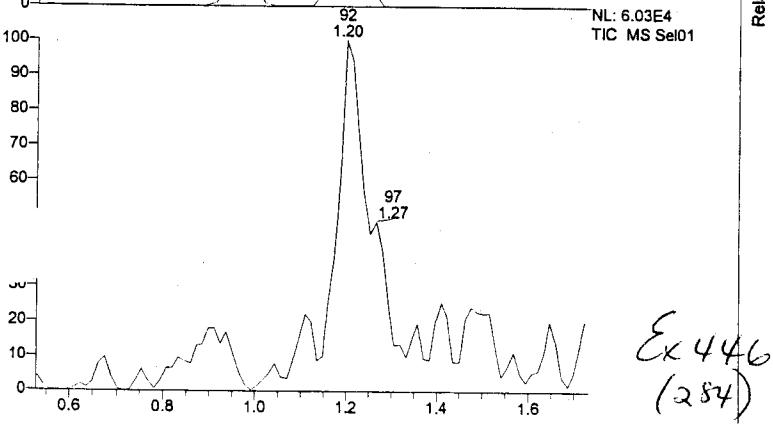


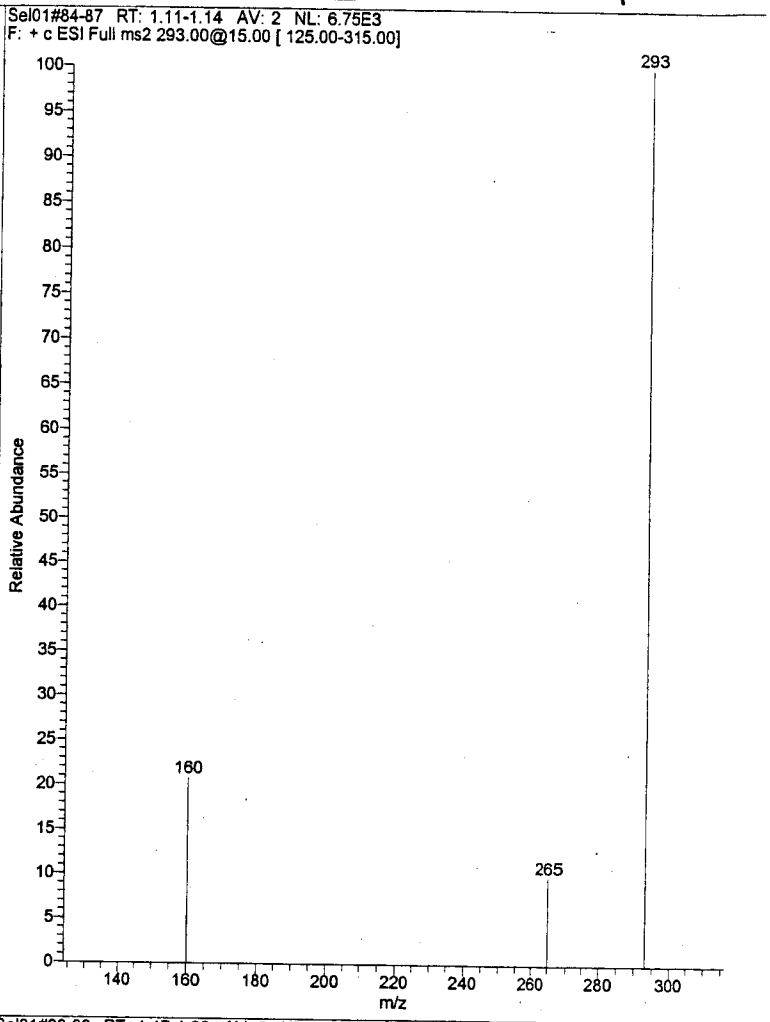


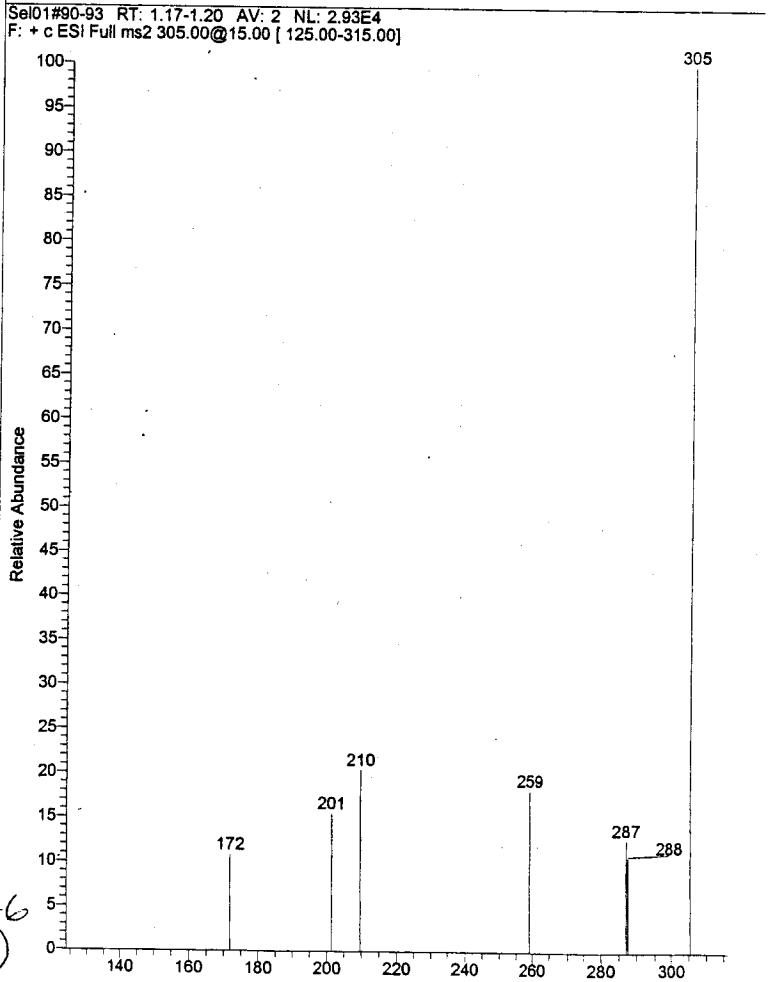








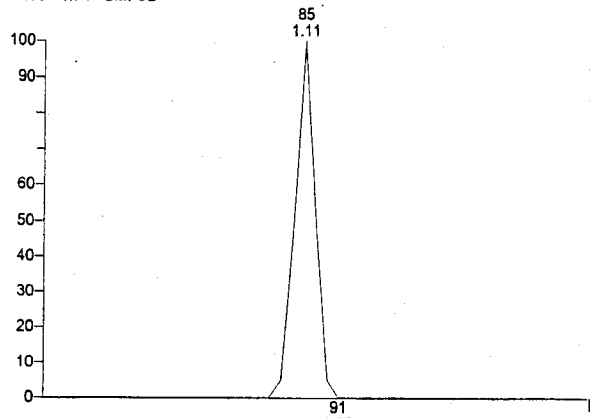


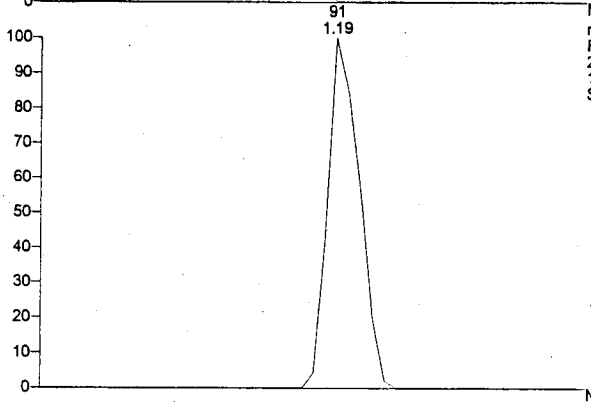


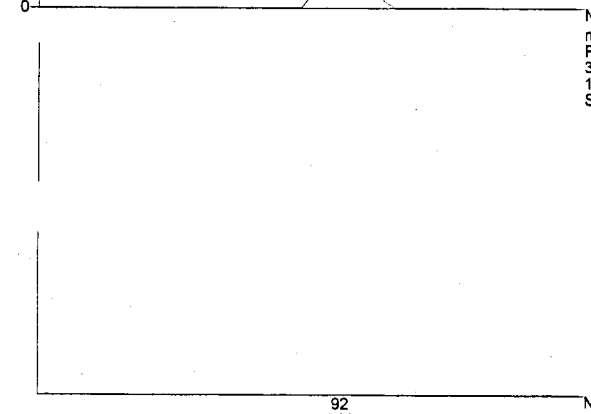
Ex 446 (284)

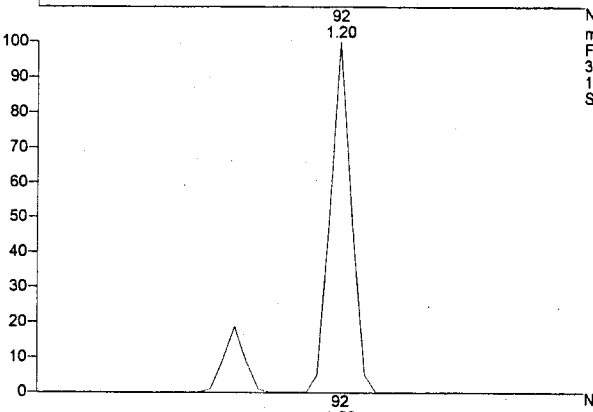
C:\Xcalibur\...\SEL_Positive\Sel01 02/09/07 10:57:05 Swab A, Case 1, Grey JDB

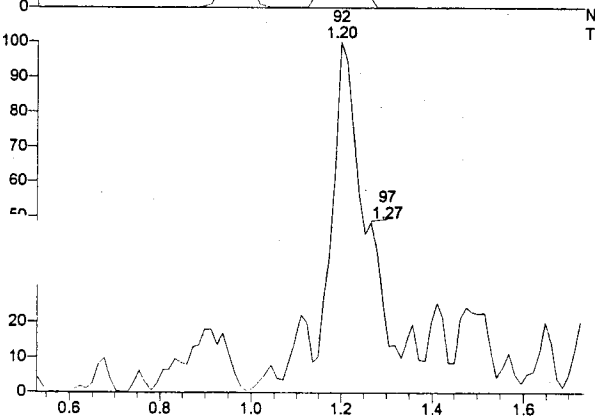
RT: 0.53 - 1.74 SM: 5G



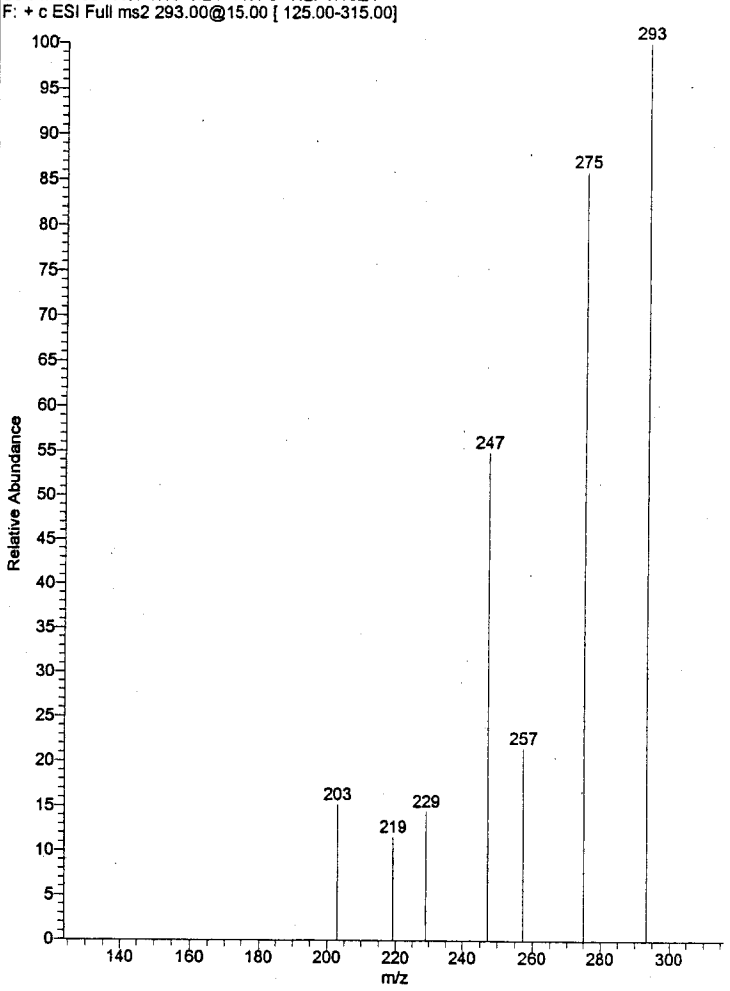




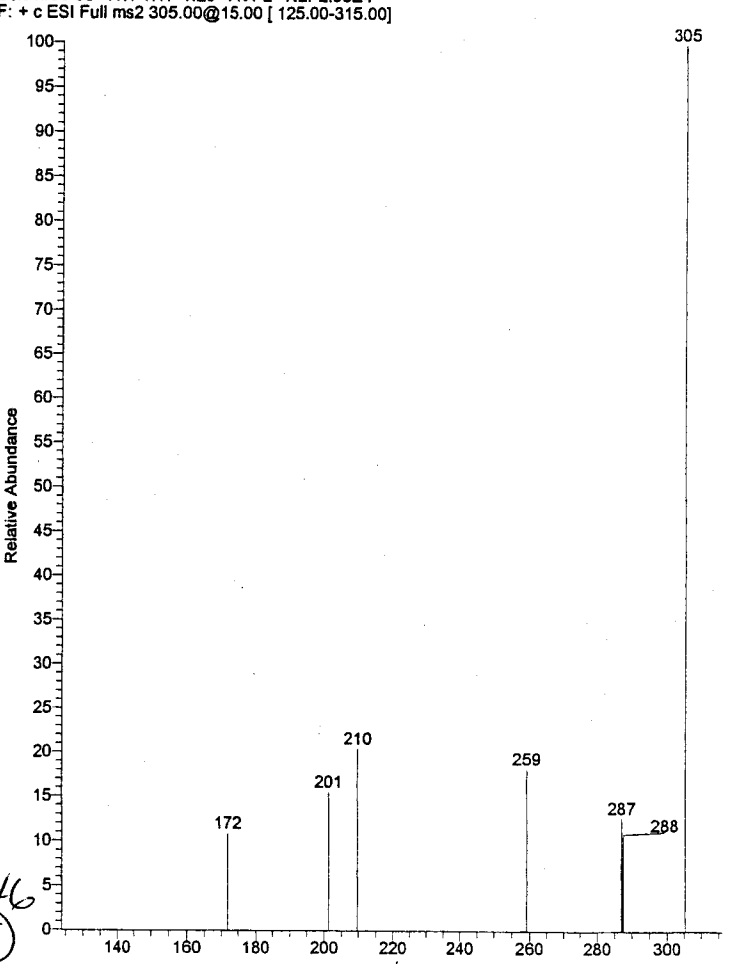




Sel01#90-95 RT: 1.19-1.24 AV: 3 NL: 1.16E4
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



Sel01#90-93 RT: 1.17-1.20 AV: 2 NL: 2.93E4
F: + c ESI Full ms2 305.00@15.00 [125.00-315.00]

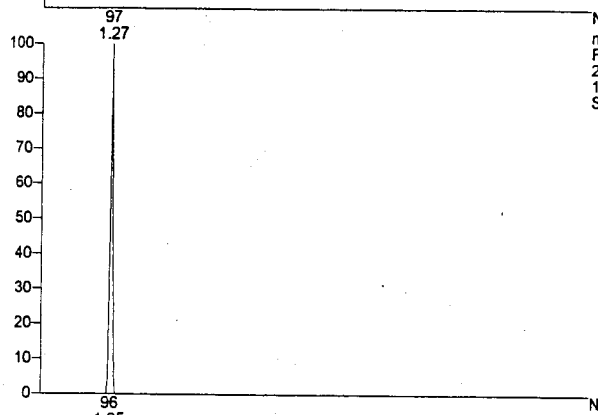


Ex 446
(285)

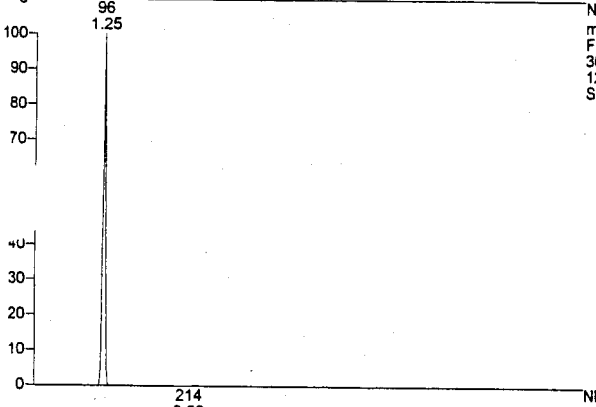
C:\Xcalibur\...\SEL_Positive\Sel02 02/09/07 11:08:17 Swab A, Case 2, Red JDB

RT: 0.00 - 10.00 SM: 5G

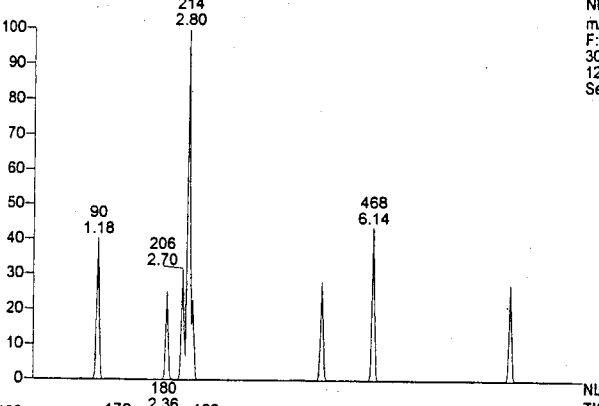
NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel02



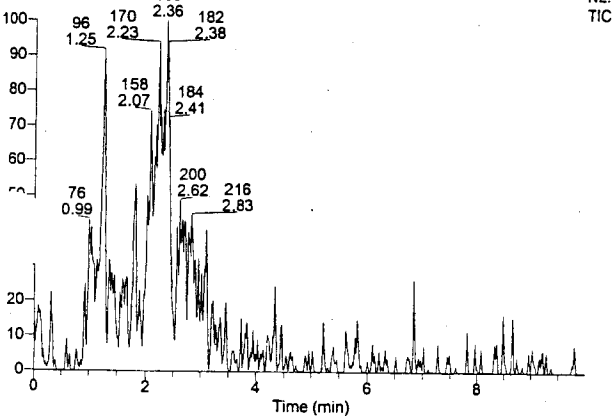
NL: 4.89E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel02



NL: 4.68E3
m/z= 167.5-168.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel02

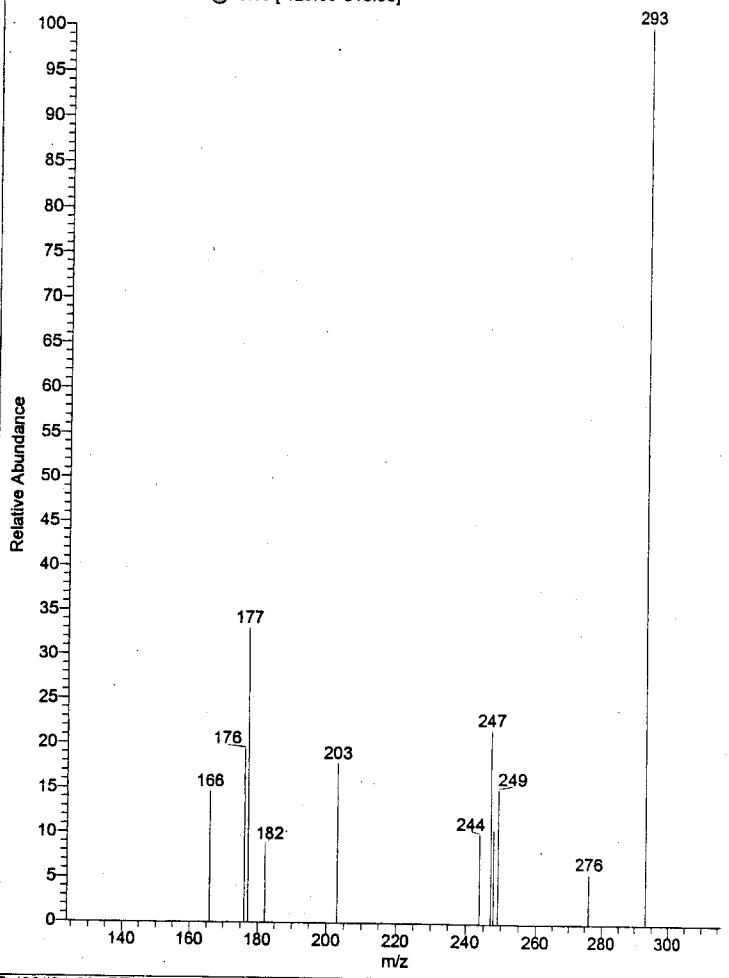


NL: 7.66E3
m/z= 258.5-259.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel02

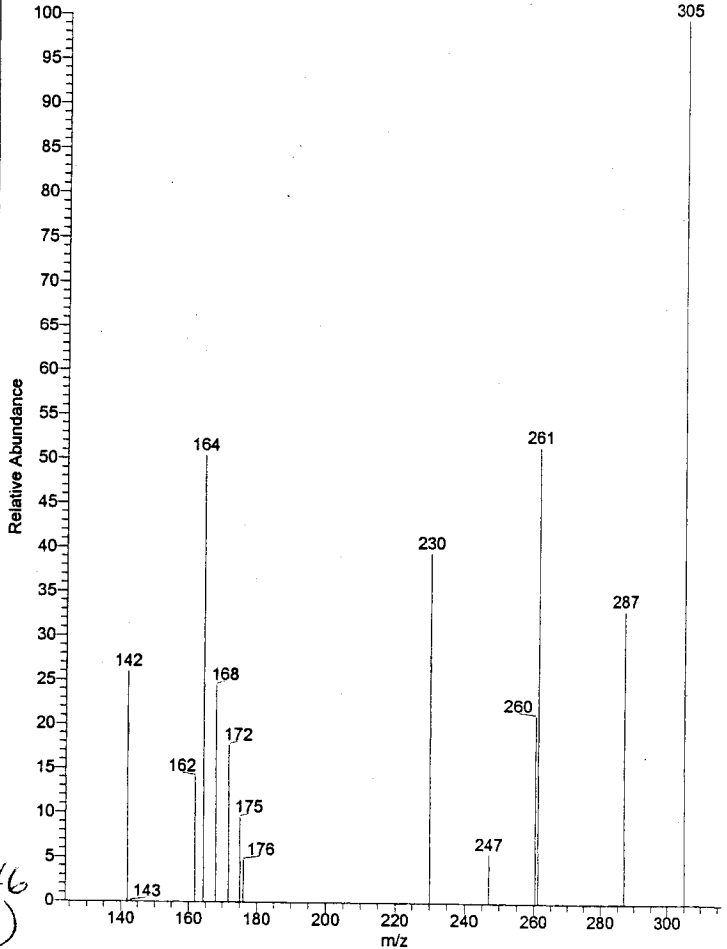


NL: 6.62E4
TIC MS Sel02

Sel02#95-99 RT: 1.24-1.29 AV: 3 NL: 1.54E4
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



Sel02#94-98 RT: 1.23-1.28 AV: 3 NL: 1.30E4
F: + c ESI Full ms2 305.00@15.00 [125.00-315.00]



Ex 446
(282)

C:\Xcalibur\...\SEL_Positive\Sel03 02/09/07 11:19:06 Swab B, Case 2, Grey JOB

RT: 0.00 - 10.00 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel03

NL: 0
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel03

NL: 0
m/z= 167.5-168.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel03

Relative

NL: 2.51E3
m/z= 258.5-259.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel03

538
7.06

NL: 2.99E4
TIC MS Sel03

158
2.07
160
2.10
184
2.41
148
1.94
142
1.86
90
1.18
265
3.48
543
7.13

Time (min)

Sel03#62-70 RT: 0.82-0.90 AV: 4 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

Relative Abundance

m/z

Sel03#82-85 RT: 1.07-1.10 AV: 2 NL: 2.89E3
F: + c ESI Full ms2 305.00@15.00 [125.00-315.00]

305

259

Relative Abundance

m/z

Ex 446
(287)

C:\Xcalibur\...\SEL_Positive\Sel04 02/09/07 11:29:58 Swab C, Case 3-1, Grey JDF

RT: 0.00 - 9.99 SM: 5G

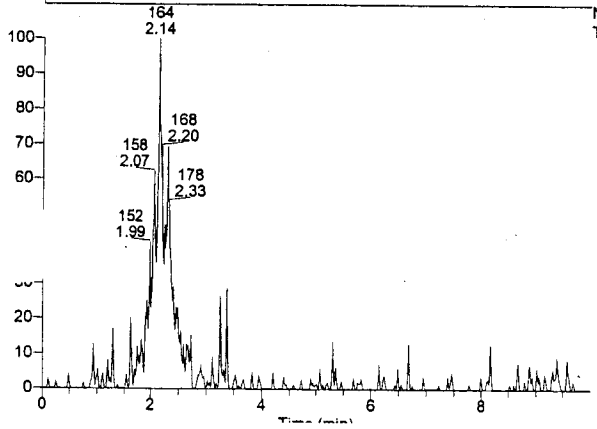
NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel04

NL: 0
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel04

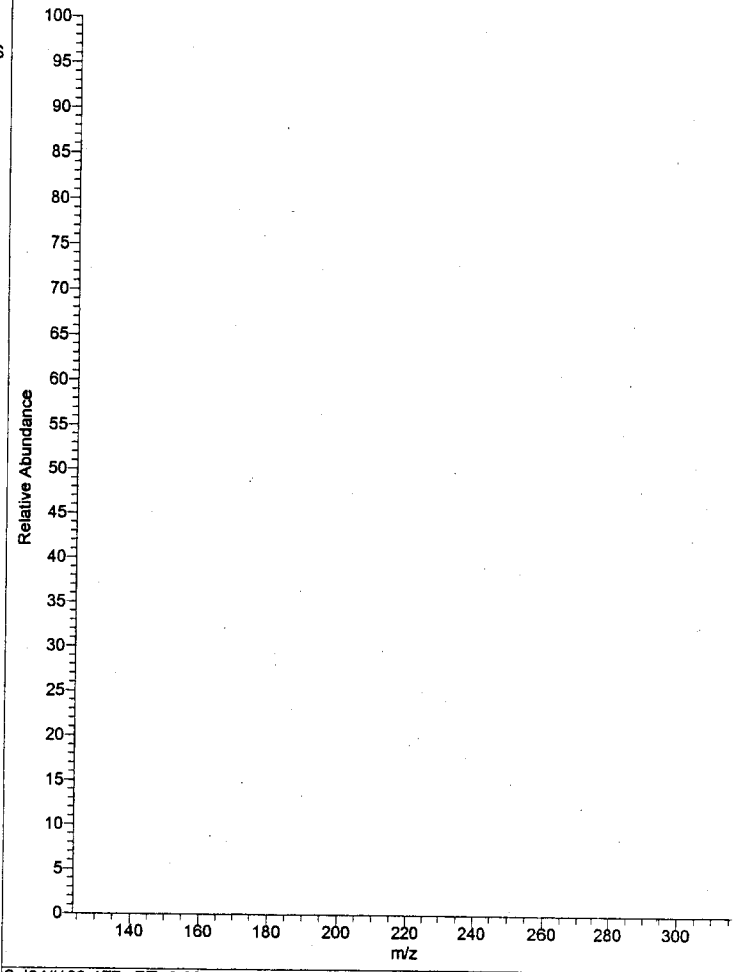
NL: 0
m/z= 167.5-168.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel04

NL: 0
m/z= 258.5-259.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel04

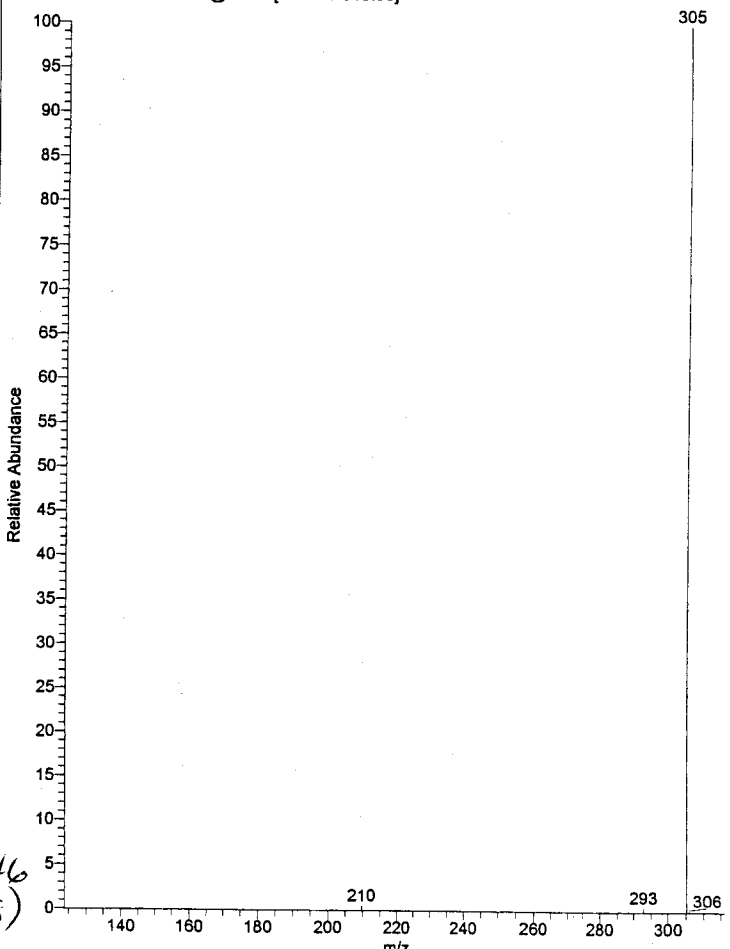
NL: 9.15E4
TIC MS Sel04



Sel04#62-70 RT: 0.82-0.90 AV: 4 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



Sel04#160-177 RT: 2.09-2.31 AV: 18 NL: 4.92E4
T: + c ESI Full ms2 305.00@15.00 [125.00-315.00]



E4446
(285)

C:\Xcalibur\...\SEL_Positive\Sel05 02/09/07 11:40:49 Swab C, Case 3-2, Red JDB

RT: 0.00 - 10.00 SM: 5G

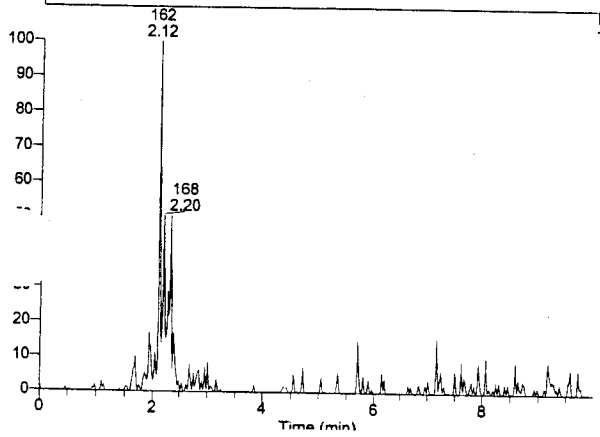
NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel05

NL: 0
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel05

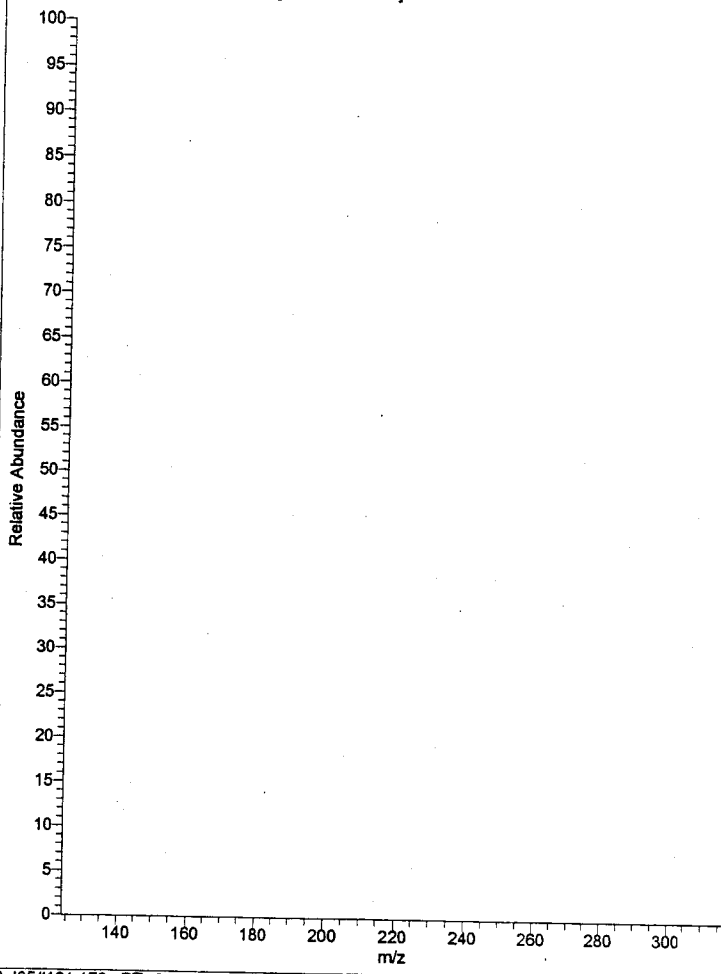
NL: 0
m/z= 167.5-168.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel05

NL: 0
m/z= 258.5-259.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel05

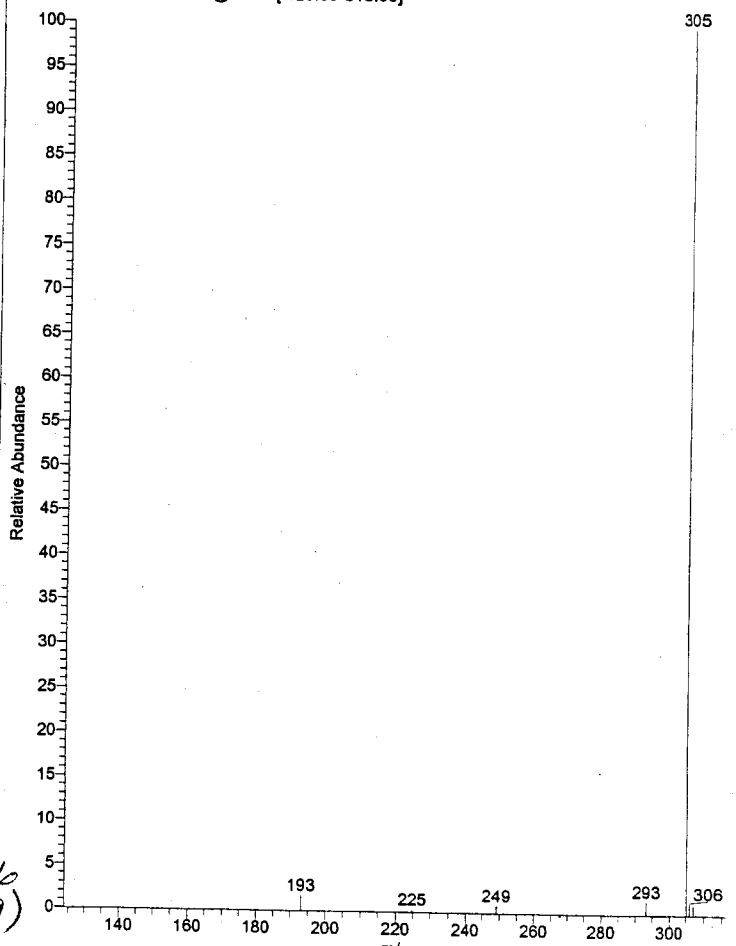
NL: 1.13E5
TIC MS Sel05



Sel05#62-70 RT: 0.82-0.90 AV: 4 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



Sel05#161-178 RT: 2.11-2.33 AV: 18 NL: 3.64E4
T: + c ESI Full ms2 305.00@15.00 [125.00-315.00]



Ex 446
(259)

C:\Xcalibur\...\SEL_Positive\Sel06 02/09/07 11:51:40 Swab C, Case 3-3, Grey JDB

RT: 0.00 - 10.00 SM: 5G

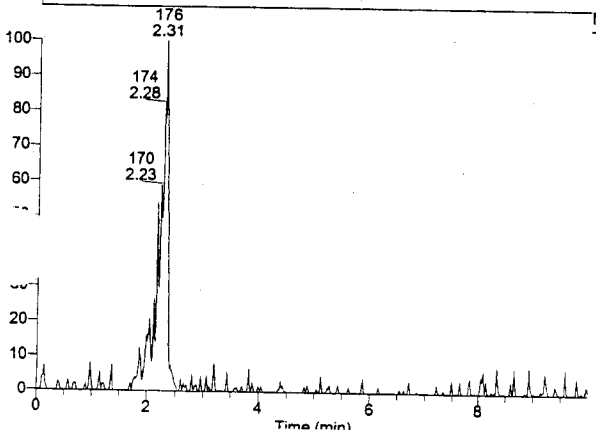
NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel06

NL: 0
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel06

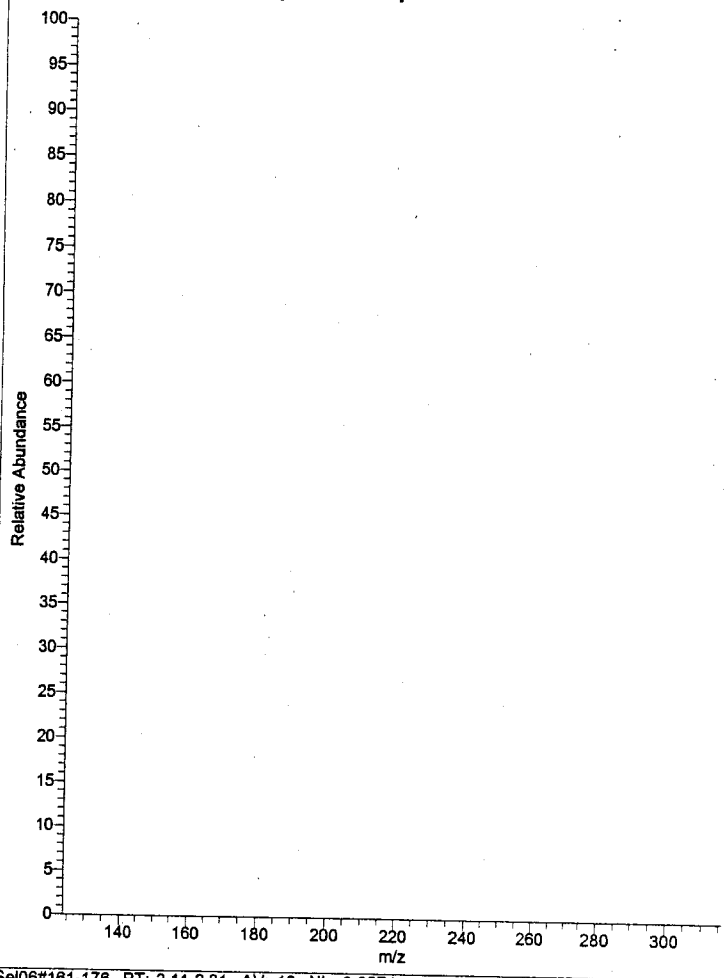
NL: 0
m/z= 167.5-168.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel06

NL: 0
m/z= 258.5-259.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel06

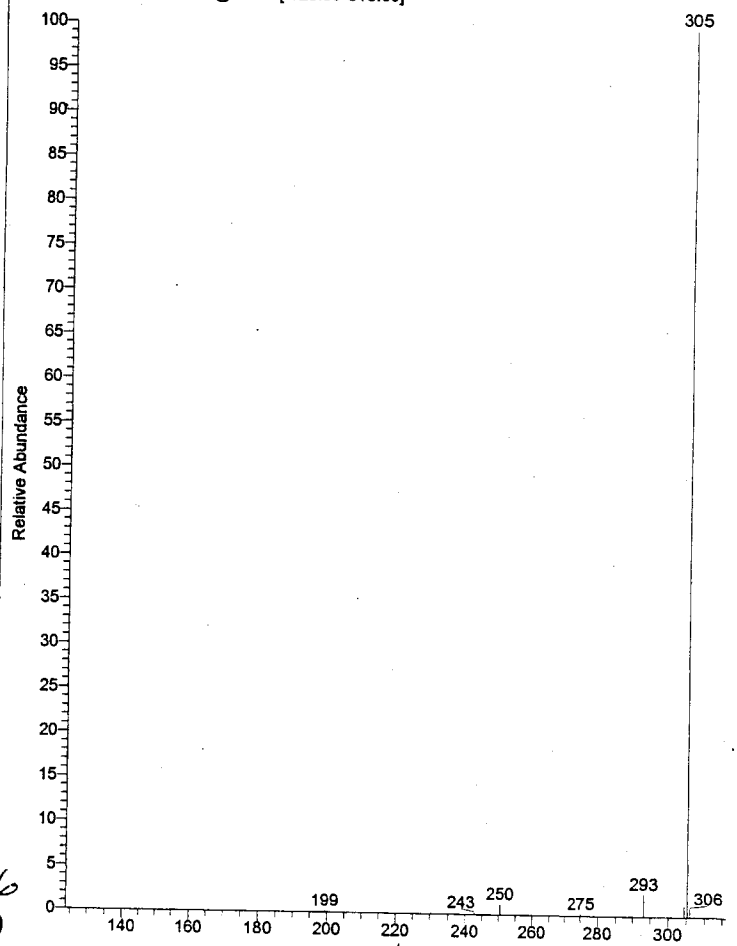
NL: 1.37E5
TIC MS Sel06



Sel06#62-70 RT: 0.82-0.90 AV: 4 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



Sel06#161-176 RT: 2.11-2.31 AV: 16 NL: 6.25E4
T: + c ESI Full ms2 305.00@15.00 [125.00-315.00]



Ex 446 (29c)

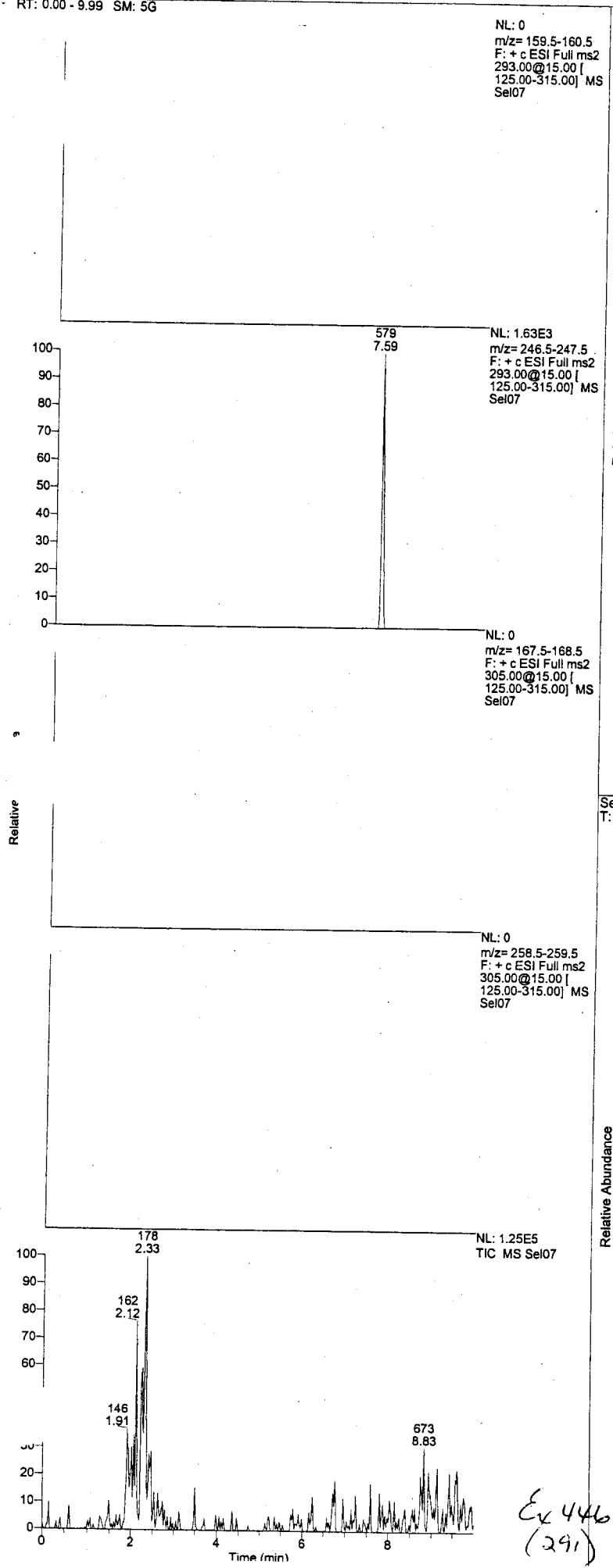
C:\Xcalibur\...\SEL_Positive\Sel07

02/09/07 12:02:31

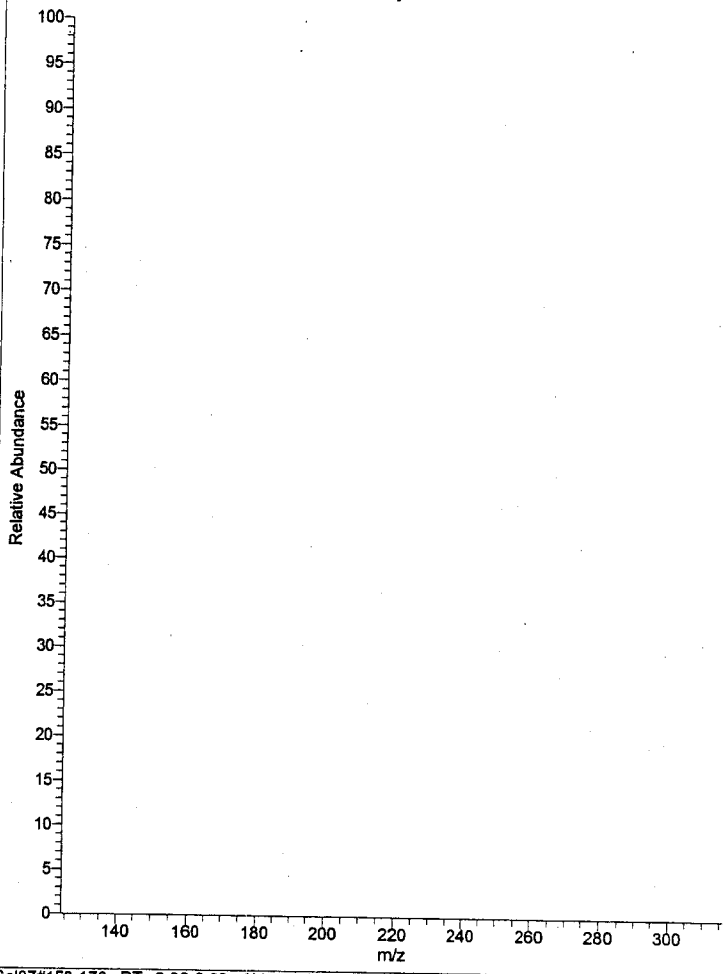
Swab D, Case 3-4, Grey

JDB

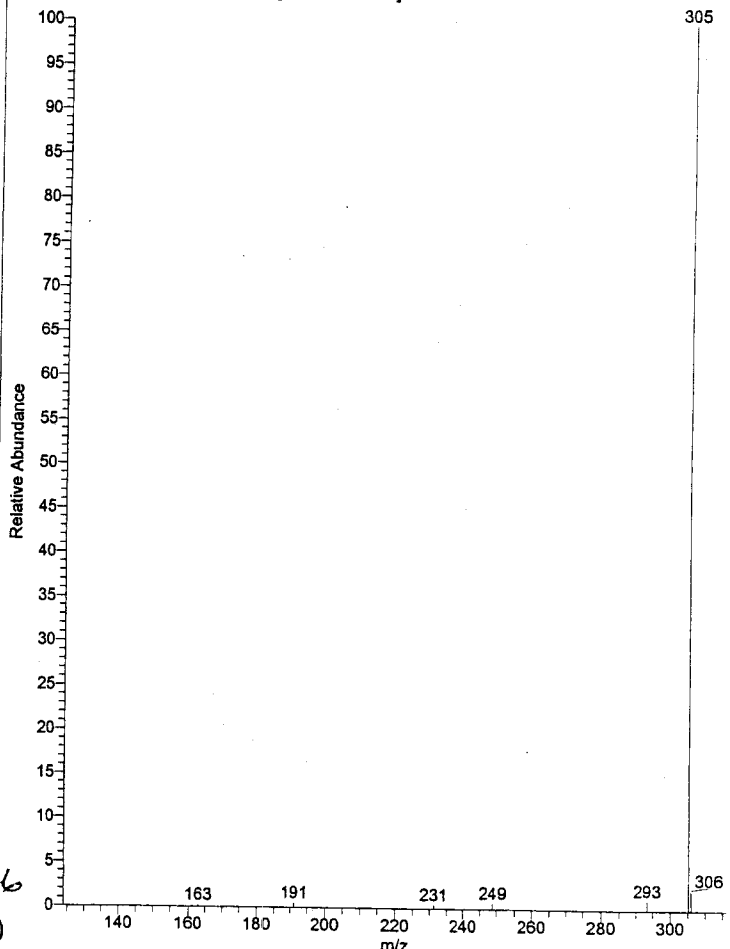
RT: 0.00 - 9.99 SM: 5G



Sel07#62-70 RT: 0.82-0.90 AV: 4 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



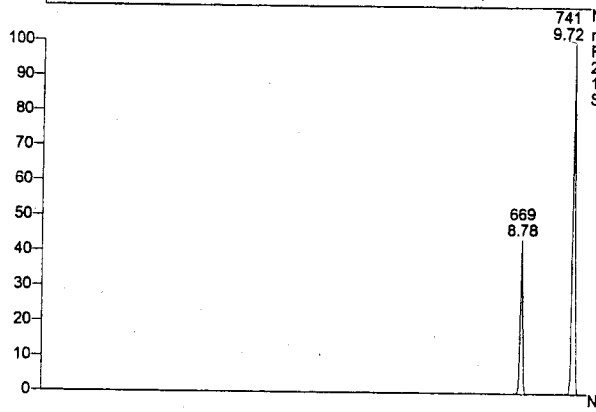
Sel07#153-176 RT: 2.00-2.30 AV: 24 NL: 3.77E4
T: + c ESI Full ms2 305.00@15.00 [125.00-315.00]



Ex 446
(291)

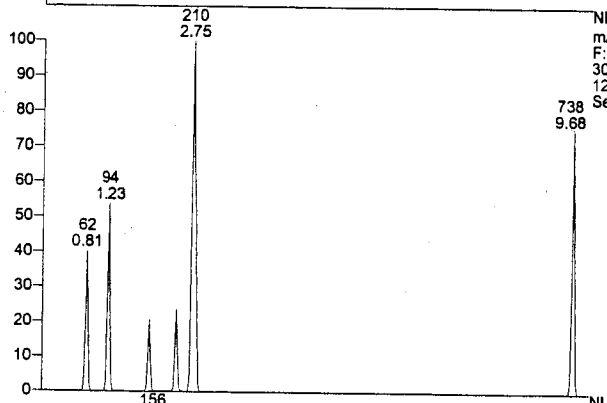
RT: 0.00 - 10.00 SM: 5G

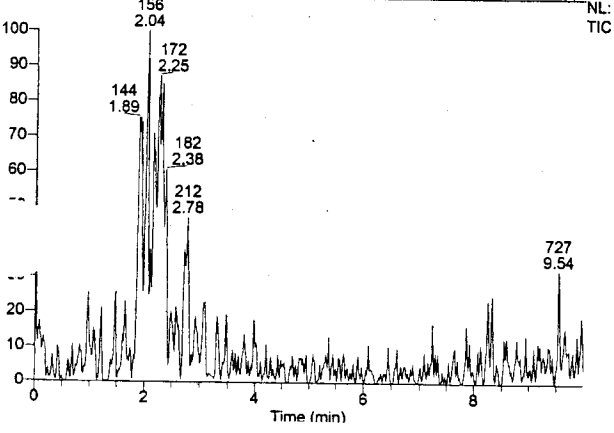
NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel08



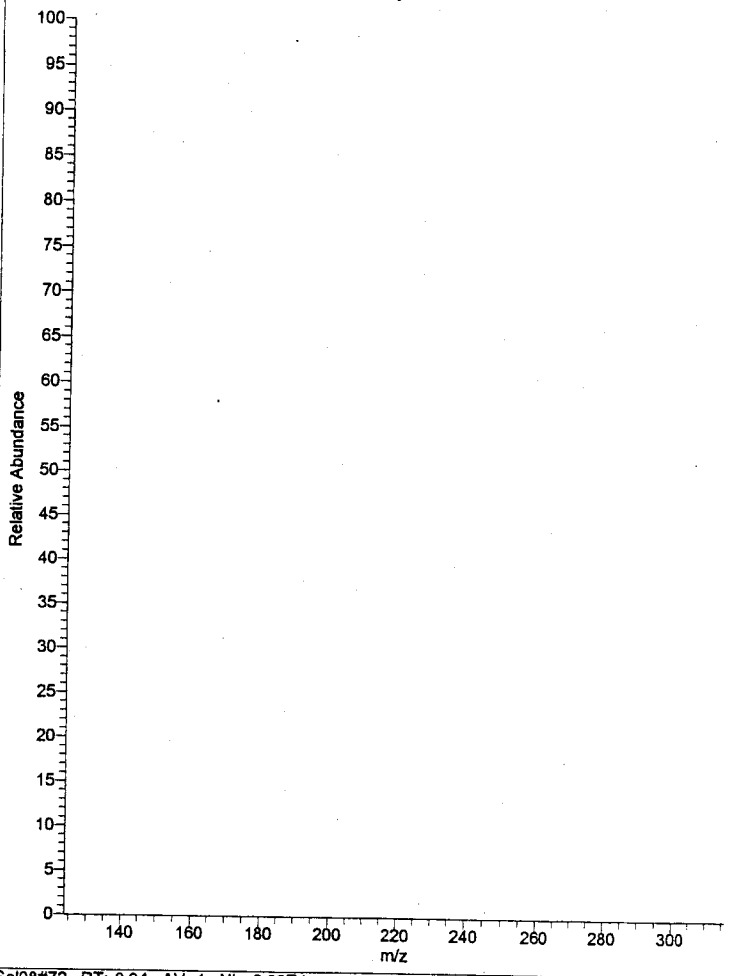
NL: 0
m/z= 167.5-168.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel08

Relativ

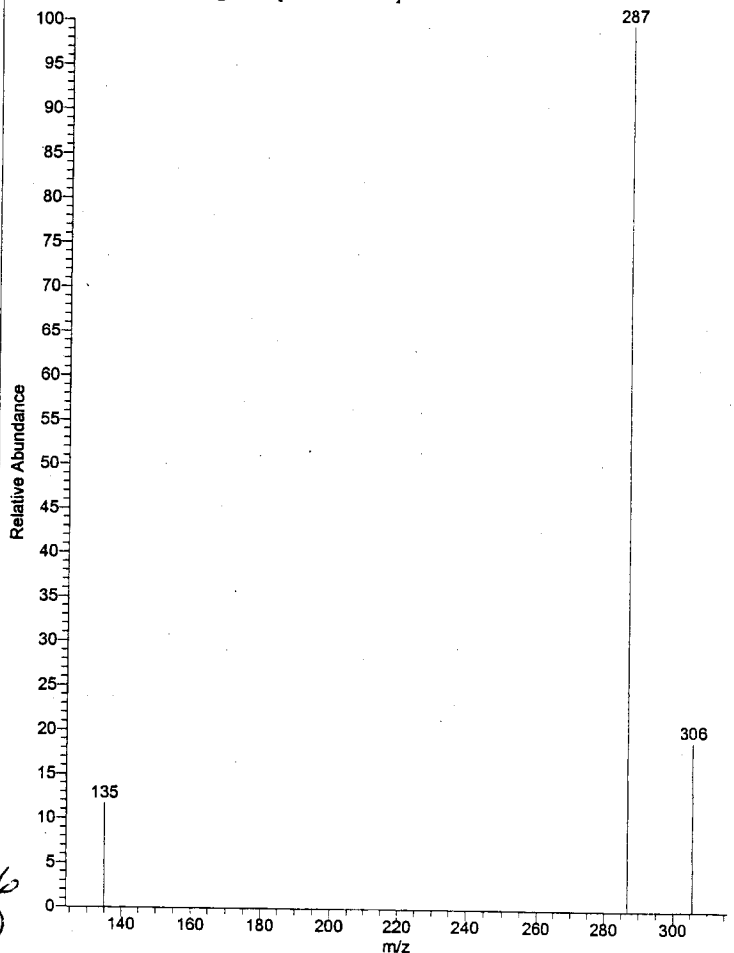




Sel08#71 RT: 0.93 AV: 1 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



Sel08#72 RT: 0.94 AV: 1 NL: 2.62E4
F: + c ESI Full ms2 305.00@15.00 [125.00-315.00]

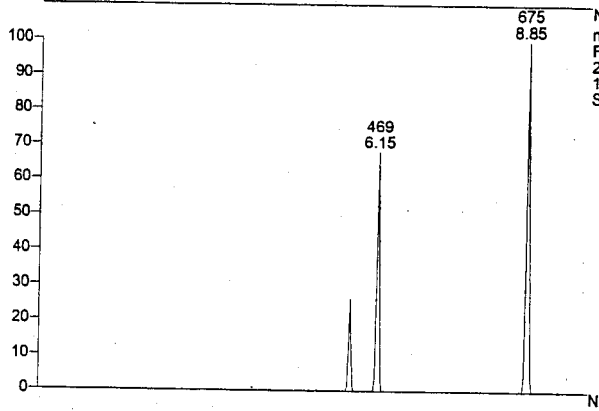


Ex 446
(293)

C:\Xcalibur\...\SEL_Positive\Sel09 02/09/07 12:24:13 Swab D, Case 5, Red JOB

RT: 0.00 - 10.00 SM: 5G

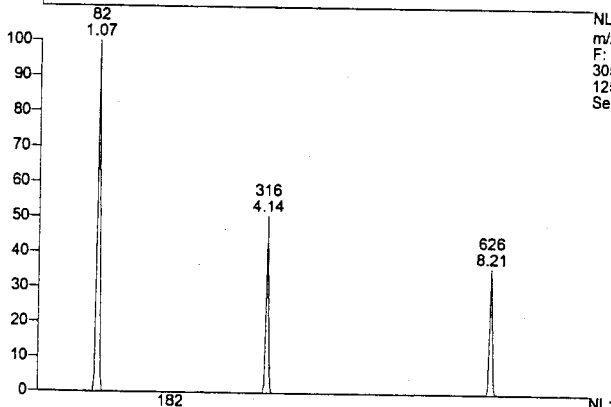
NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel09



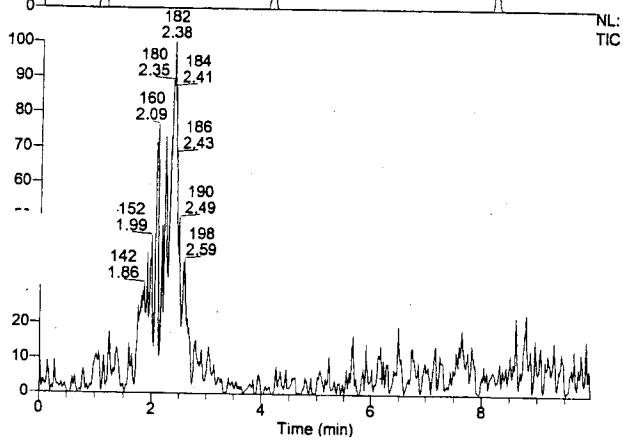
NL: 3.24E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel09

NL: 0
m/z= 167.5-168.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel09

Relativ



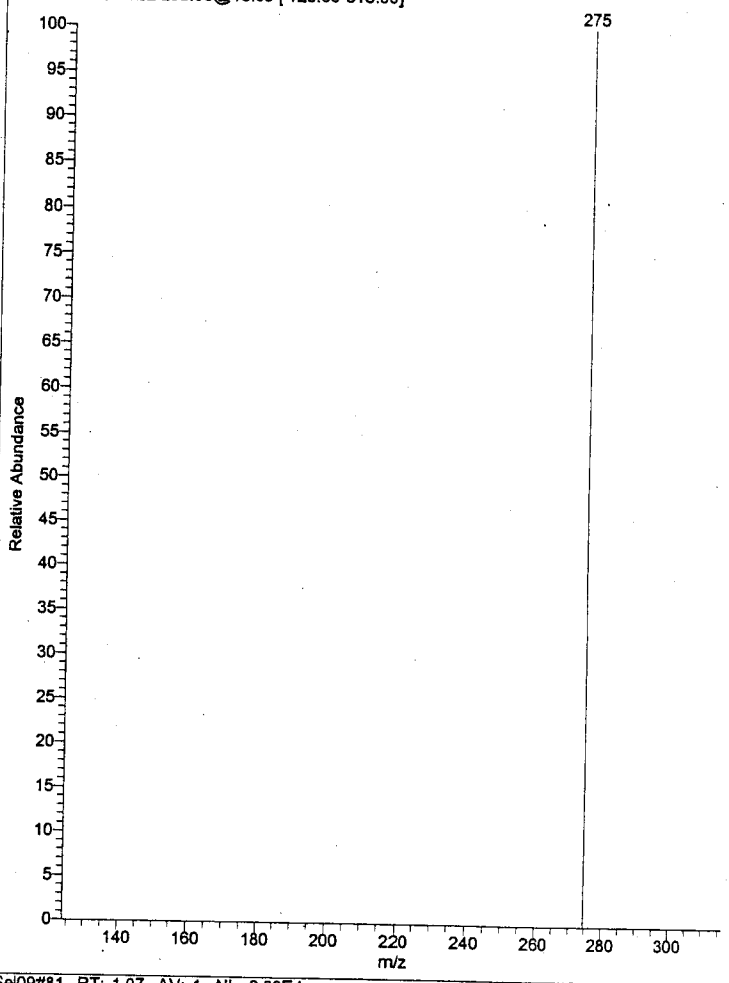
NL: 6.96E3
m/z= 258.5-259.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel09



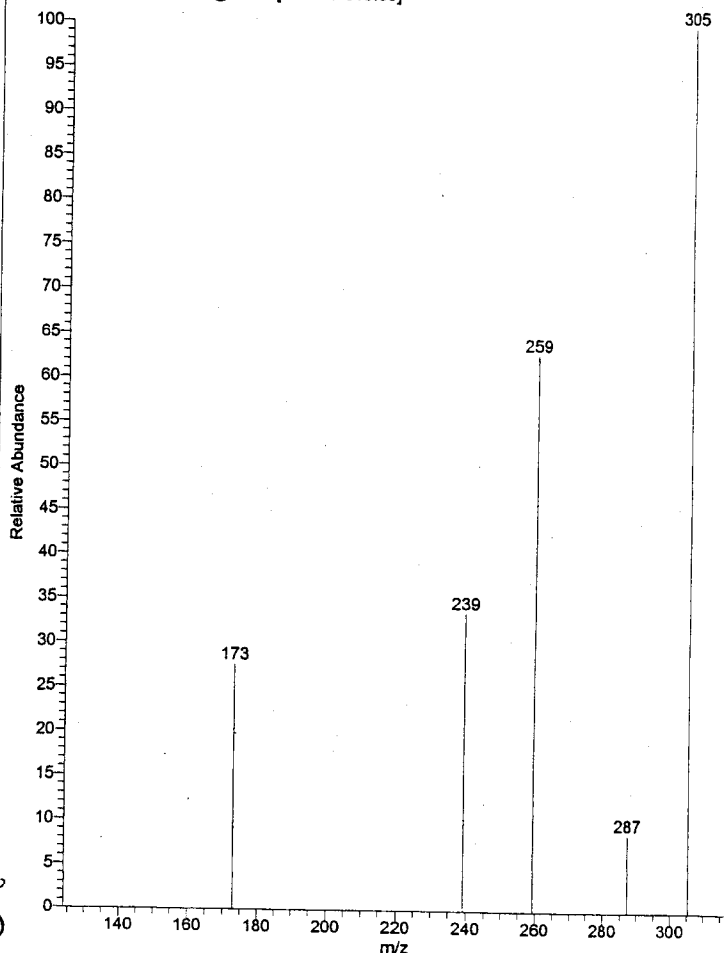
NL: 2.45E5
TIC MS Sel09

Ex 446
(293)

Sel09#71 RT: 0.92 AV: 1 NL: 5.07E3
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

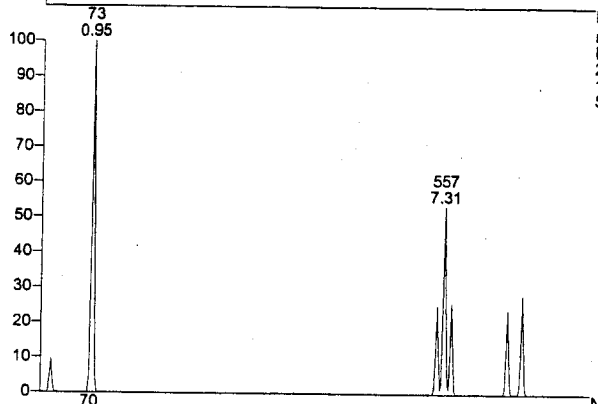


Sel09#81 RT: 1.07 AV: 1 NL: 2.26E4
F: + c ESI Full ms2 305.00@15.00 [125.00-315.00]

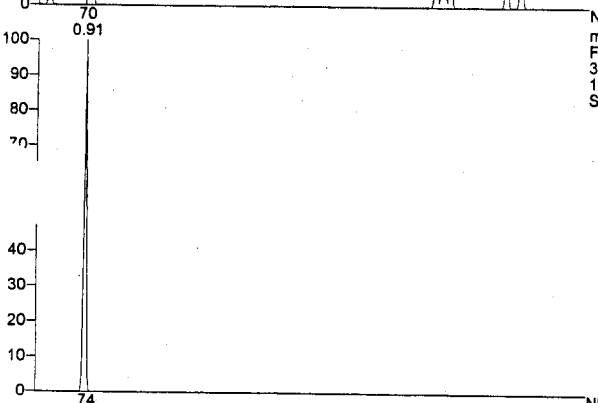


RT: 0.00 - 10.00 SM: 5G

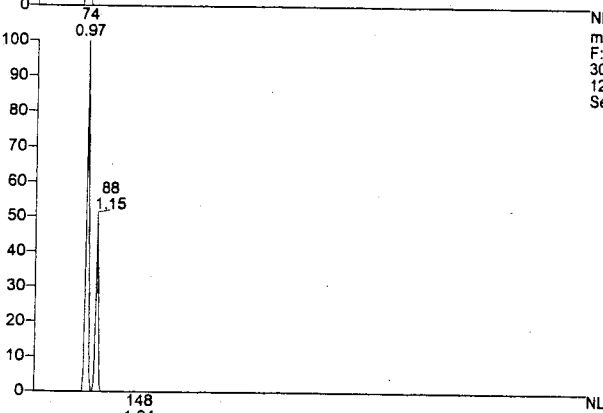
NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel10



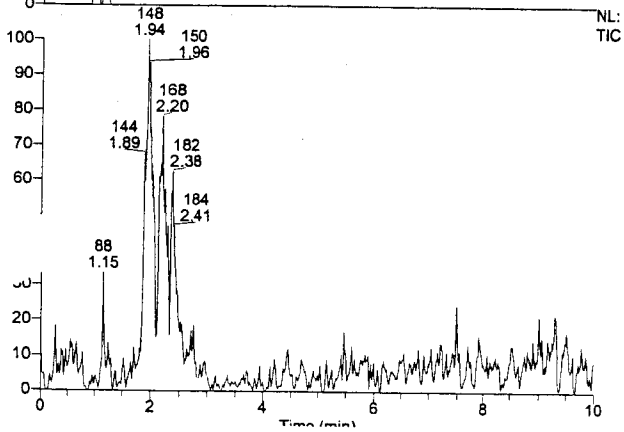
NL: 8.14E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel10



NL: 9.97E2
m/z= 167.5-168.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel10

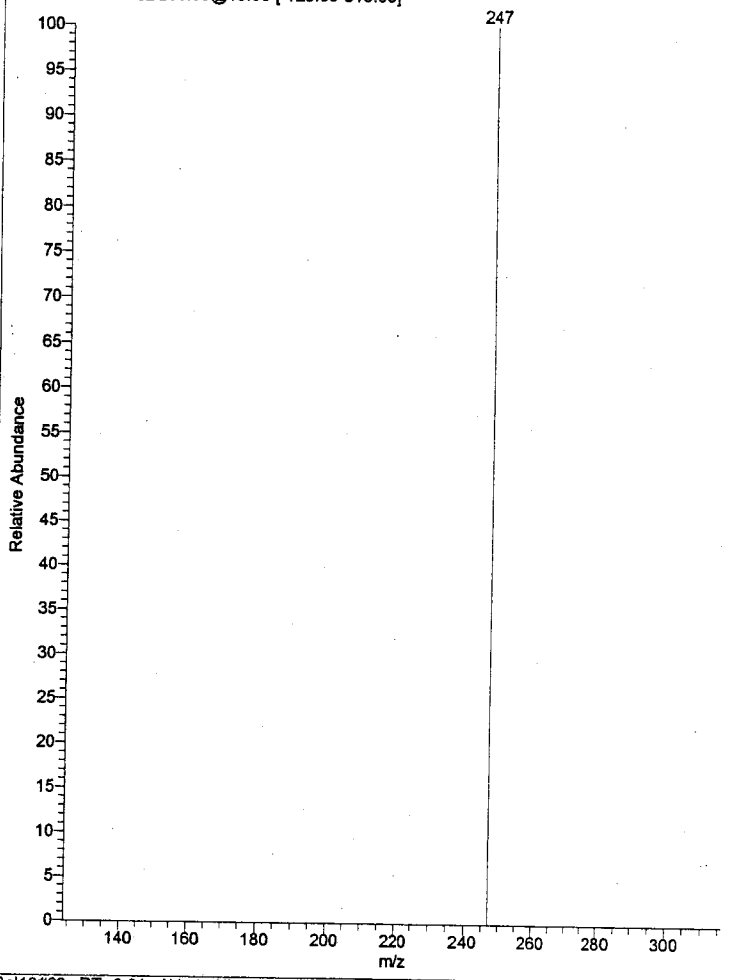


NL: 2.67E3
m/z= 258.5-259.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel10

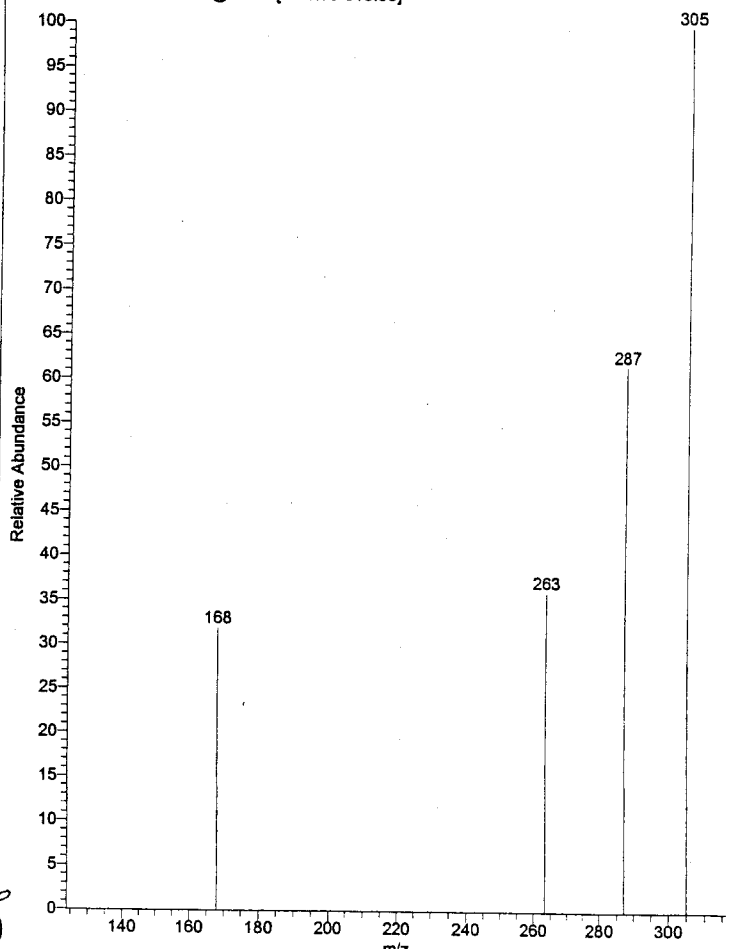


NL: 3.01E5
TIC MS Sel10

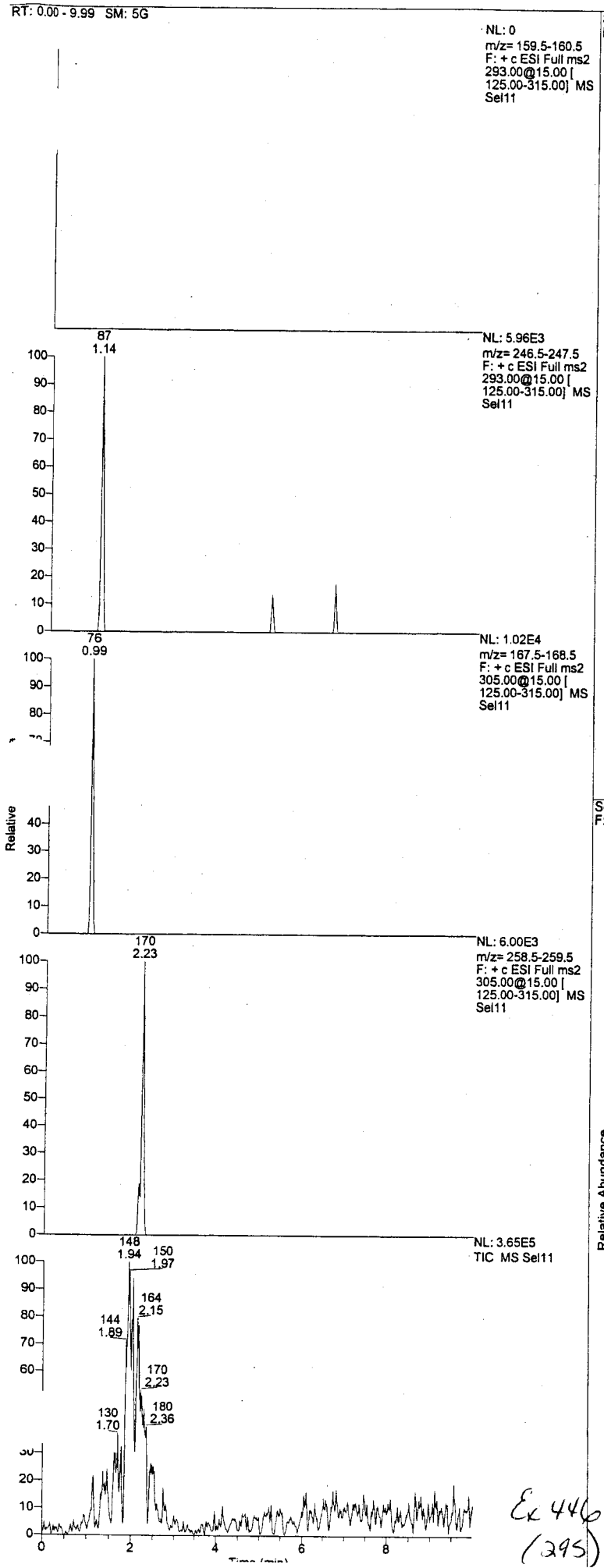
Sel10#72-75 RT: 0.95-0.98 AV: 2 NL: 8.33E3
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

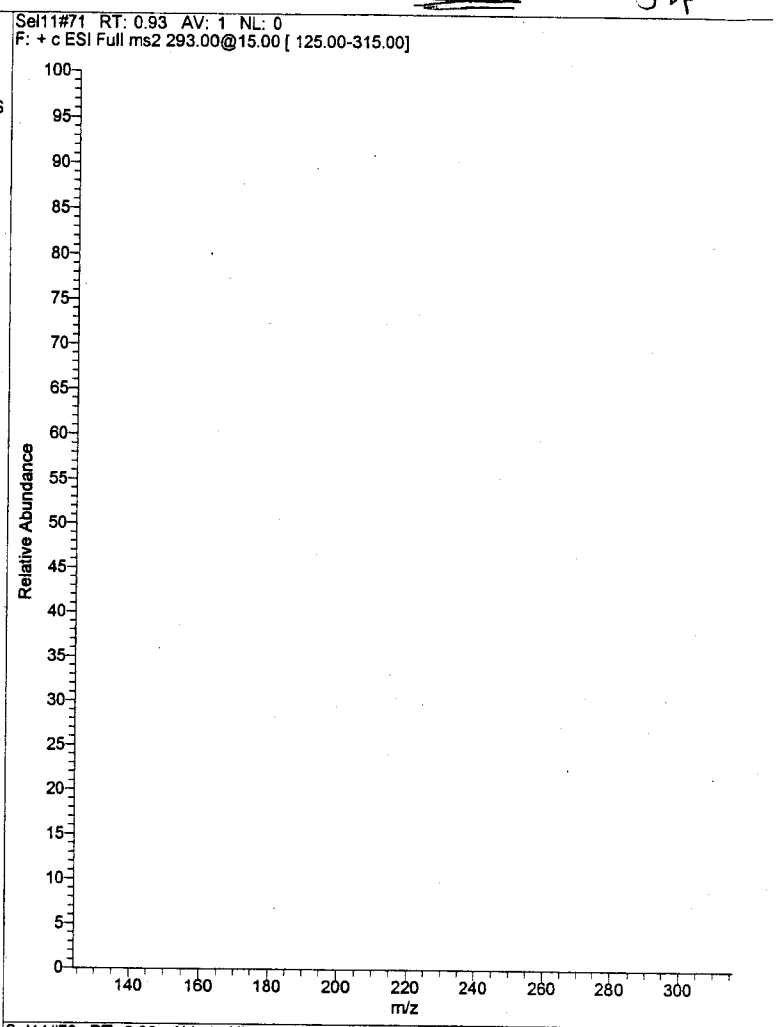


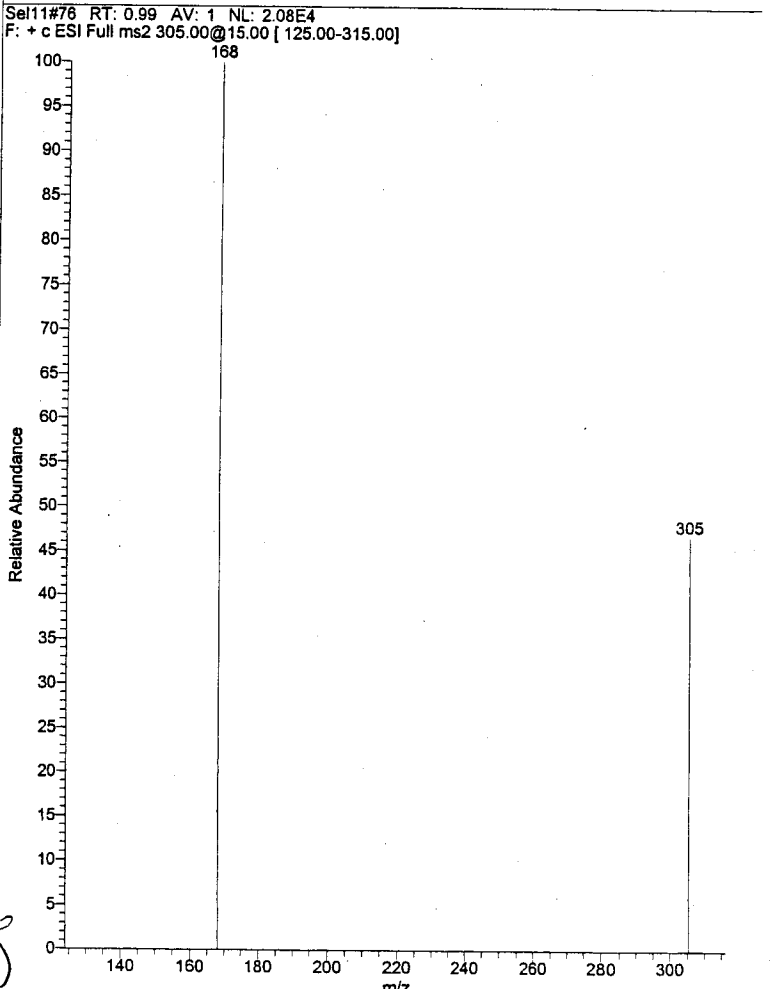
Sel10#69 RT: 0.91 AV: 1 NL: 6.40E3
F: + c ESI Full ms2 305.00@15.00 [125.00-315.00]



Ex 446
(294)

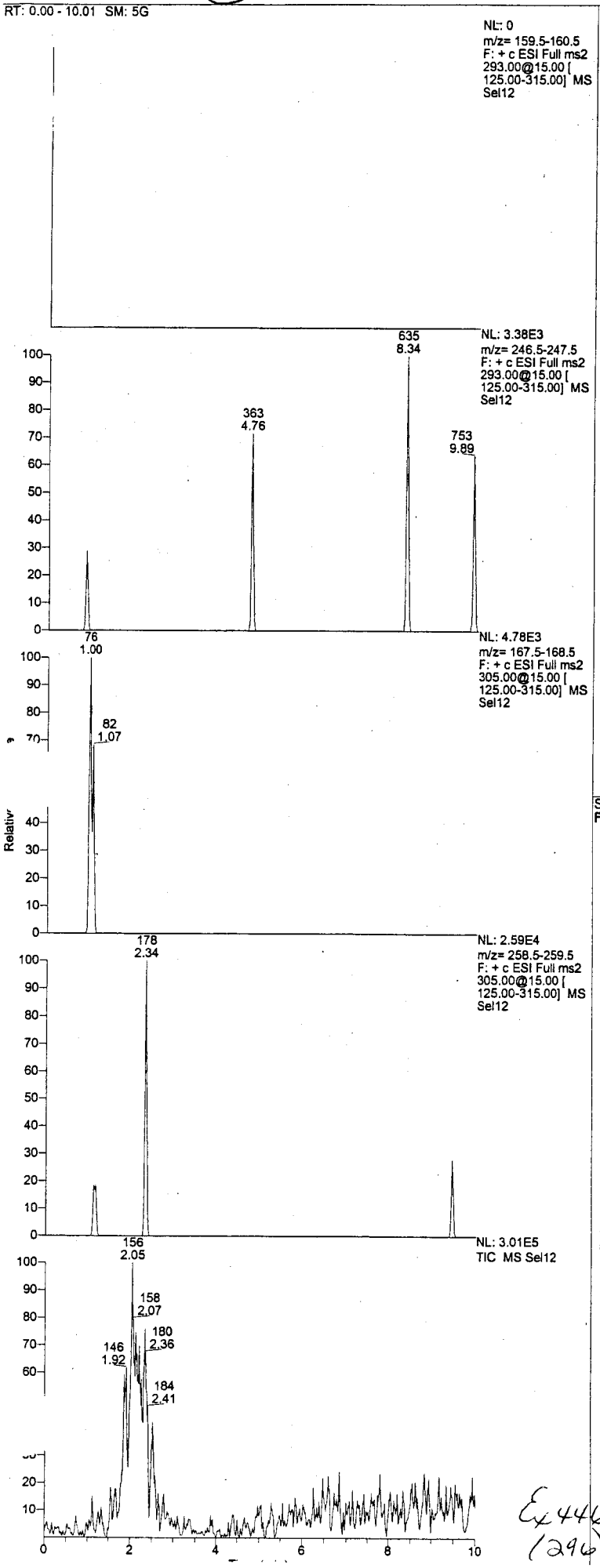

RT: 0.00 - 9.99 SM: 5G
NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel11
87
1.14
NL: 5.96E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Sel11
76
0.99
NL: 1.02E4
m/z= 167.5-168.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel11
Relative
170
2.23
NL: 6.00E3
m/z= 258.5-259.5
F: + c ESI Full ms2
305.00@15.00 [
125.00-315.00] MS
Sel11
148
1.94
150
1.97
164
2.15
144
1.89
170
2.23
180
2.36
130
1.70
NL: 3.65E5
TIC MS Sel11
Time (min)


Sel11#71 RT: 0.93 AV: 1 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]
Relative Abundance
m/z

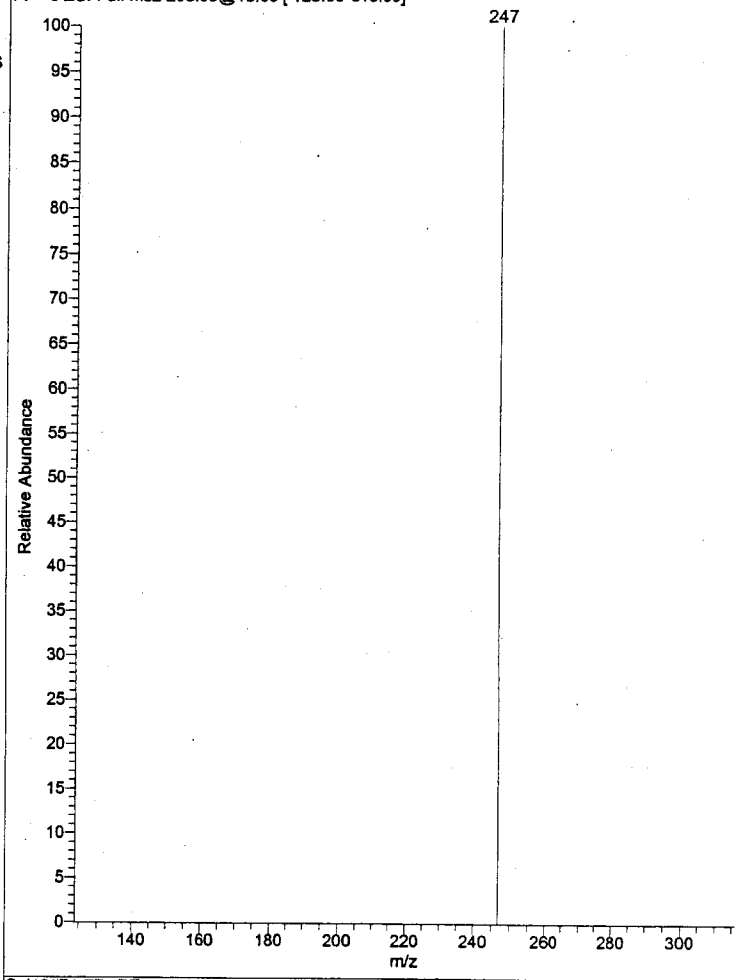

Sel11#76 RT: 0.99 AV: 1 NL: 2.08E4
F: + c ESI Full ms2 305.00@15.00 [125.00-315.00]
168
305
Relative Abundance
m/z

Ex 446
(295)

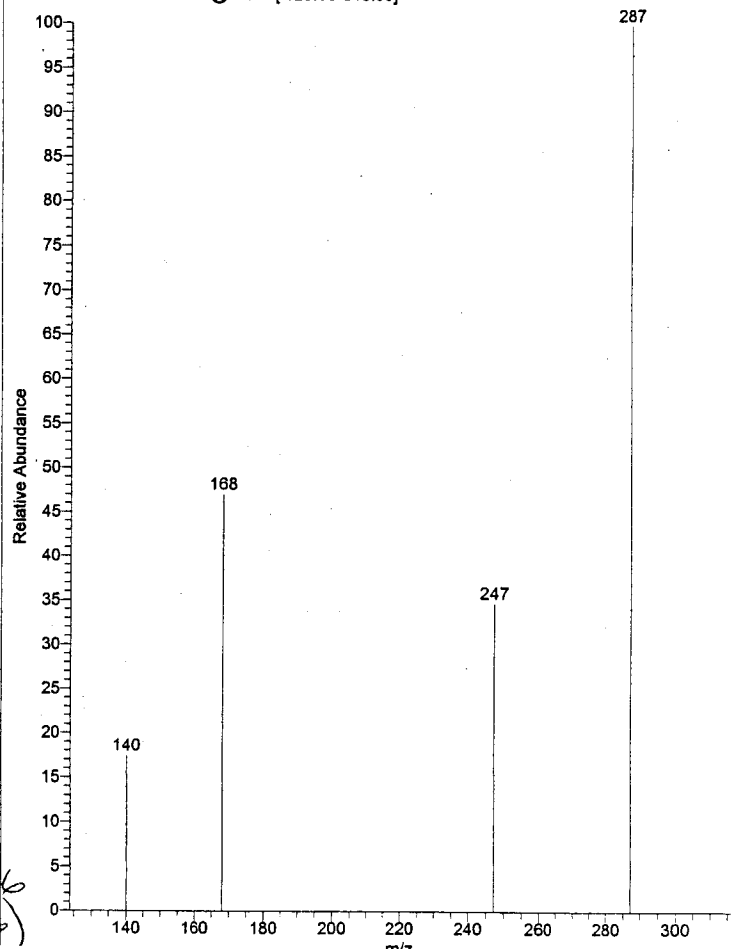
RT: 0.00 - 10.01 SM: 5G



Sel12#67 RT: 0.88 AV: 1 NL: 2.00E3
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



Sel12#74-77 RT: 0.97-1.00 AV: 2 NL: 1.22E4
F: + c ESI Full ms2 305.00@15.00 [125.00-315.00]



Ex 446 (296)

Sequence---select_neg.sld [Open]

Sample Name: Swab A, Case 1, Grey

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Sel01	1	C:\Xcalibur\Data\EDTA\Brewer\SEL_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Swab A, Case 2, Red

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Sel02	02	C:\Xcalibur\Data\EDTA\Brewer\SEL_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			2	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JPB
2/12/07

Ex 446
(297)

Sequence---select_neg.sld [Open]

Sample Name: Swab B, Case 2, Grey

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Sel03	03	C:\Xcalibur\Data\EDTA\Brewer\SEL_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			3	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Swab C, Case 3-1, Grey

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Sel04	04	C:\Xcalibur\Data\EDTA\Brewer\SEL_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			4	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

TOP 2/12/07

Ex 446
(298)

Sequence---select_neg.sld [Open]

Sample Name: Swab C, Case 3-2, Red

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Sel05	05	C:\Xcalibur\Data\EDTA\Brewer\SEL_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			5	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Swab C, Case 3-3, Grey

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Sel06	06	C:\Xcalibur\Data\EDTA\Brewer\SEL_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			6	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(299)

JOB
2/12/07

Sequence---select_neg.sld [Open]

Sample Name: Swab D, Case 3-4, Grey

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Sel07	07	C:\Xcalibur\Data\EDTA\Brewer\SEL_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			7	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Swab D, Case 4-1, Red

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Sel08	08	C:\Xcalibur\Data\EDTA\Brewer\SEL_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			8	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(300)

JDP
2/14/07

Sequence---select_neg.sld [Open]

Sample Name: Swab D, Case 5, Red

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Sel09	09	C:\Xcalibur\Data\EDTA\Brewer\SEL_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			9	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Swab A, MAL, Yellow

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Sel10	10	C:\Xcalibur\Data\EDTA\Brewer\SEL_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			10	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(301)

JoP
2/12/07

Sequence---select_neg.sld [Open]

Sample Name: Swab A, MAL, Yellow, d12 spiked

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Sel11	11	C:\Xcalibur\Data\EDTA\Brewer\SEL_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Swab D, MAL, Yellow, d12 spiked

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Sel12	12	C:\Xcalibur\Data\EDTA\Brewer\SEL_Negative

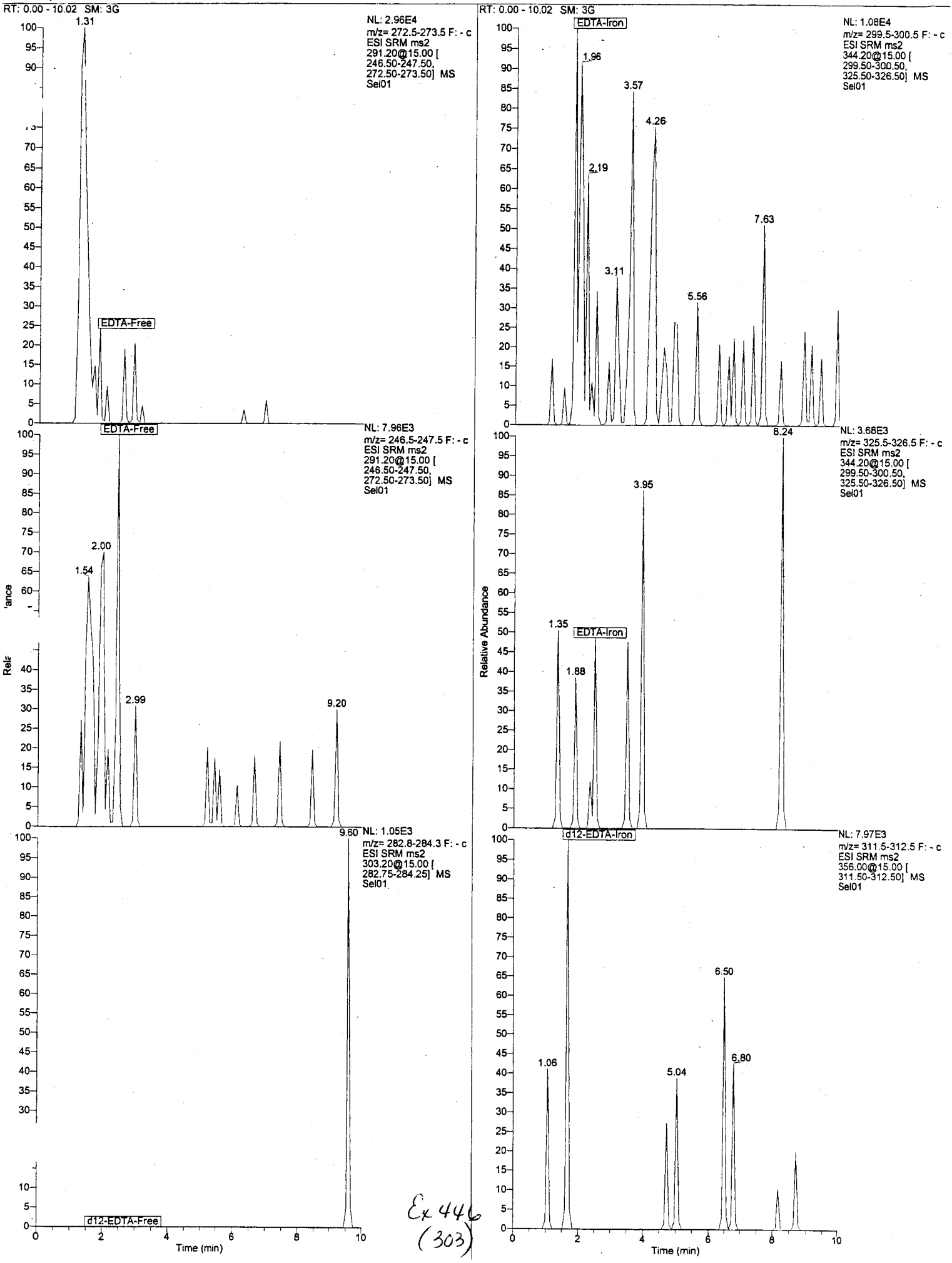
Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

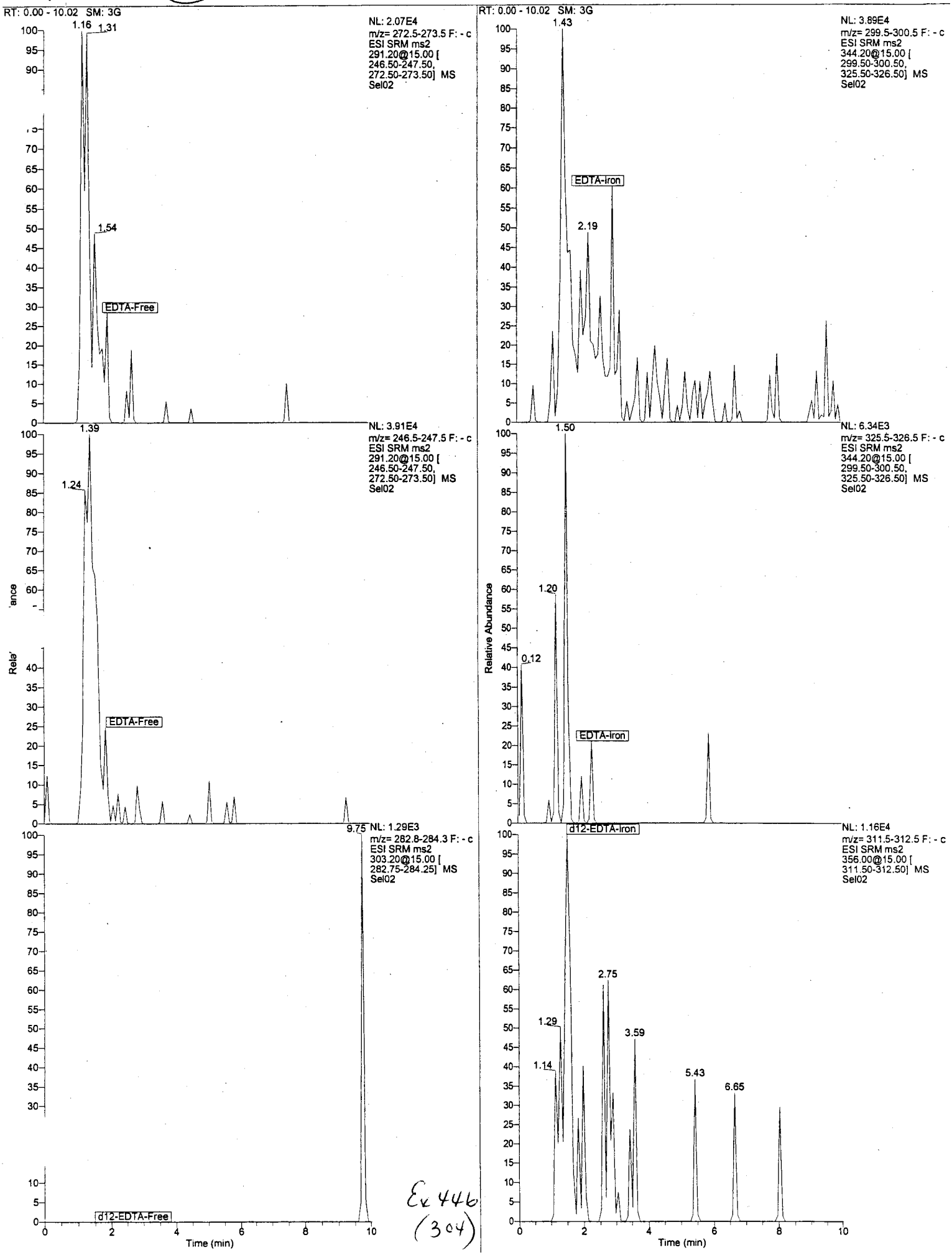
****

Ex 446
(302)

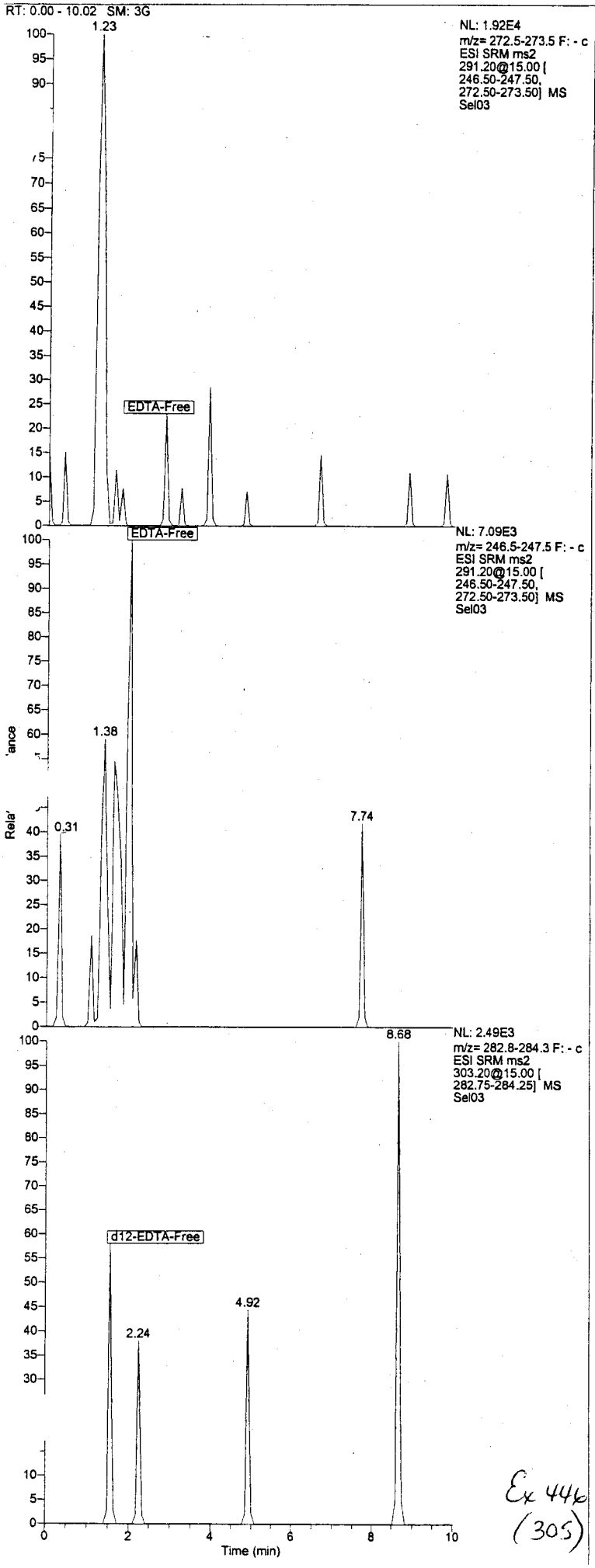
JBP
2/12/07

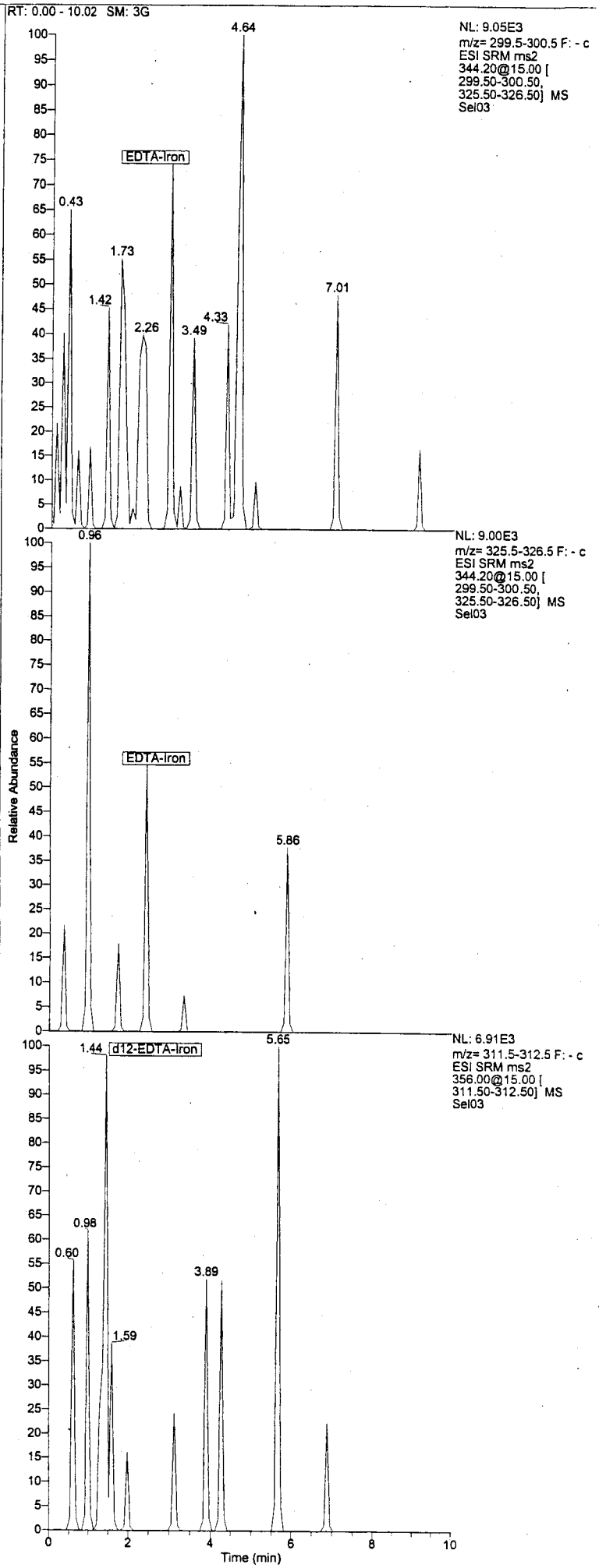

RT: 0.00 - 10.02 SM: 3G
NL: 2.96E4
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel01
1.31
EDTA-Free
RT: 0.00 - 10.02 SM: 3G
NL: 1.08E4
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel01
EDTA-Iron
1.96
3.57
4.26
2.19
7.63
3.11
5.56
NL: 7.96E3
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel01
EDTA-Free
2.00
1.54
2.99
9.20
NL: 3.68E3
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel01
8.24
3.95
1.35
EDTA-Iron
1.88
Relative Abundance
NL: 1.05E3
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Sel01
9.60
d12-EDTA-Free
NL: 7.97E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Sel01
d12-EDTA-Iron
6.50
1.06
6.80
5.04
Time (min)
Time (min)

Ex 446
(303)


RT: 0.00 - 10.02 SM: 3G
NL: 2.07E4
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel02
1.16
1.31
1.54
EDTA-Free
RT: 0.00 - 10.02 SM: 3G
NL: 3.89E4
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel02
1.43
EDTA-Iron
2.19
NL: 3.91E4
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel02
1.39
1.24
EDTA-Free
NL: 6.34E3
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel02
1.50
1.20
0.12
EDTA-Iron
Relative Abundance
NL: 1.29E3
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Sel02
9.75
d12-EDTA-Free
NL: 1.16E4
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Sel02
d12-EDTA-Iron
2.75
1.29
3.59
1.14
5.43
6.65
Time (min)
Time (min)

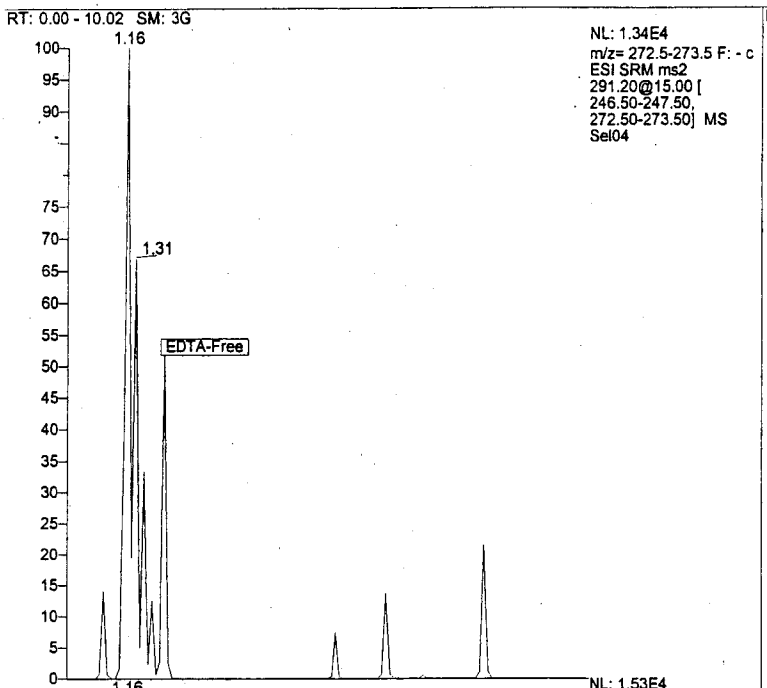
Ex 446
(304)

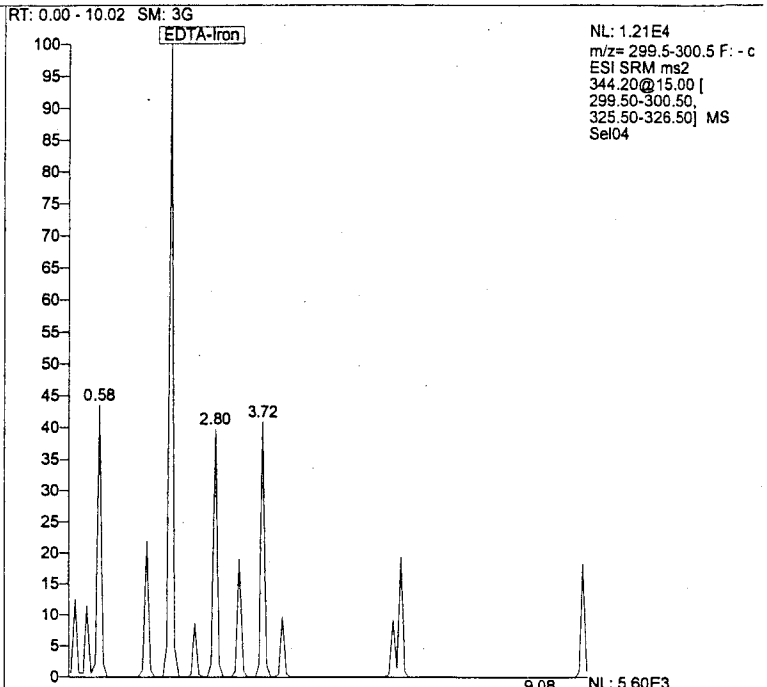

RT: 0.00 - 10.02 SM: 3G
NL: 1.92E4
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel03
1.23
EDTA-Free
NL: 7.09E3
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel03
EDTA-Free
1.38
0.31
7.74
NL: 2.49E3
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Sel03
8.68
d12-EDTA-Free
2.24
4.92
Time (min)

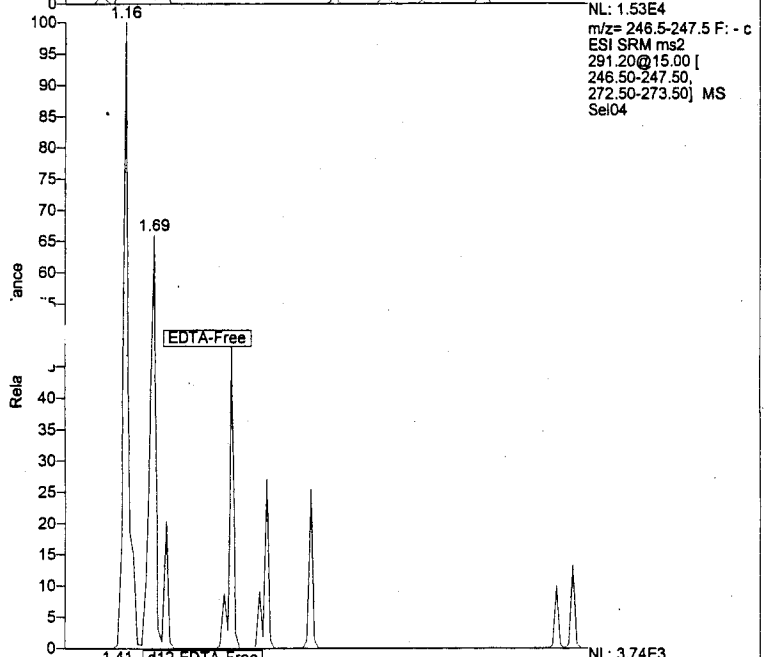

RT: 0.00 - 10.02 SM: 3G
NL: 9.05E3
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel03
4.64
EDTA-Iron
0.43
1.73
1.42
2.26
3.49
4.33
7.01
NL: 9.00E3
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel03
0.96
EDTA-Iron
5.86
Relative Abundance
NL: 6.91E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Sel03
1.44
d12-EDTA-Iron
5.65
0.98
0.60
3.89
1.59
Time (min)

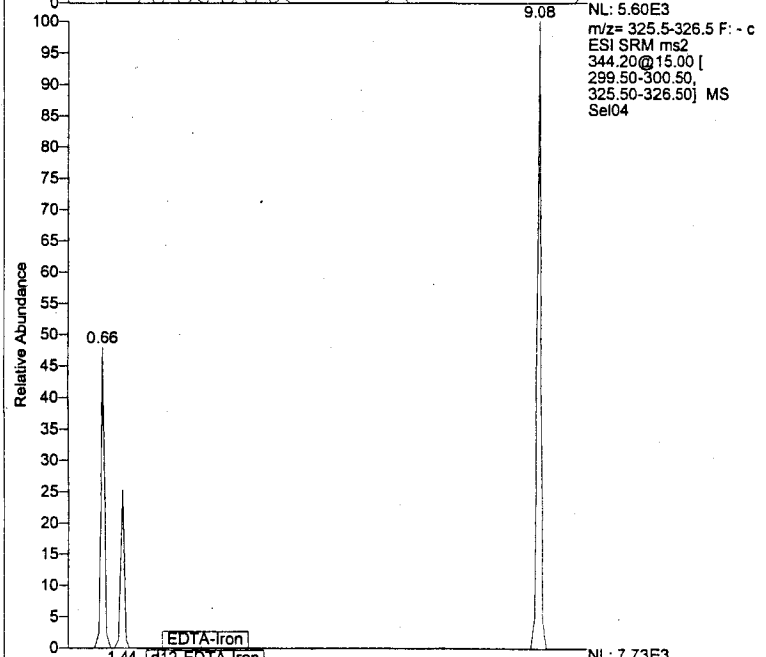
Ex 446
(305)

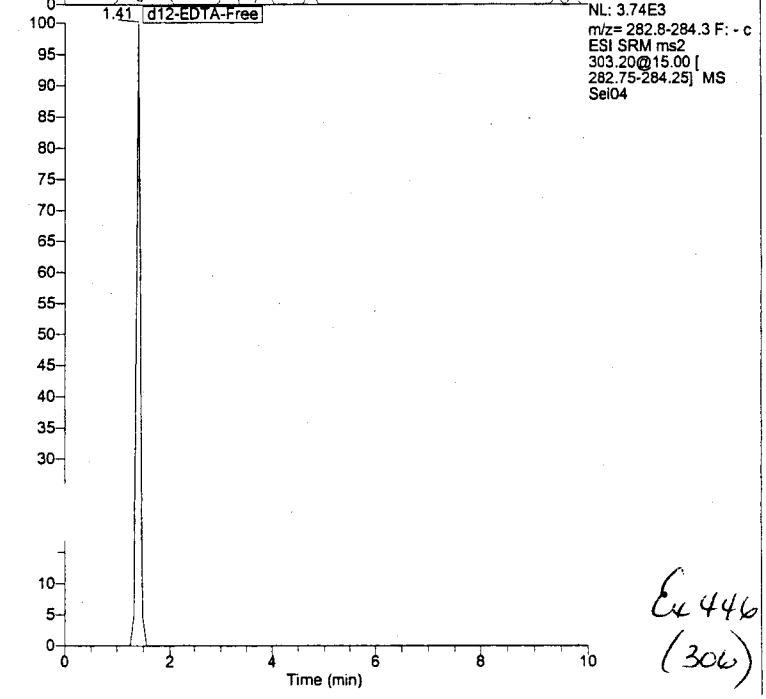
C:\Xcalibur\...\SEL_Negative\Sel04 02/12/07 10:54:09 Swab C, Case 3-1, Grey JVB

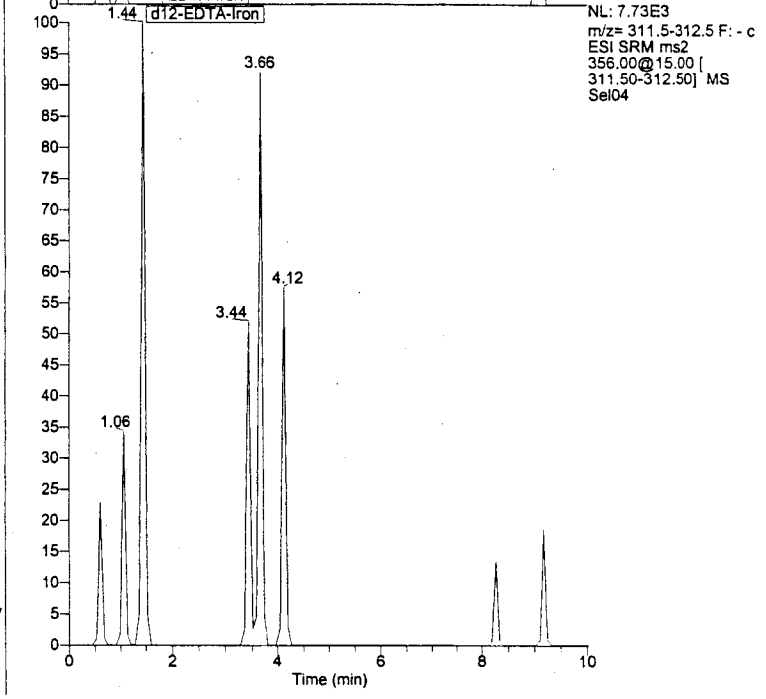




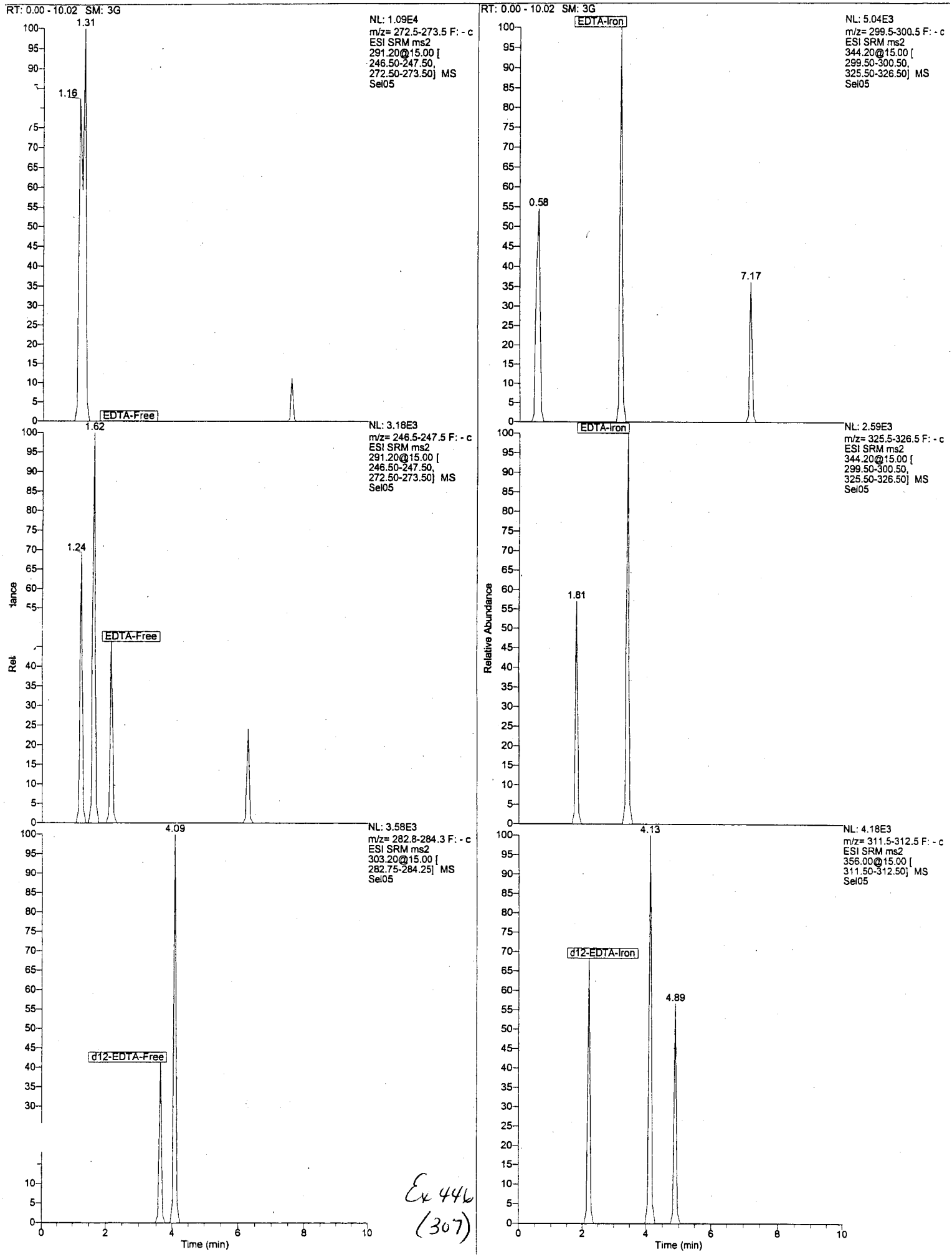




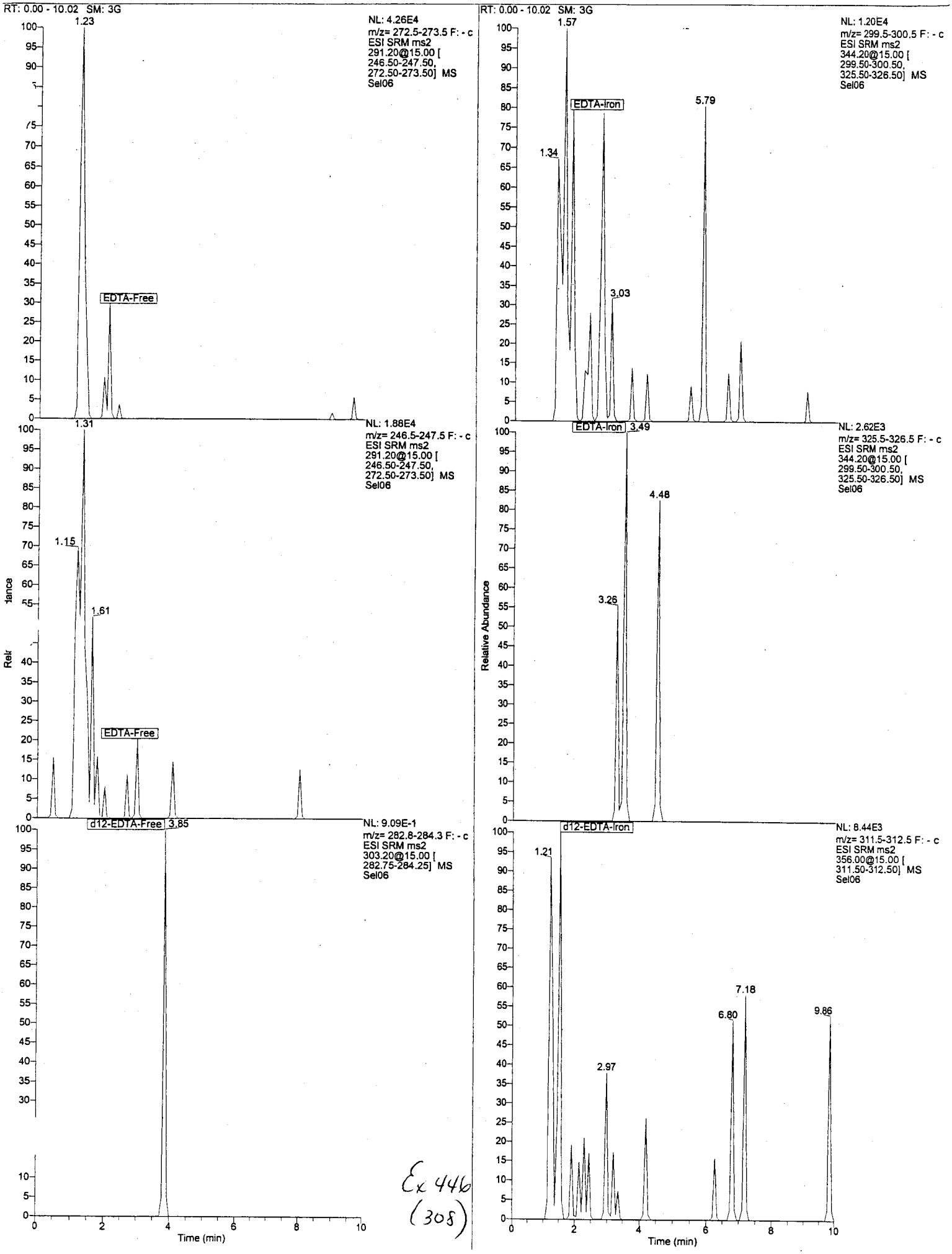




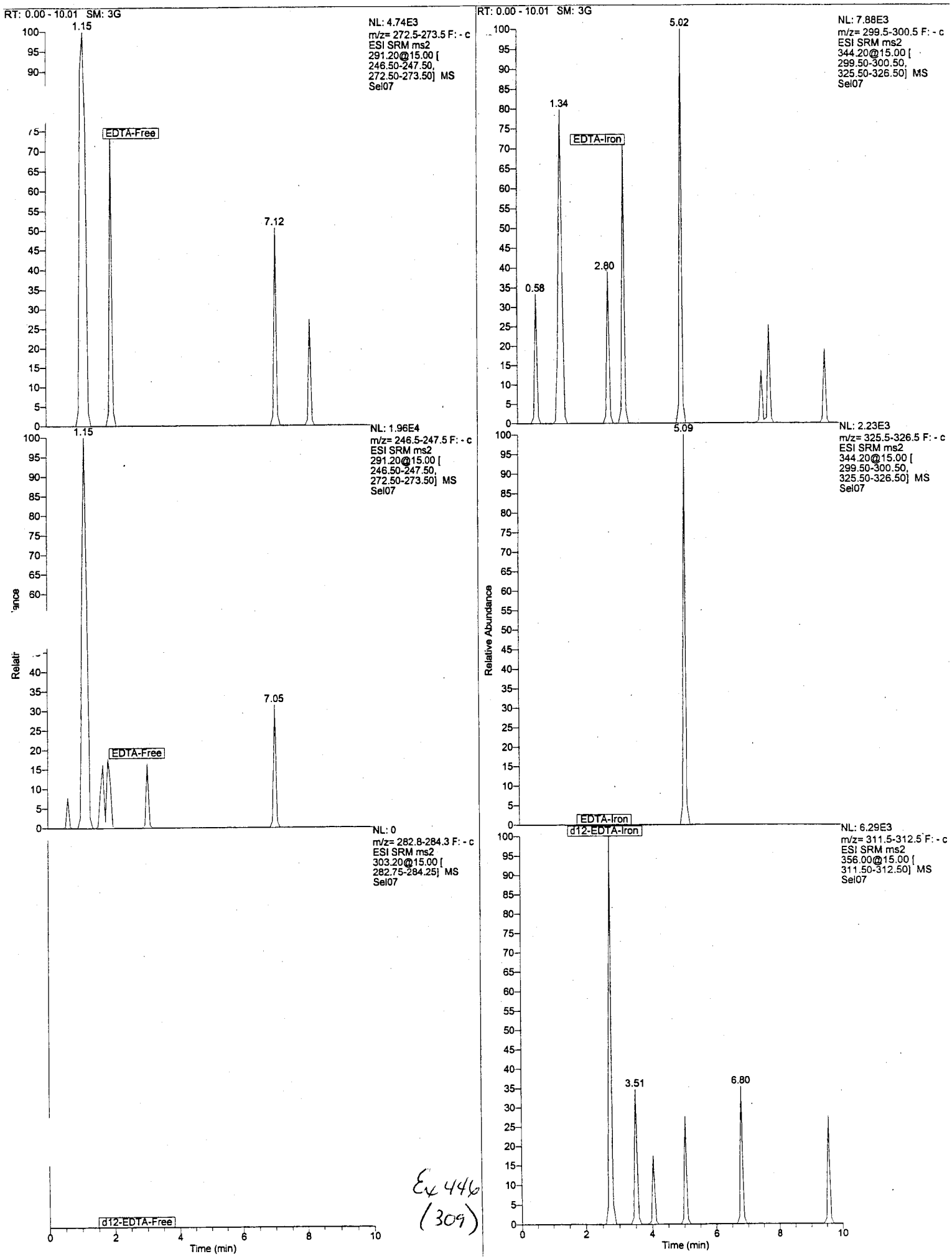
Ex 446
(306)


RT: 0.00 - 10.02 SM: 3G
NL: 1.09E4
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel05
1.31
1.16
EDTA-Free
NL: 5.04E3
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel05
EDTA-Iron
0.58
7.17
NL: 3.18E3
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel05
1.62
1.24
EDTA-Free
NL: 2.59E3
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel05
EDTA-Iron
1.81
Relative Abundance
NL: 3.58E3
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Sel05
4.09
d12-EDTA-Free
NL: 4.18E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Sel05
4.13
d12-EDTA-Iron
4.89
Time (min)
Time (min)

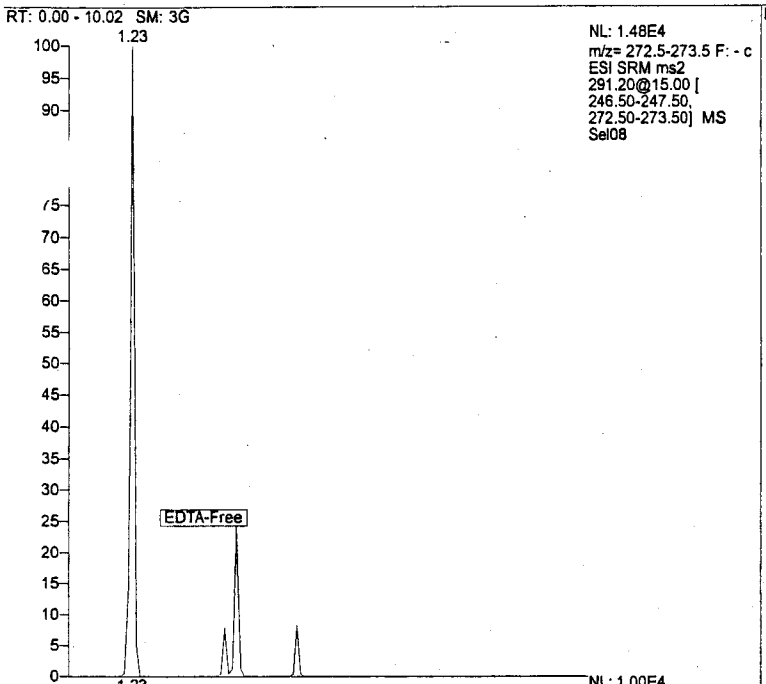
Ex 446
(307)

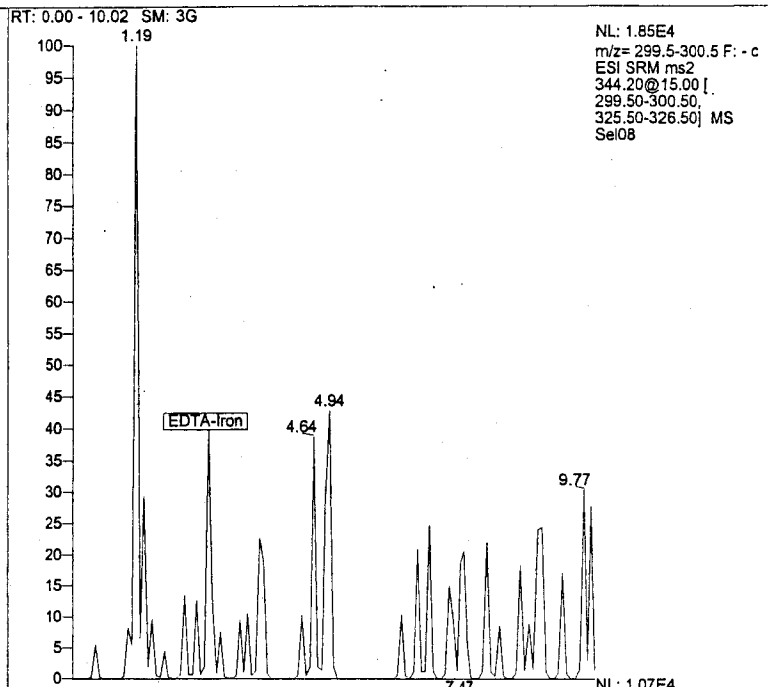

RT: 0.00 - 10.02 SM: 3G
NL: 4.26E4
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel06
1.23
EDTA-Free
NL: 1.88E4
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel06
1.31
1.15
1.61
EDTA-Free
NL: 9.09E-1
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Sel06
d12-EDTA-Free 3.85
Time (min)
NL: 1.20E4
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel06
1.57
EDTA-Iron
5.79
1.34
3.03
NL: 2.62E3
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel06
EDTA-Iron 3.49
4.48
3.26
NL: 8.44E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Sel06
d12-EDTA-Iron
1.21
7.18
6.80
9.86
2.97
Time (min)
Relative Abundance

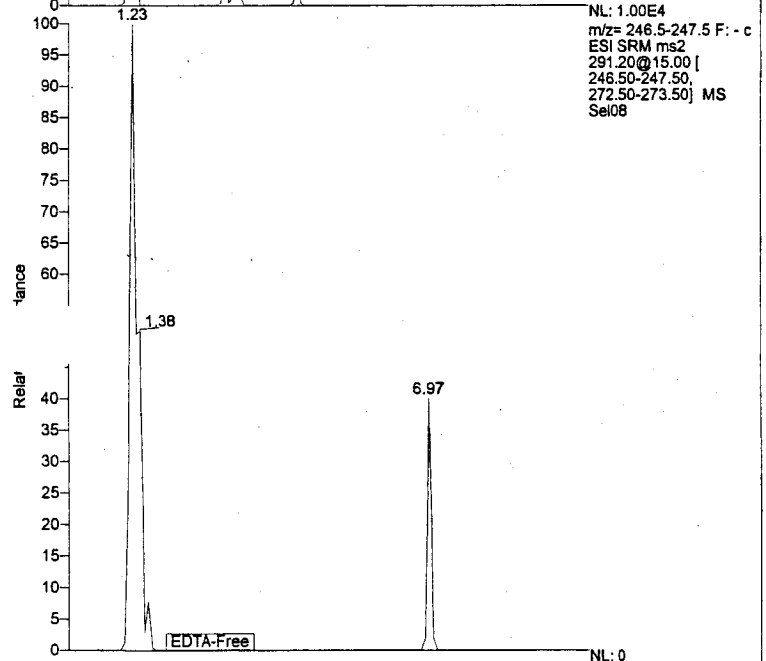
Ex 446 (308)

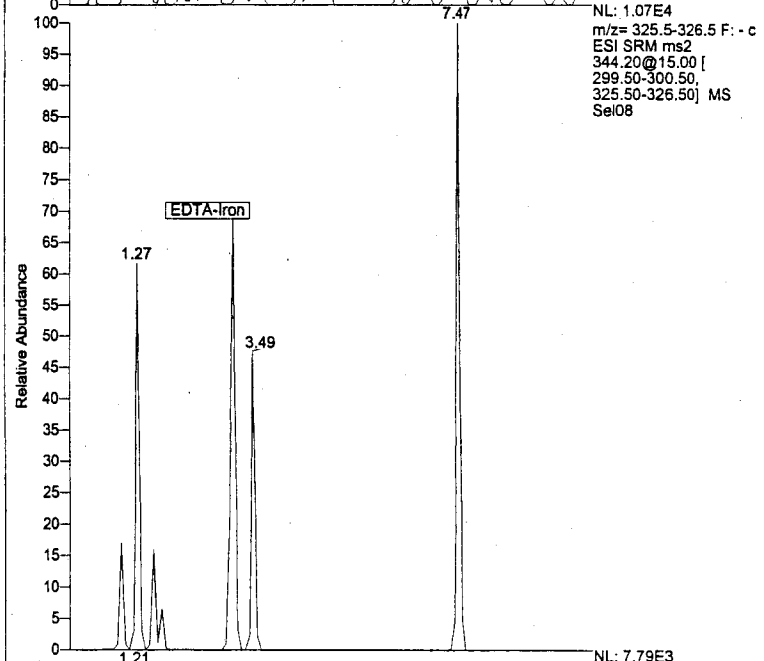

RT: 0.00 - 10.01 SM: 3G
NL: 4.74E3
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel07
1.15
EDTA-Free
7.12
NL: 1.96E4
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel07
1.15
7.05
EDTA-Free
NL: 0
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Sel07
d12-EDTA-Free
Relati
ance
Time (min)
RT: 0.00 - 10.01 SM: 3G
NL: 7.88E3
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel07
5.02
1.34
EDTA-Iron
2.80
0.58
NL: 2.23E3
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel07
5.09
EDTA-Iron
d12-EDTA-Iron
NL: 6.29E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Sel07
3.51
6.80
Relative Abundance
Time (min)

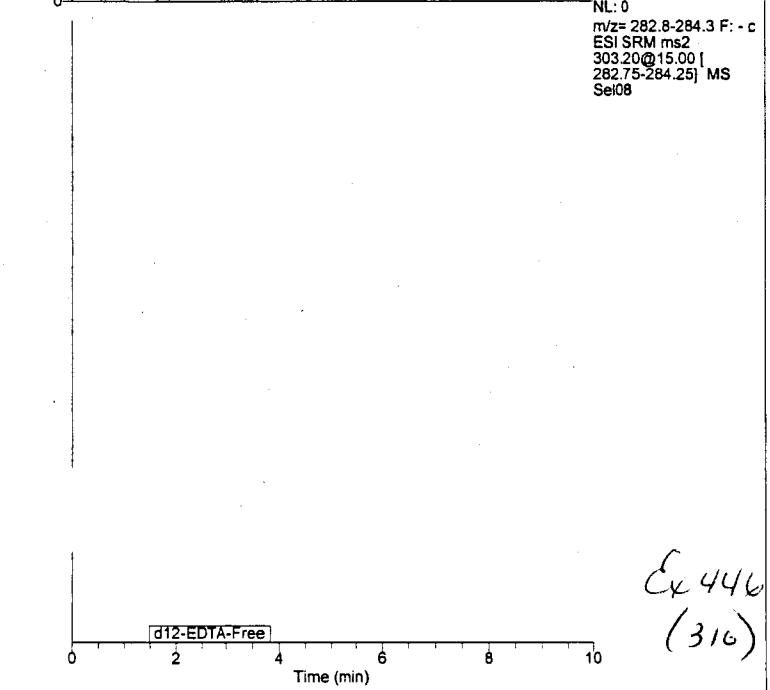
Ex 446
(309)

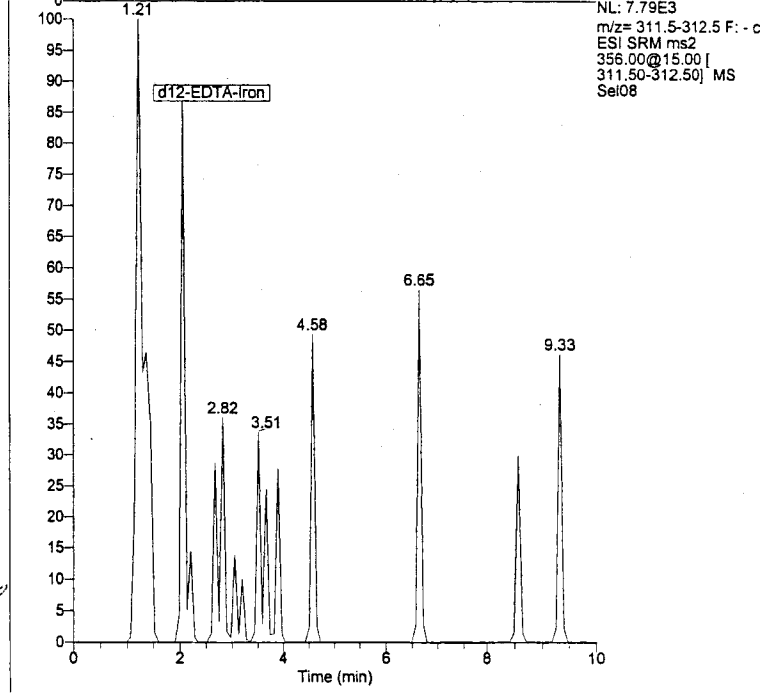

RT: 0.00 - 10.02 SM: 3G
NL: 1.48E4
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel08
1.23
EDTA-Free


RT: 0.00 - 10.02 SM: 3G
NL: 1.85E4
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel08
1.19
EDTA-Iron
4.64
4.94
9.77

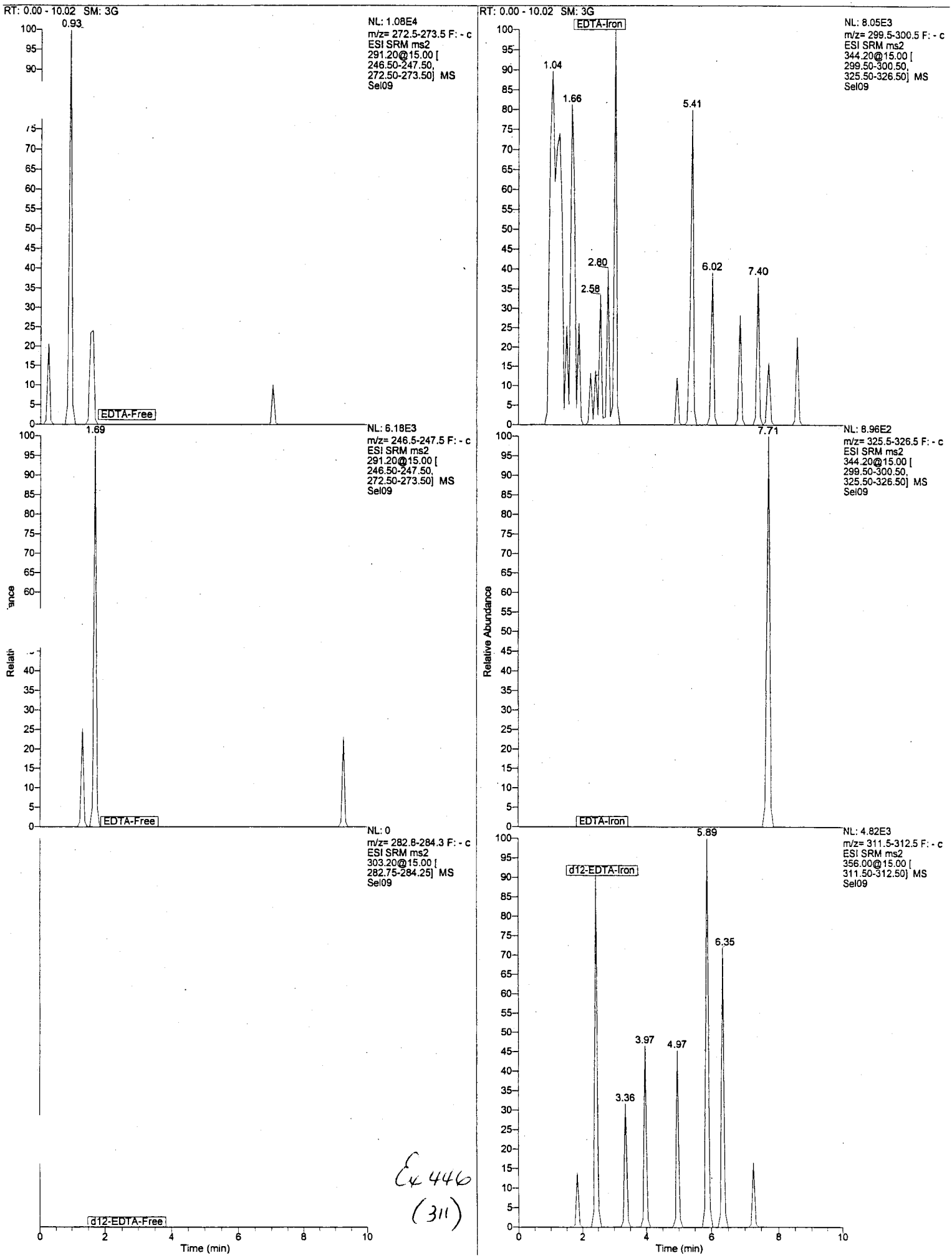

NL: 1.00E4
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel08
1.23
1.38
6.97
EDTA-Free
Relat
tance


NL: 1.07E4
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel08
7.47
EDTA-Iron
1.27
3.49
Relative Abundance

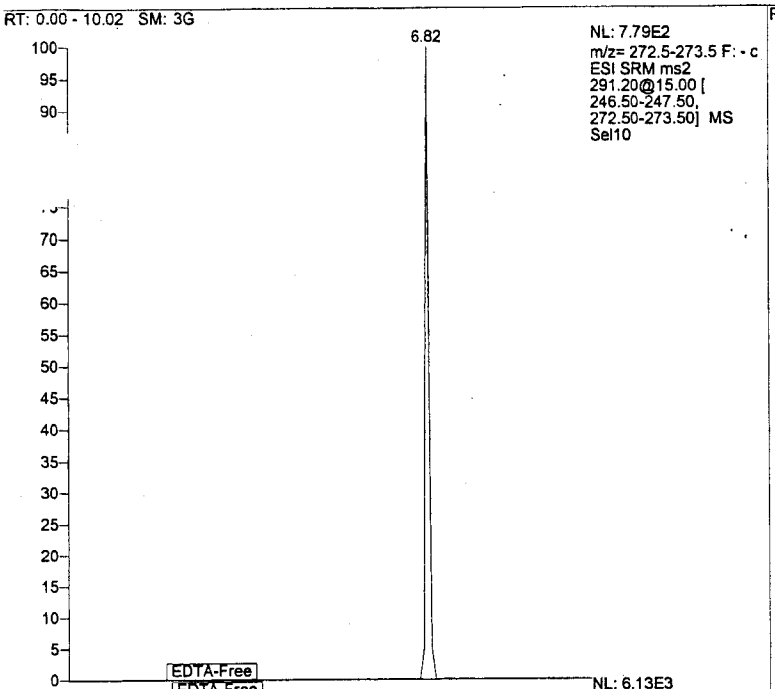

NL: 0
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Sel08
d12-EDTA-Free
0
2
4
6
8
10
Time (min)

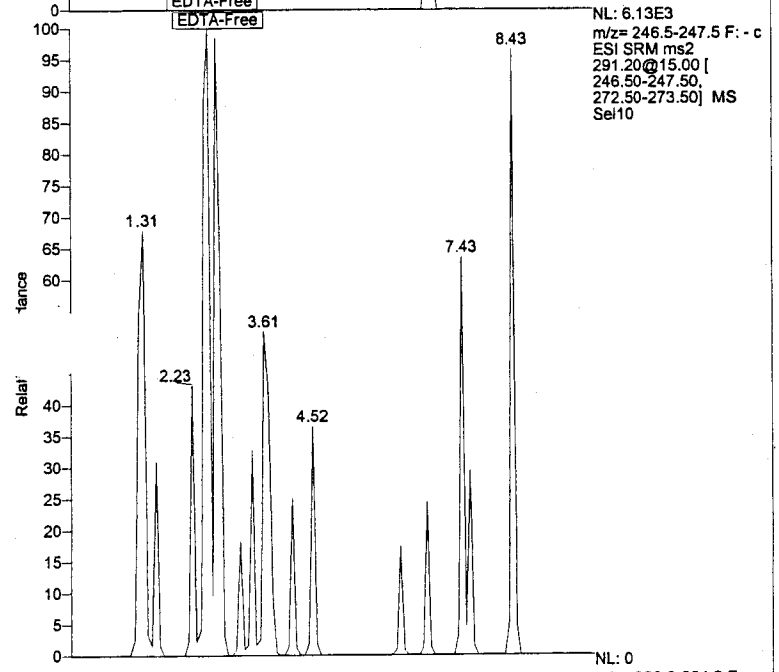

NL: 7.79E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Sel08
1.21
d12-EDTA-Iron
2.82
3.51
4.58
6.65
9.33
0
2
4
6
8
10
Time (min)

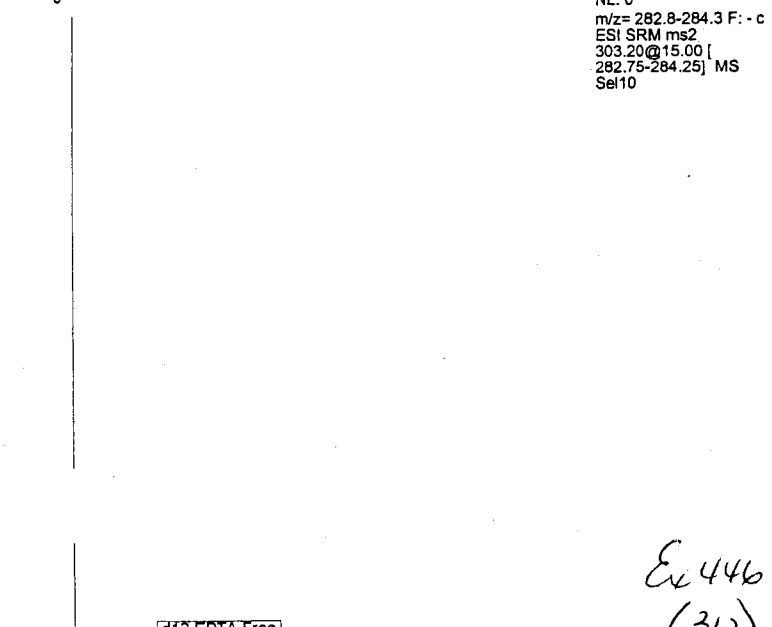
Ex 446
(316)


RT: 0.00 - 10.02 SM: 3G
EDTA-Free
NL: 1.08E4
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel09
0.93
EDTA-Free
NL: 6.18E3
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel09
1.69
EDTA-Free
NL: 0
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Sel09
d12-EDTA-Free
Relati
ance
Time (min)
RT: 0.00 - 10.02 SM: 3G
EDTA-Iron
NL: 8.05E3
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel09
1.04
1.66
2.80
2.58
5.41
6.02
7.40
EDTA-Iron
NL: 8.96E2
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel09
7.71
EDTA-Iron
NL: 4.82E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Sel09
5.89
d12-EDTA-Iron
6.35
3.97
4.97
3.36
Relative Abundance
Time (min)

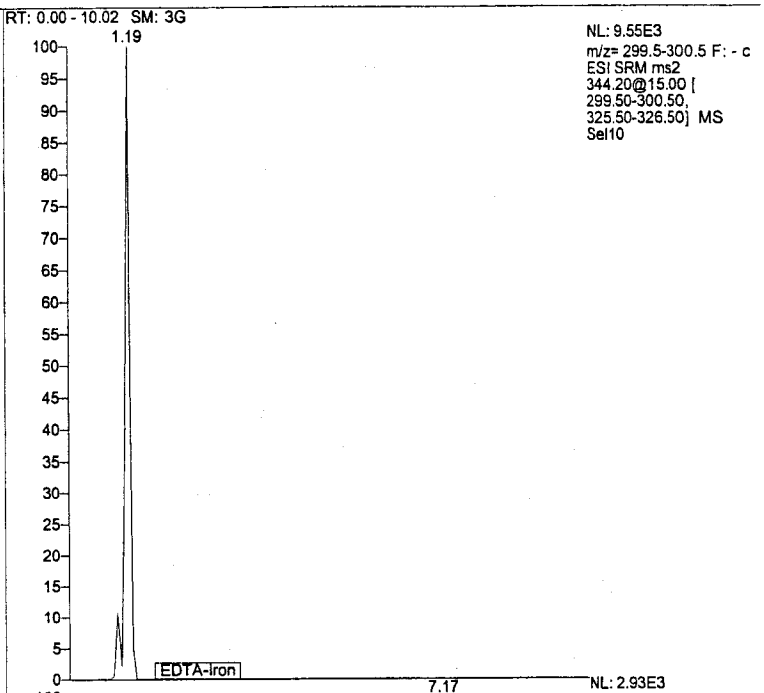
Ex 446
(311)

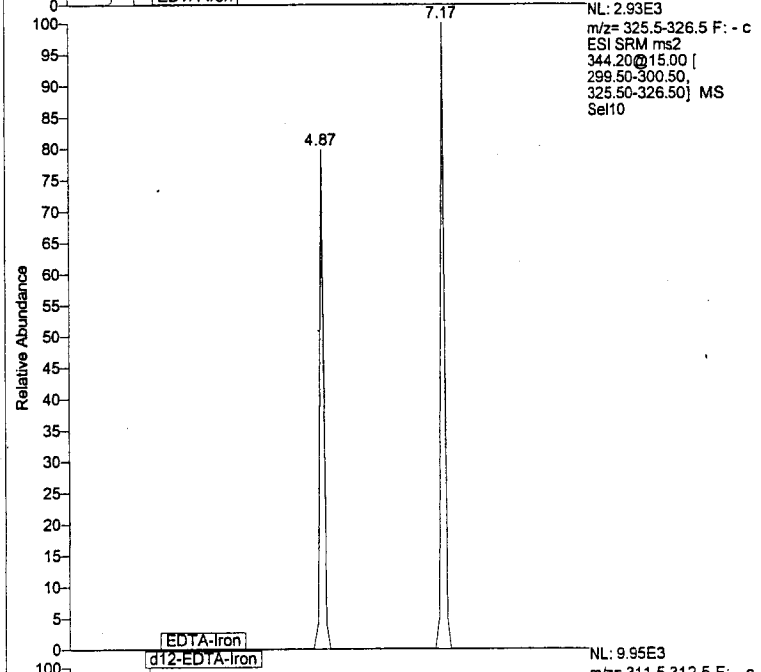

RT: 0.00 - 10.02 SM: 3G
6.82
NL: 7.79E2
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel10
EDTA-Free

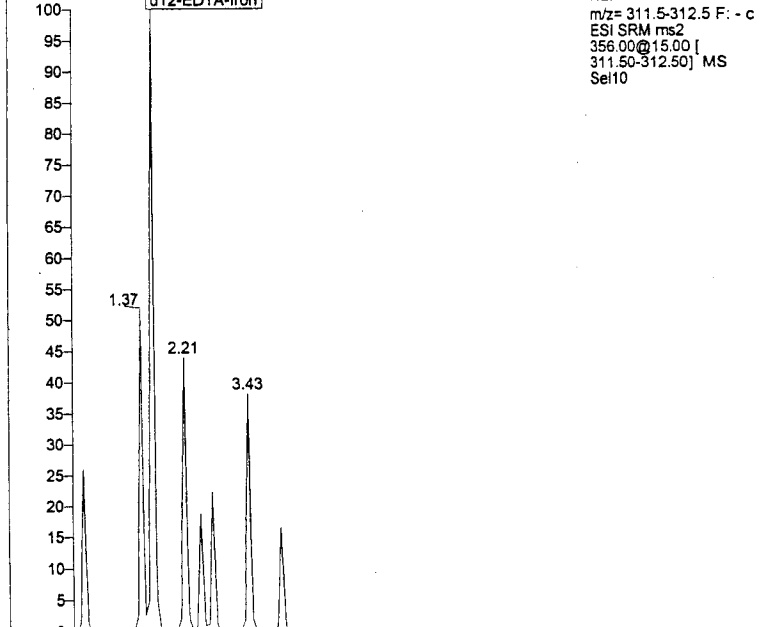

EDTA-Free
NL: 6.13E3
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel10
1.31
2.23
3.61
4.52
7.43
8.43
Relat
tance

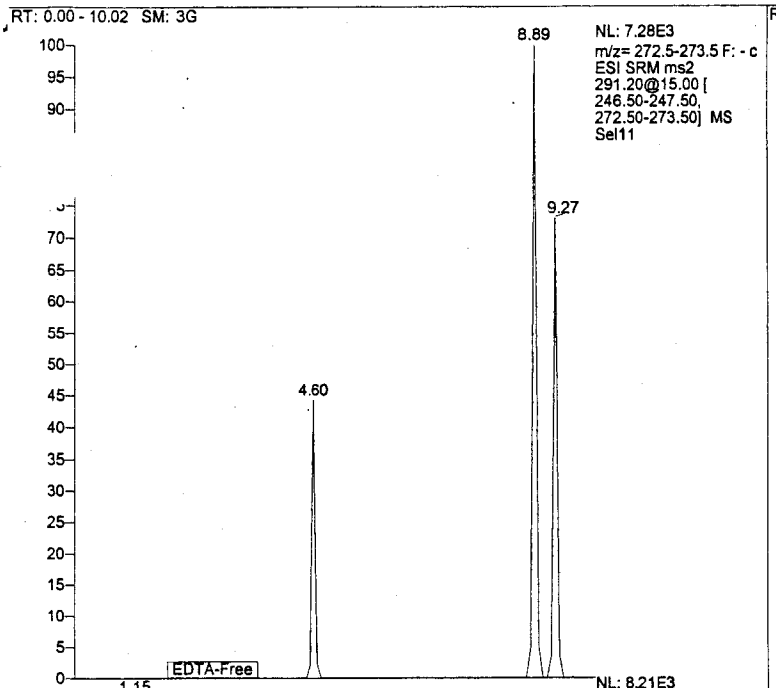

NL: 0
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Sel10
d12-EDTA-Free
0
2
4
6
8
10
Time (min)

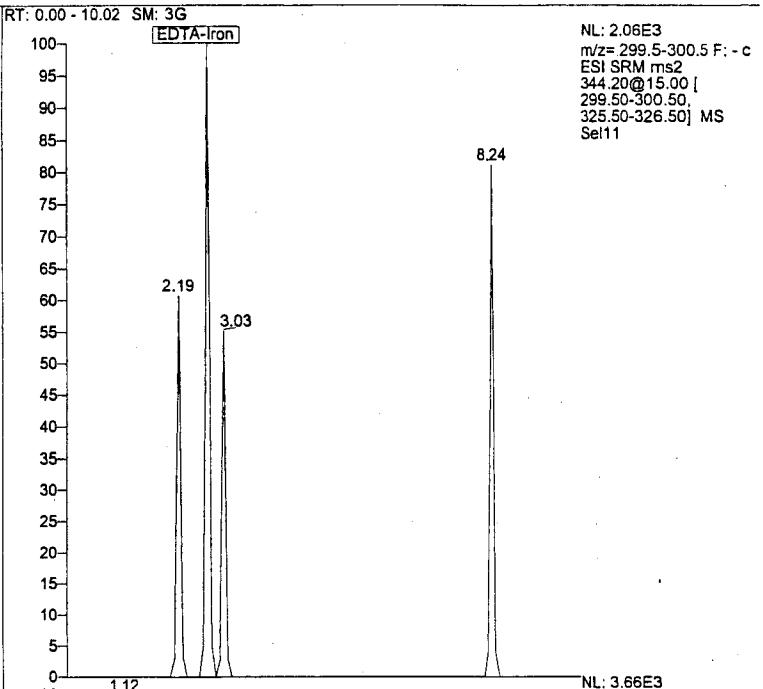
Ex 446 (312)

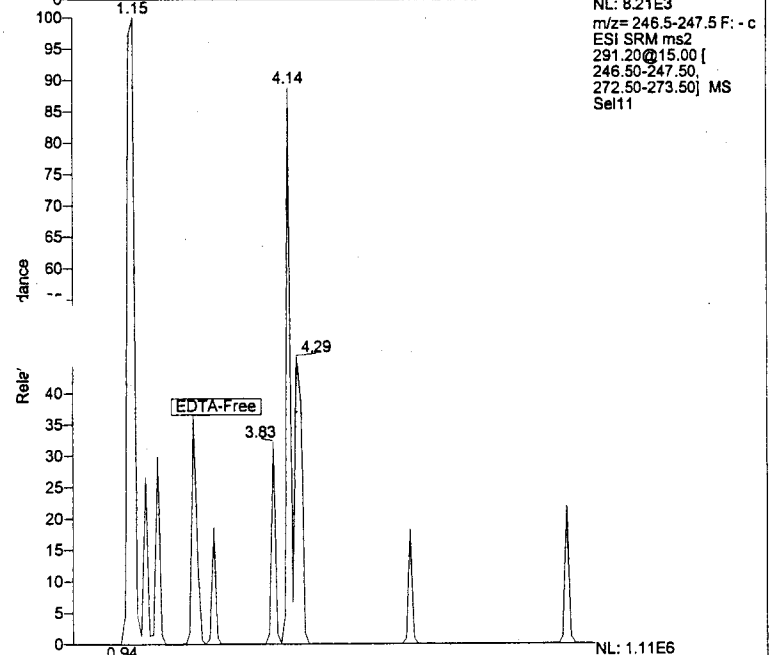

RT: 0.00 - 10.02 SM: 3G
1.19
NL: 9.55E3
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel10
EDTA-Iron

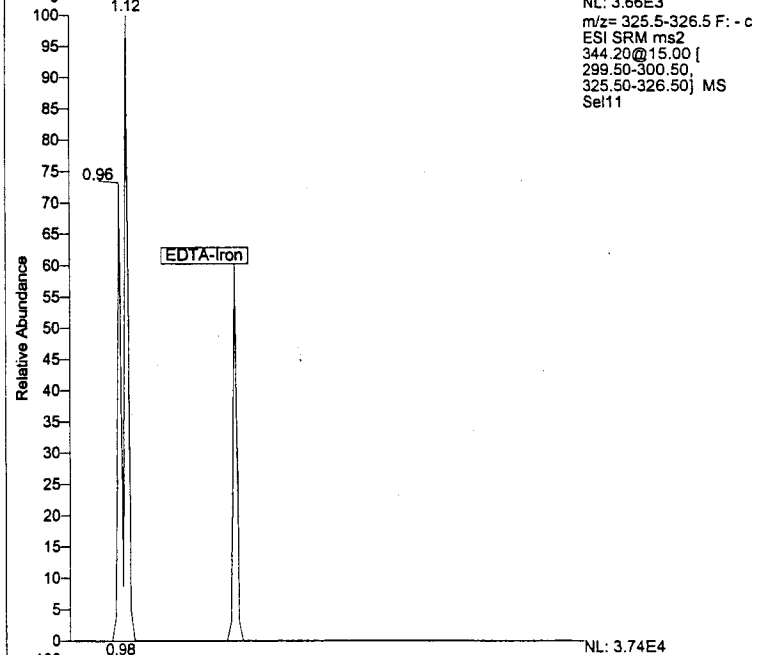

NL: 2.93E3
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel10
4.87
7.17
Relative Abundance

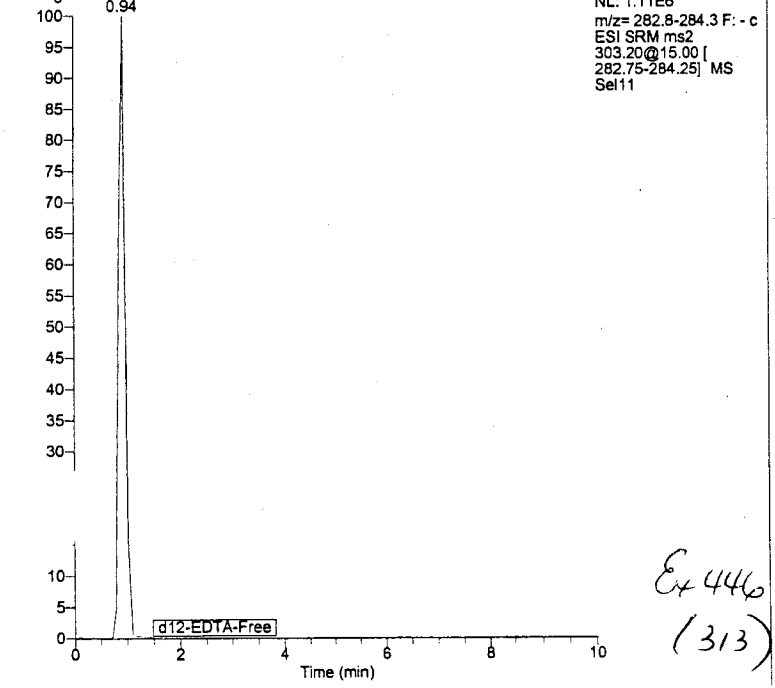

EDTA-Iron
d12-EDTA-Iron
NL: 9.95E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Sel10
1.37
2.21
3.43
0
2
4
6
8
10
Time (min)

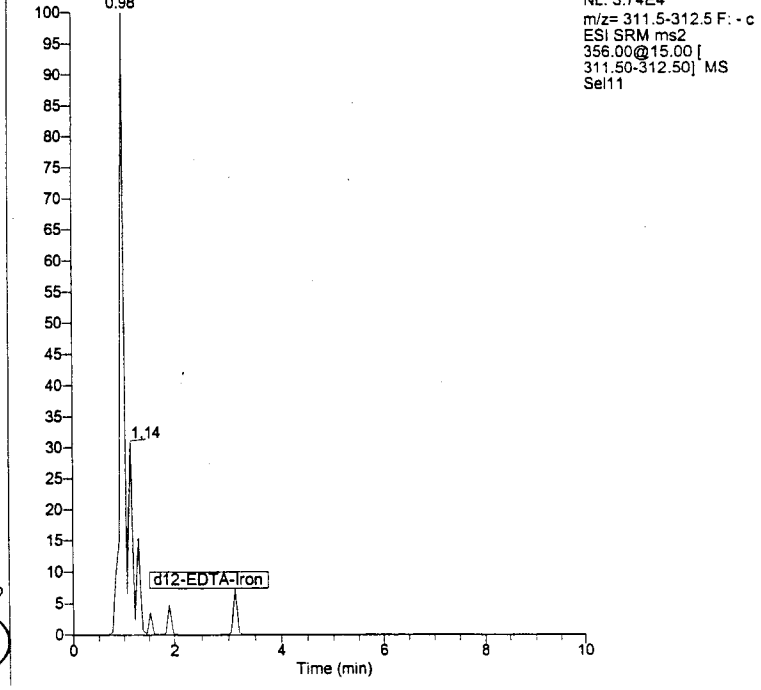

RT: 0.00 - 10.02 SM: 3G
NL: 7.28E3
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel11
8.89
9.27
4.60
EDTA-Free


RT: 0.00 - 10.02 SM: 3G
NL: 2.06E3
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel11
EDTA-Iron
8.24
2.19
3.03

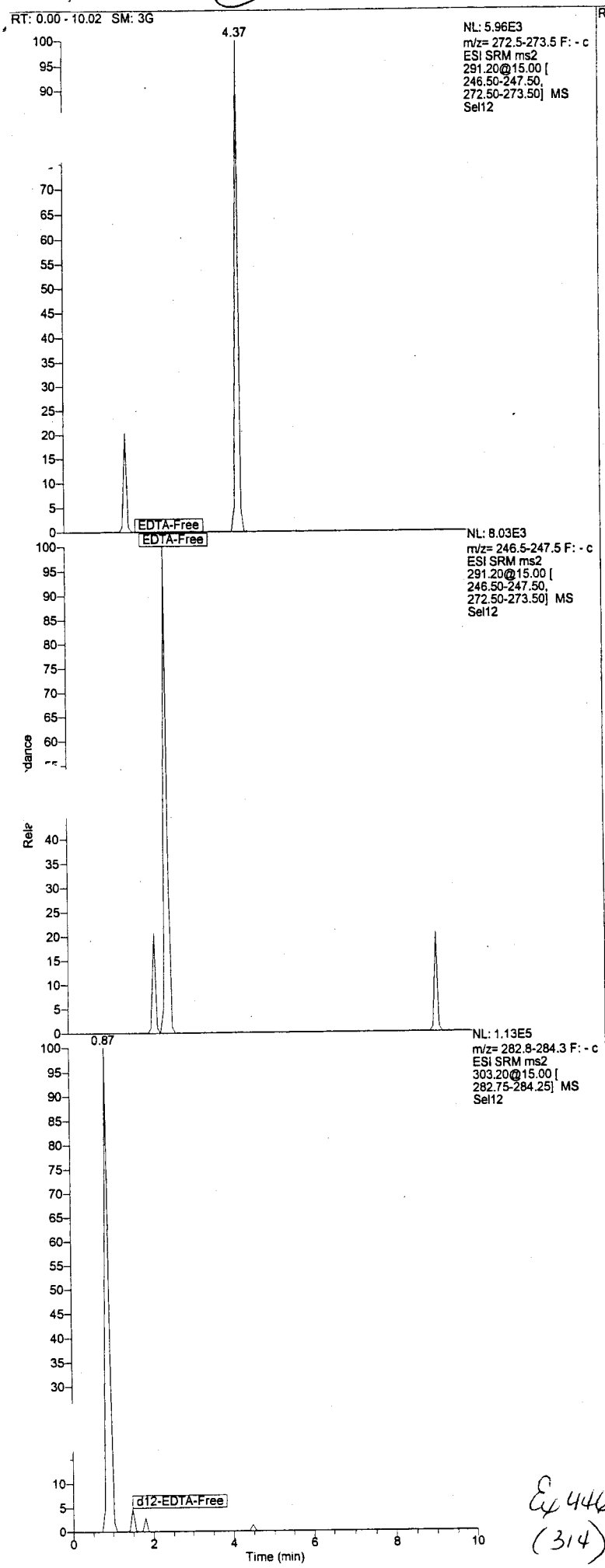

NL: 8.21E3
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel11
1.15
4.14
4.29
3.83
EDTA-Free

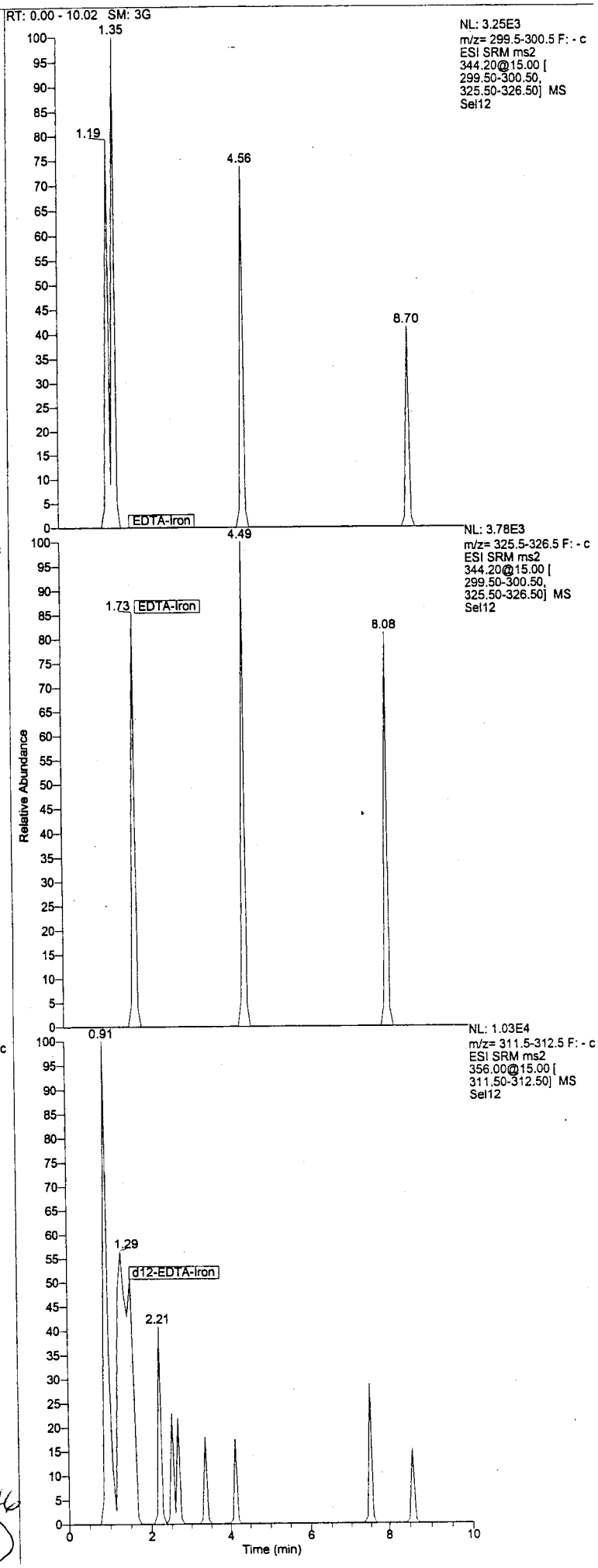

NL: 3.66E3
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel11
1.12
0.96
EDTA-Iron
Relative Abundance


NL: 1.11E6
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Sel11
0.94
d12-EDTA-Free
Time (min)


NL: 3.74E4
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Sel11
0.98
1.14
d12-EDTA-Iron
Time (min)

Ex 446
(313)


RT: 0.00 - 10.02 SM: 3G
NL: 5.96E3
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel12
4.37
EDTA-Free
NL: 8.03E3
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Sel12
EDTA-Free
NL: 1.13E5
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Sel12
0.87
d12-EDTA-Free
Time (min)


RT: 0.00 - 10.02 SM: 3G
NL: 3.25E3
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel12
1.35
1.19
4.56
8.70
EDTA-Iron
NL: 3.78E3
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Sel12
4.49
1.73
EDTA-Iron
8.08
Relative Abundance
NL: 1.03E4
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Sel12
0.91
1.29
d12-EDTA-Iron
2.21
Time (min)

Eq 446 (314)

L.O.D.

Ex 446
(315)

Sequence---LOD_neg.sld [Open]

Sample Name: 100 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD01	1	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 100 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD02	01	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(316)

JDP
2/12/07

Sequence---LOD_neg.sld [Open]

Sample Name: 100 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD03	01	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD04	02	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		2	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(317)

JDB
2/12/07

Sequence---LOD_neg.sld [Open]

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD05	02	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		2	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD06	02	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		2	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(318)

JOB
2/12/07

Sequence---LOD_neg.sld [Open]

Sample Name: 25 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD07	03	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		3	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 25 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD08	03	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		3	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(319)

JnP
2/12/07

Sequence---LOD_neg.sld [Open]

Sample Name: 25 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD09	03	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		3	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 13 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD10	04	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		4	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(320)

JPB
2/12/07

Sequence---LOD_neg.sld [Open]

Sample Name: 13 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD11	04	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		4	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 13 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD12	04	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		4	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(321)

JDB
2/12/07

Sequence---LOD_neg.sld [Open]

Sample Name: 6 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD13	05	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		5	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 6 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD14	05	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		5	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(322)

JOB
2/12/07

Sequence---LOD_neg.sld [Open]

Sample Name: 6 ppm EDTA

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD15	05	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

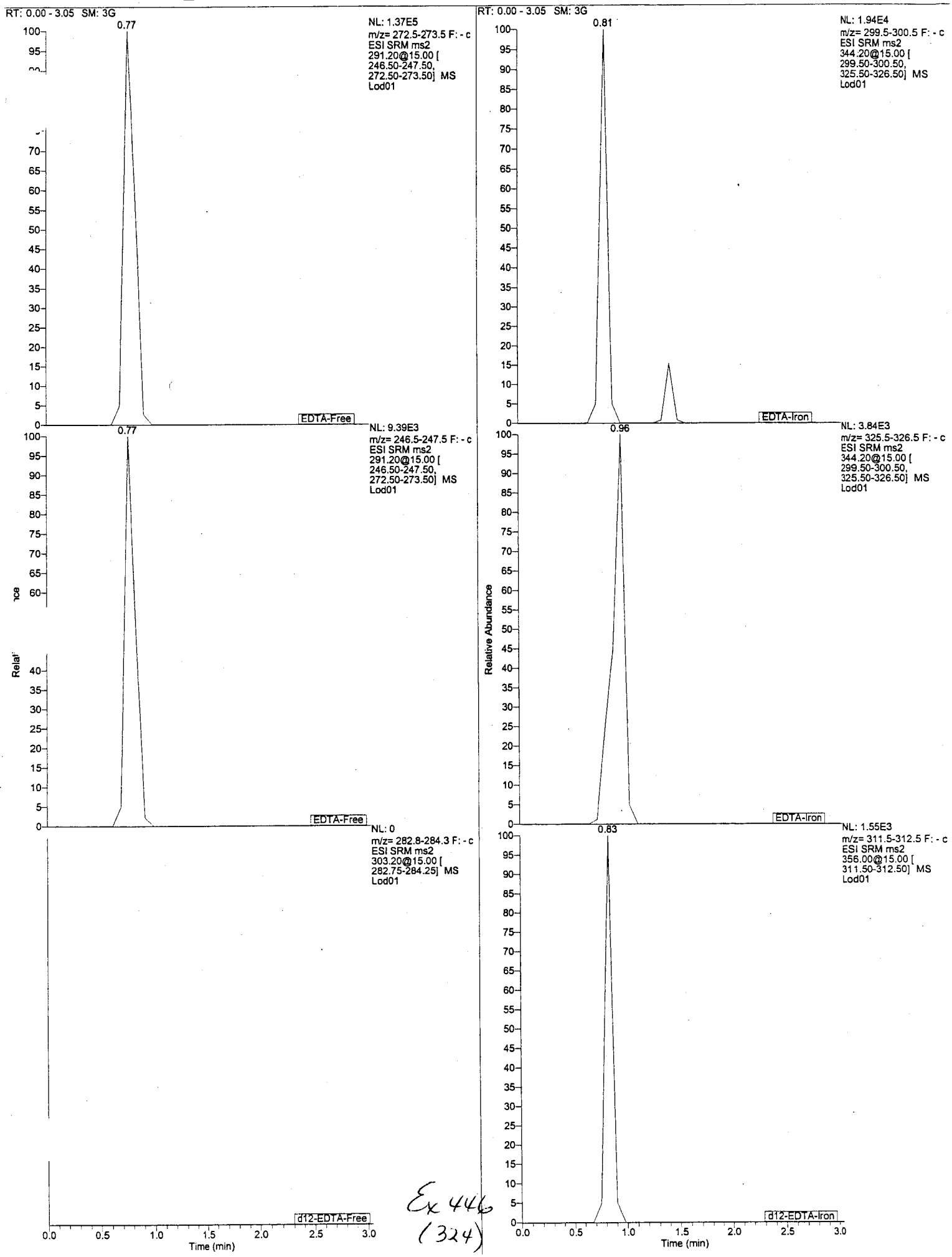
Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		5	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

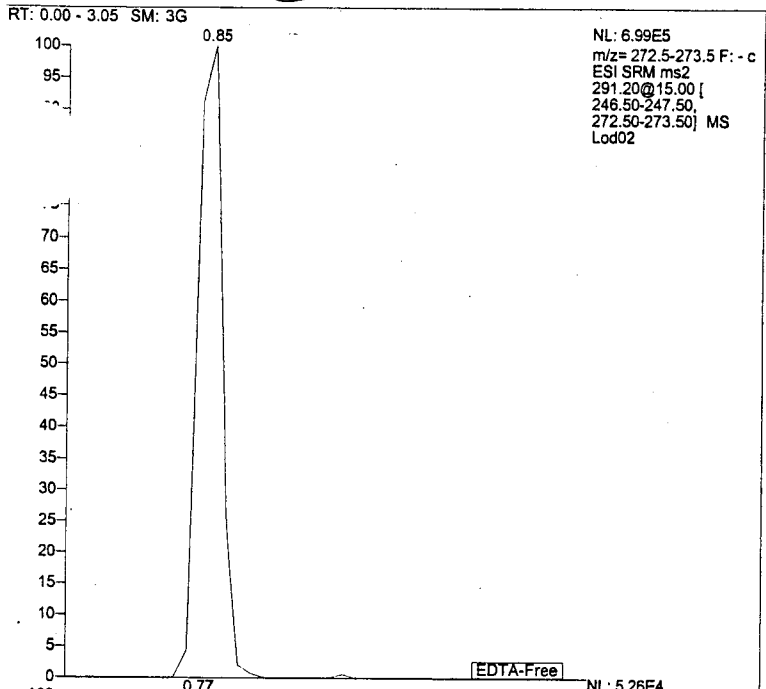
Ex 446
(323)

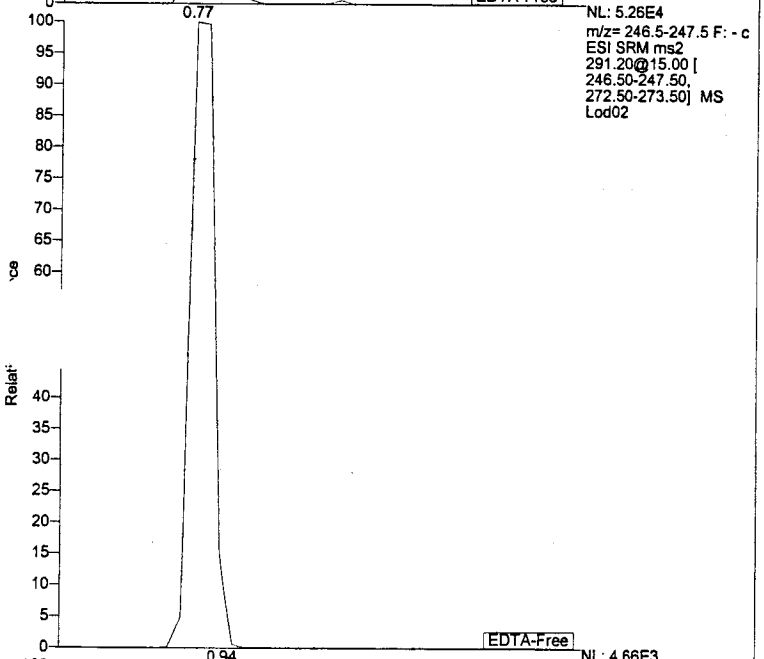
JDP
2/12/07

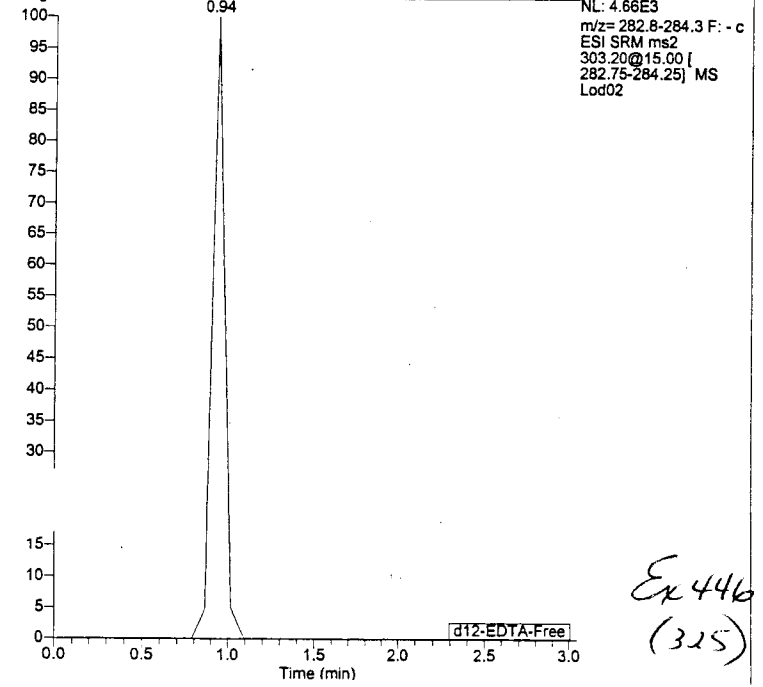

RT: 0.00 - 3.05 SM: 3G
0.77
NL: 1.37E5
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Lod01
EDTA-Free
NL: 9.39E3
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Lod01
EDTA-Free
NL: 0
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Lod01
d12-EDTA-Free
Time (min)
RT: 0.00 - 3.05 SM: 3G
0.81
NL: 1.94E4
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Lod01
EDTA-Iron
0.96
NL: 3.84E3
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Lod01
EDTA-Iron
Relative Abundance
0.83
NL: 1.55E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Lod01
d12-EDTA-Iron
Time (min)

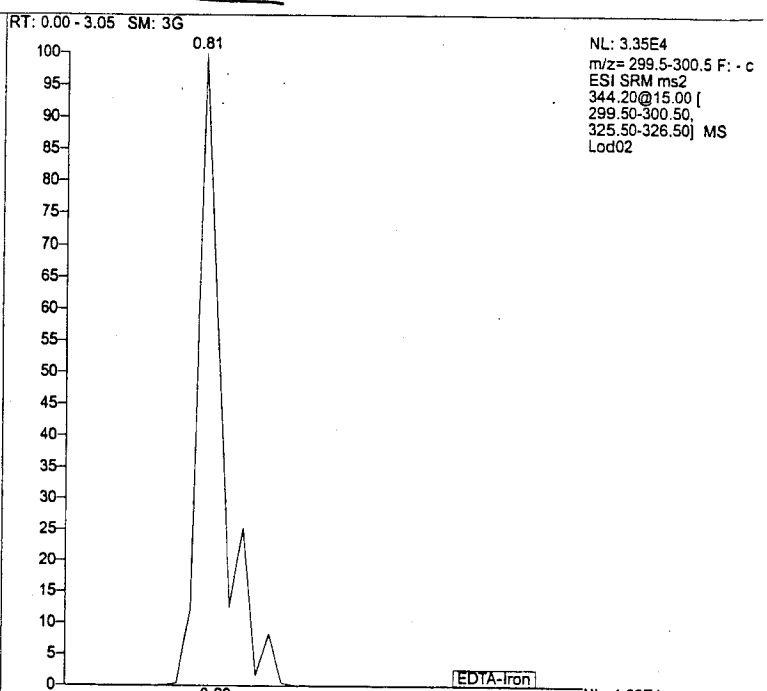
Ex 446
(324)

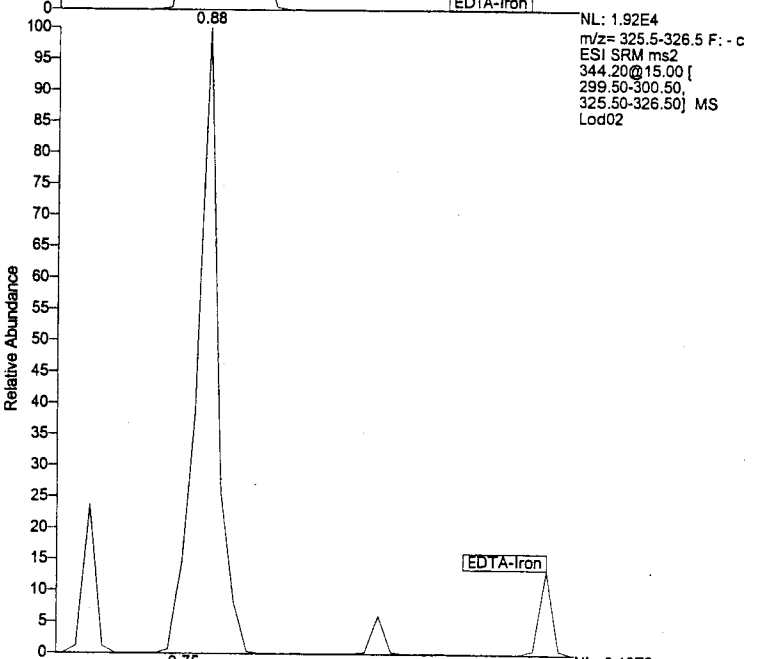
C:\Xcalibur\...\LOD_Negative\Lod02 02/12/07 12:45:27 100 ppm EDTA

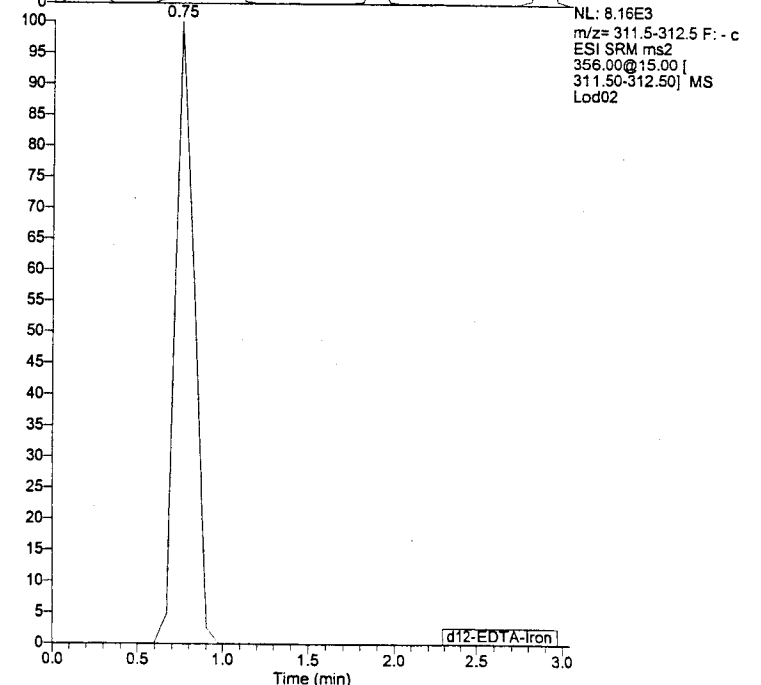






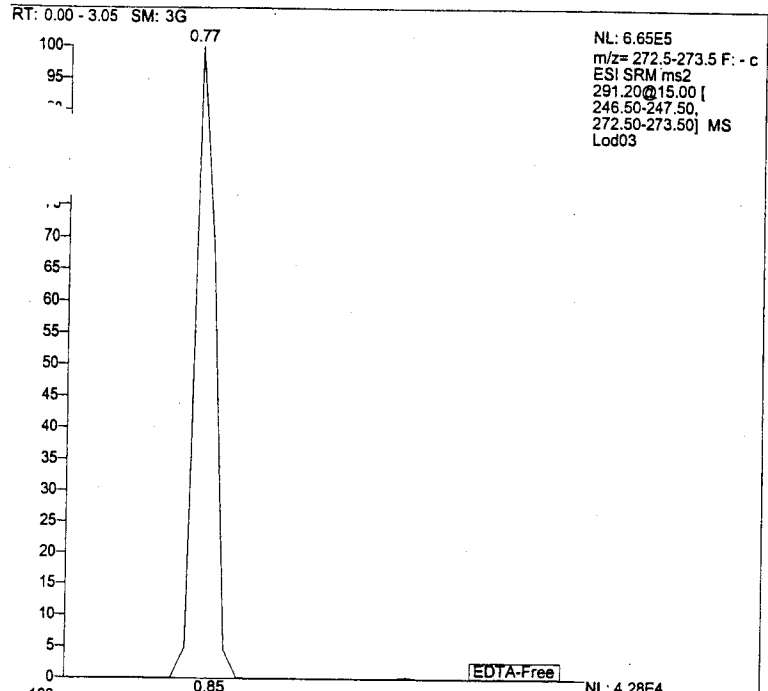


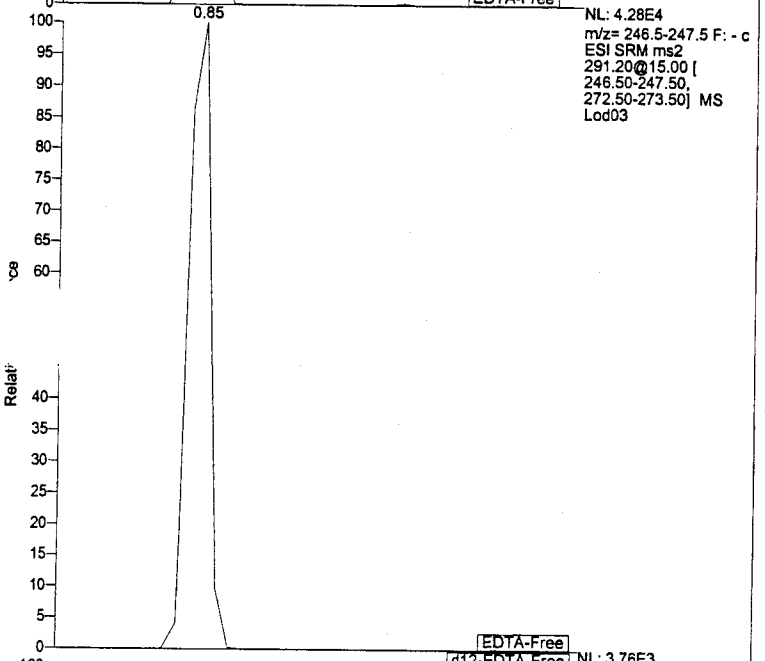


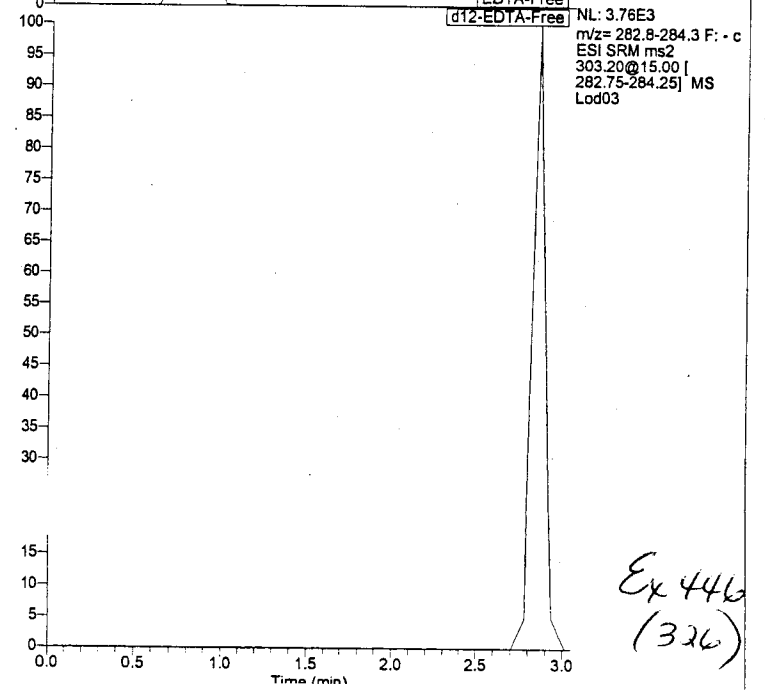


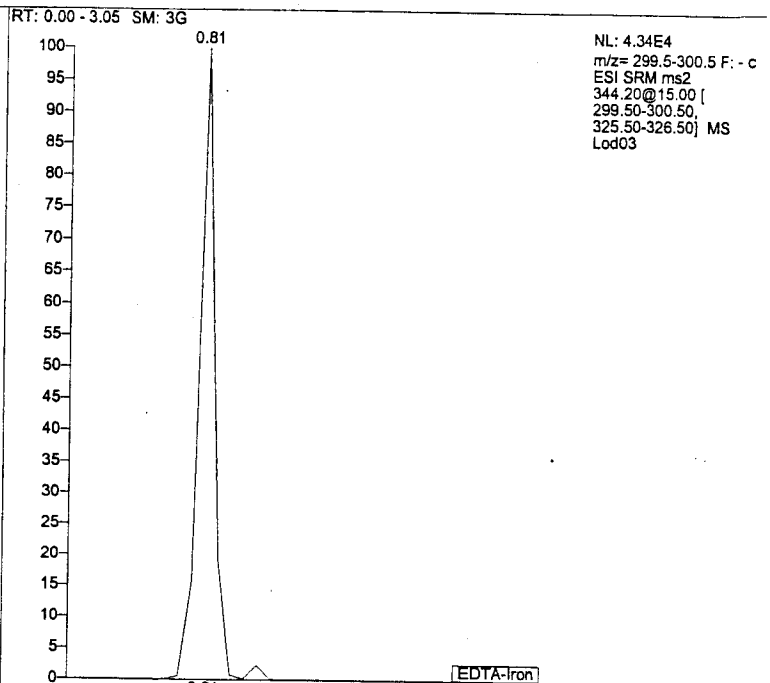
Ex 446
(325)

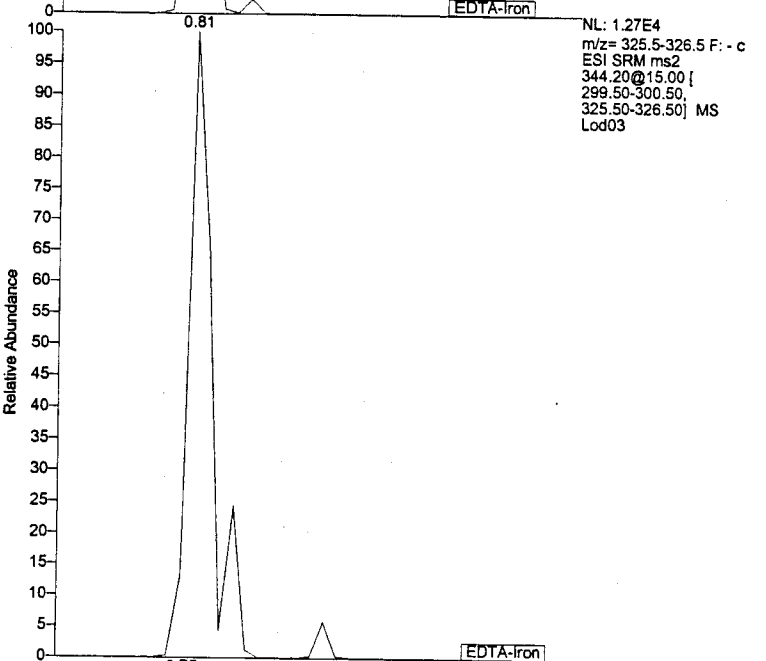
C:\Xcalibur\...\LOD_Negative\Lod03 02/12/07 12:49:20 100 ppm EDTA JDB

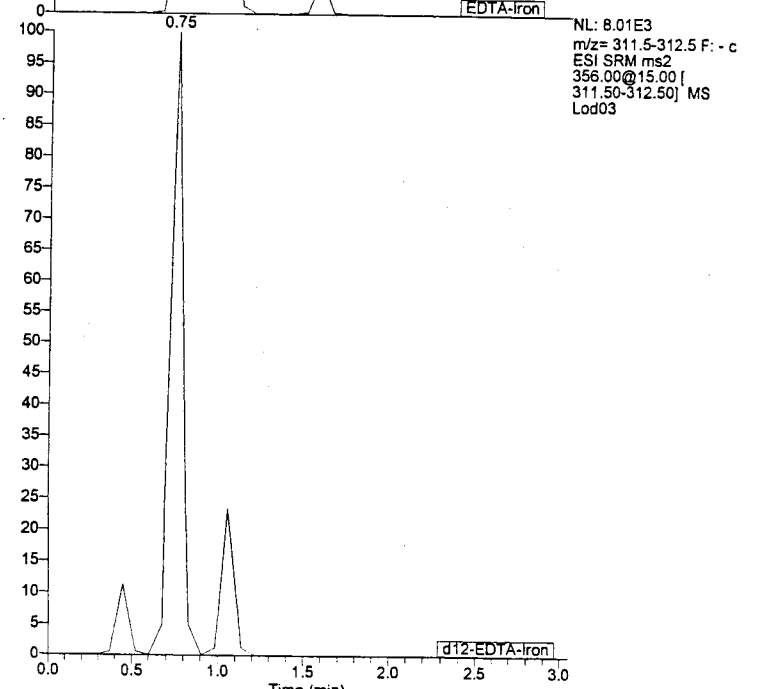






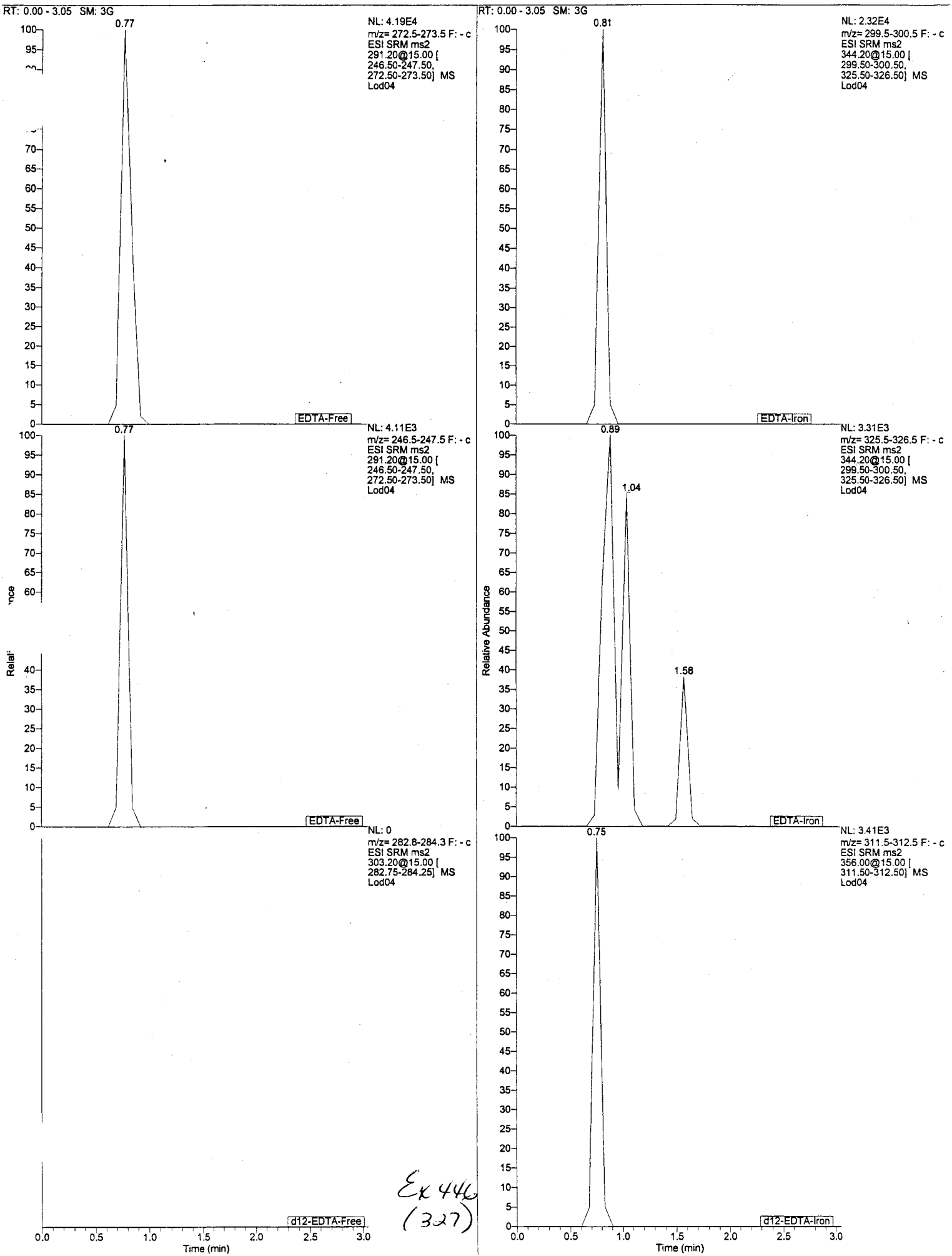






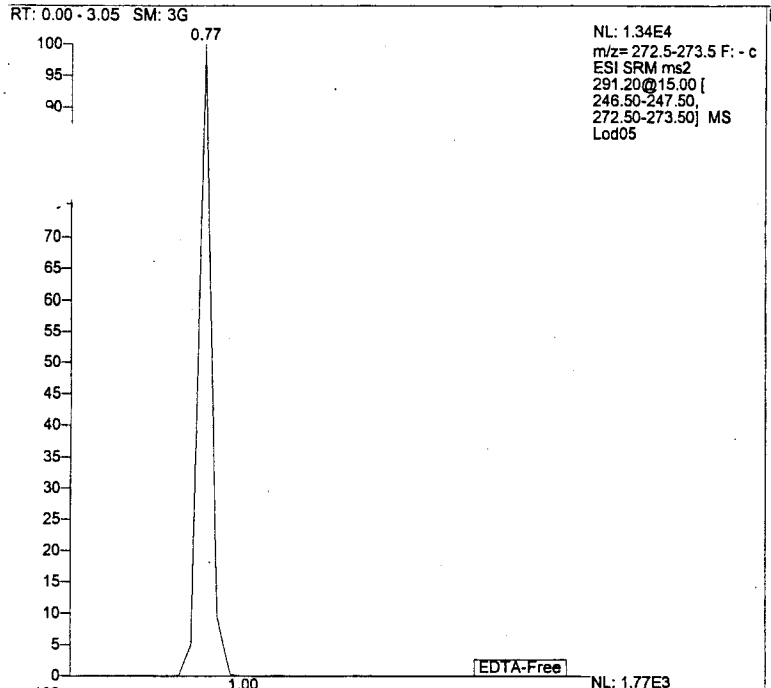
Ex 446
(326)

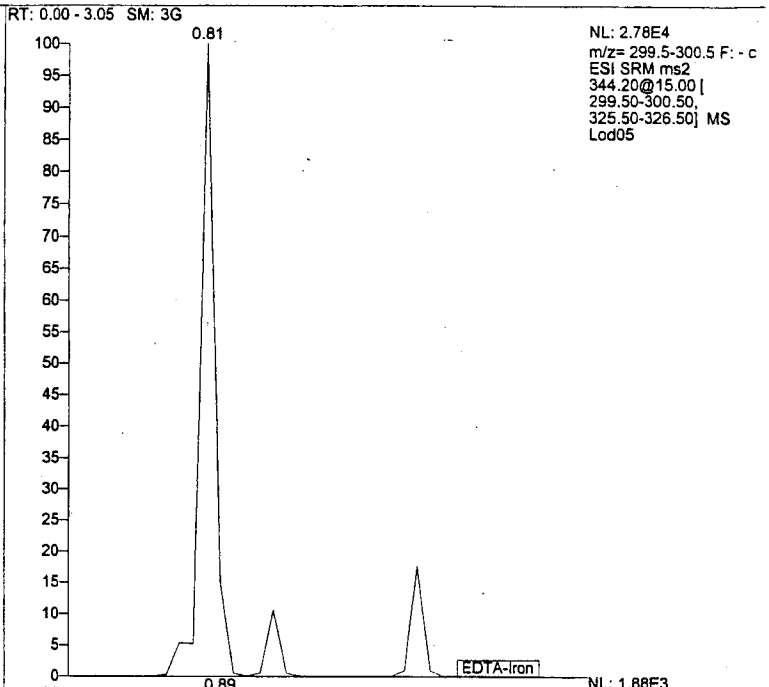
C:\Xcalibur\...\LOD_Negative\Lod04 02/12/07 12:53:15 50 ppm EDTA JPB

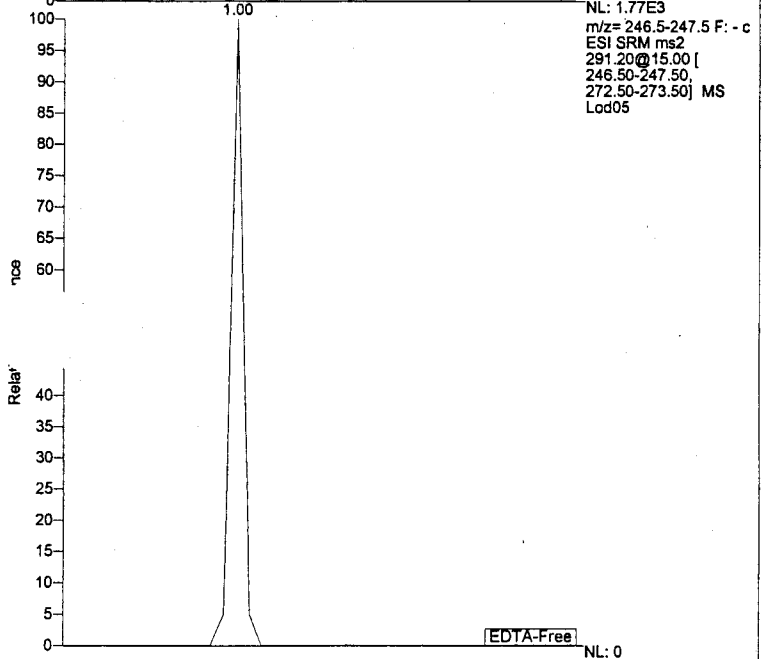


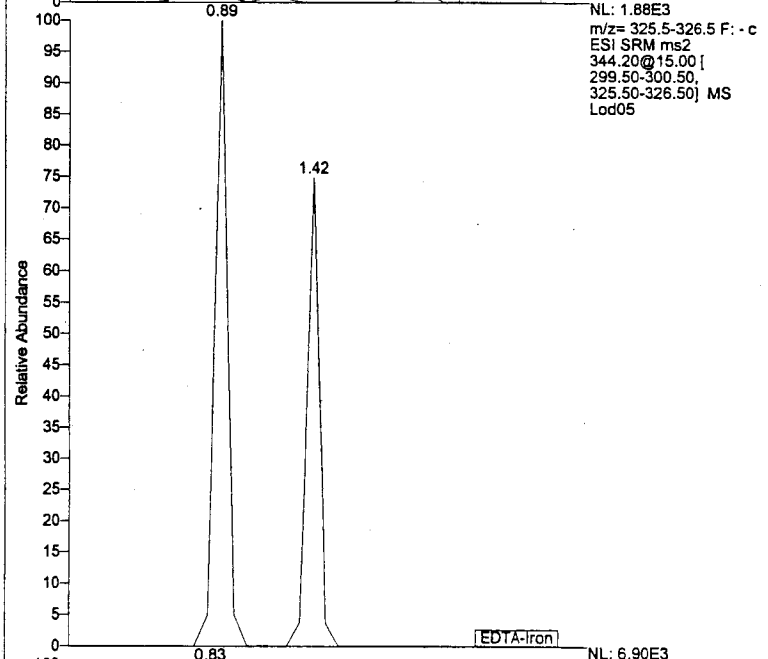
Ex 446
(327)

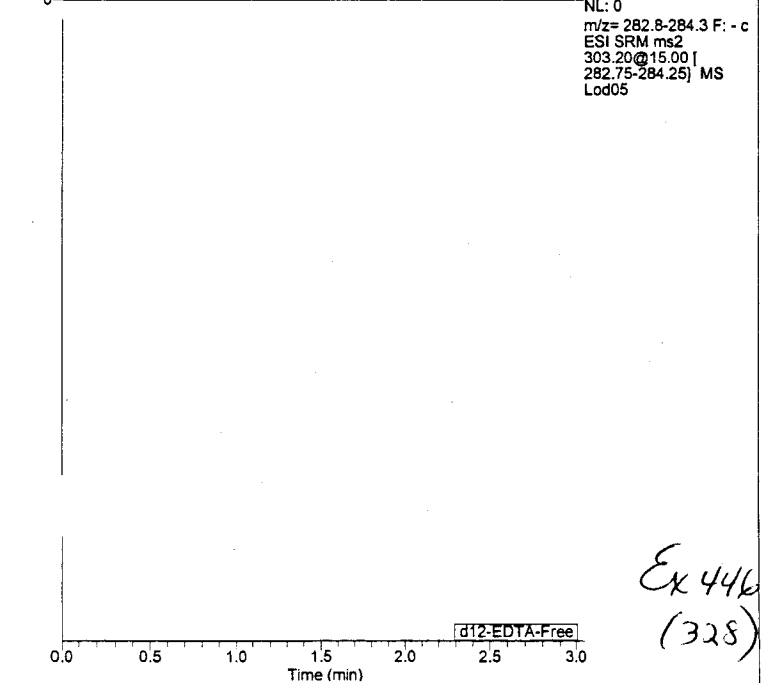
C:\Xcalibur\...\LOD_Negative\Lod05 02/12/07 12:57:07 50 ppm EDTA JOP

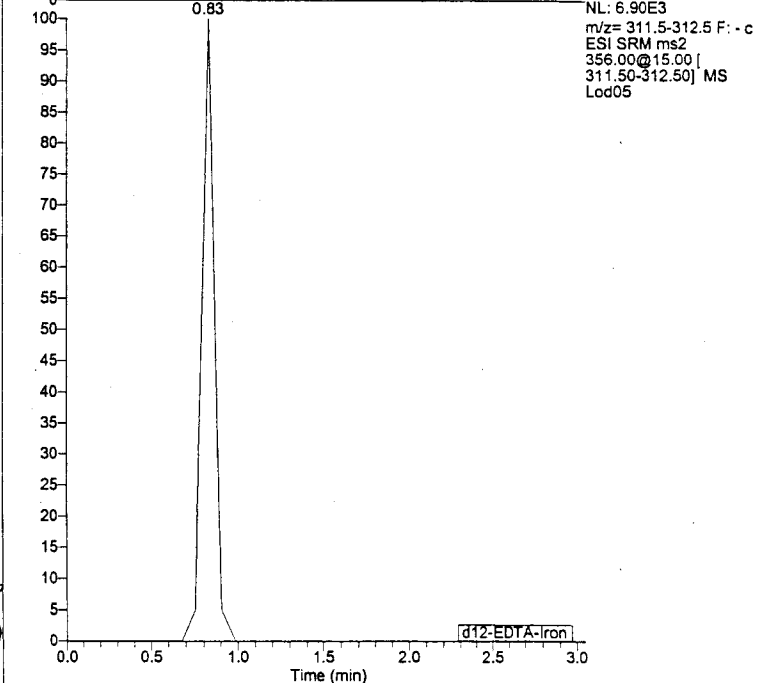






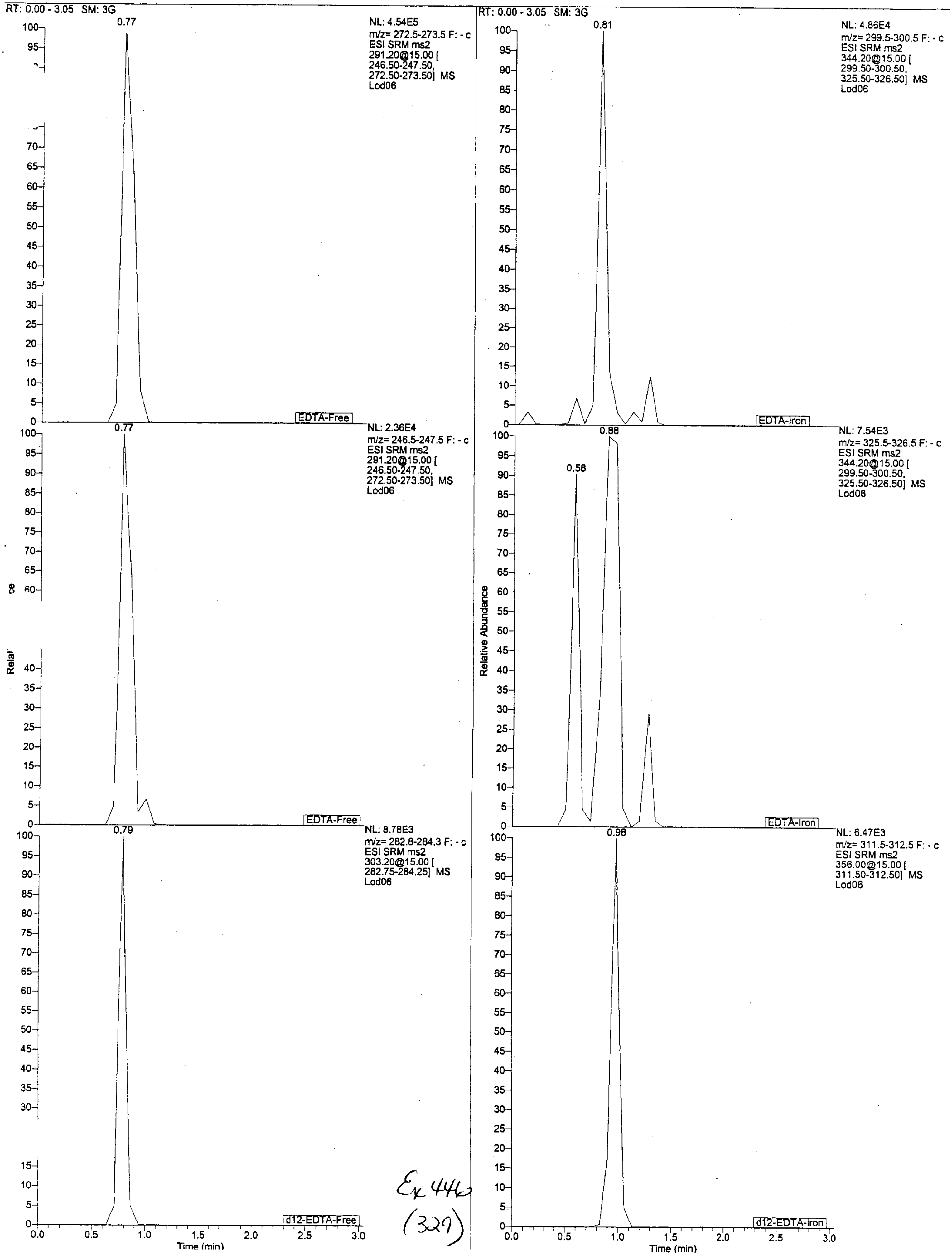






Ex 446
(328)

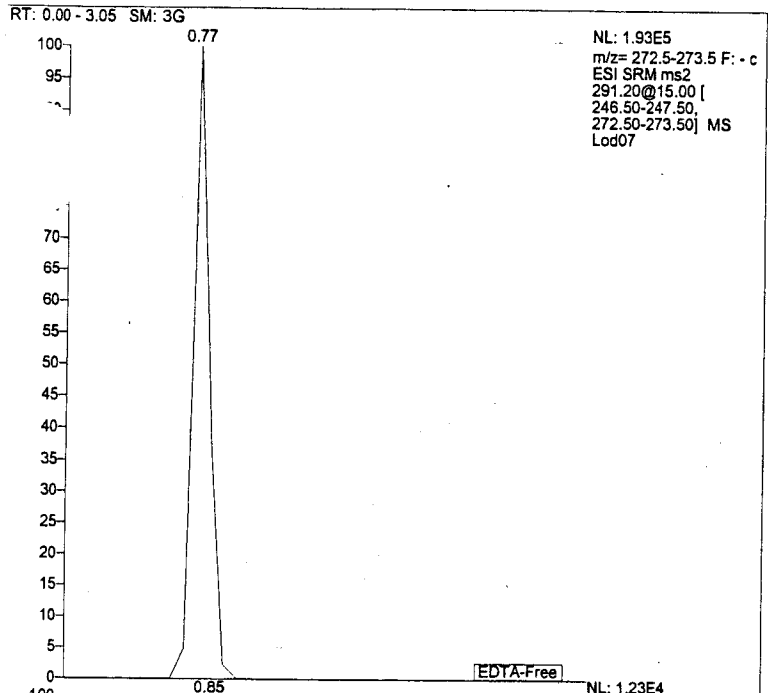
C:\Xcalibur\...\LOD_Negative\Lod06 02/12/07 01:01:00 50 ppm EDTA JbP

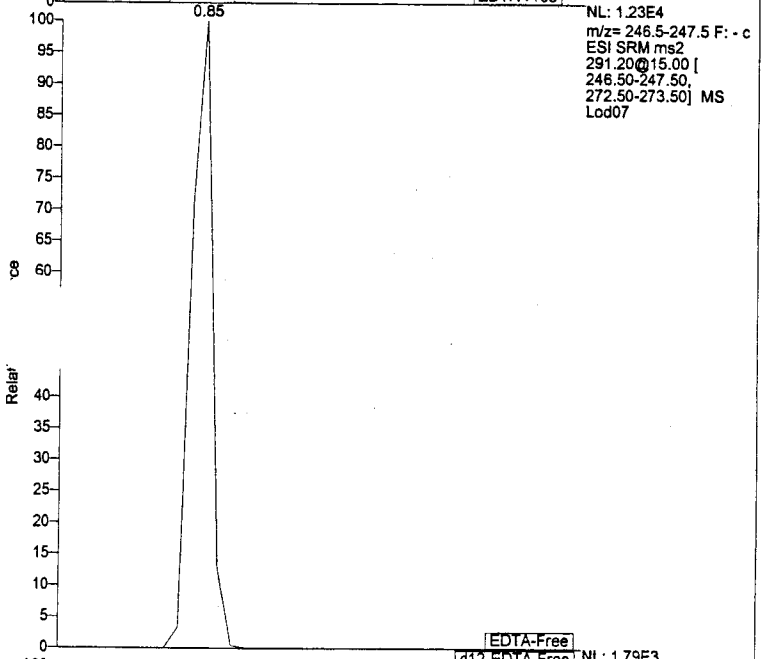


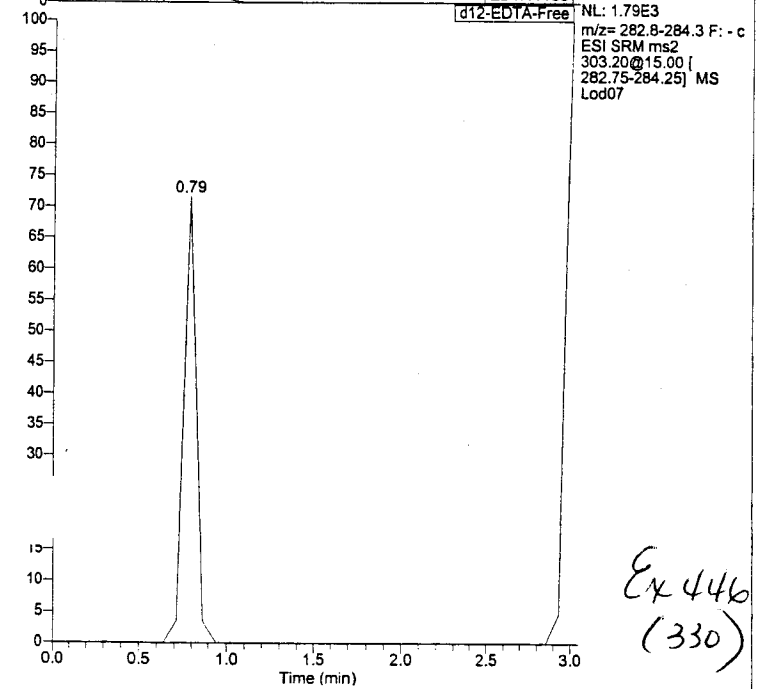
Ex 446

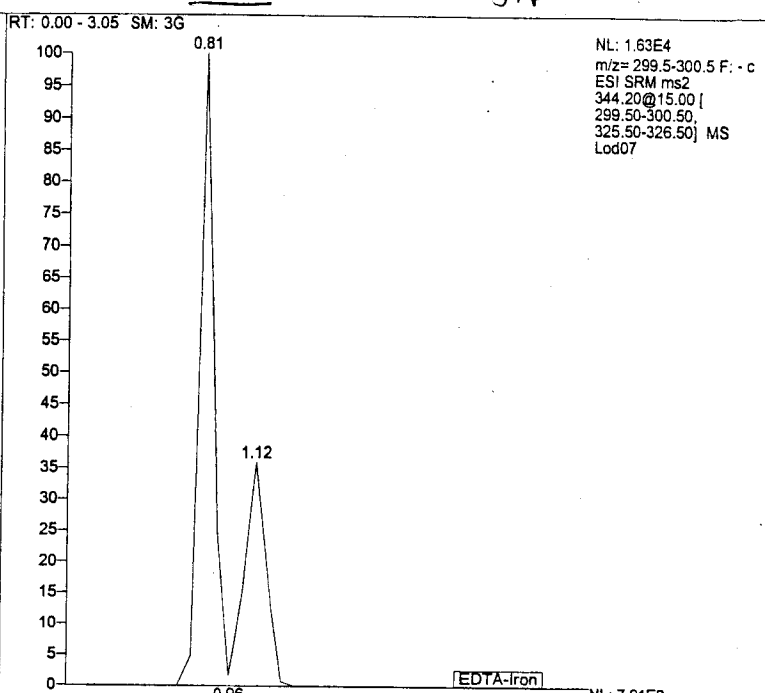
(329)

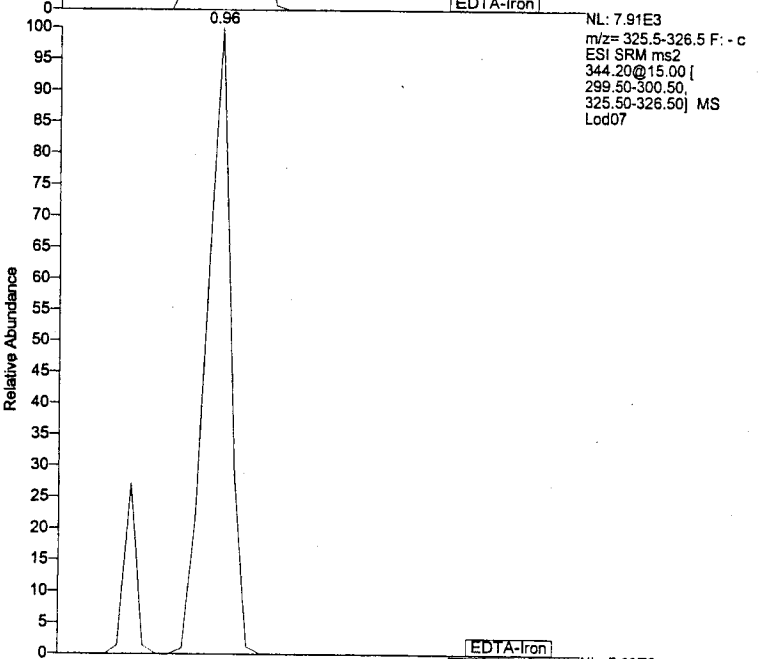
C:\Xcalibur\...\LOD_Negative\Lod07 02/12/07 01:04:53 25 ppm EDTA JPB

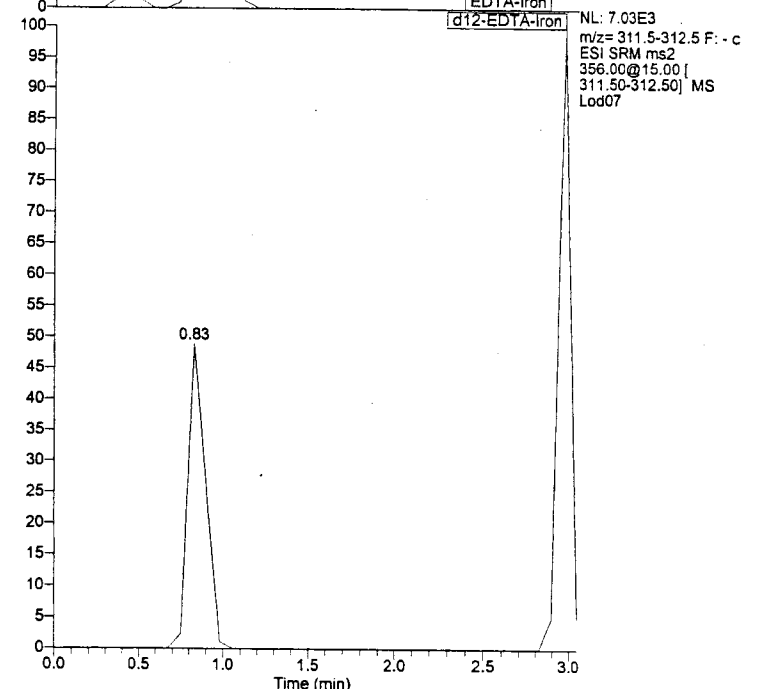






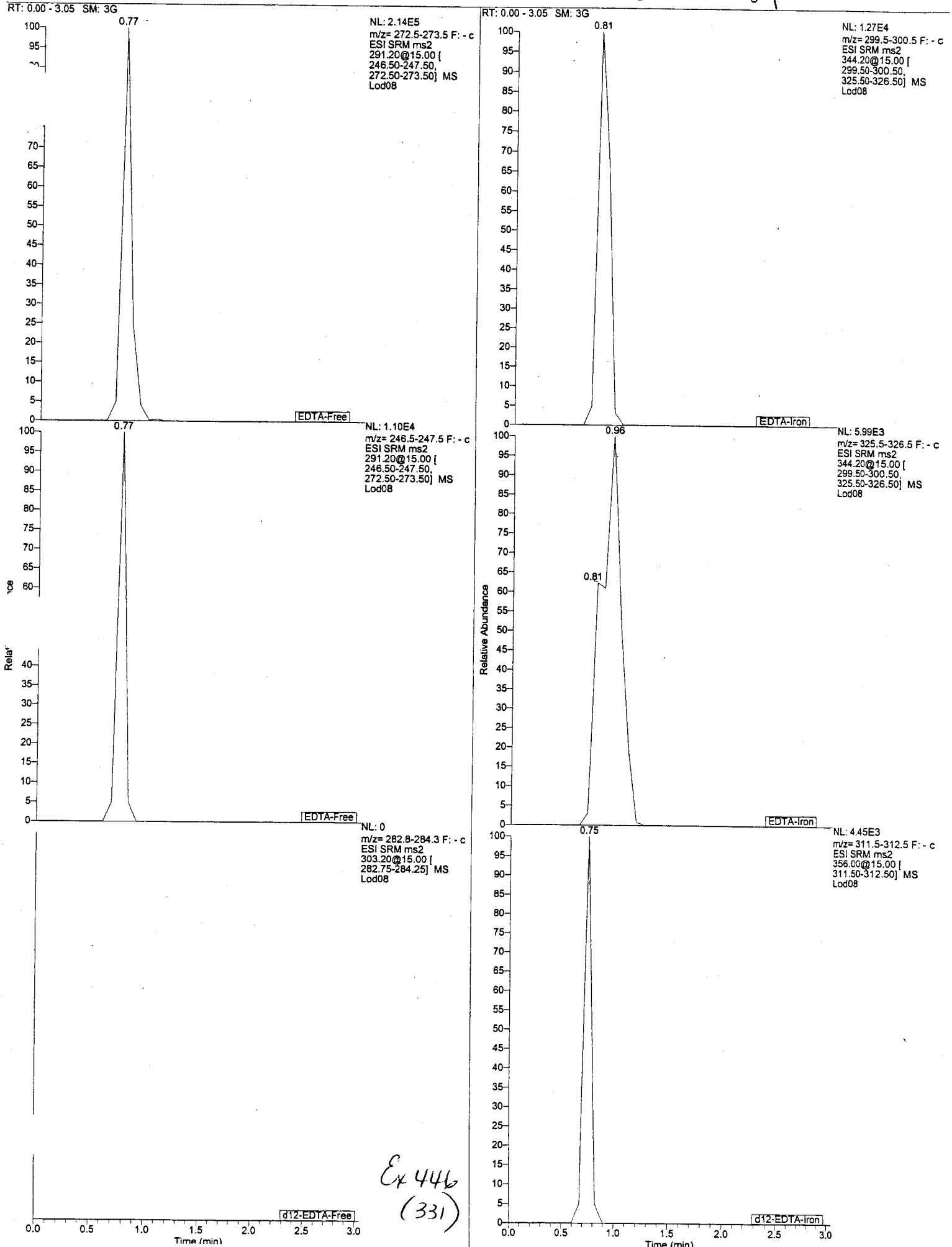






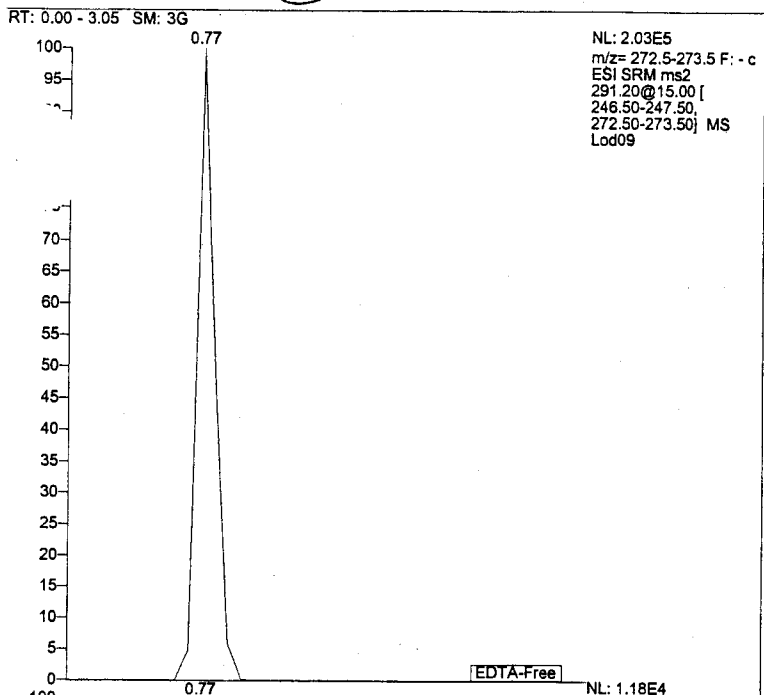
Ex 446
(330)

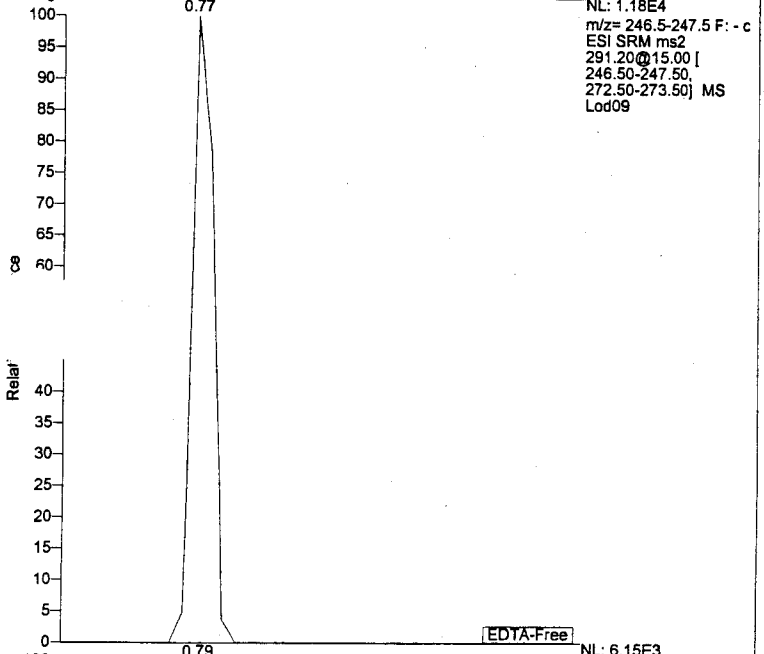
C:\Xcalibur\...\LOD_Negative\Lod08 02/12/07 01:08:47 25 ppm EDTA JDB

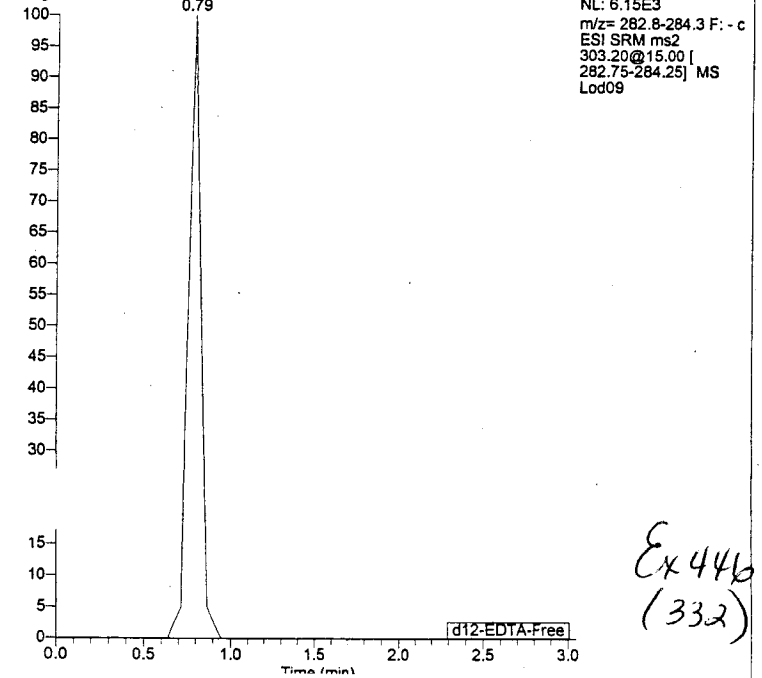


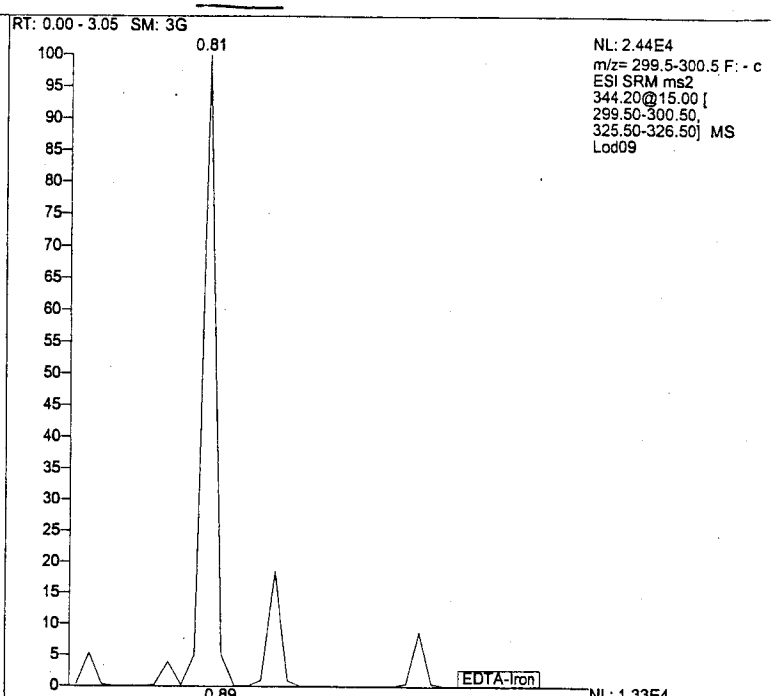
Ex 446
(331)

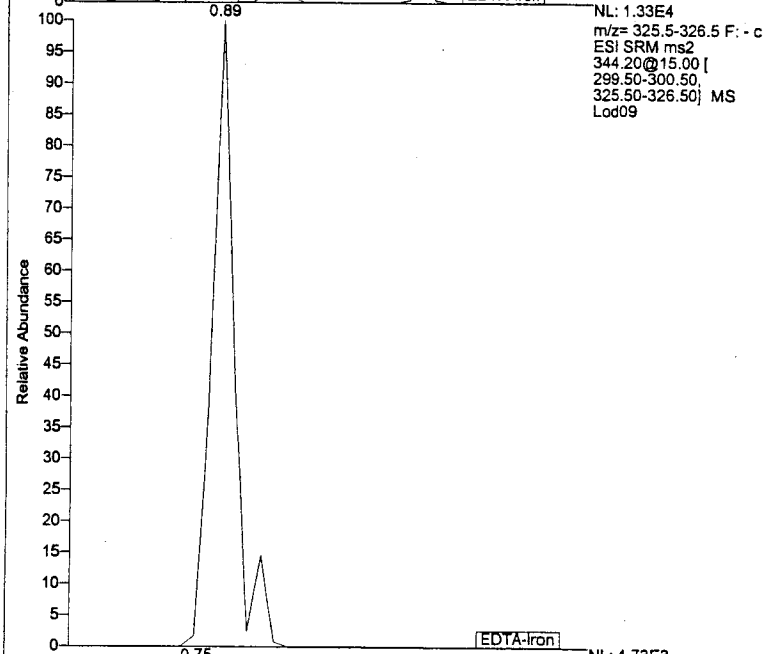
C:\Xcalibur\...\LOD_Negative\Lod09 02/12/07 01:12:45 25 ppm EDTA JDB

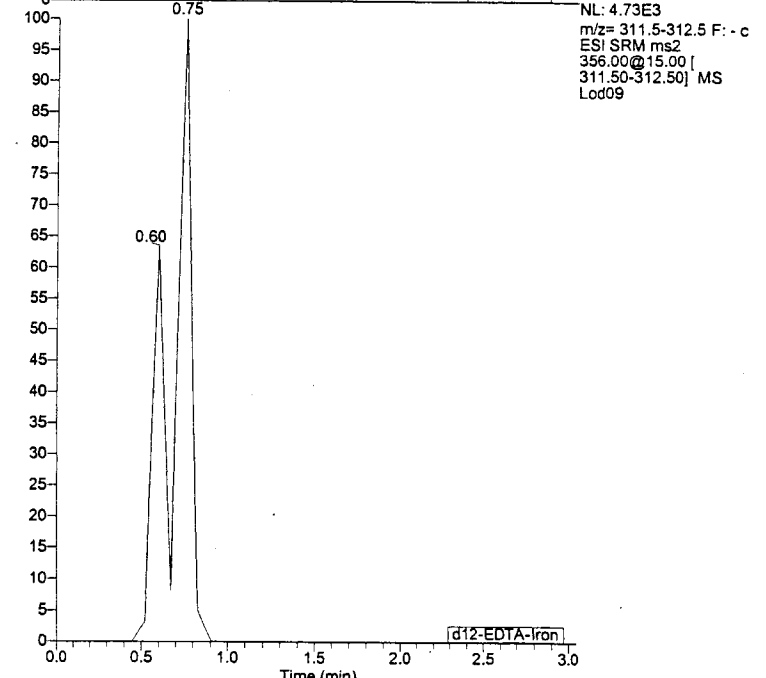






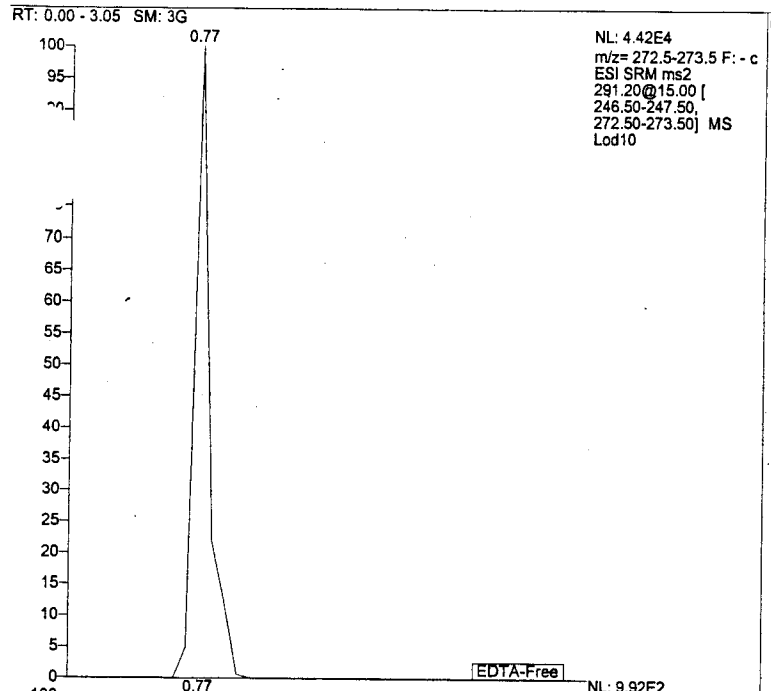


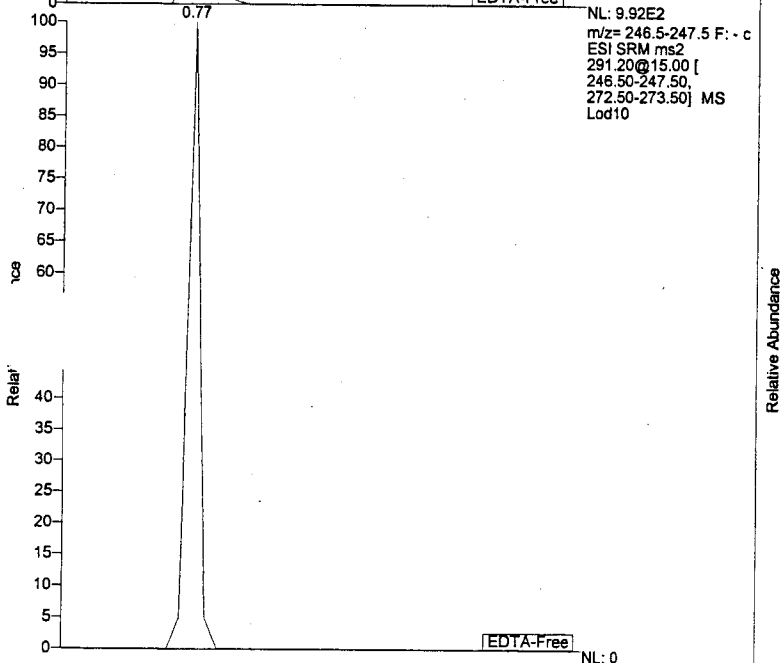


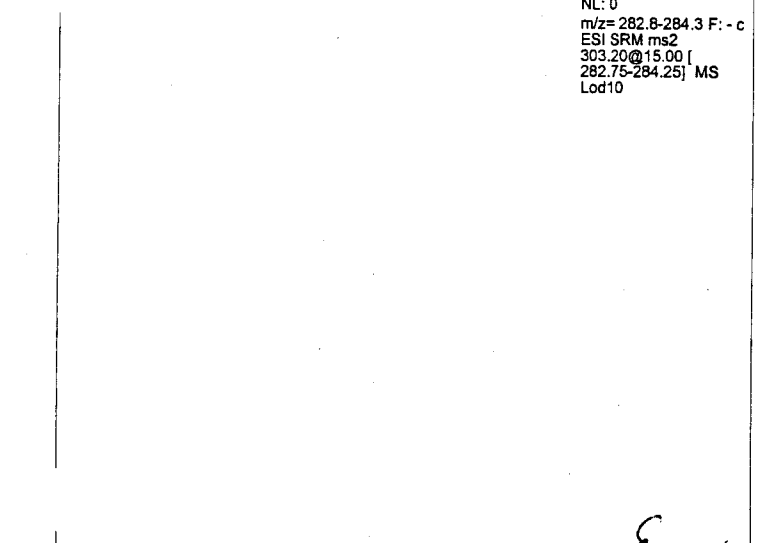


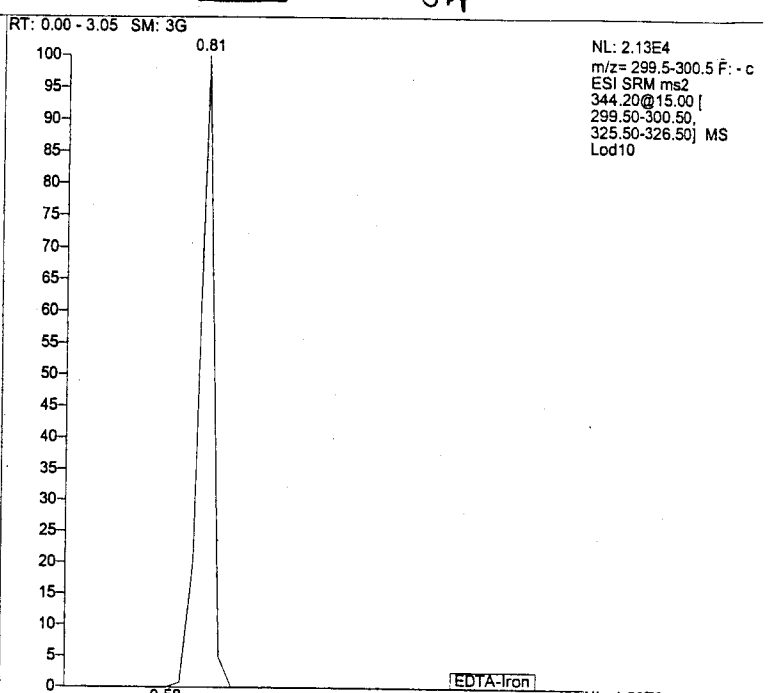
Ex 446
(332)

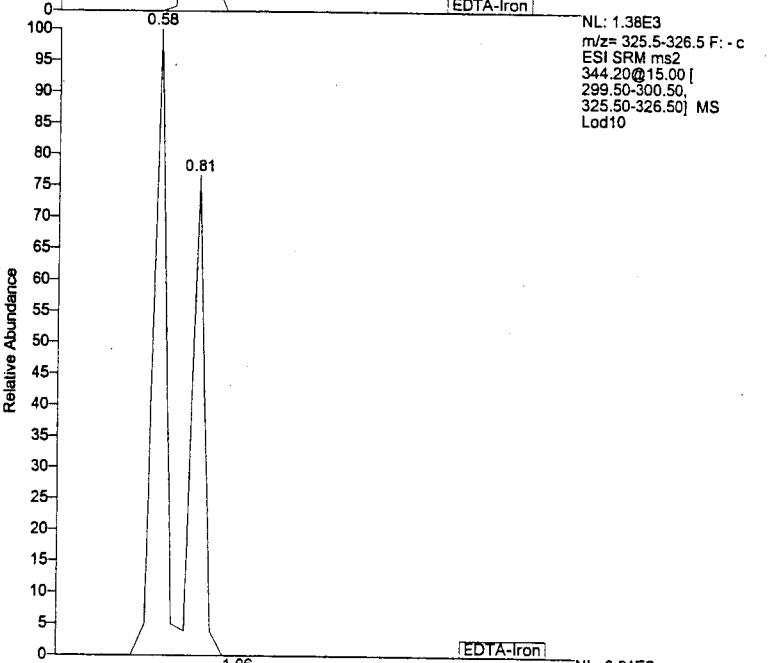
C:\Xcalibur\...\LOD_Negative\Lod10 02/12/07 01:16:37 13 ppm EDTA JDB

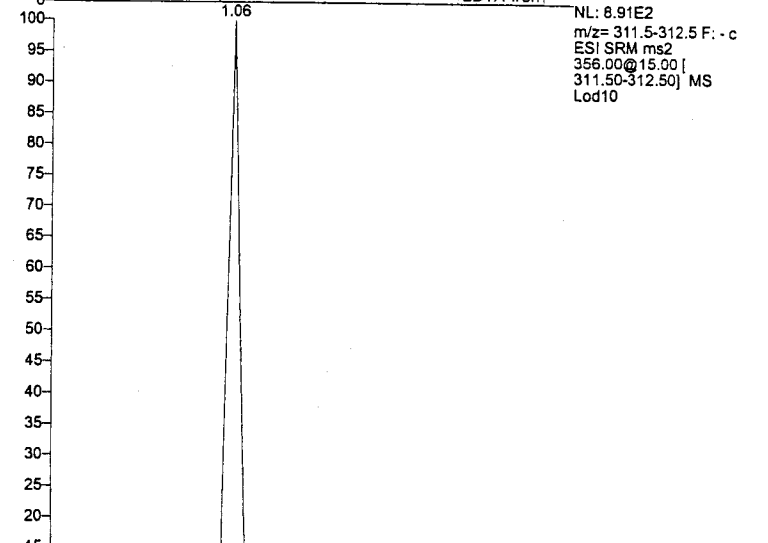






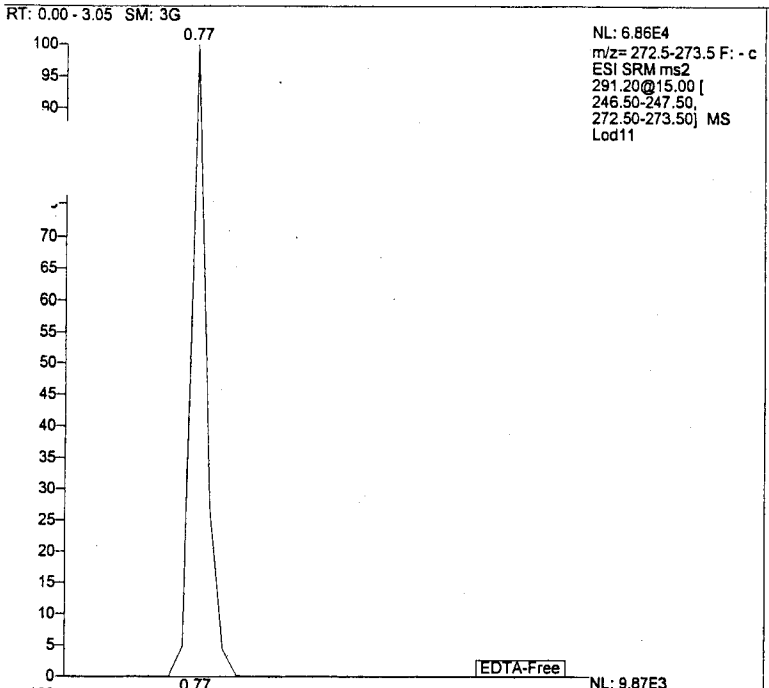


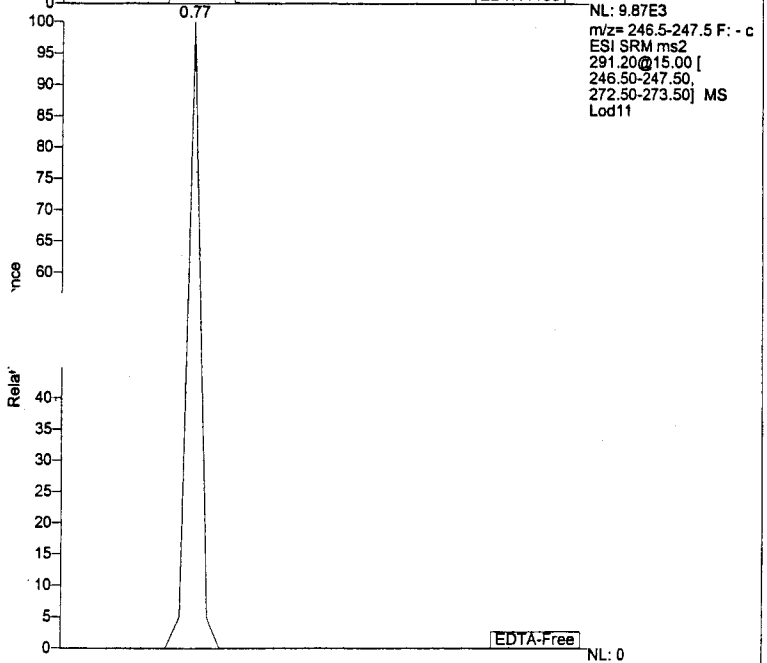


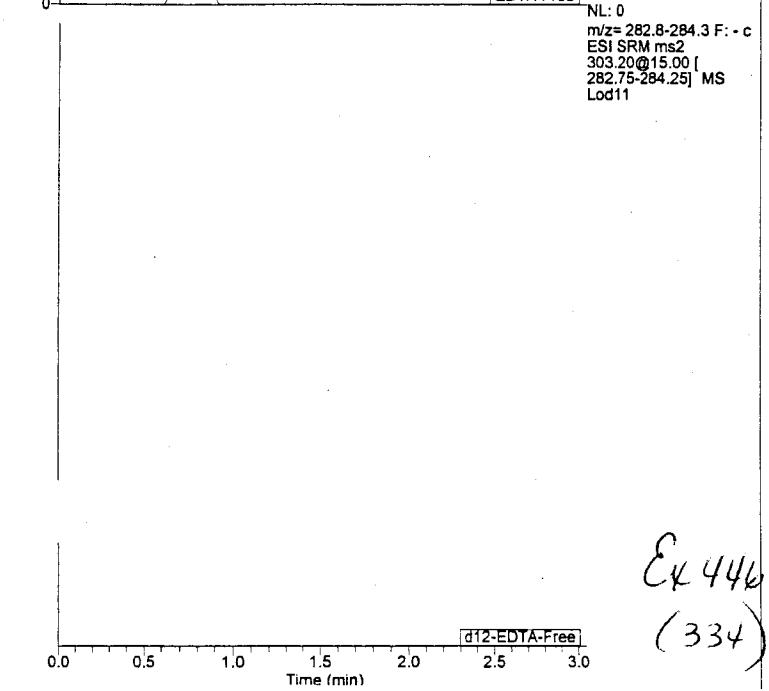


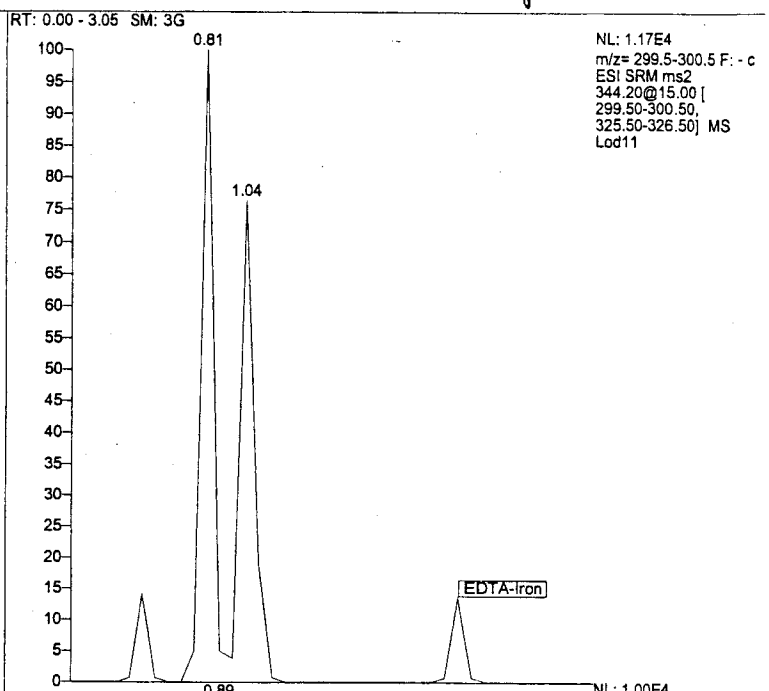
Ex 446
(333)

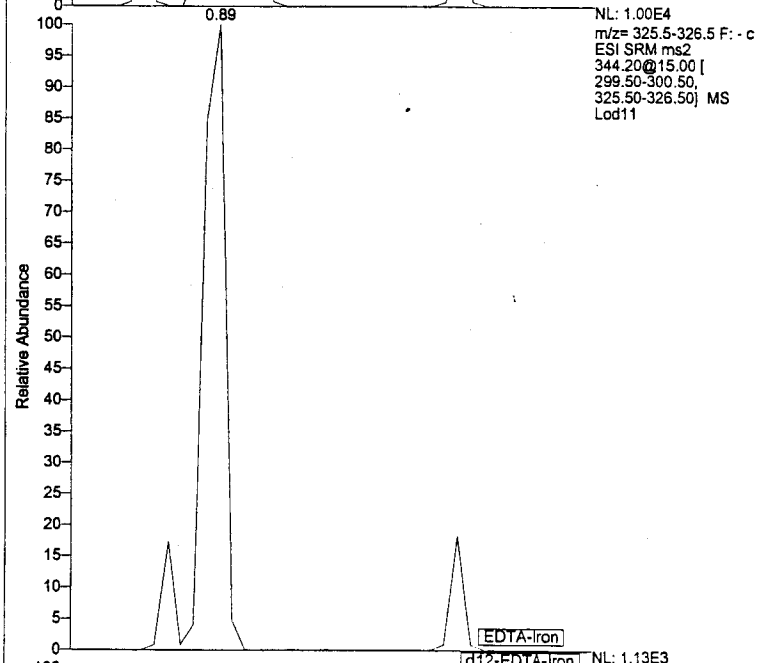
C:\Xcalibur\...\LOD_Negative\Lod11 02/12/07 01:20:32 13 ppm EDTA JDB

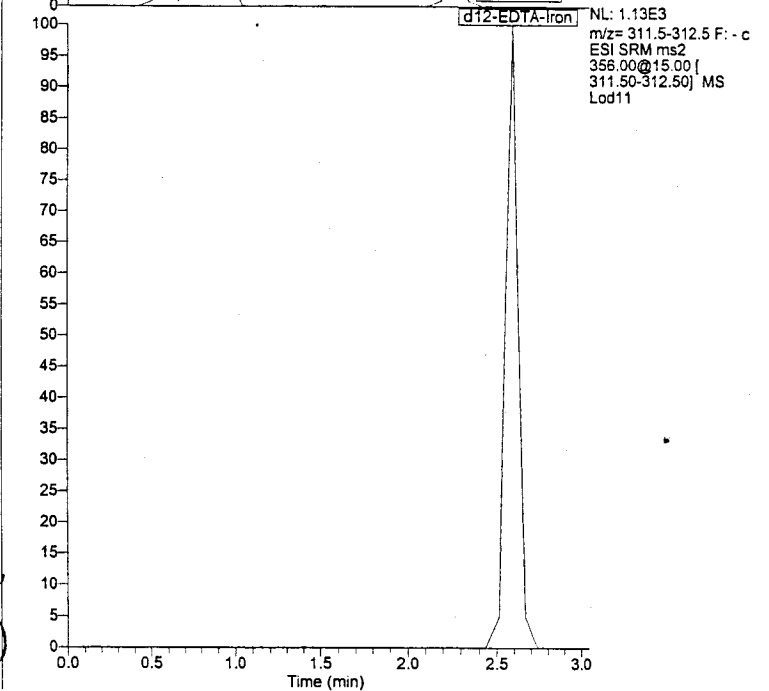






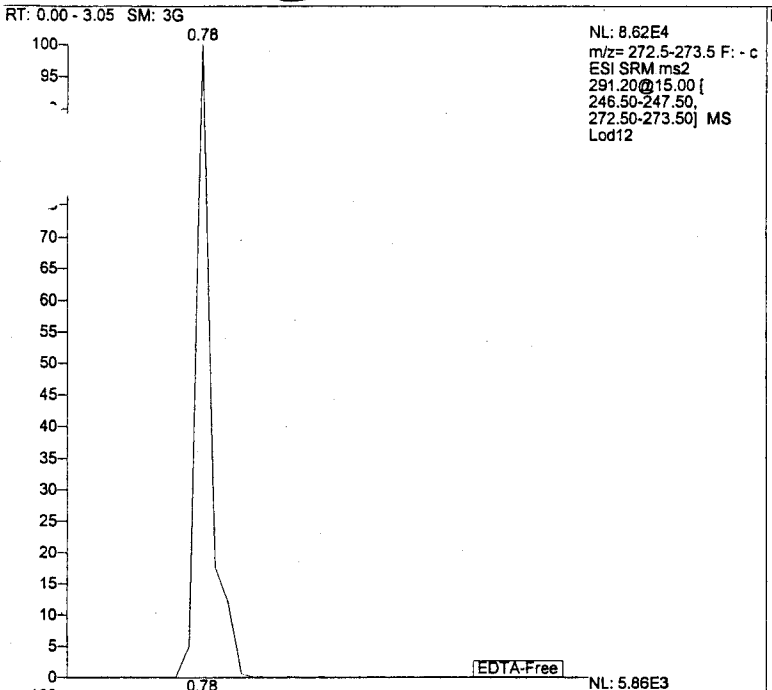


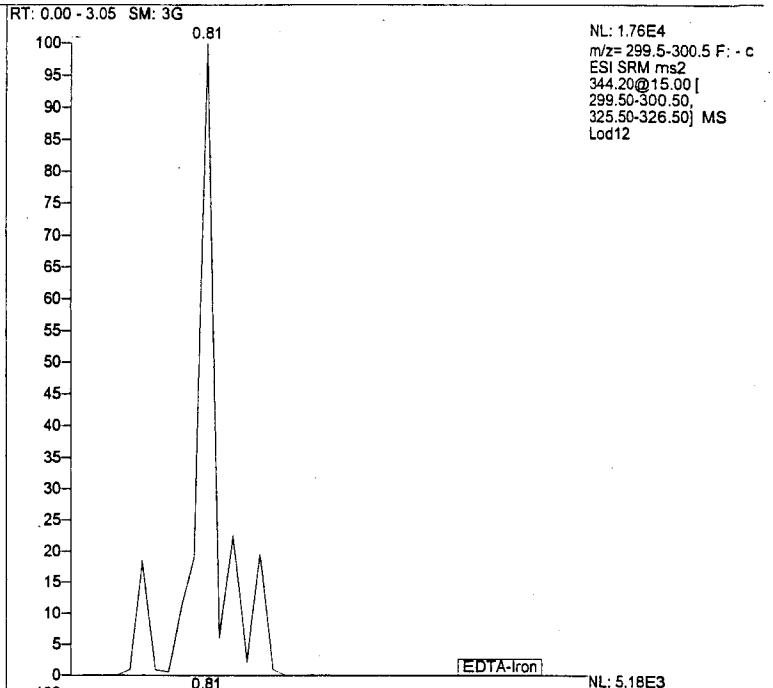


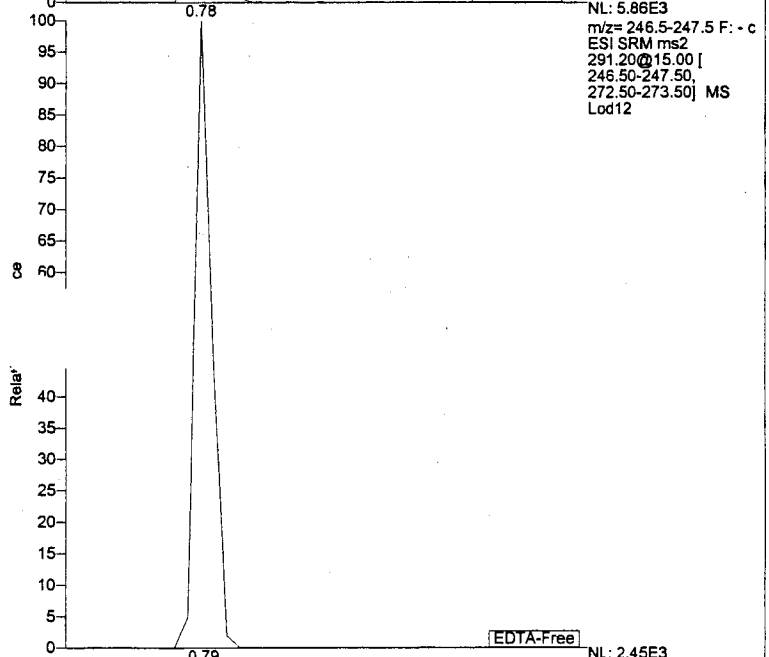


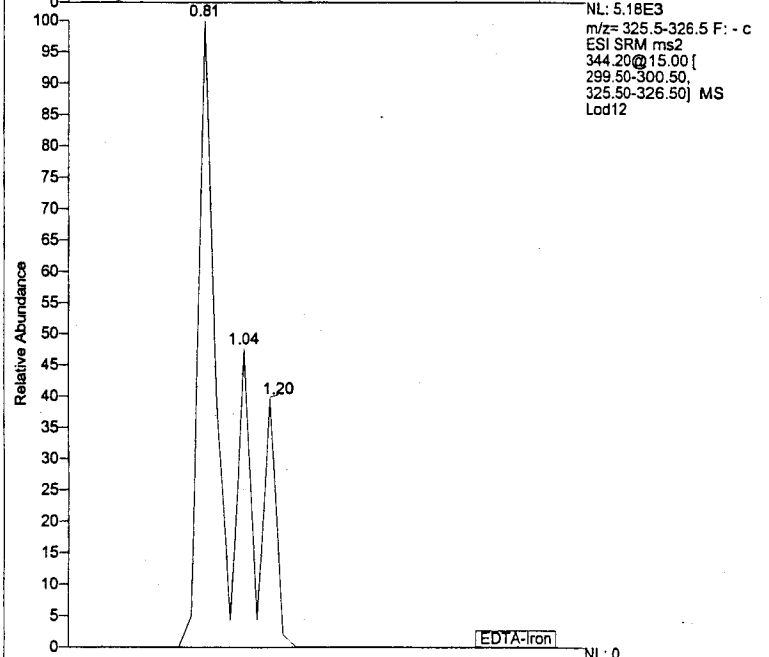
Ex 446
(334)

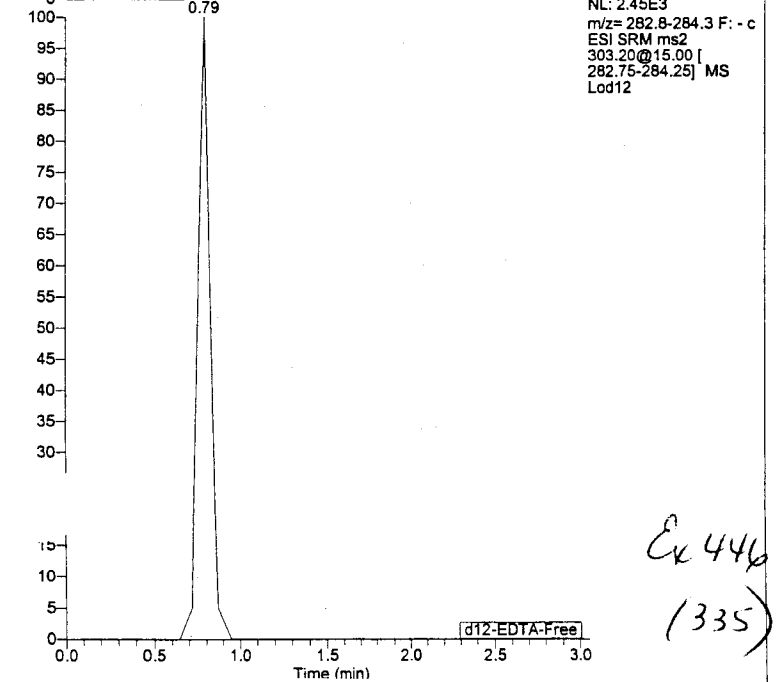
C:\Xcalibur\...\LOD_Negative\Lod12 02/12/07 01:24:25 13 ppm EDTA JOB

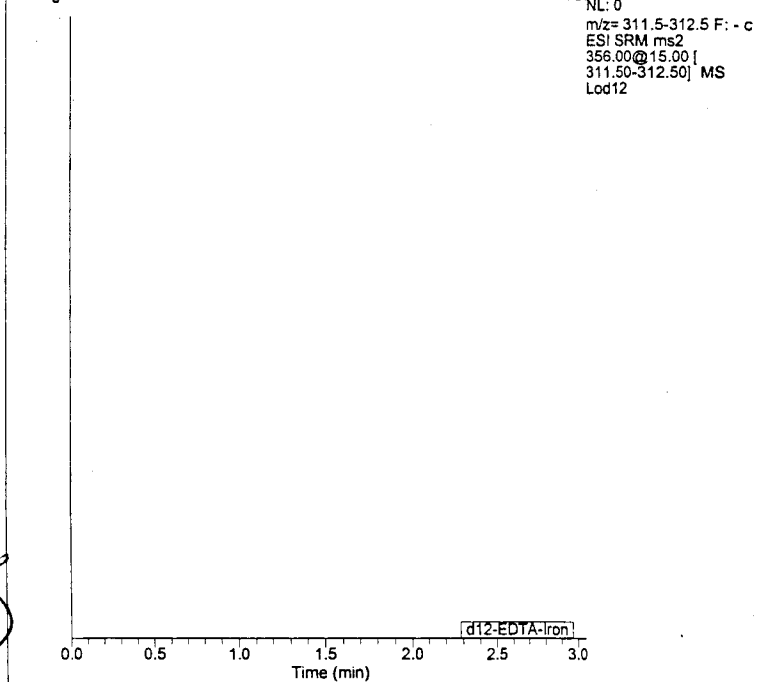






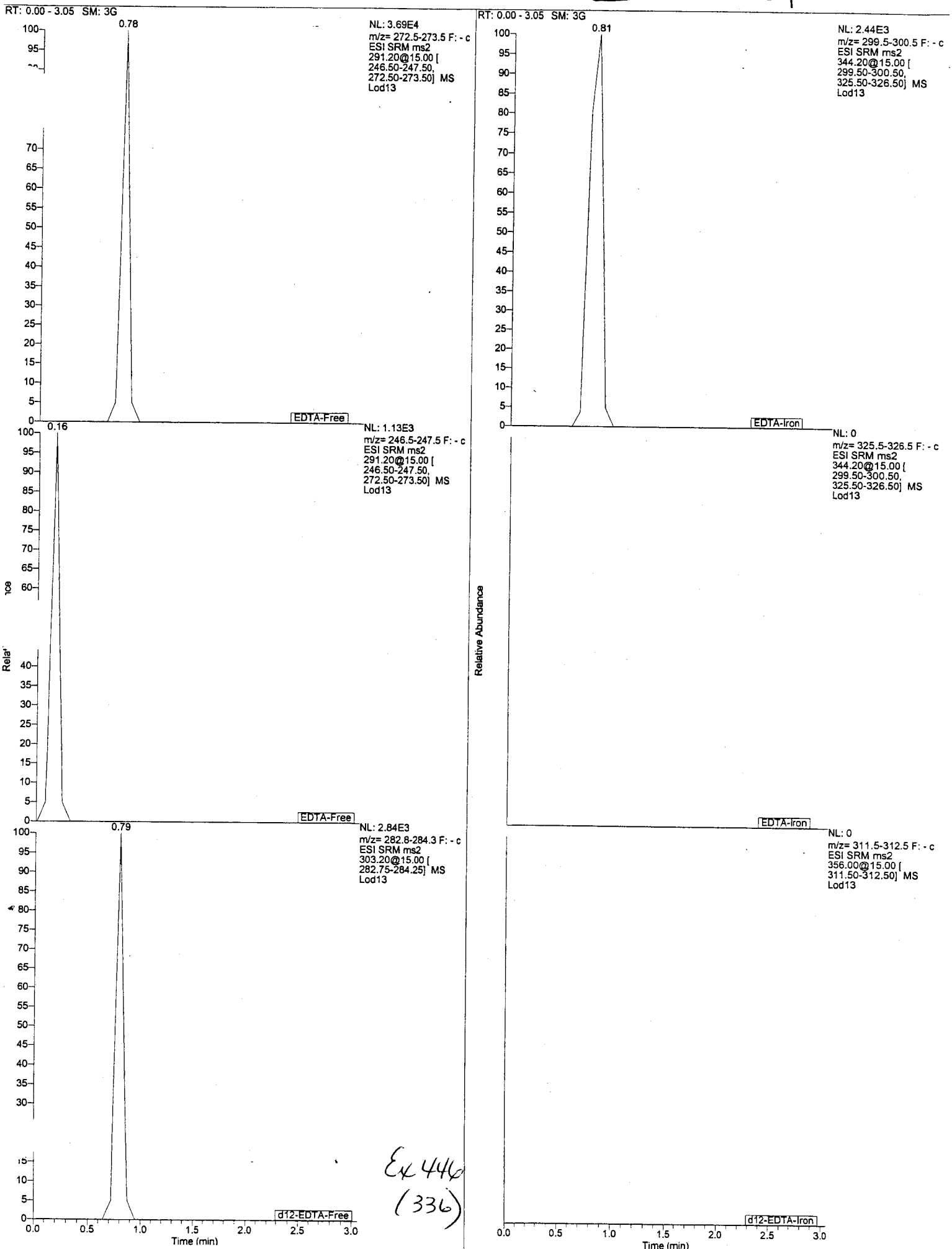






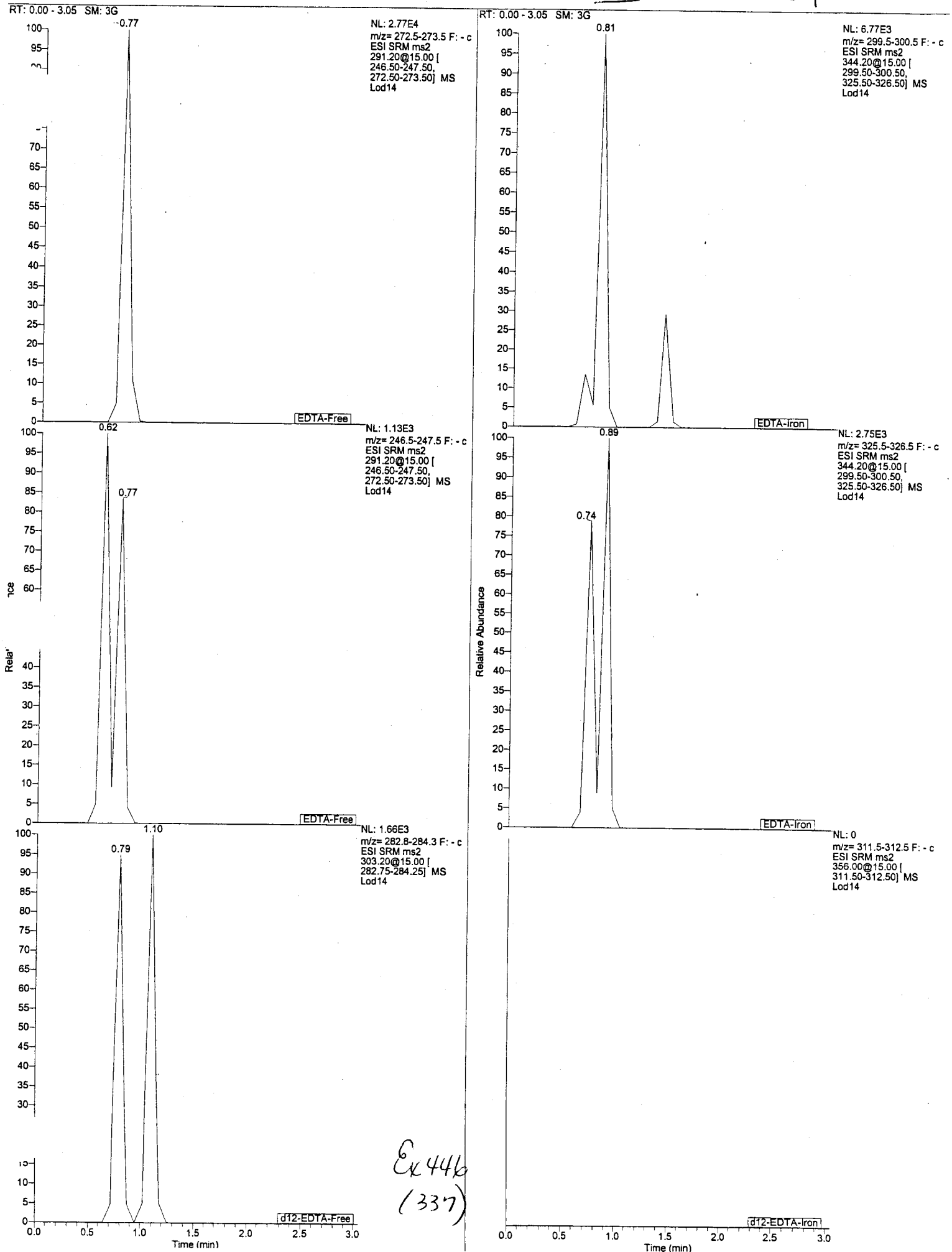
Ex 446
(335)

C:\Xcalibur\...\LOD_Negative\Lod13 02/12/07 01:28:18 6 ppm EDTA JDB



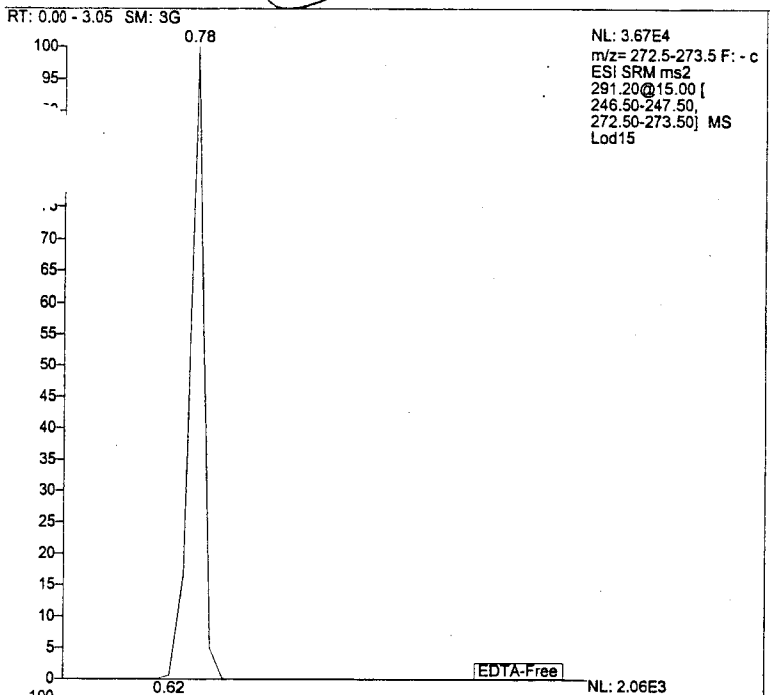
Ex 446
(336)

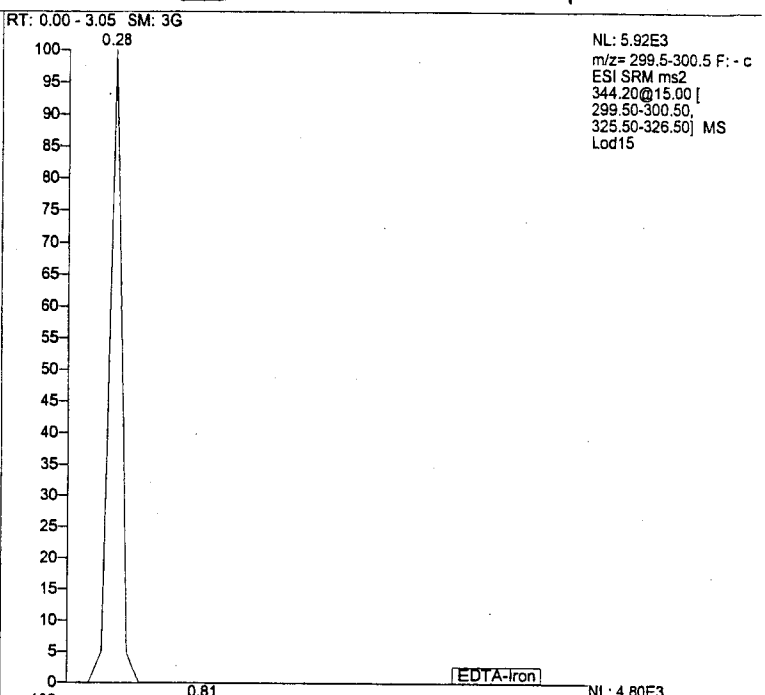
C:\Xcalibur\...\LOD_Negative\Lod14 02/12/07 01:32:10 6 ppm EDTA JDB

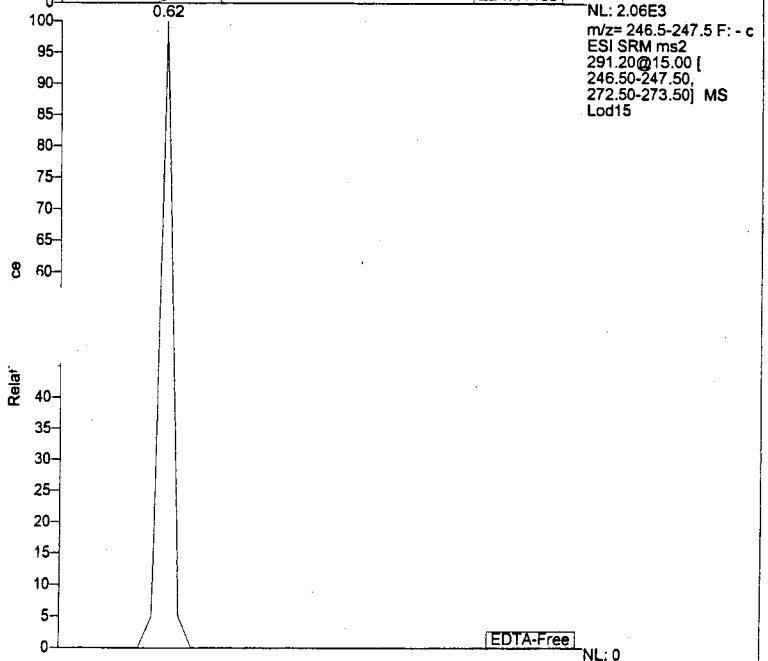


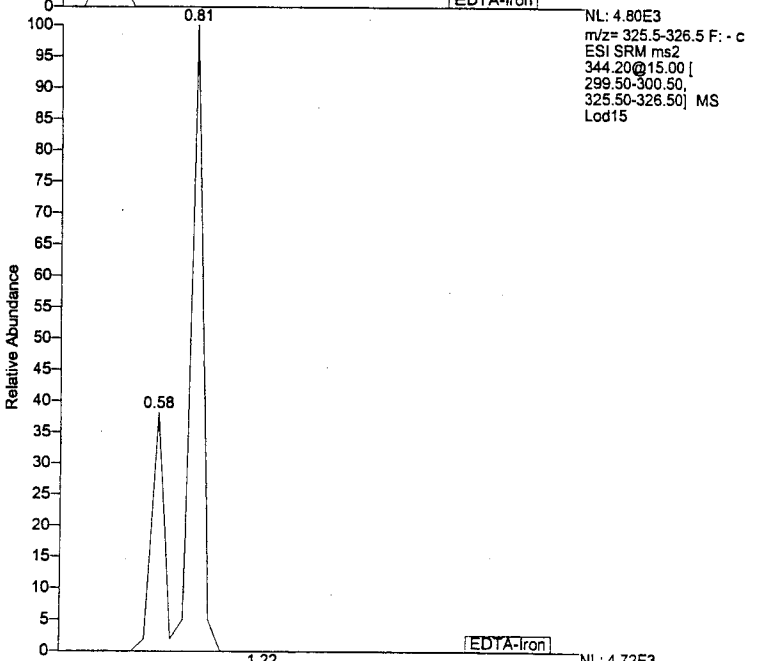
Ex 446
(337)

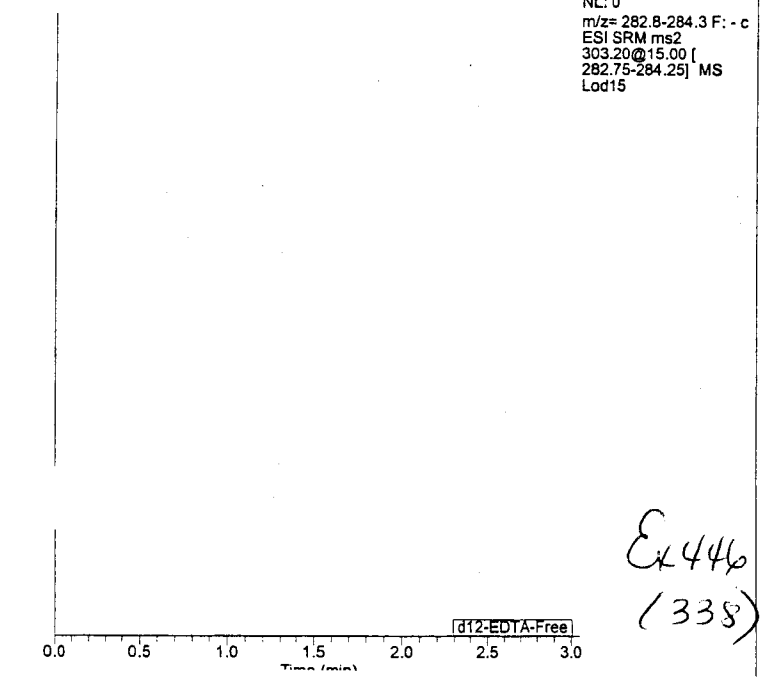
C:\Xcalibur\...\LOD_Negative\Lod15 02/12/07 01:36:03 6 ppm EDTA JMB

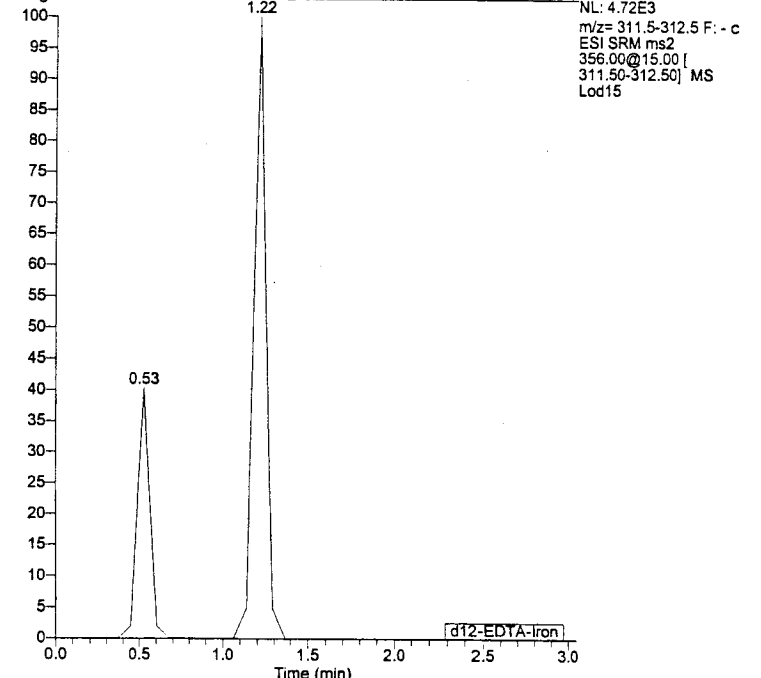












Ex 446
(338)

Sequence---LOD2_neg.sld [Open]

Sample Name: 3 ppm EDTA
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD16	05	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		6	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 3 ppm EDTA
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD17	05	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		6	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(339)

JDP
4/14/07

Sequence---LOD2_neg.sld [Open]

Sample Name: 3 ppm EDTA
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD18	05	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		6	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 1.6 ppm EDTA
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD19	05	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		7	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(340)

JDP
2/12/07

Sequence---LOD2_neg.sld [Open]

Sample Name: 1.6 ppm EDTA

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD20	05	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		7	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 1.6 ppm EDTA

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD21	05	C:\Xcalibur\Data\EDTA\Brewer\LOD_Negative	C:\Xcalibur\methods\EDTA_Neg

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		7	5.0		0.000	0.000	0.000

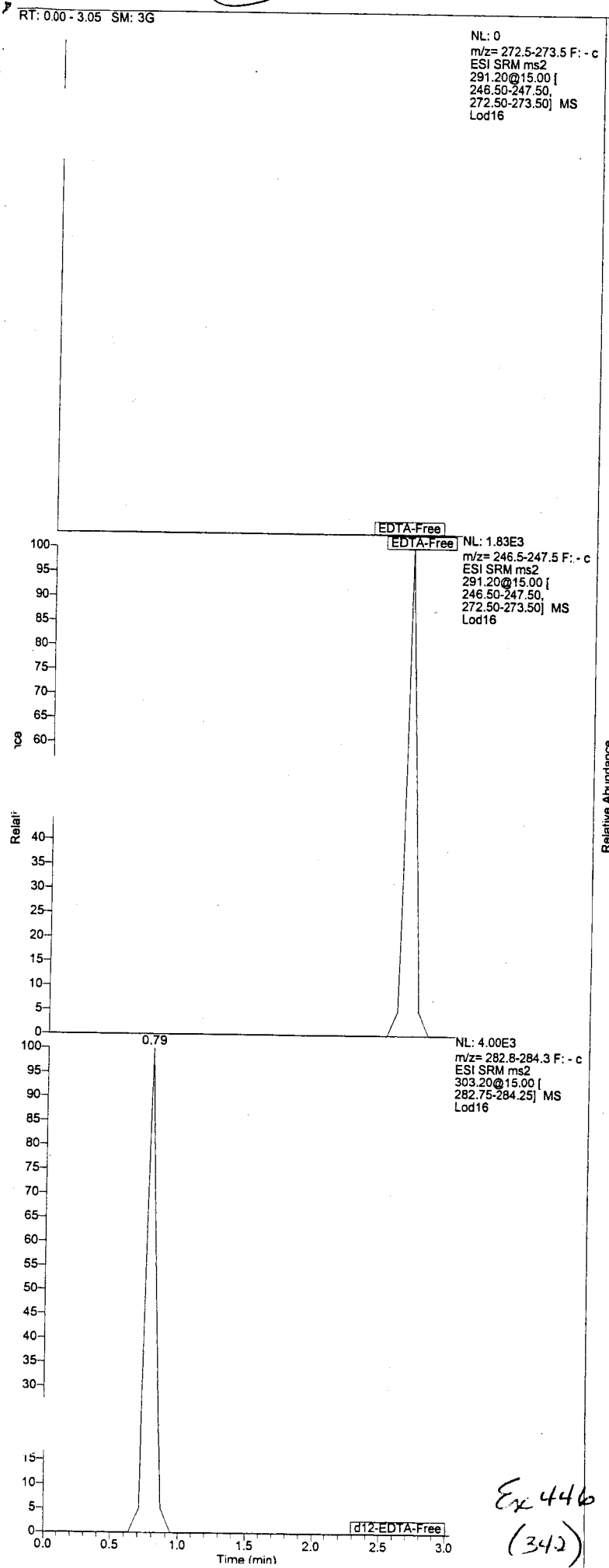
Dil Factor
1.000

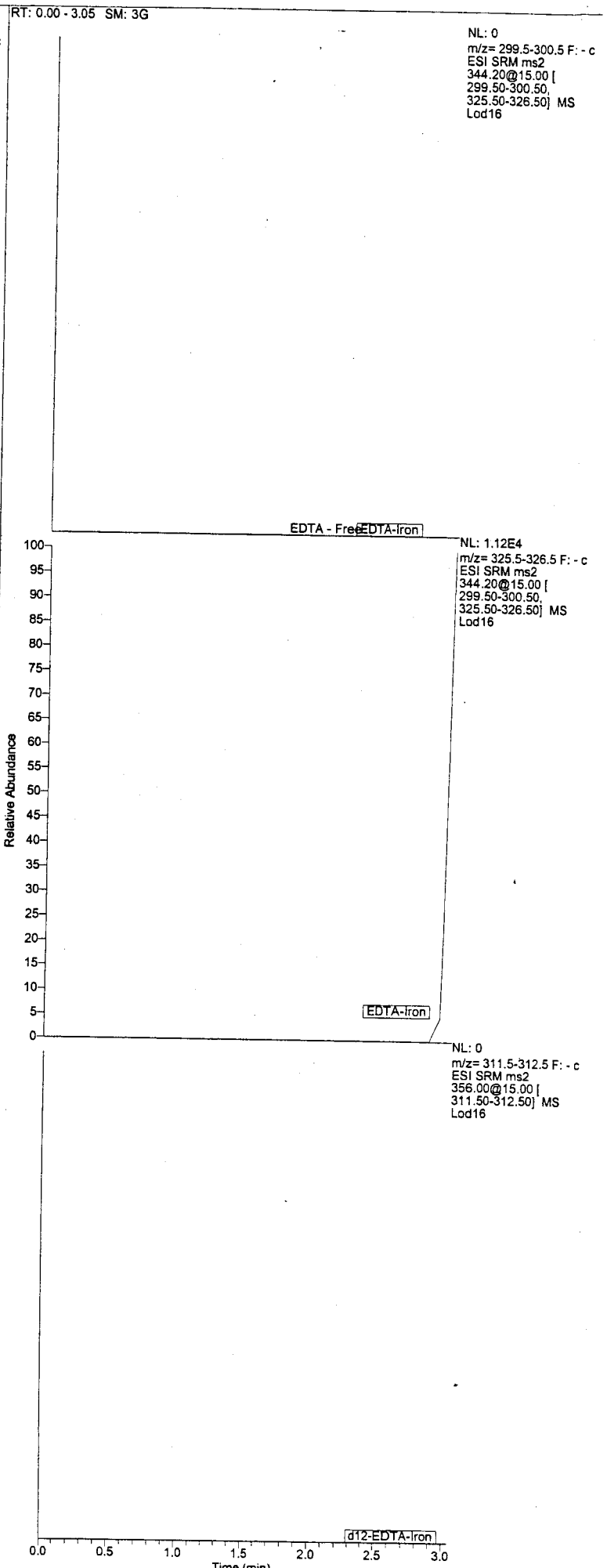
****

JoB
2/12/07

Ex 446
(341)

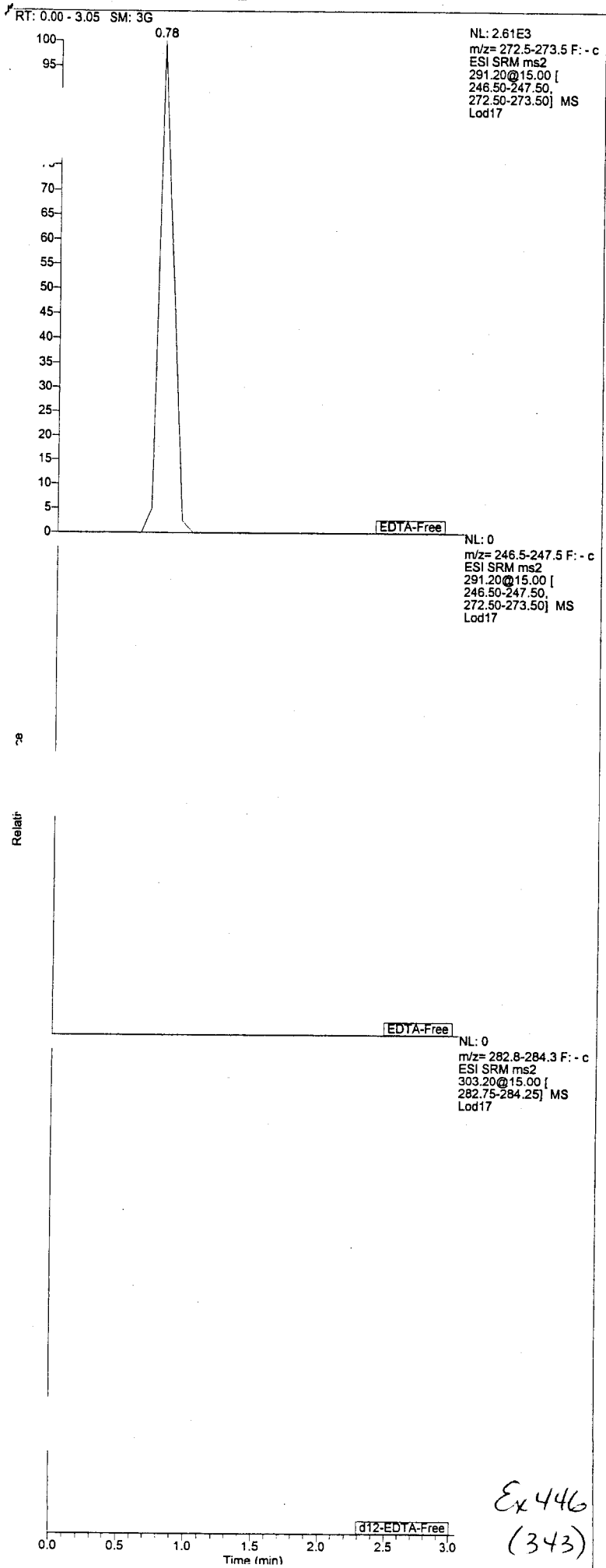
C:\Xcalibur\...\LOD_Negative\Lod16 02/12/07 01:46:39 3 ppm EDTA JDB

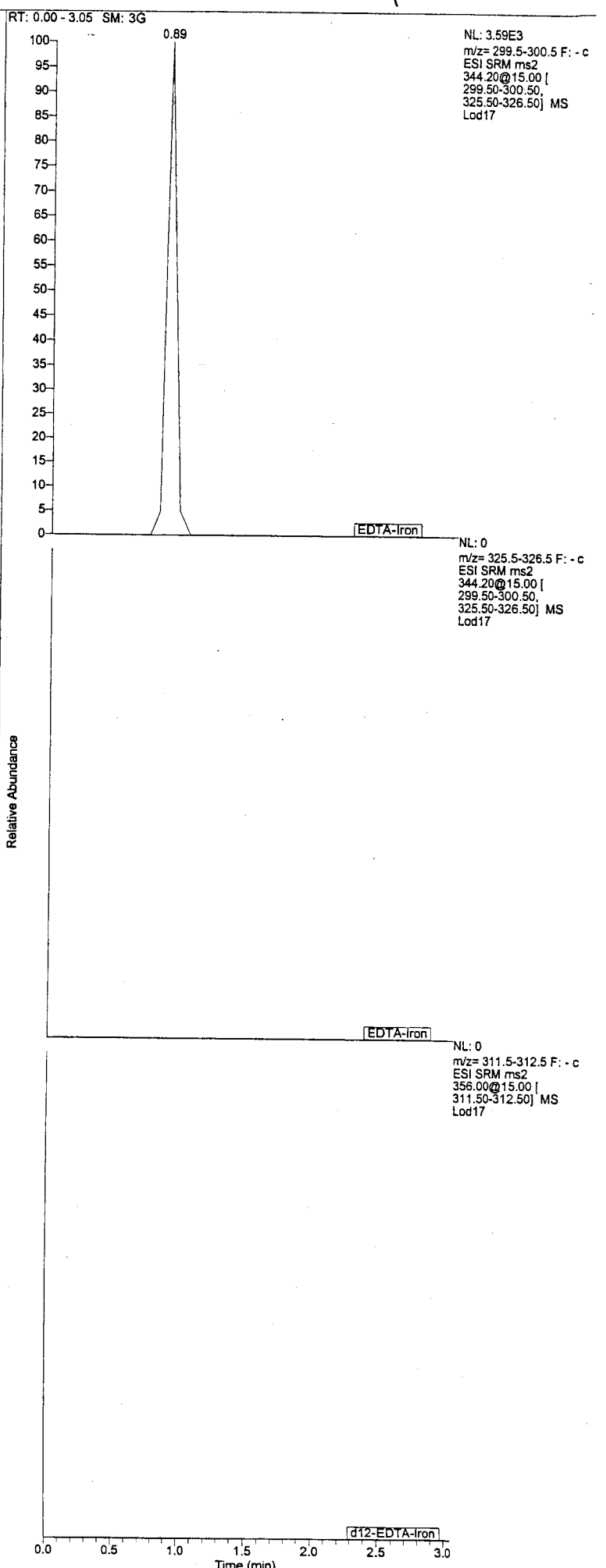




Ex 446
(342)

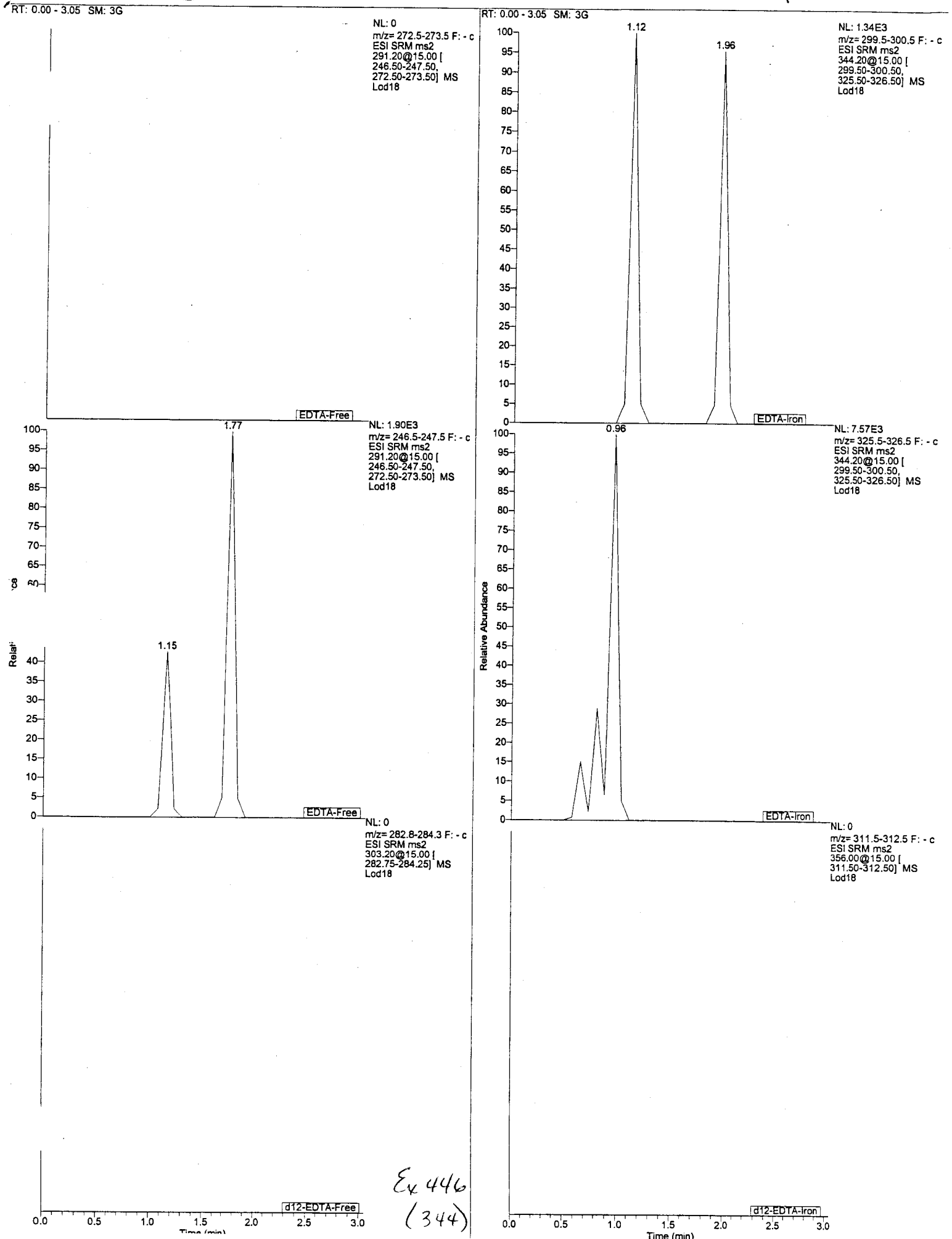
C:\Xcalibur\...\LOD_Negative\Lod17 02/12/07 01:50:58 3 ppm EDTA JDB





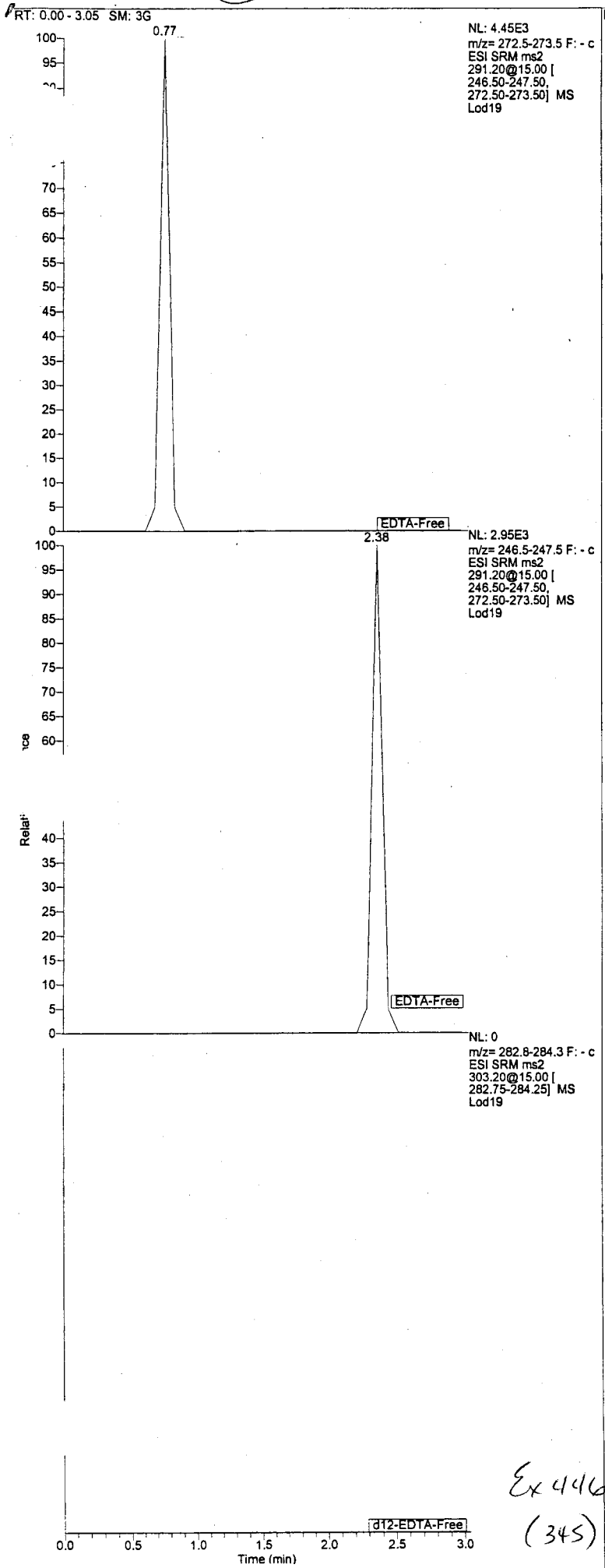
Ex 446
(343)

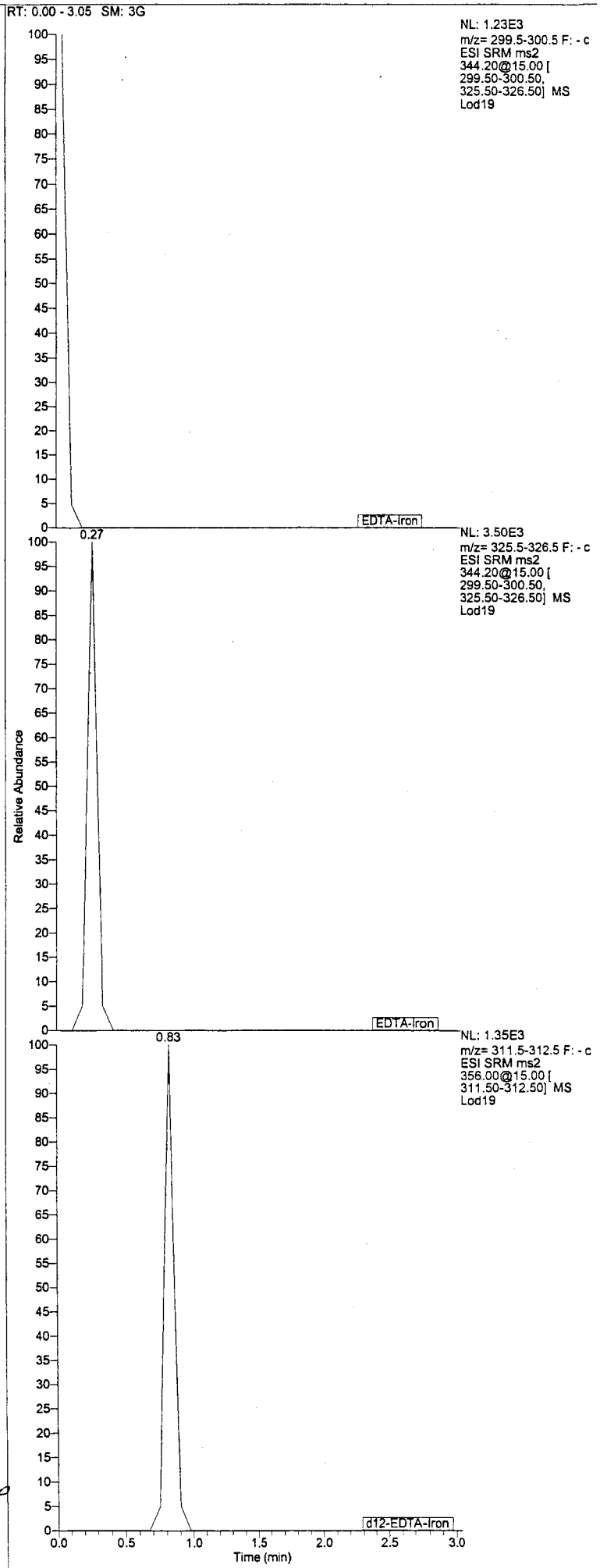
C:\Xcalibur\...\LOD_Negative\Lod18 02/12/07 01:54:50 3 ppm EDTA JDB



Ex 446
(344)

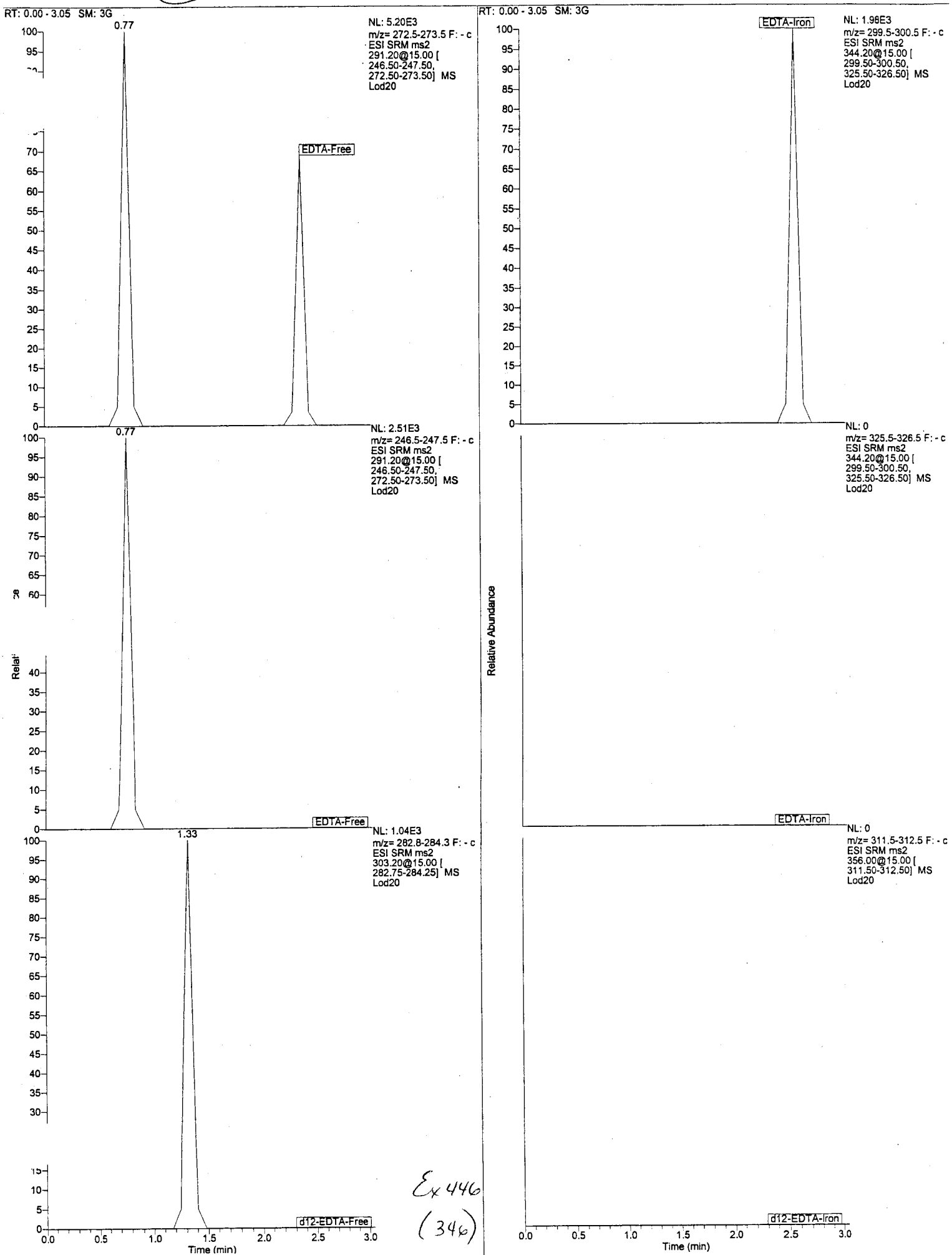
C:\Xcalibur\...\LOD_Negative\Lod19 02/12/07 01:58:43 1.6 ppm EDTA JDB





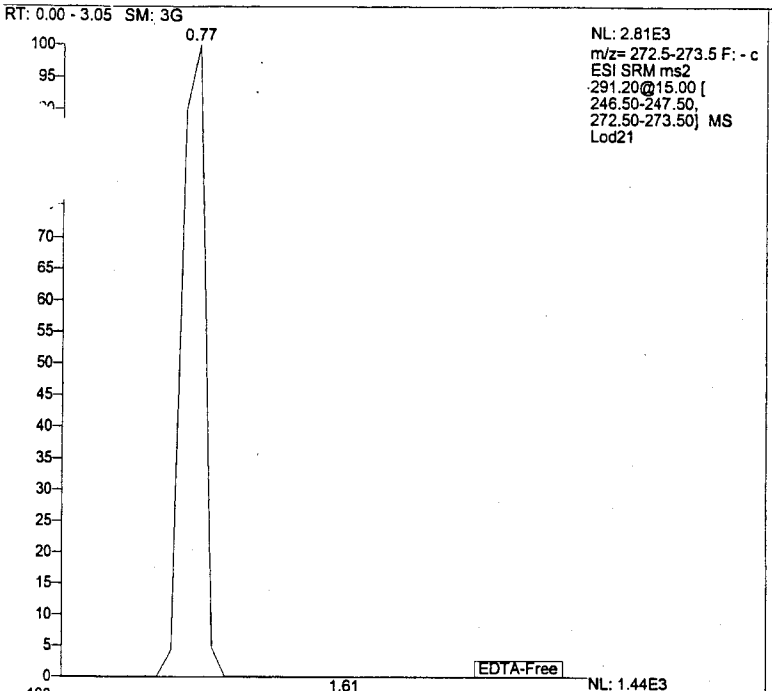
Ex 4416
(345)

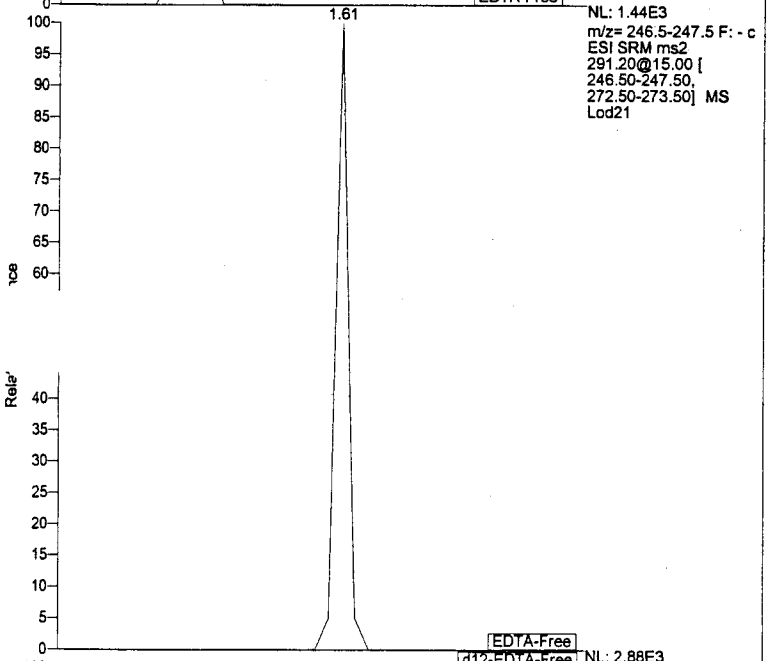
C:\Xcalibur\...\LOD_Negative\Lod20 02/12/07 02:02:36 1.6 ppm EDTA

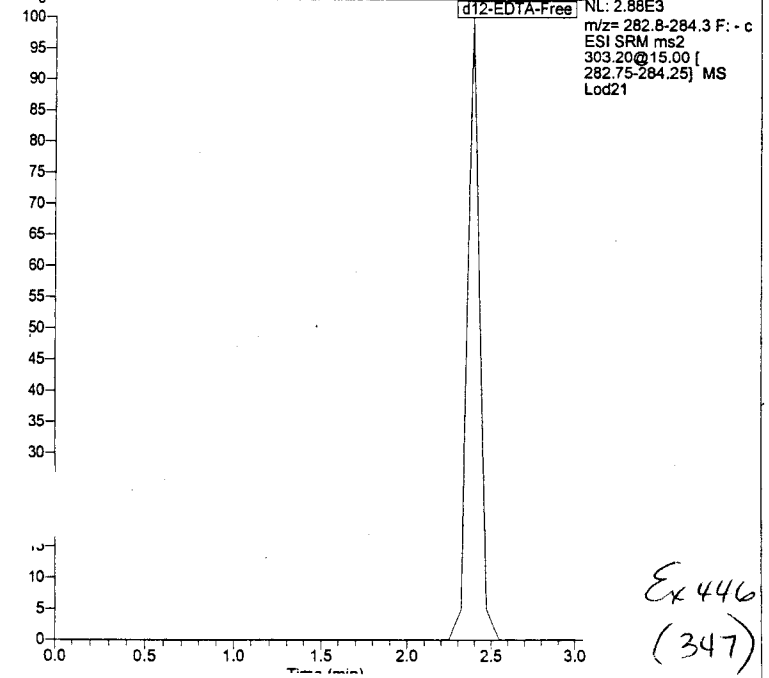


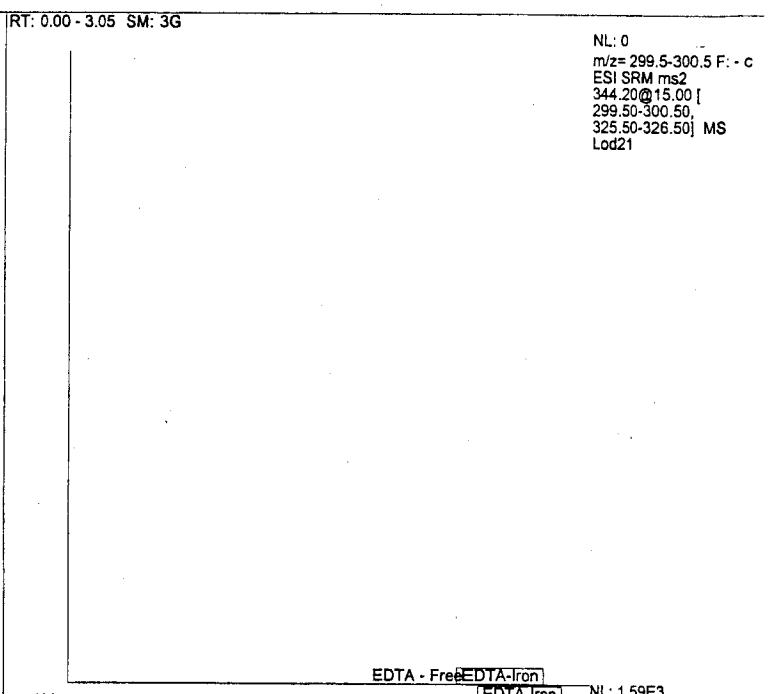
Ex 446
(346)

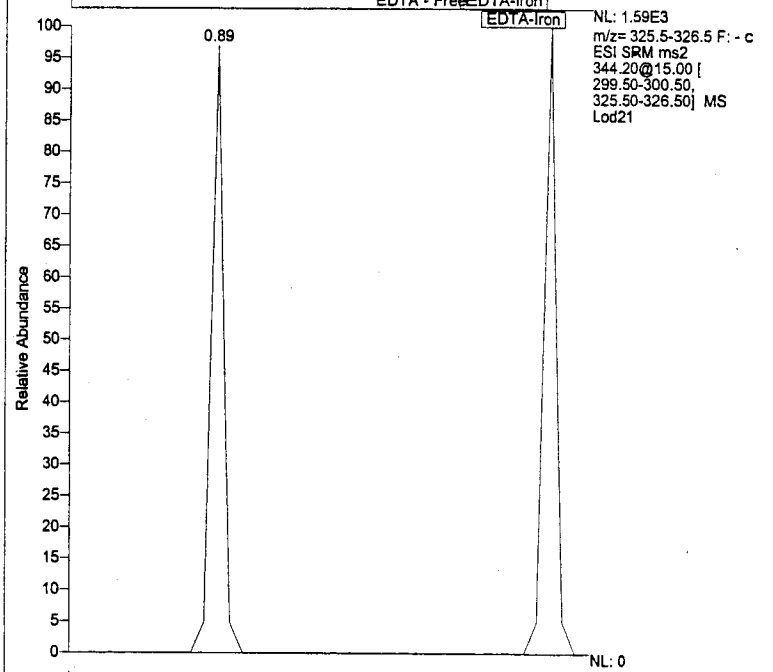
C:\Xcalibur\...\LOD_Negative\Lod21 02/12/07 02:06:29 1.6 ppm EDTA JDB

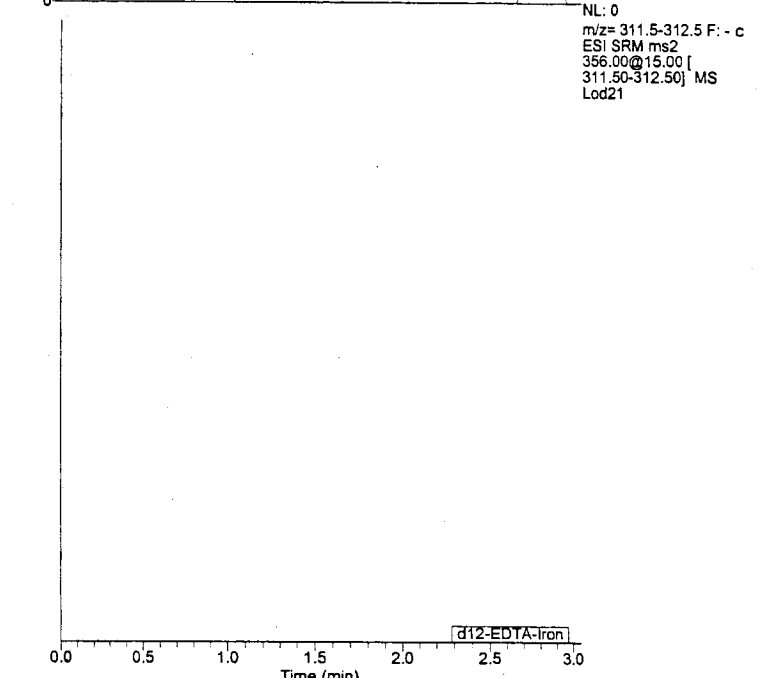












Ex 446
(347)

Sequence---LOD_pos.sld [Open]

Sample Name: 100 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD01	1	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 100 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD02	01	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(345)

JOB
2/12/07

Sequence---LOD_pos.sld [Open]

Sample Name: 100 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD03	01	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		1	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD04	02	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		2	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(349)

JBP
2/12/07

Sequence---LOD_pos.sld [Open]

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD05	02	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		2	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD06	02	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		2	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(350)

JDB
2/12/07

Sequence---LOD_pos.sld [Open]

Sample Name: 25 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD07	03	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		3	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 25 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD08	04	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		3	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(351)

JDB
2/12/07

Sequence---LOD_pos.sld [Open]

Sample Name: 25 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD09	03	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		3	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 13 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD10	04	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		4	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(352)

JDB
2/14/07

Sequence---LOD_pos.sld [Open]

Sample Name: 13 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD11	04	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		4	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 13 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD12	04	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		4	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(353)

JOP
2/12/07

Sequence---LOD_pos.sld [Open]

Sample Name: 6 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD13	05	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		5	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 6 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD14	05	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		5	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(354)

JDB
2/12/07

Sequence---LOD_pos.sld [Open]

Sample Name: 6 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD15	05	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		5	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 3 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD16	06	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		6	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(355)

JDB
2/12/07

Sequence---LOD_pos.sld [Open]

Sample Name: 3 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD17	06	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		6	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 3 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD18	06	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		6	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(356)

JDP
2/12/07

Sequence---LOD_pos.sld [Open]

Sample Name: 1.6 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD19	07	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		7	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Sample Name: 1.6 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD20	07	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		7	5.0		0.000	0.000	0.000

Dil Factor
1.000

****

Ex 446
(357)

JDB
1/11/07

Sequence---LOD_pos.sld [Open]

Sample Name: 1.6 ppm EDTA

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path	Inst Method
Unknown	LOD21	07	C:\Xcalibur\Data\EDTA\Brewer\LOD_Positive	C:\Xcalibur\methods\EDTA_Pos

Proc Method	Cal File	Position	InjVol	Level	Sample Wt	Sample Vol	ISTD Amt
		7	5.0		0.000	0.000	0.000

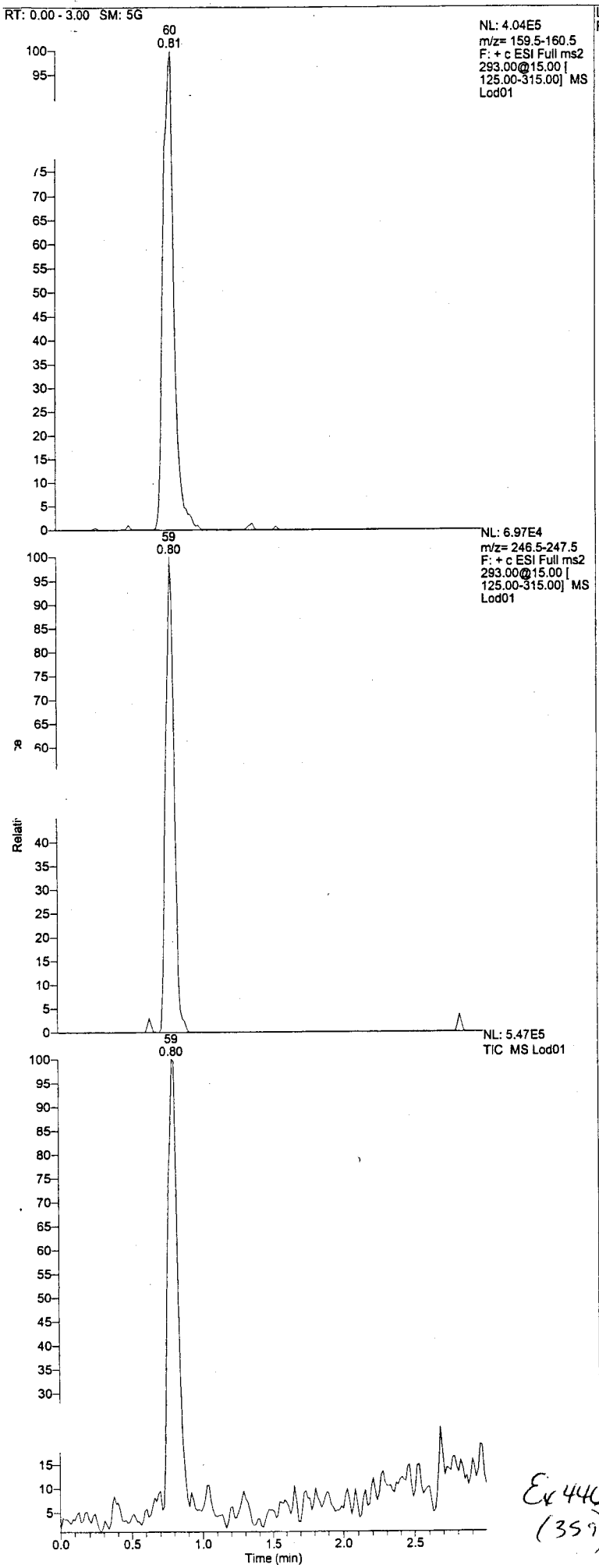
Dil Factor
1.000

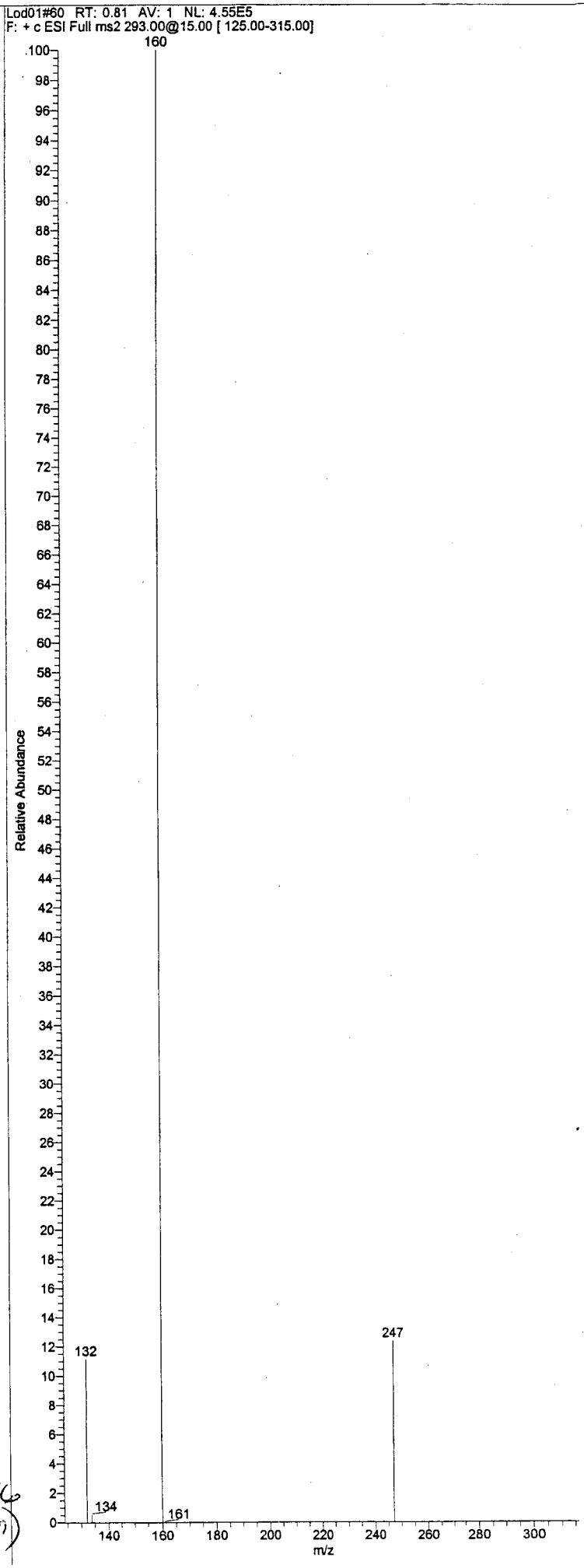
****

Ex 446
(358)

JHP
4/12/07

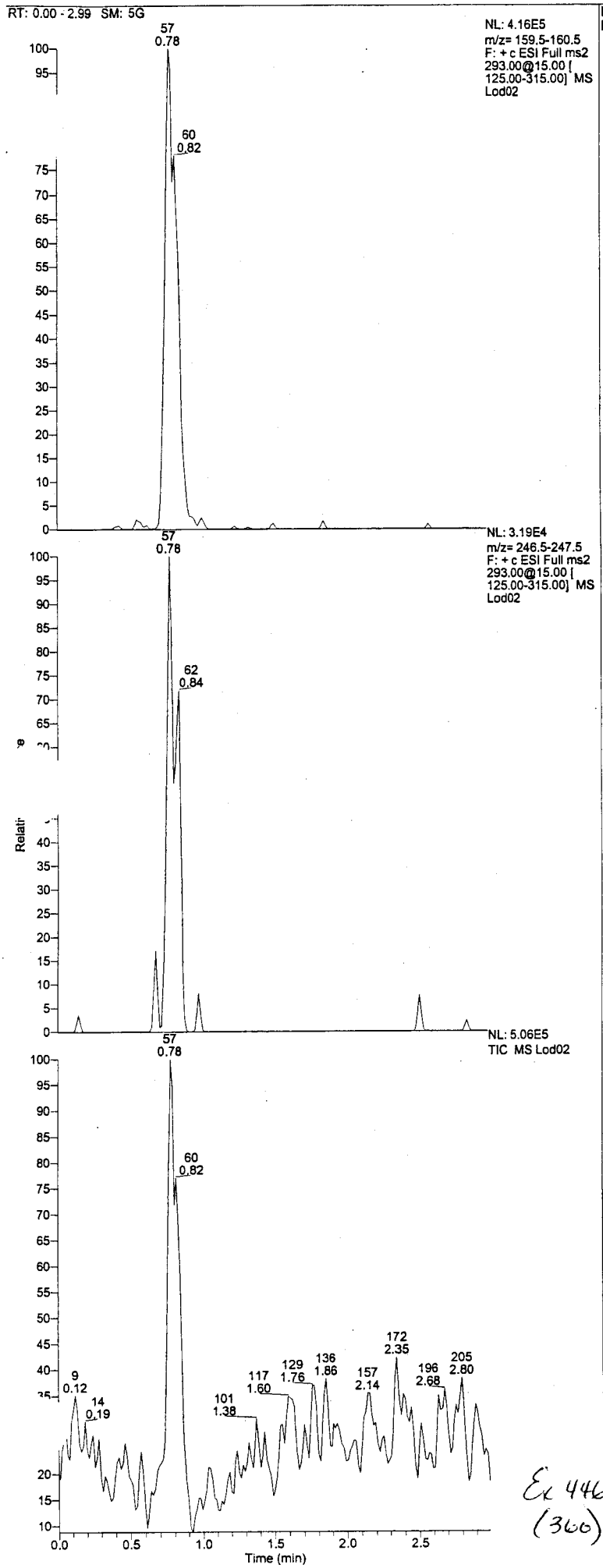
C:\Xcalibur\...\LOD_Positive\Lod01 02/12/07 02:43:57 100 ppm EDTA JMB

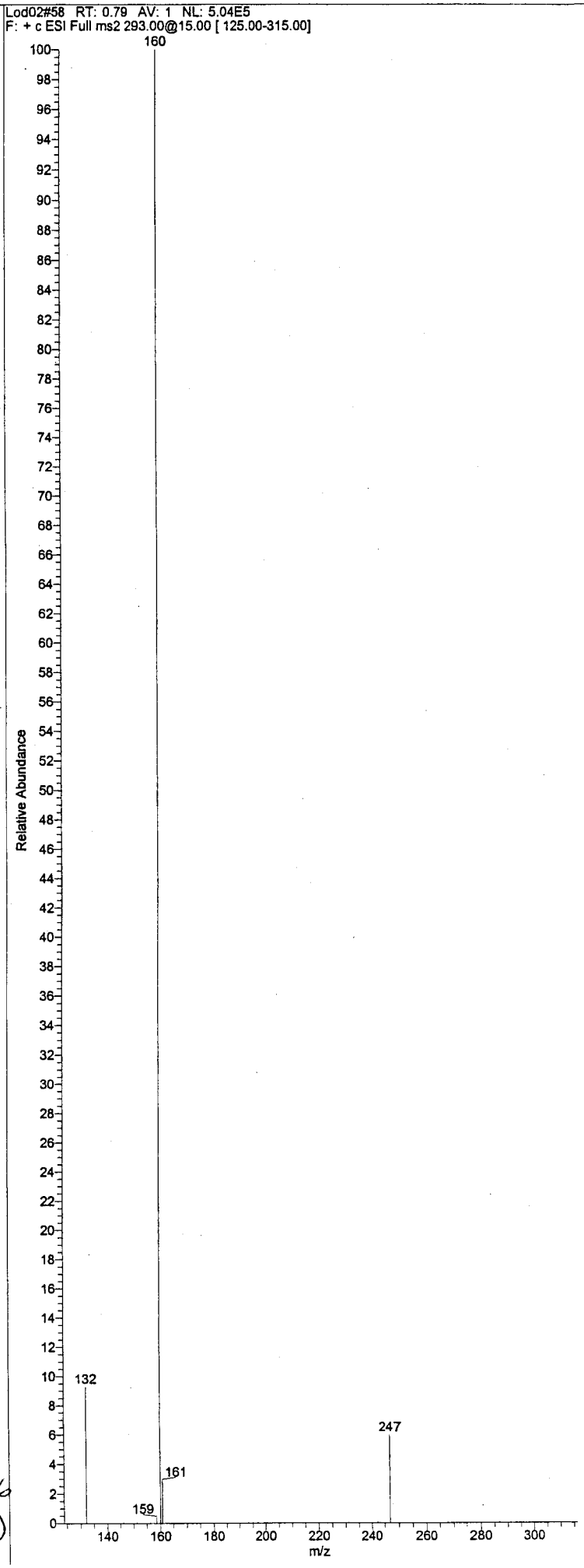




Ex 446
(359)

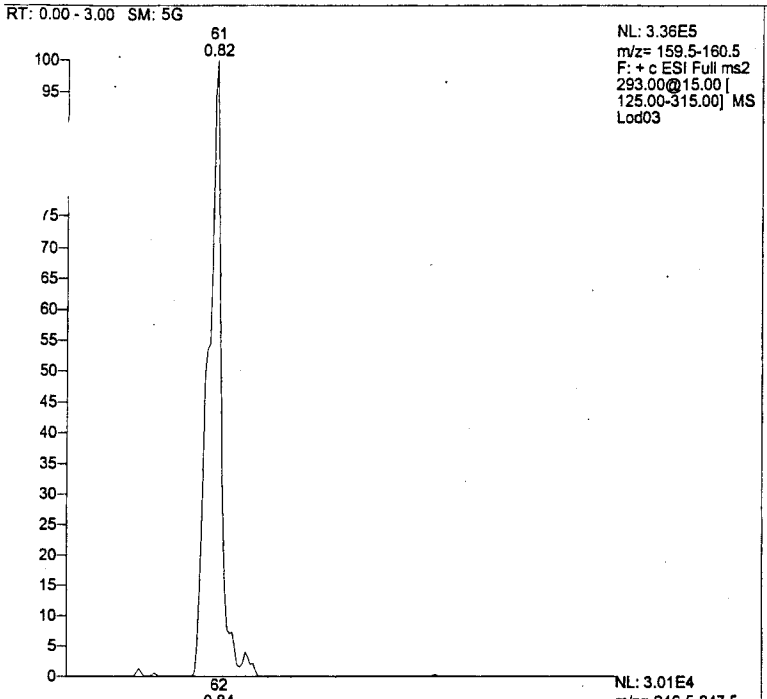
C:\Xcalibur\...\LOD_Positive\Lod02 02/12/07 02:47:51 100 ppm EDTA JDP

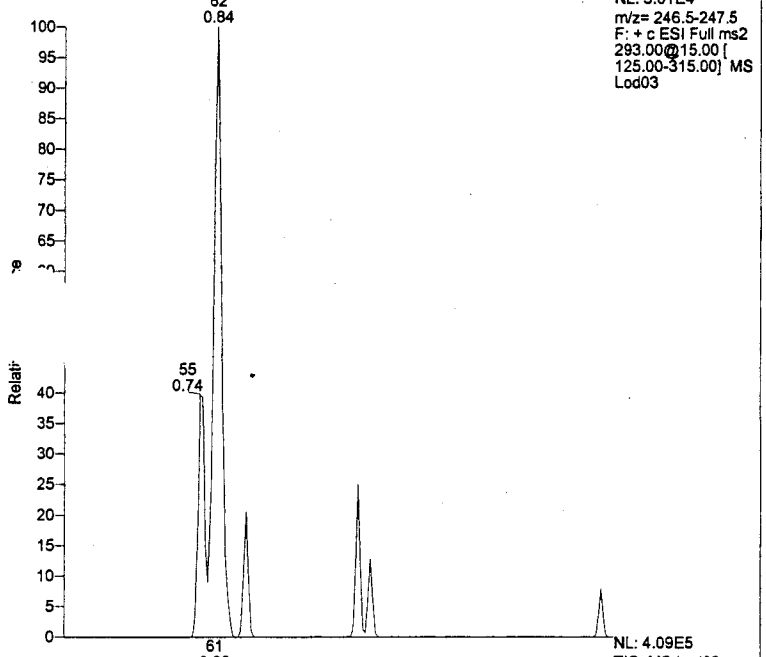


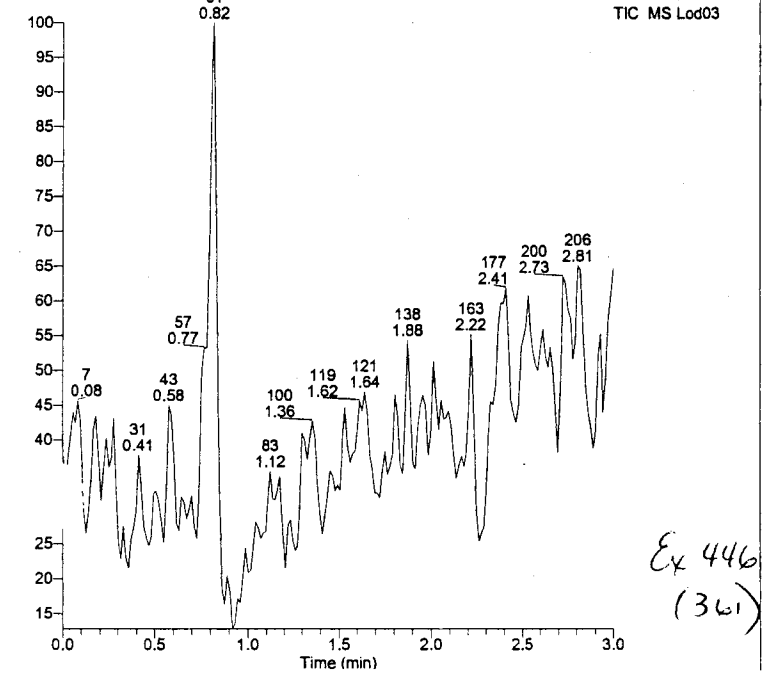


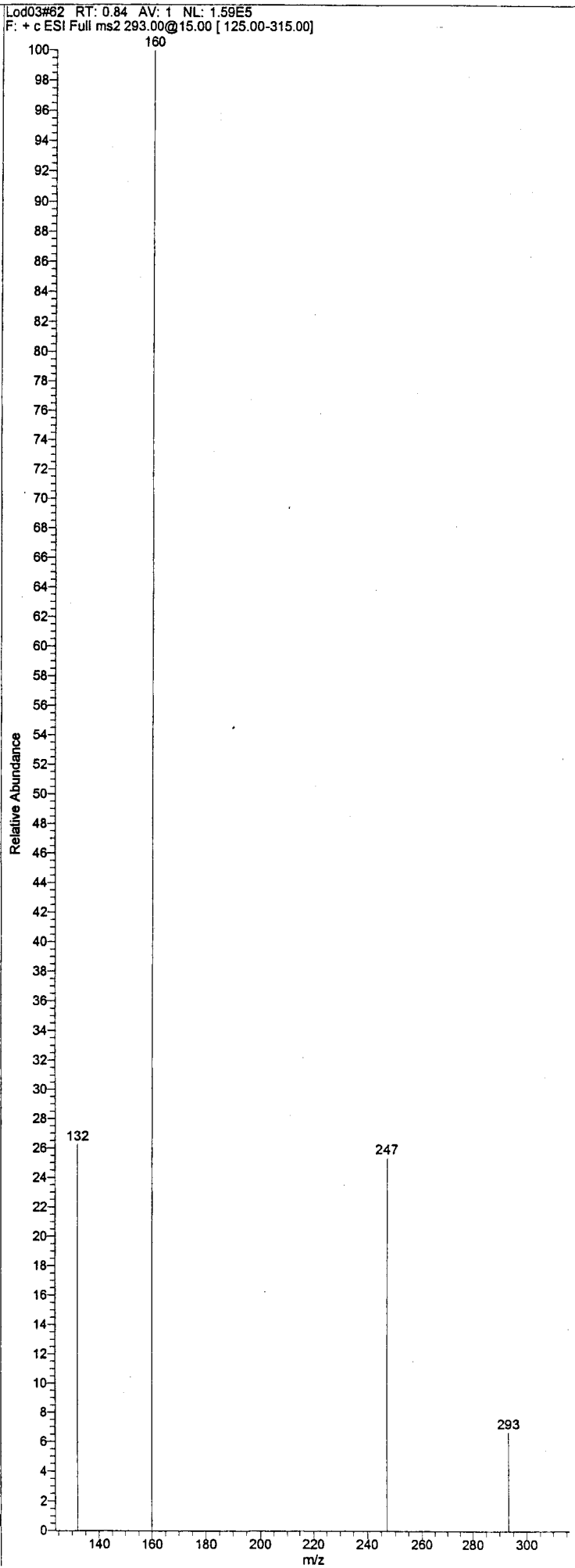
Ex 446 (360)

C:\Xcalibur\...\LOD_Positive\Lod03 02/12/07 02:51:40 100 ppm EDTA JJB



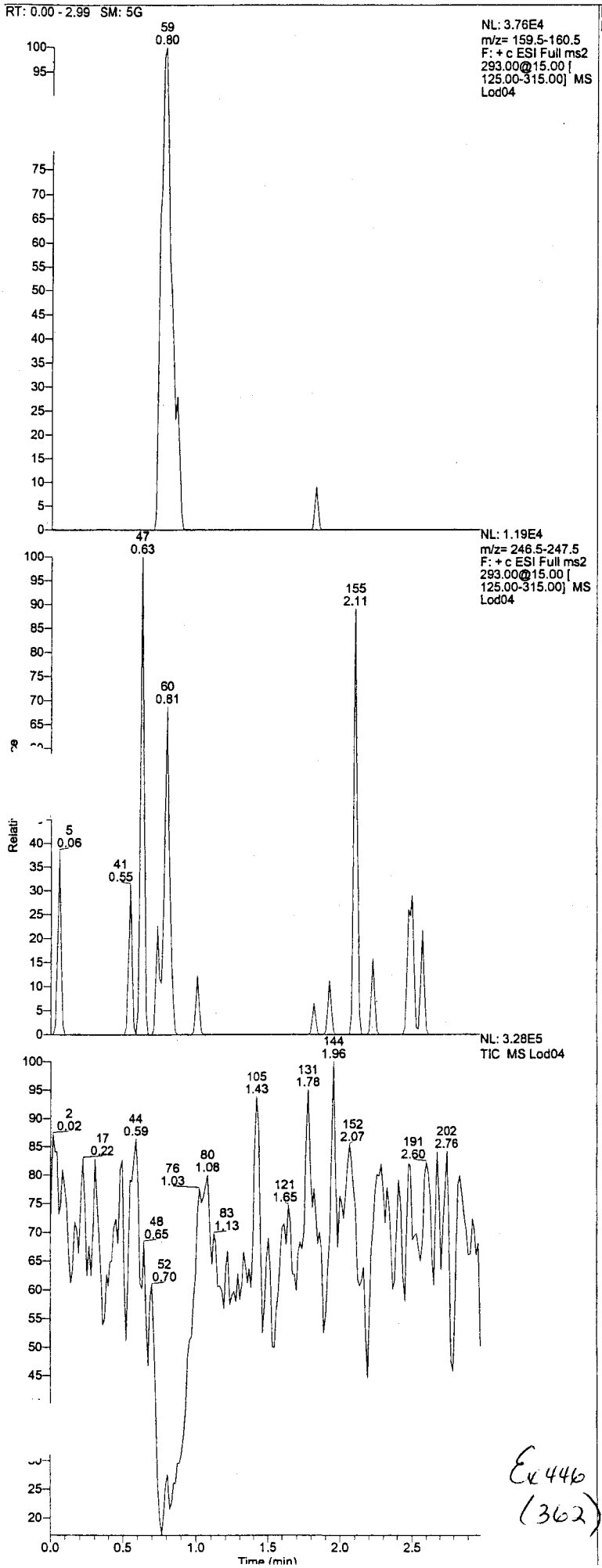


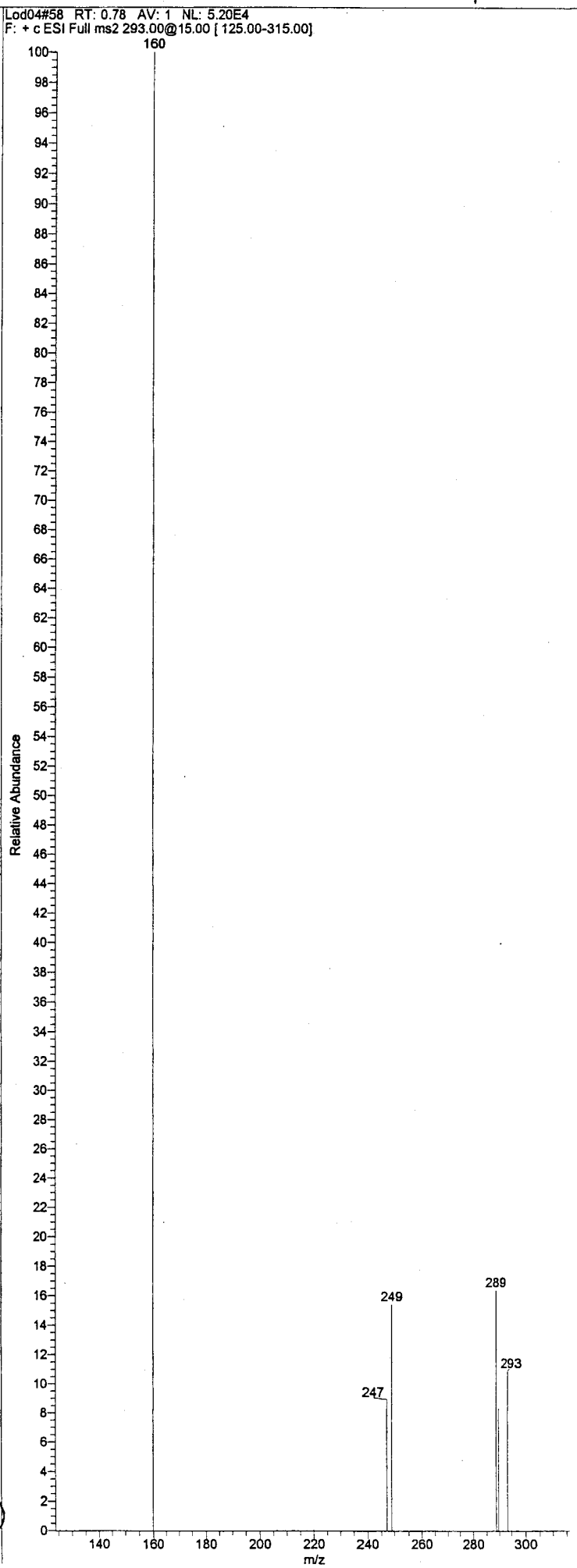




Ex 446
(361)

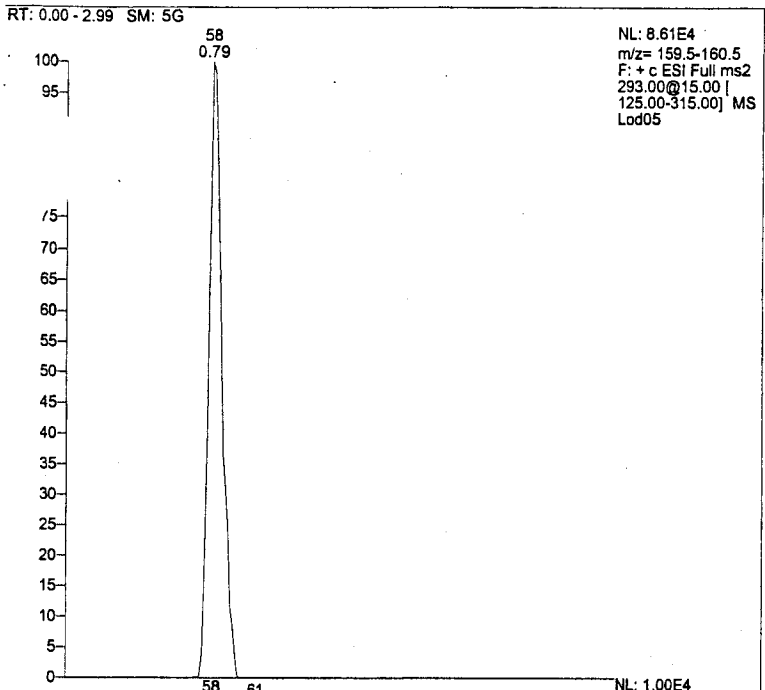
C:\Xcalibur\...\LOD_Positive\Lod04 02/12/07 02:55:29 50 ppm EDTA JDB

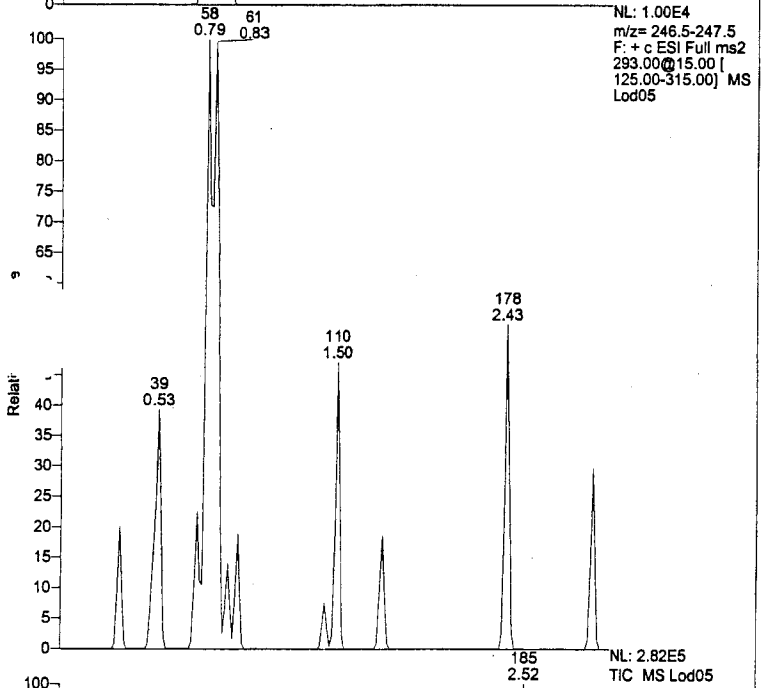


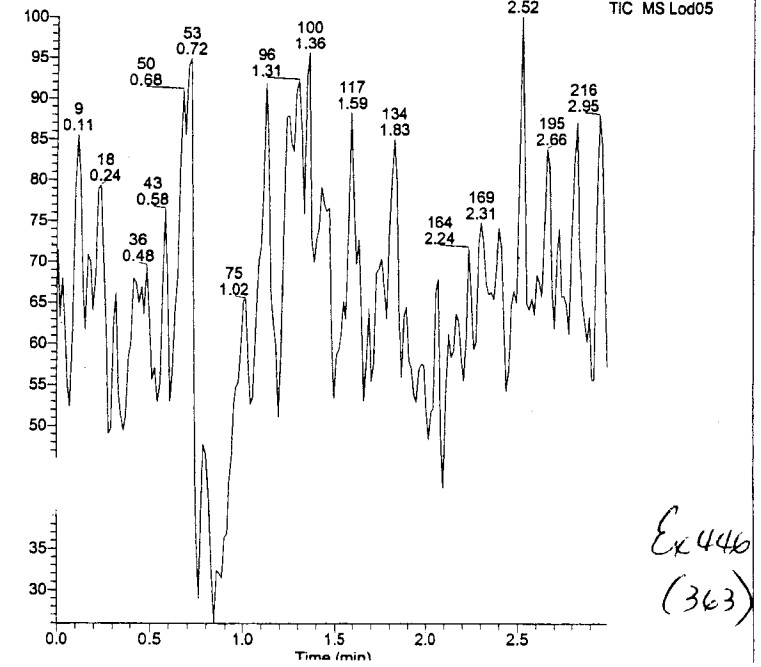


Ex 446
(362)

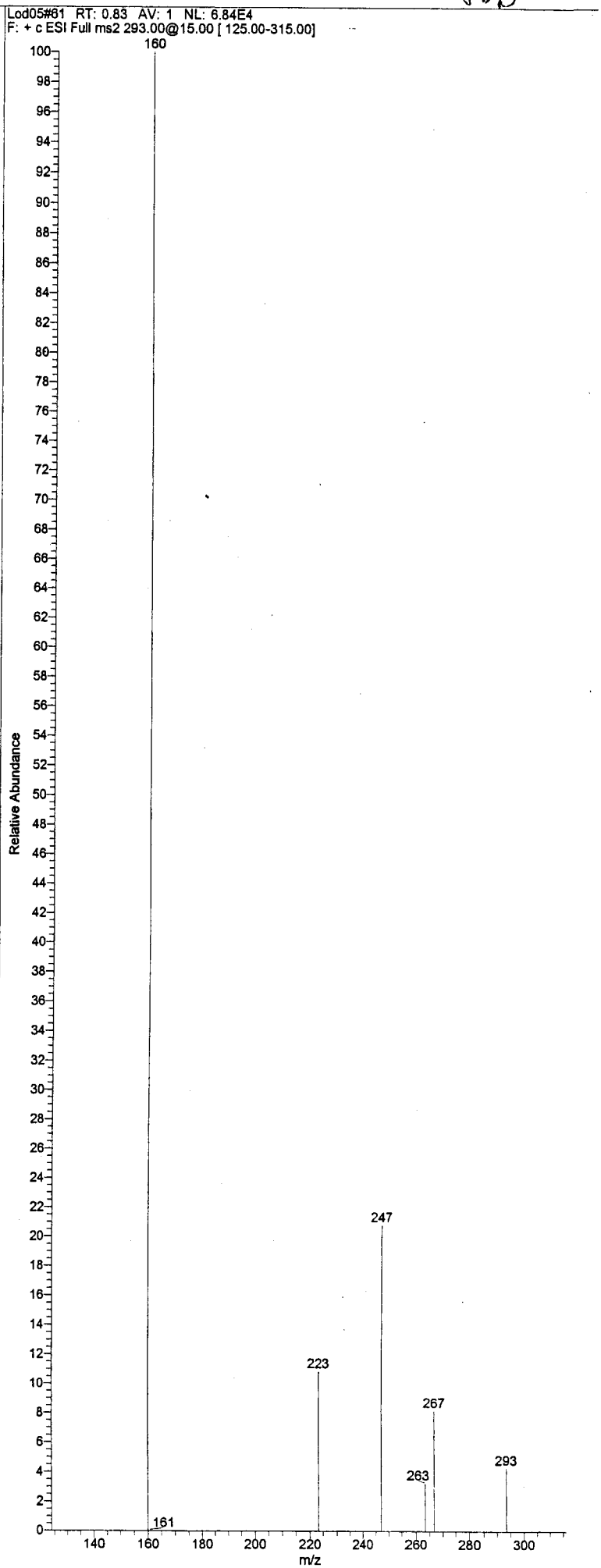
C:\Xcalibur\...\LOD_Positive\Lod05 02/12/07 02:59:15 50 ppm EDTA DDB



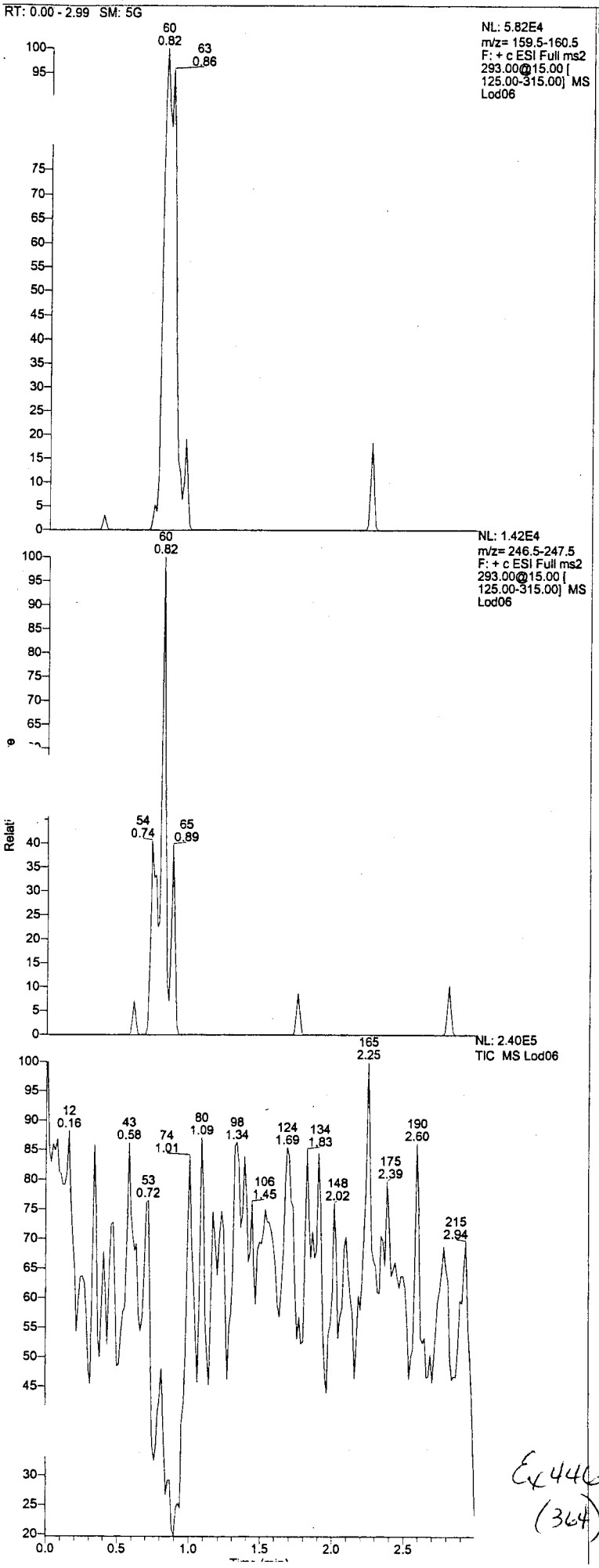


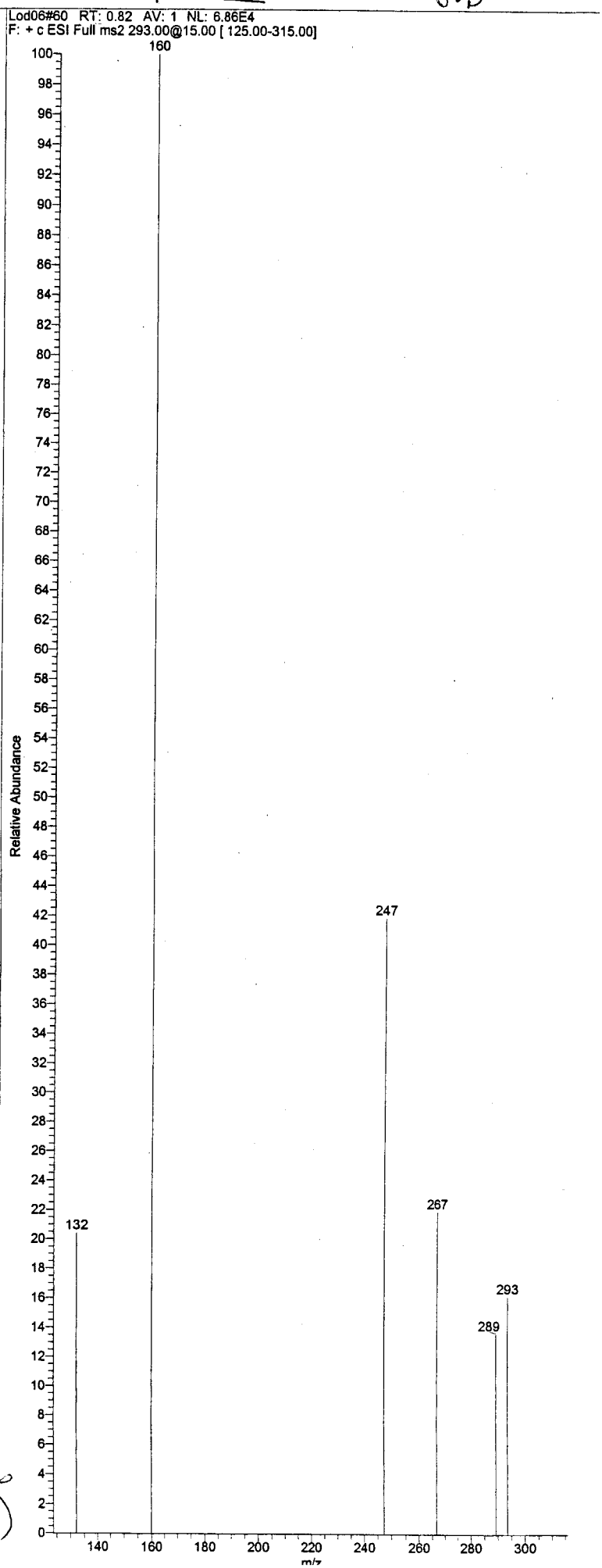


Ex 446
(363)



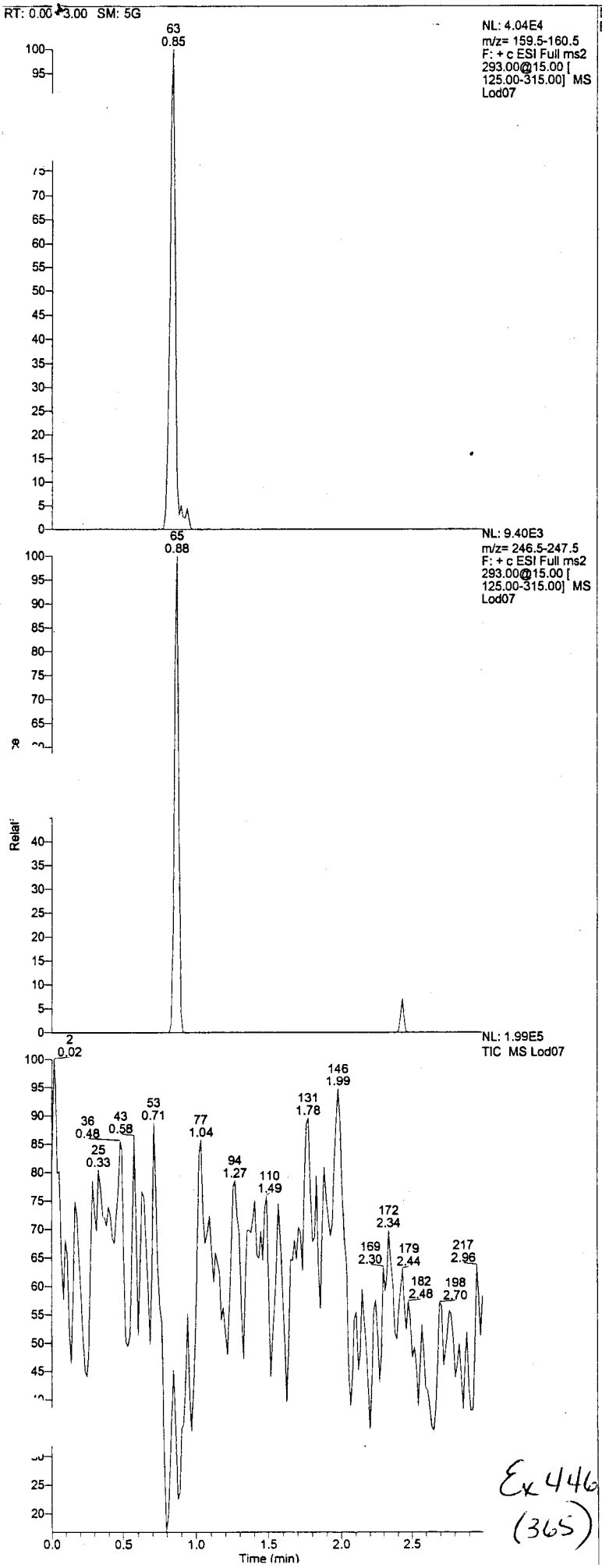
C:\Xcalibur\...\LOD_Positive\Lod06 02/12/07 03:03:06 50 ppm EDTA JDB



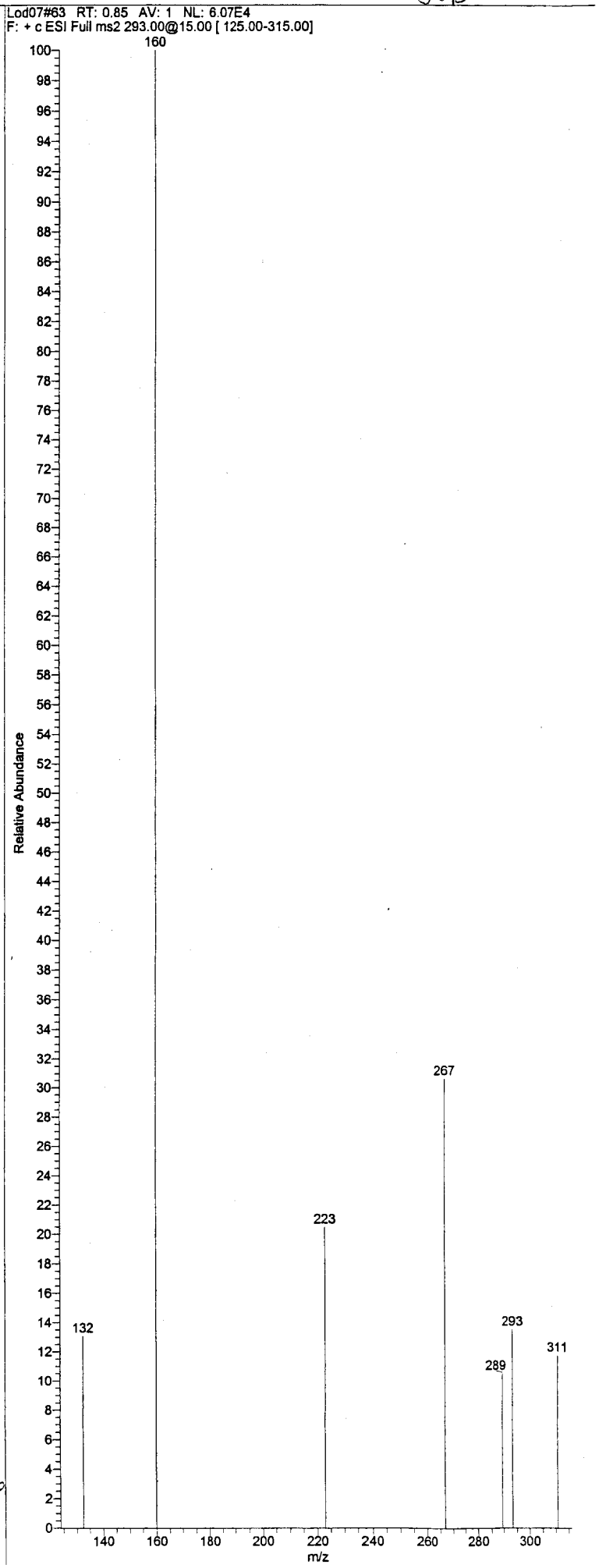


Ex 446
(364)

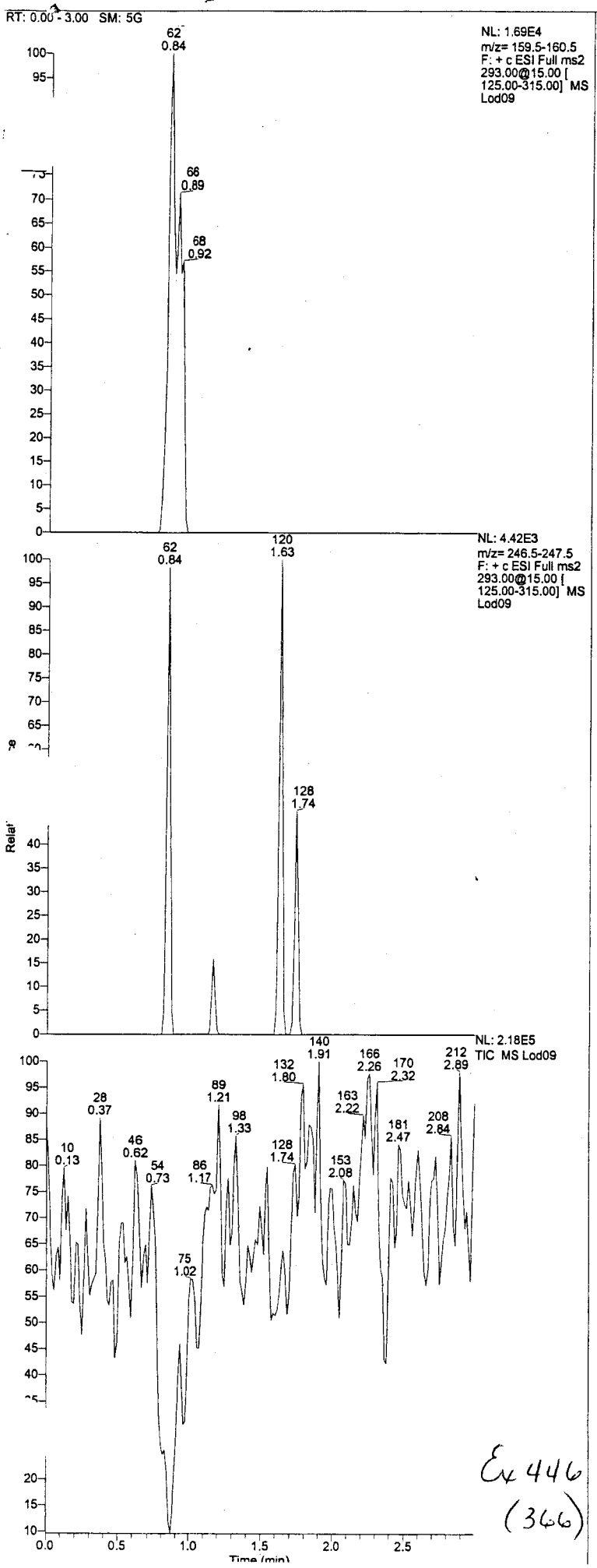
C:\Xcalibur\...\LOD_Positive\Lod07 02/12/07 03:06:59 25 ppm EDTA JDB

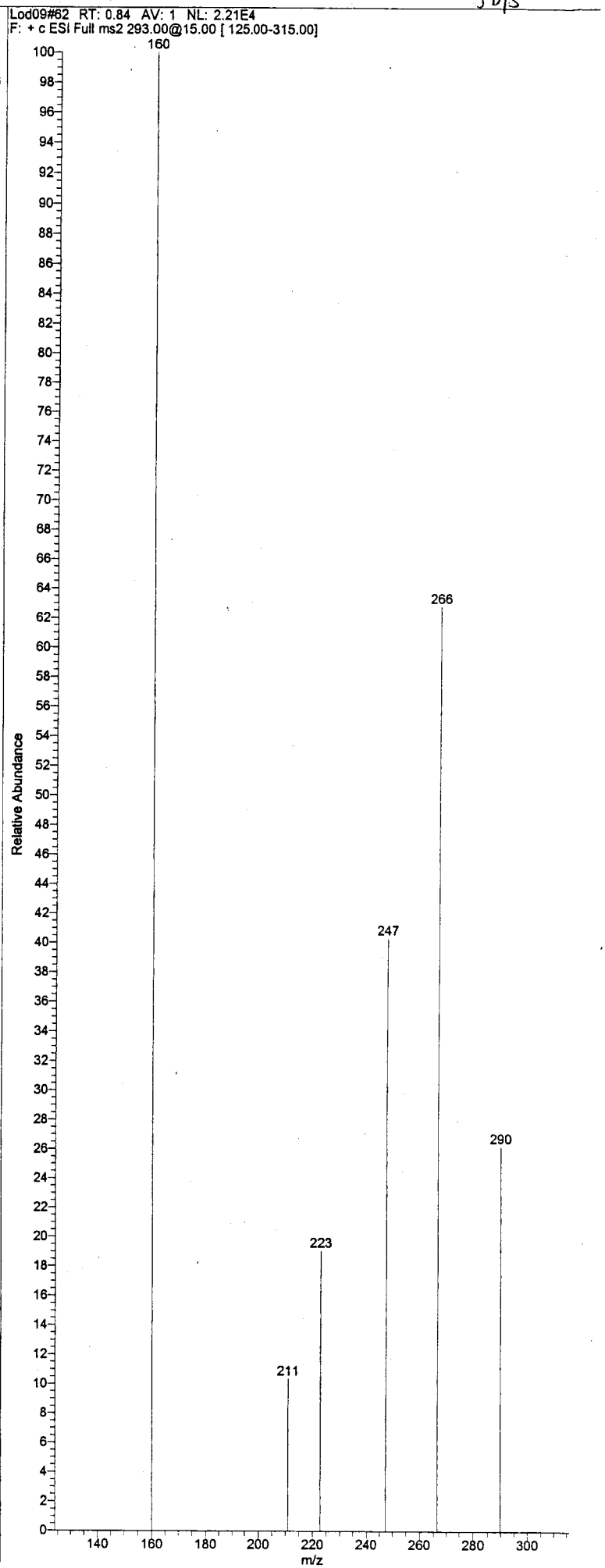


Ex 446
(365)



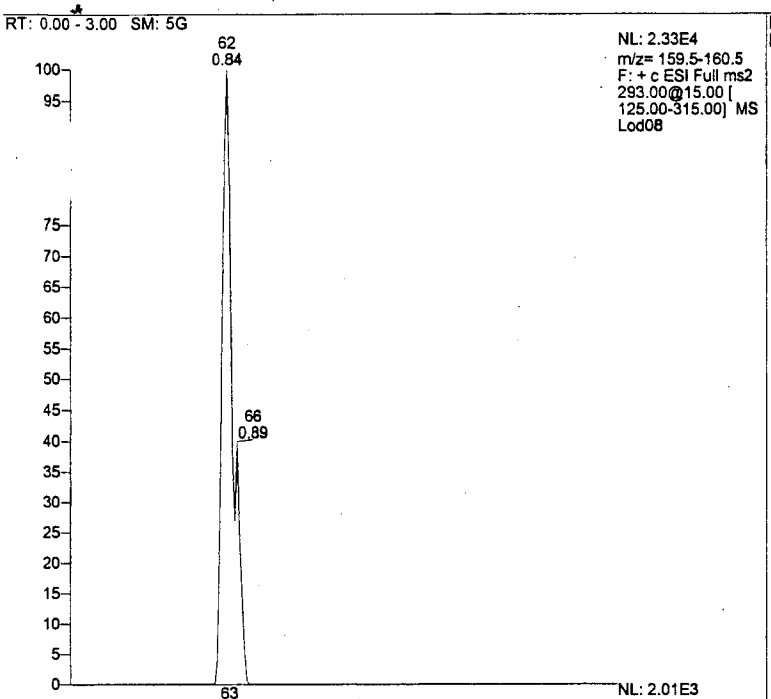
C:\Xcalibur\...\LOD_Positive\Lod09 02/12/07 03:14:36 25 ppm EDTA JDB

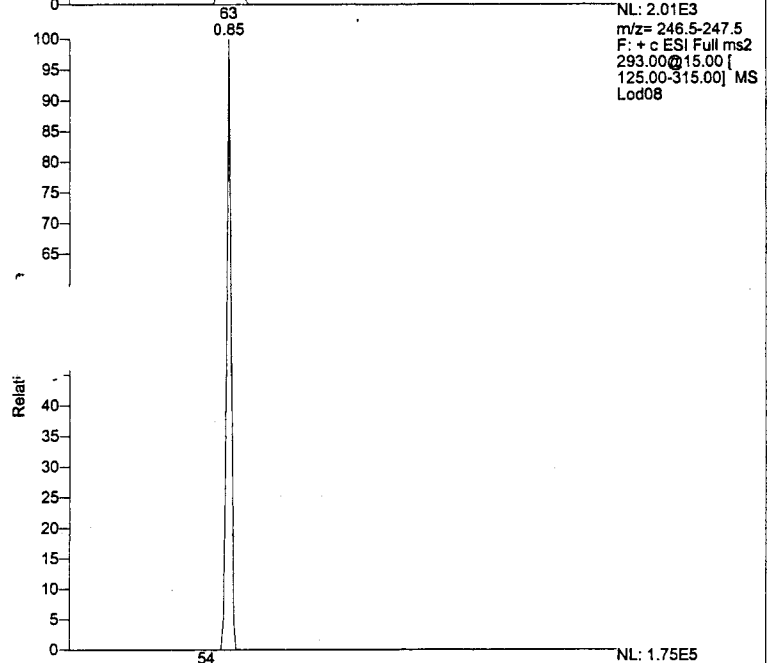


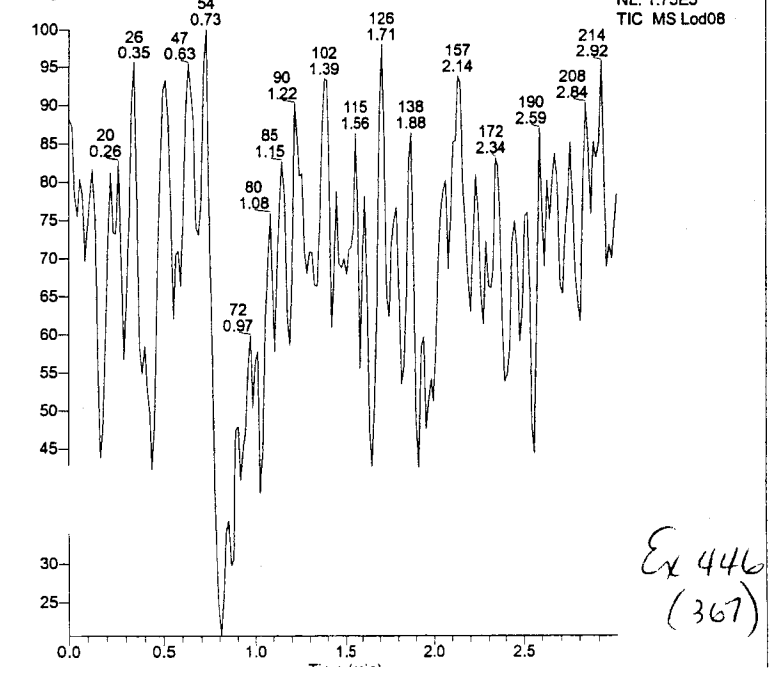


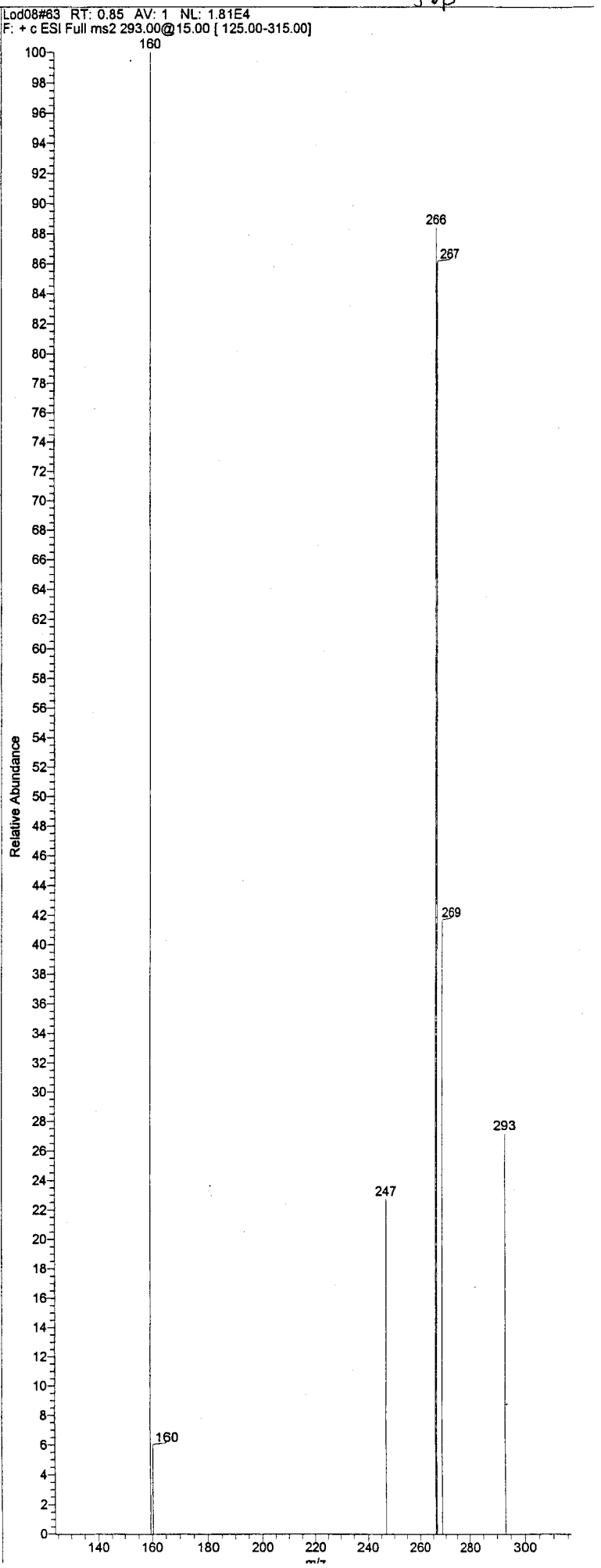
Ex 446
(366)

C:\Xcalibur\...\LOD_Positive\Lod08 02/12/07 03:10:47 25 ppm EDTA JDB



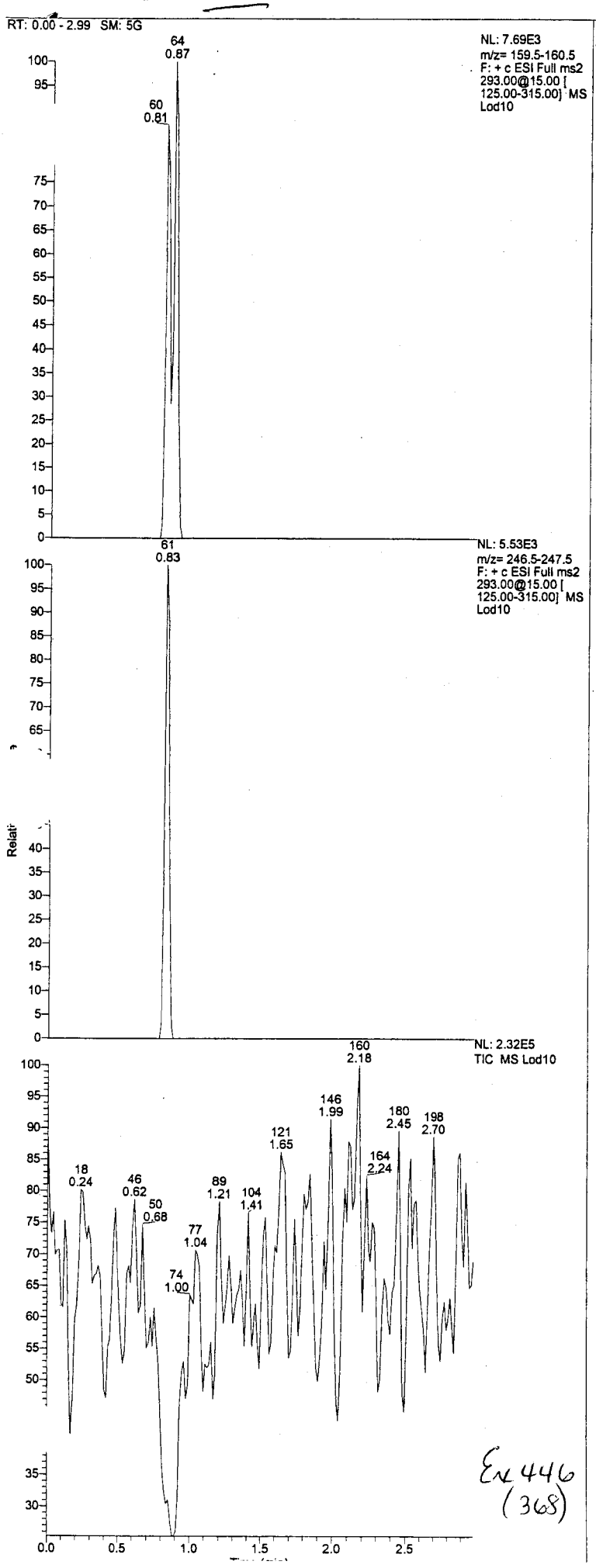




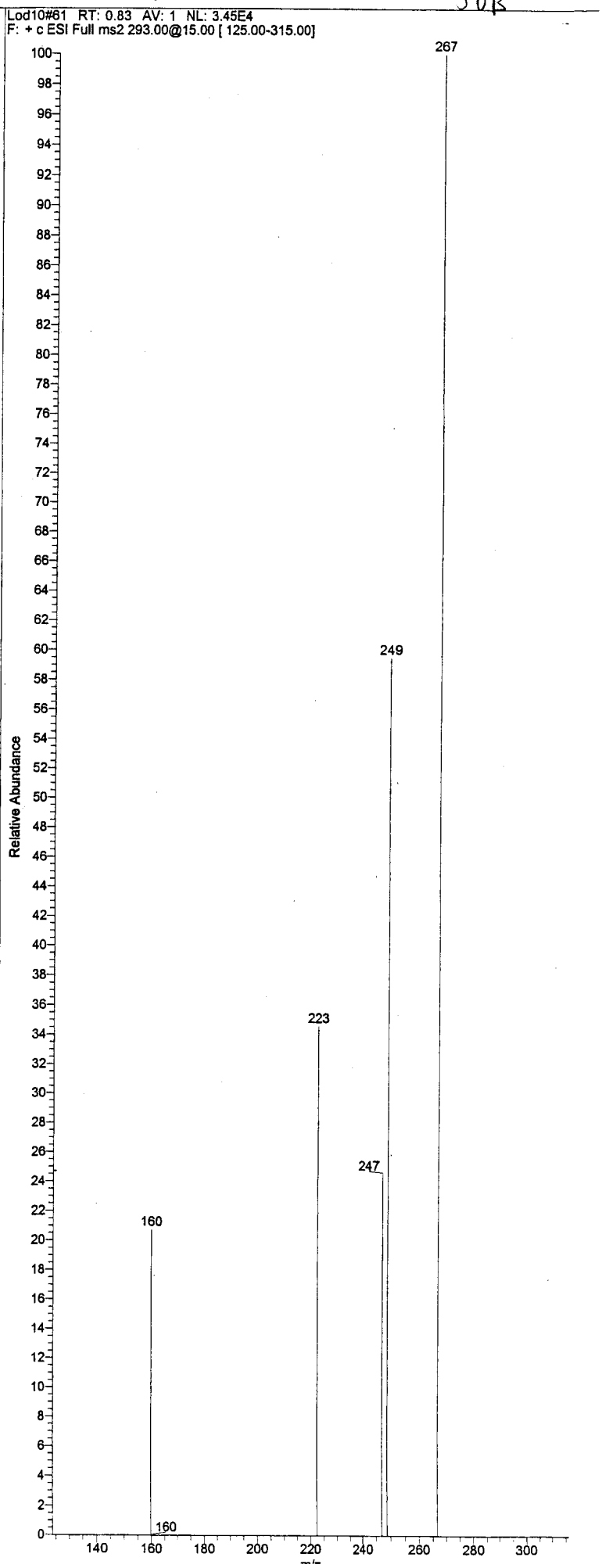


Ex 446
(367)

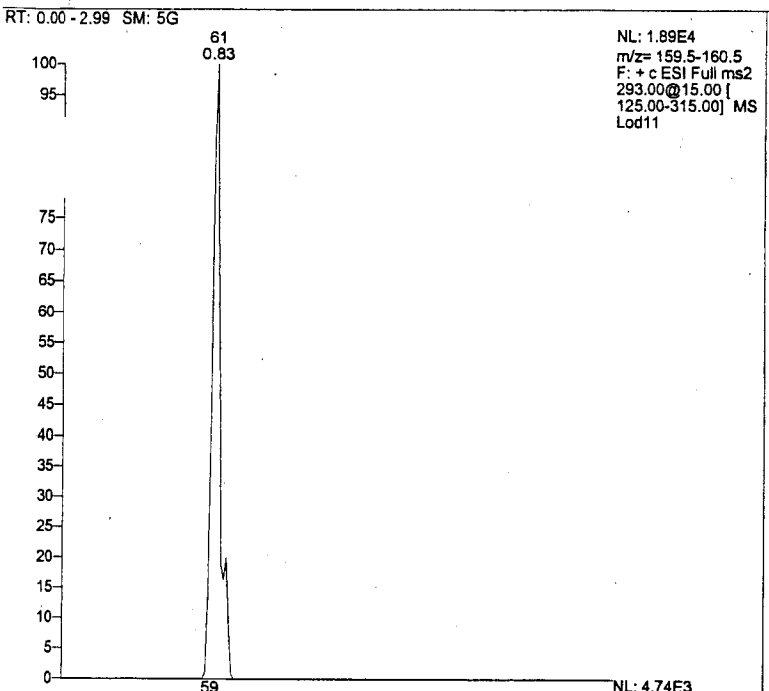
C:\Xcalibur\...\LOD_Positive\Lod10 02/12/07 03:18:25 13 ppm EDTA JDR

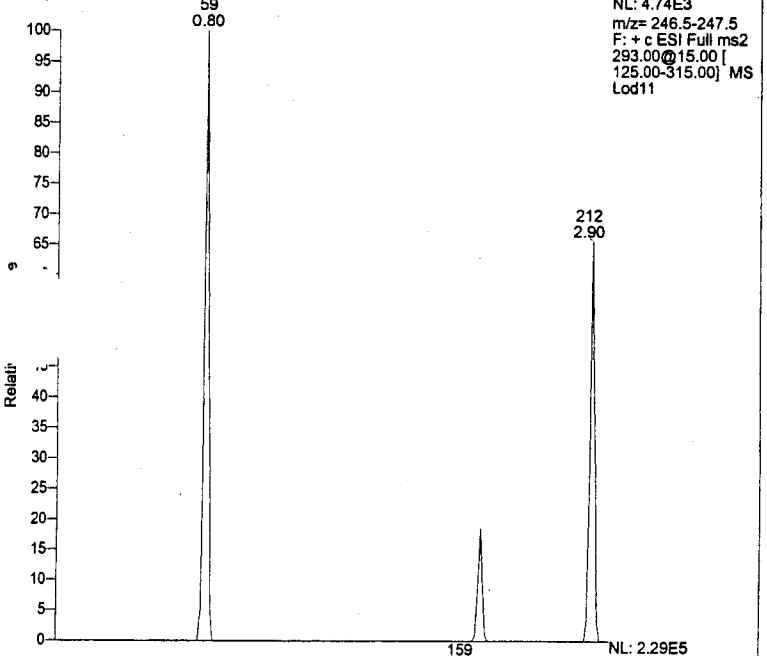


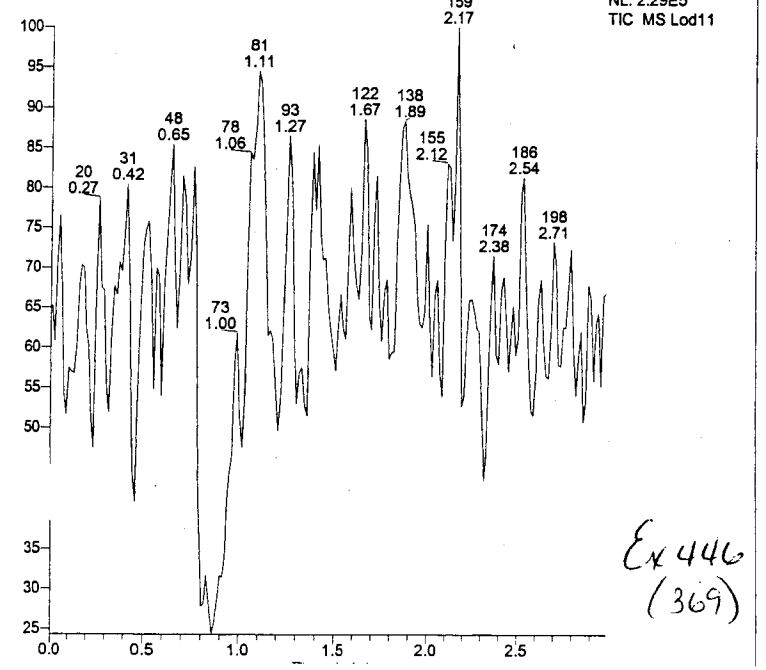
Ex 446 (368)

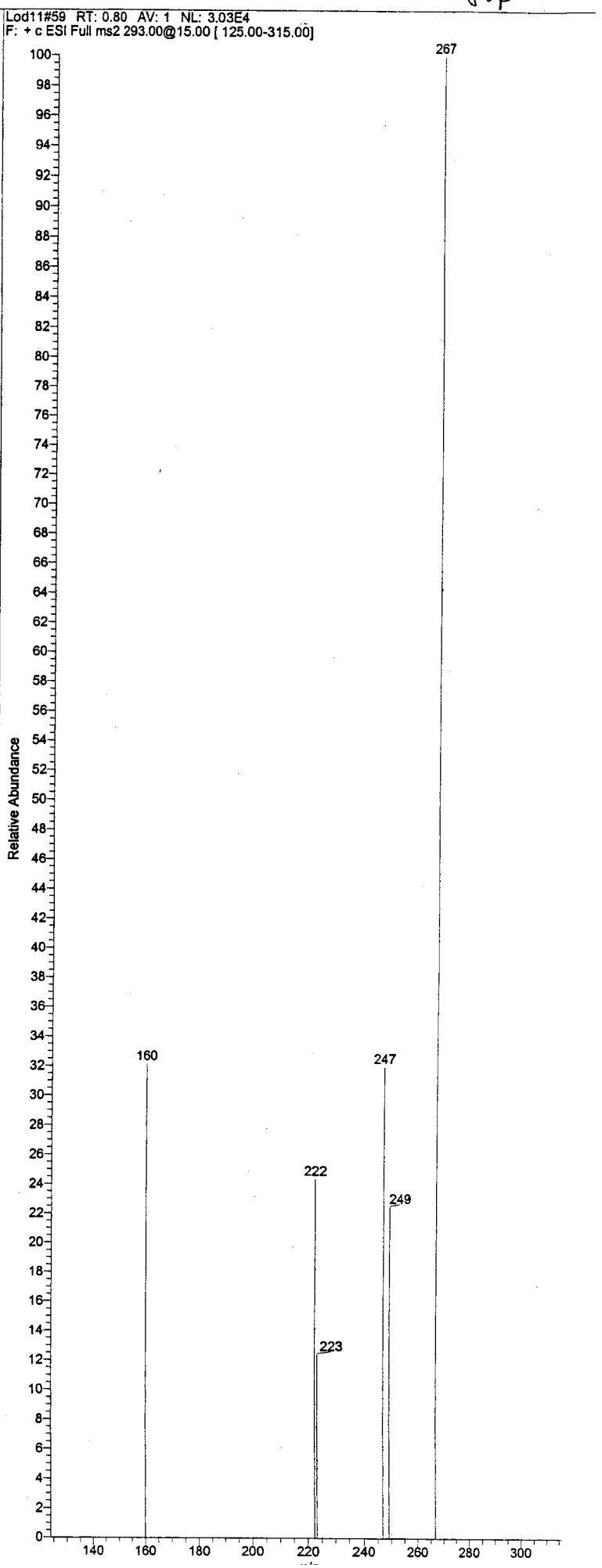


C:\Xcalibur\...\LOD_Positive\Lod11 02/12/07 03:22:11 13 ppm EDTA JDB



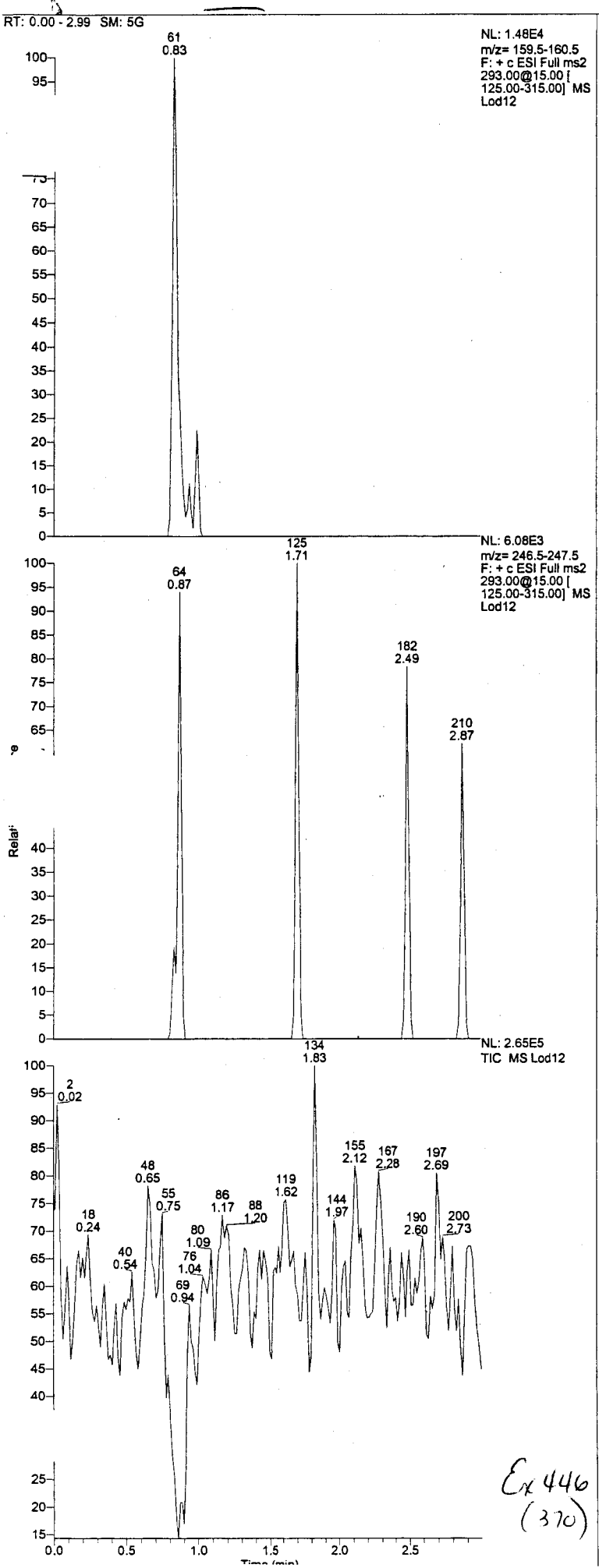


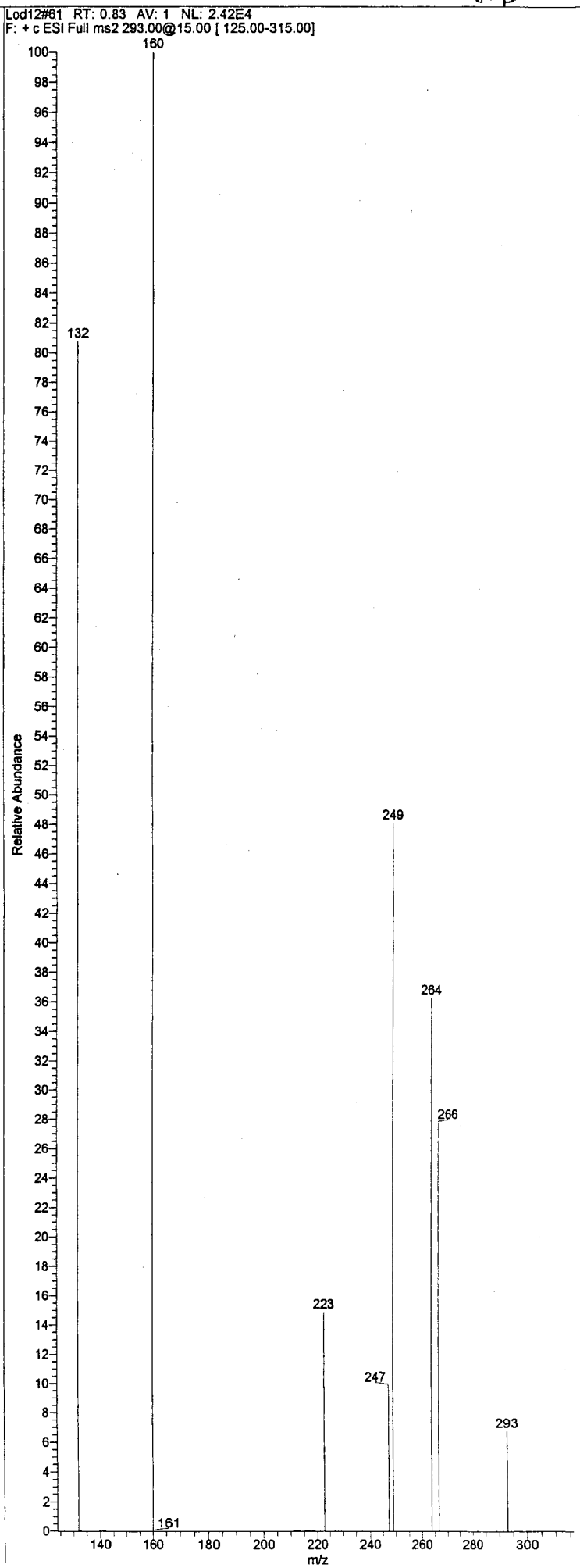




Ex 446
(369)

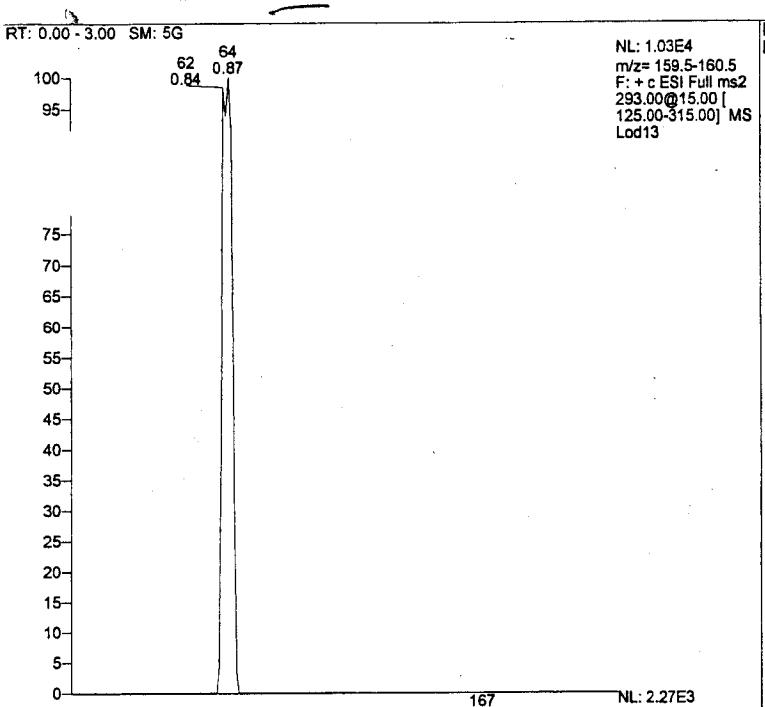
C:\Xcalibur\...\LOD_Positive\Lod12 02/12/07 03:26:02 13 ppm EDTA JDB

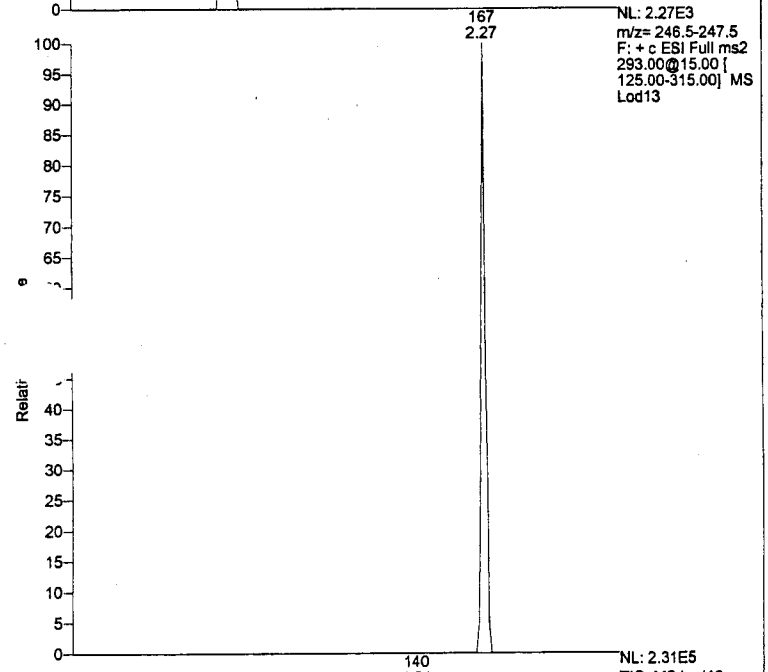


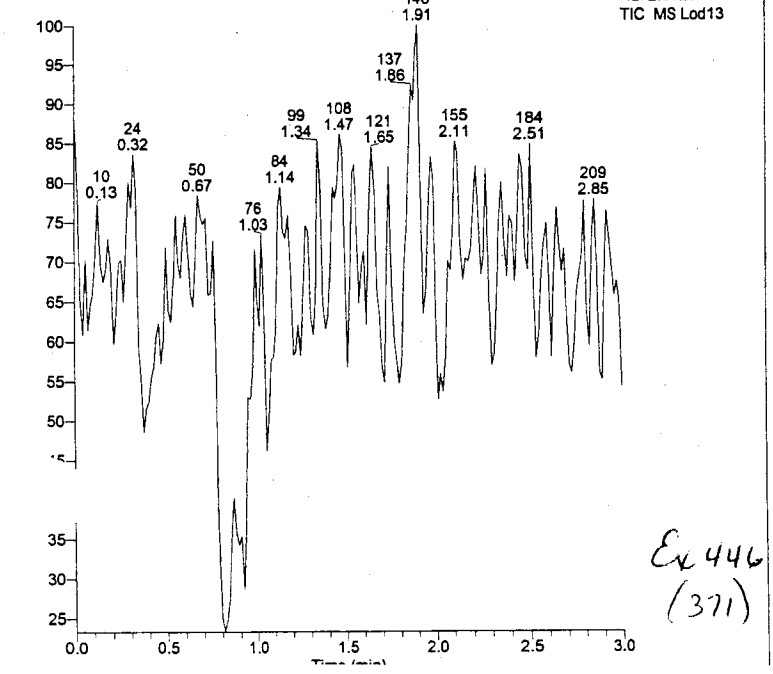


Ex 446
(370)

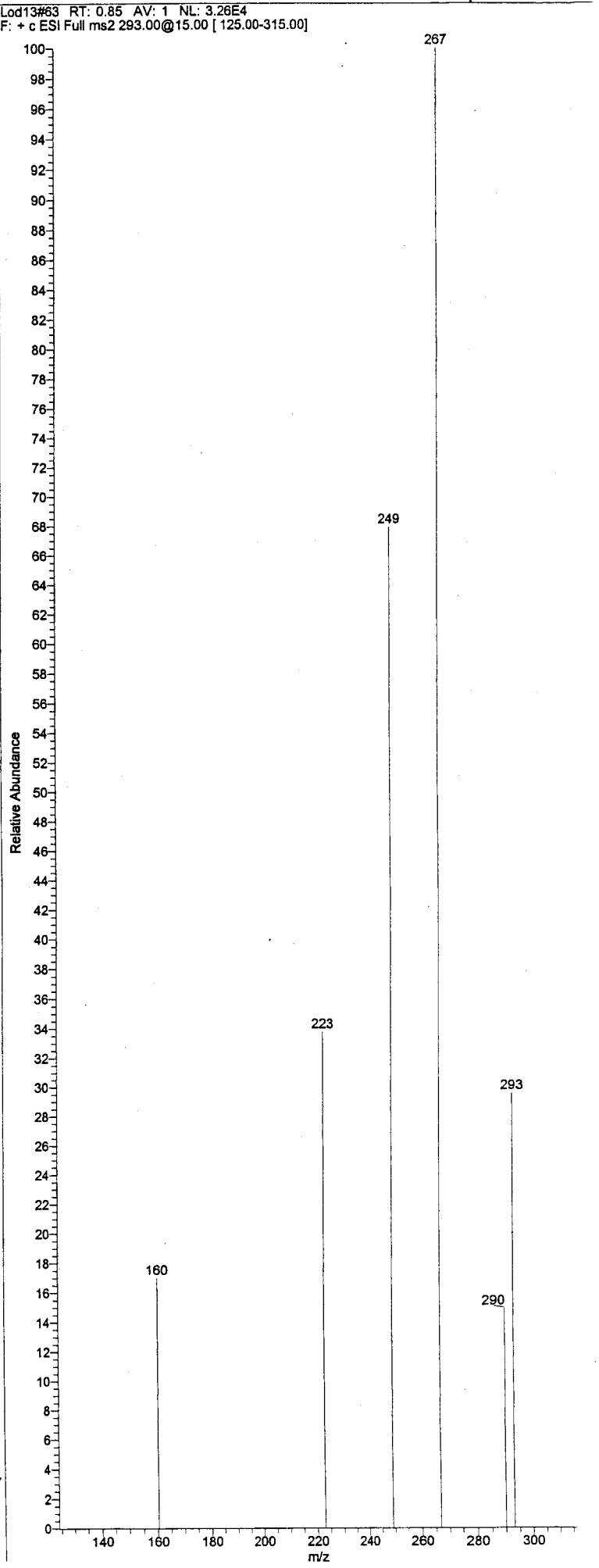
C:\Xcalibur\...\LOD_Positive\Lod13 02/12/07 03:29:50 6 ppm EDTA JDB



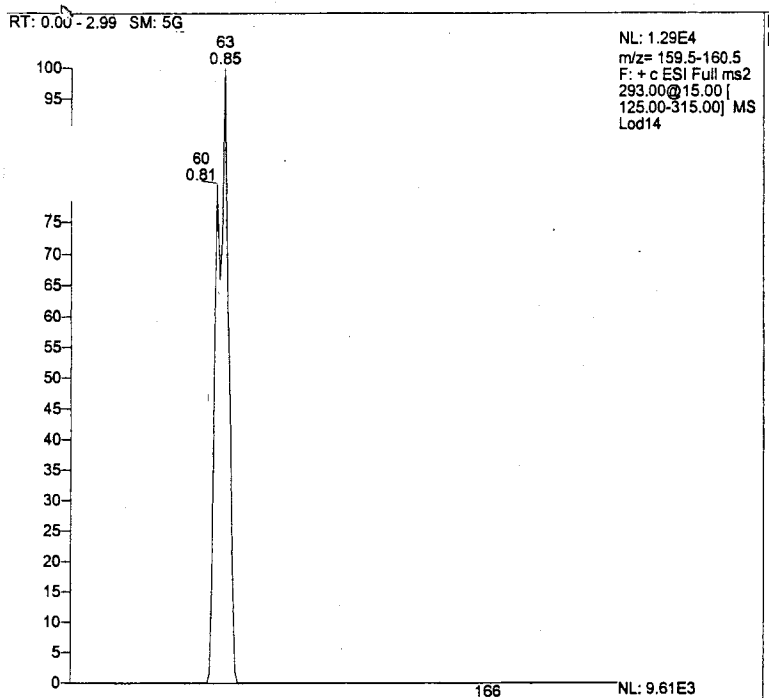


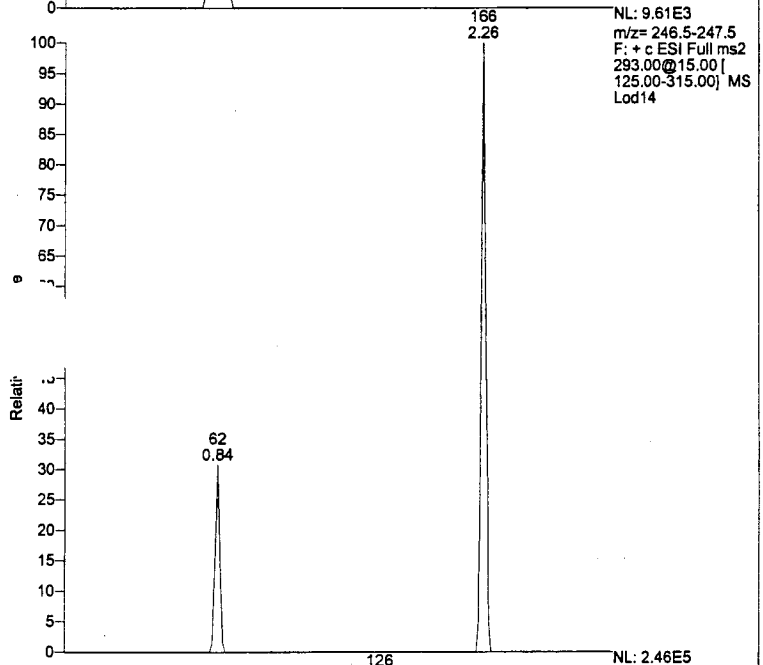


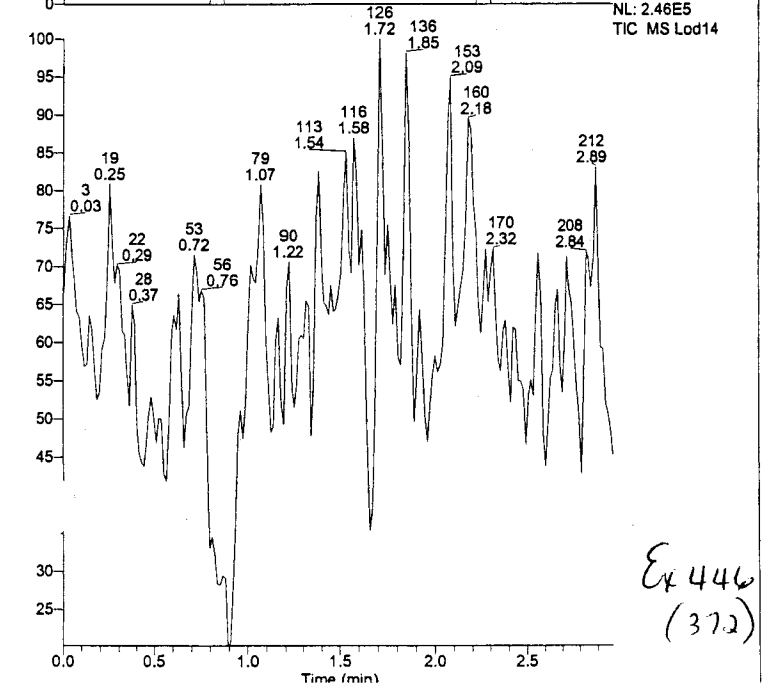
Ex 446
(371)



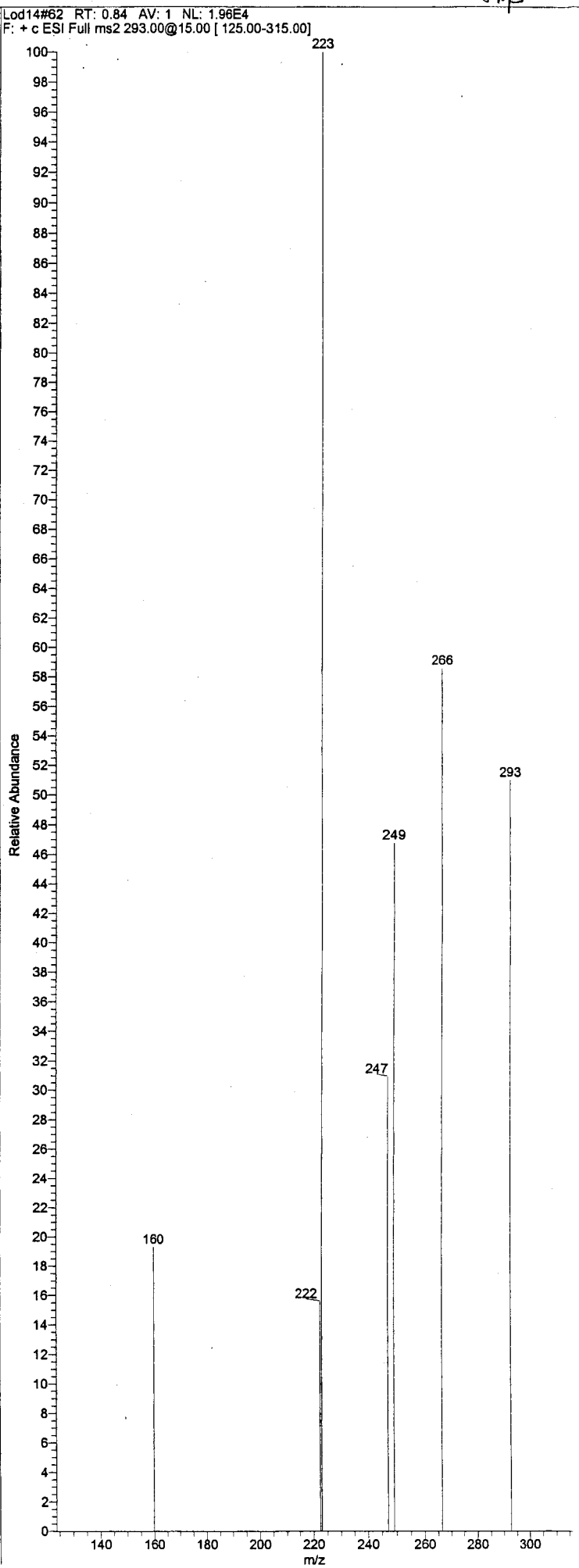
C:\Xcalibur\...\LOD_Positive\Lod14 02/12/07 03:33:39 6 ppm EDTA JDB





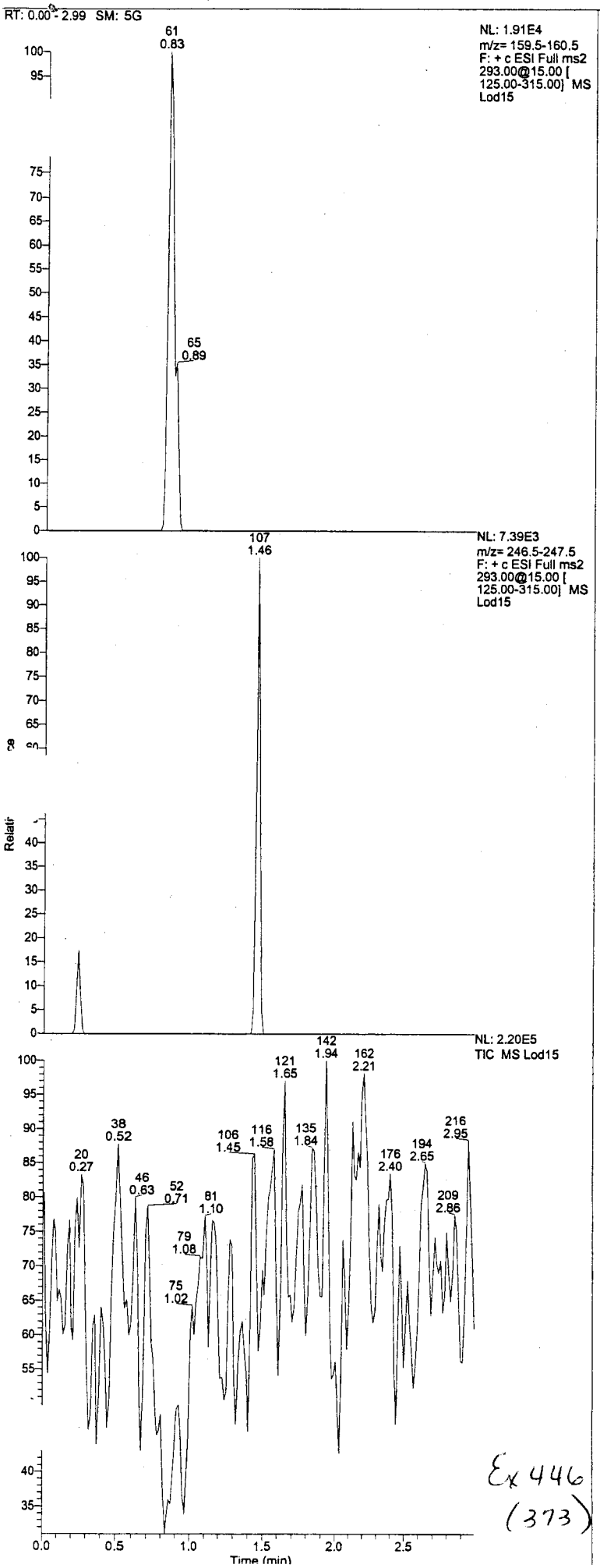


Ex 446
(372)

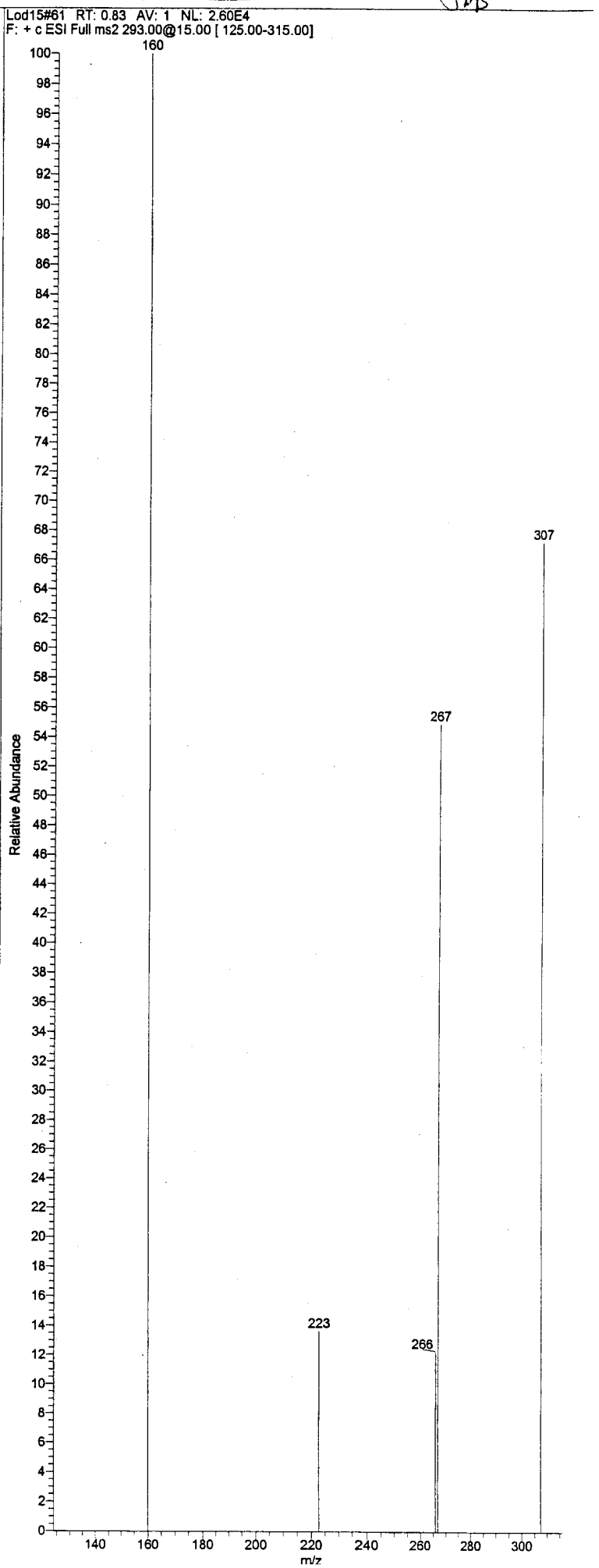


C:\Xcalibur\...\LOD_Positive\Lod15 02/12/07 03:37:26 6 ppm EDTA

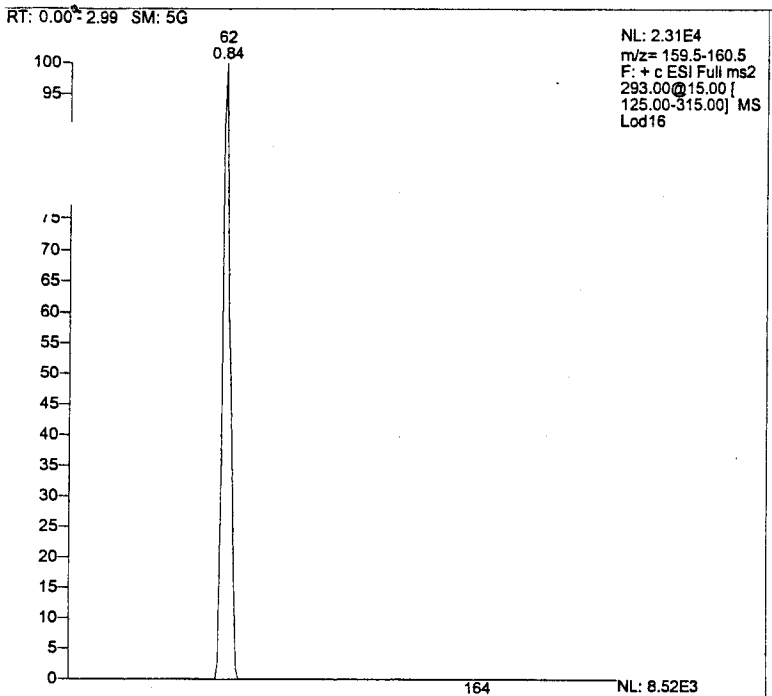
JDB

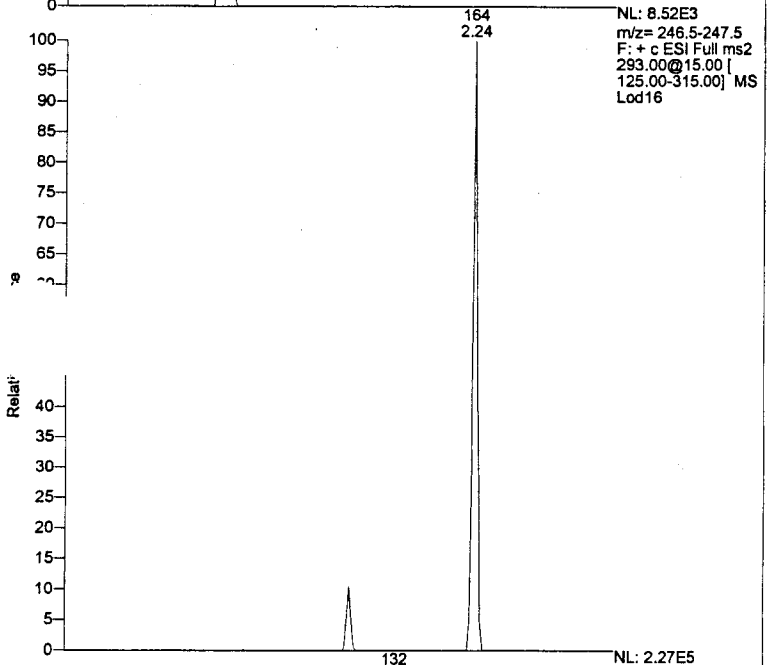


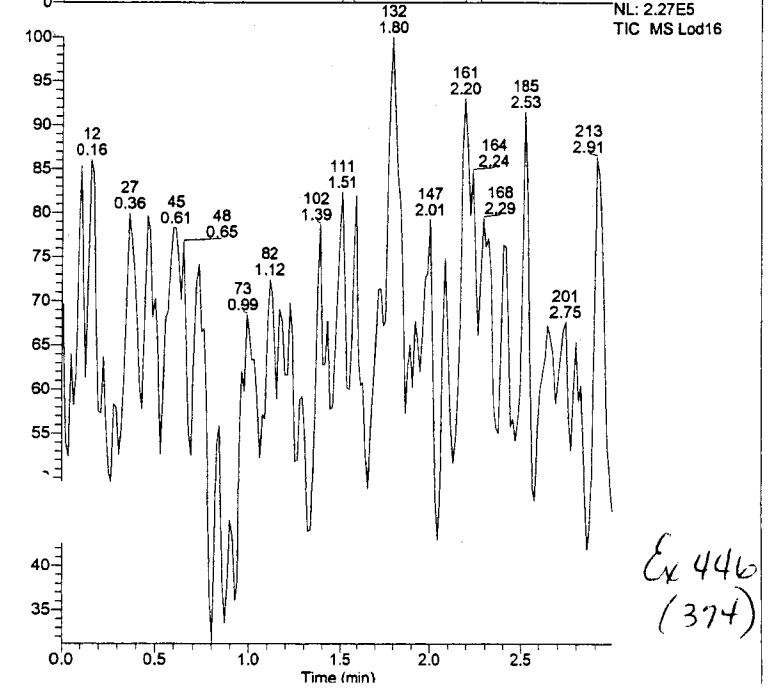
Ex 446
(373)



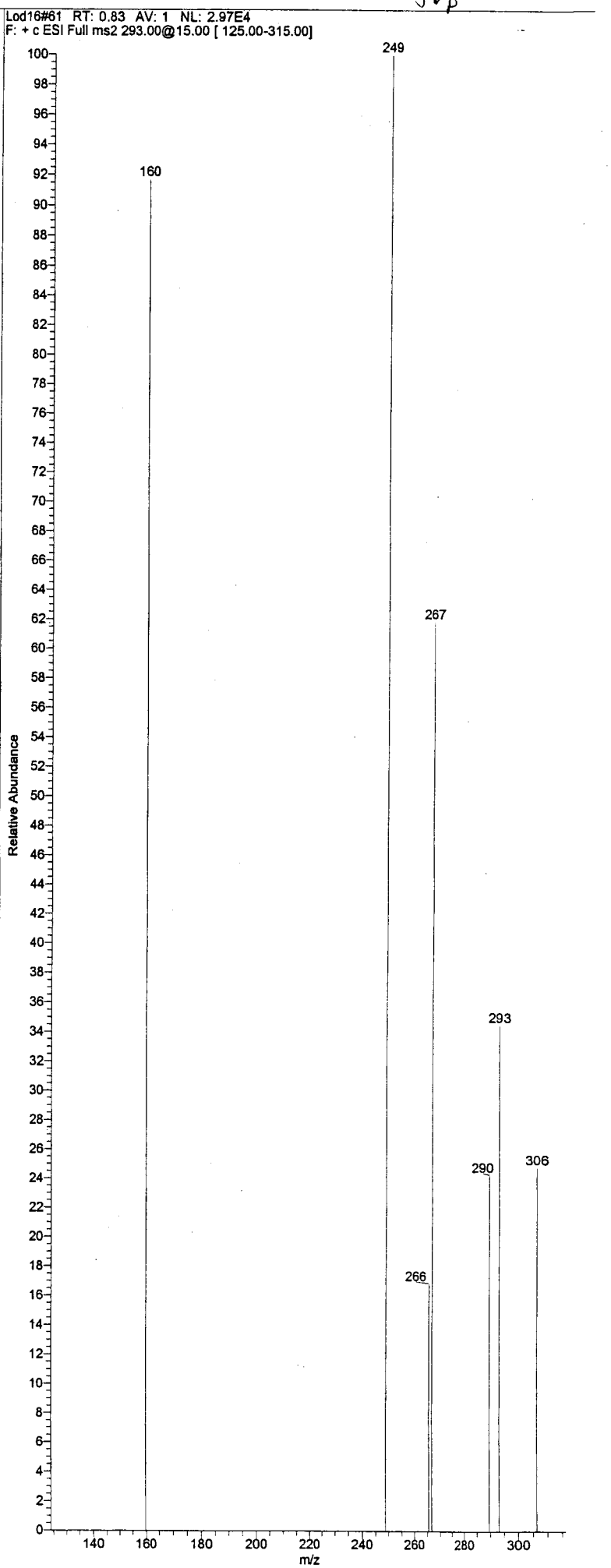
C:\Xcalibur\...\LOD_Positive\Lod16 02/12/07 03:41:16 3 ppm EDTA JDB





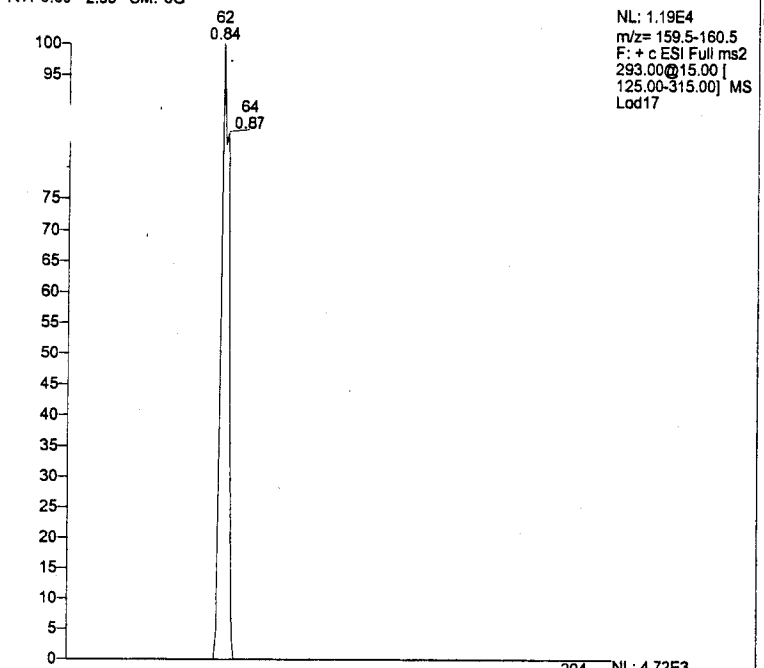


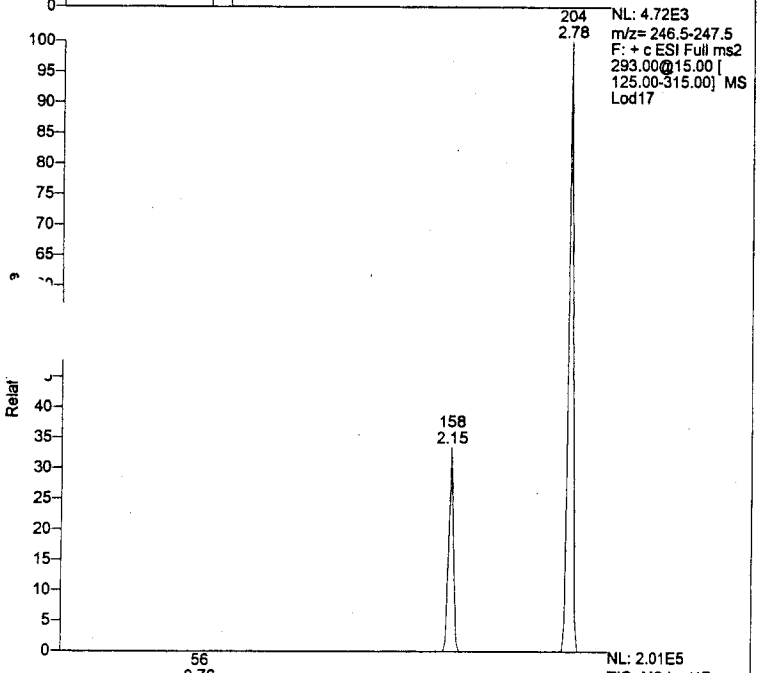
Ex 446
(374)

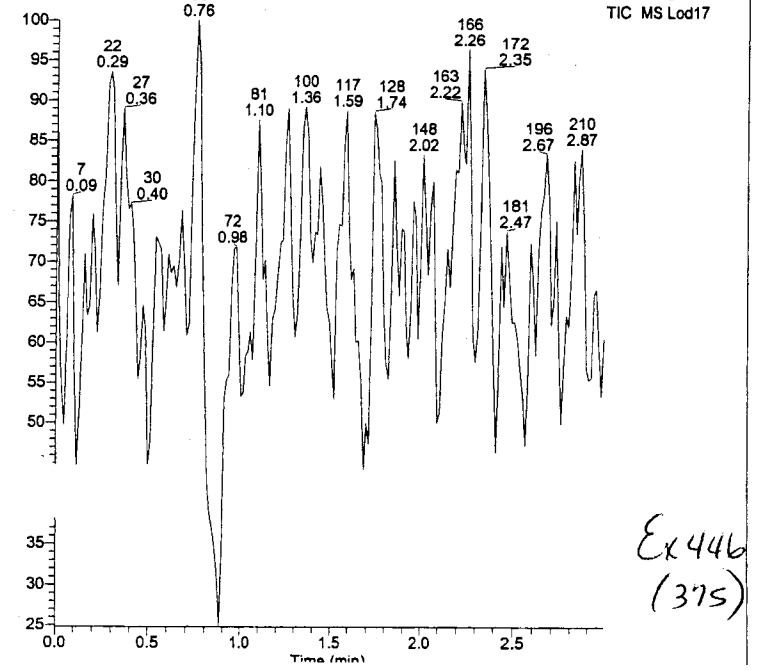


C:\Xcalibur\...\LOD_Positive\Lod17 02/12/07 03:45:05 3 ppm EDTA JDB

RT: 0.00 - 2.99 SM: 5G

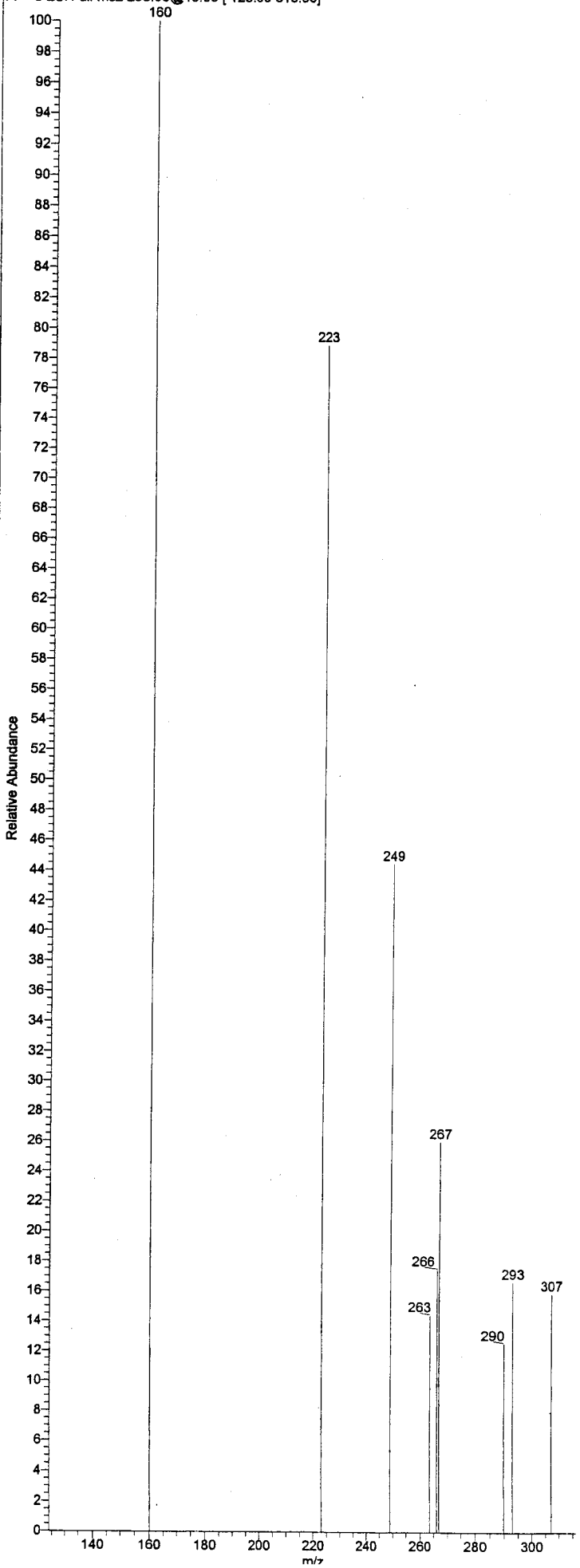




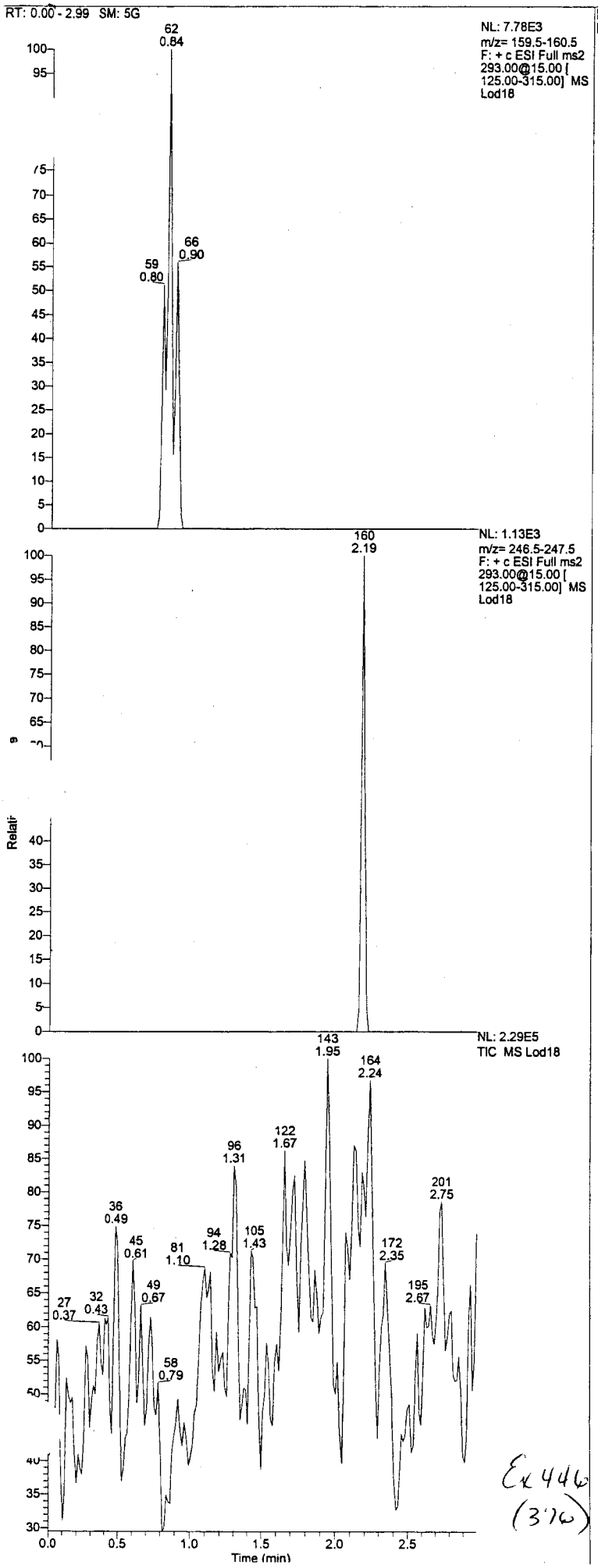


Ex 446 (375)

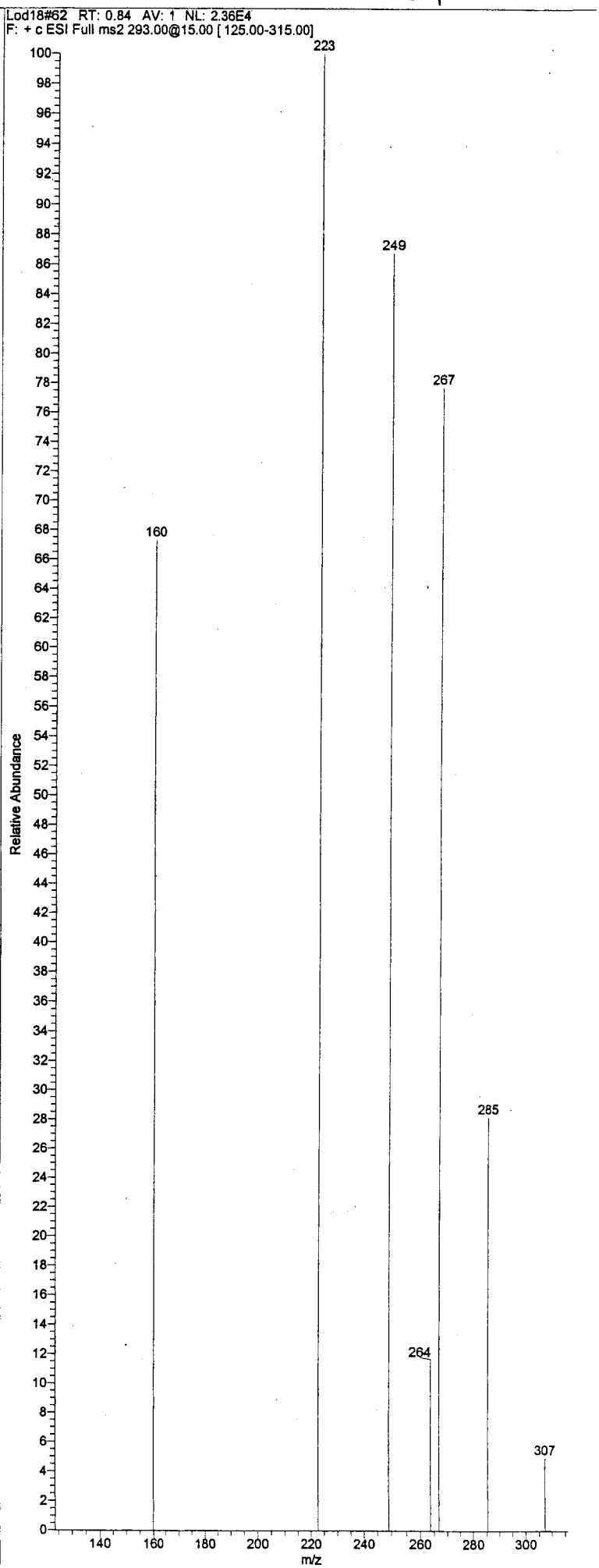
Lod17#62 RT: 0.84 AV: 1 NL: 2.33E4
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



C:\Xcalibur\...\LOD_Positive\Lod18 02/12/07 03:48:52 3 ppm EDTA JDB



Ex 446
(3%)



MATRIX

Ex 446
(377)

Sequence---MATRIX_pos.sld [Open]

Sample Name: Matrix 6 ("50 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX01	1	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			6	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Matrix 7 ("50 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX02	02	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			7	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(378)

TDP
2/12/07

Sequence---MATRIX_pos.sld [Open]

Sample Name: Matrix 8 ("50 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX03	03	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			8	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Matrix 9 ("50 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX04	04	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			9	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(379)

JnB
2/12/07

Sequence---MATRIX_pos.sld [Open]

Sample Name: Matrix 10 ("50 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX05	05	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			10	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX06	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(350)

JnB
2/12/07

Sequence---MATRIX_pos.sld [Open]

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX07	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX08	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(381)

JnP
2/12/07

Sequence---MATRIX_pos.sld [Open]

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX09	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX10	12	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(352)

JDB
2/12/07

Sequence---MATRIX_pos.sld [Open]

Sample Name: Matrix 3 ("500 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX13	03	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			3	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Matrix 4 ("500 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX14	04	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			4	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(383)

JDB
2/12/07

Sequence---MATRIX_pos.sld [Open]

Sample Name: Matrix 5 ("500 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX15	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			5	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 500 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX16	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(384)

JDP
2/12/07

Sequence---MATRIX_pos.sld [Open]

Sample Name: 500 ppm EDTA

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX17	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 500 ppm EDTA

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX18	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(355)

JnP
2/12/07

Sequence---MATRIX_pos.sld [Open]

Sample Name: 500 ppm EDTA

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX19	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 500 ppm EDTA

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX20	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(386)

JnP
2/12/07

Sequence---MATRIX_pos.sld [Open]

Sample Name: Matrix 1 ("500 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX11	01	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Matrix 2 ("500 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX12	02	C:\Xcalibur\Data\EDTA\Brewer\MAT_Positive

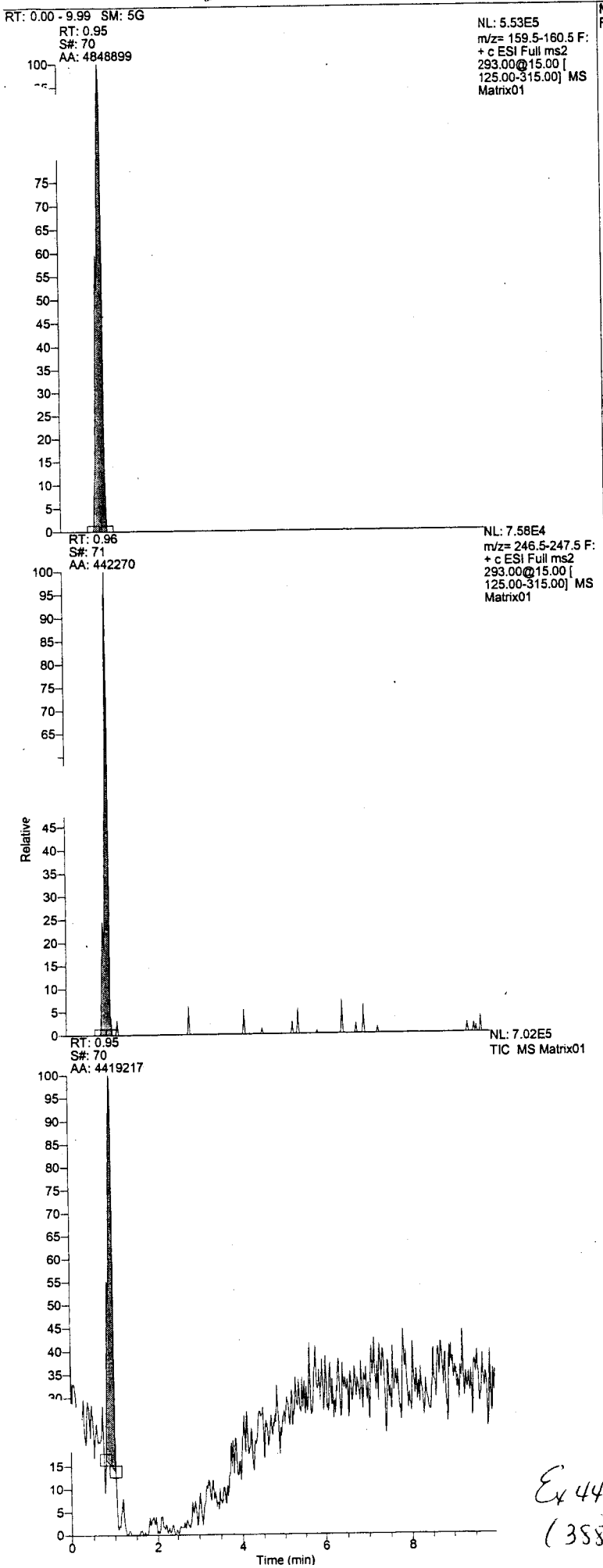
Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			2	5.0		0.000

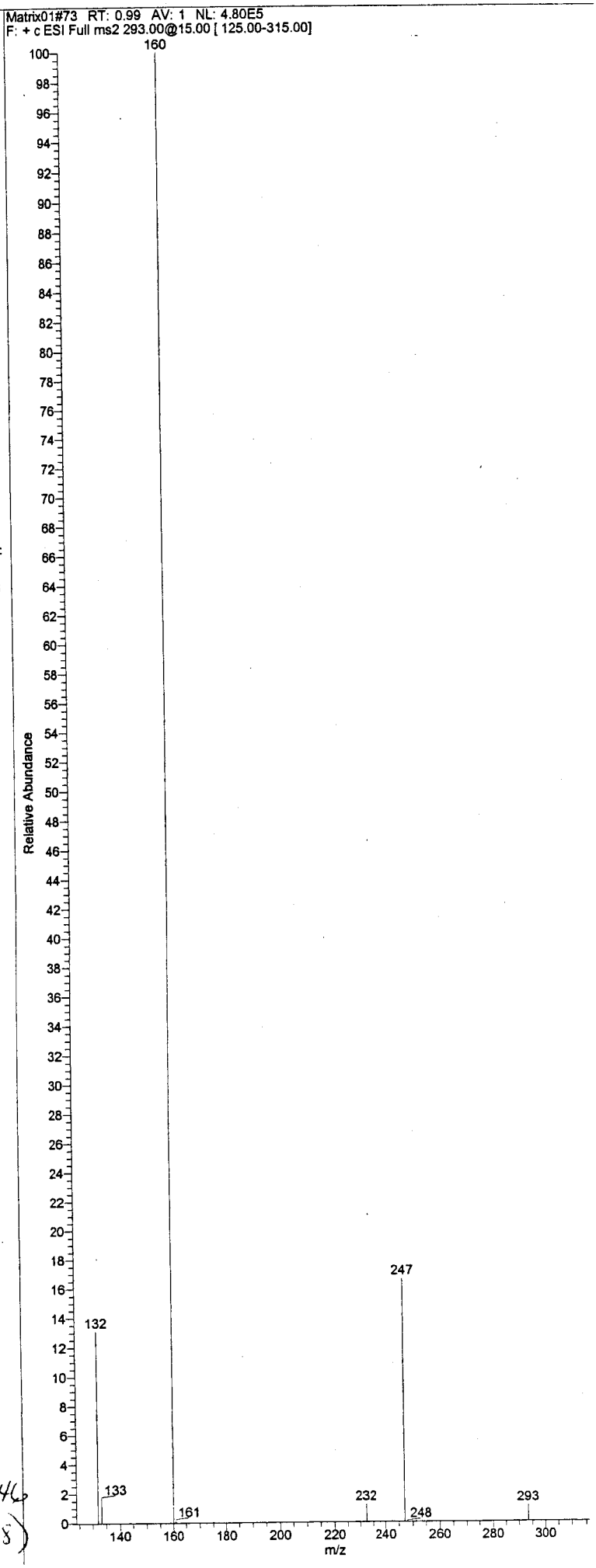
Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(351)

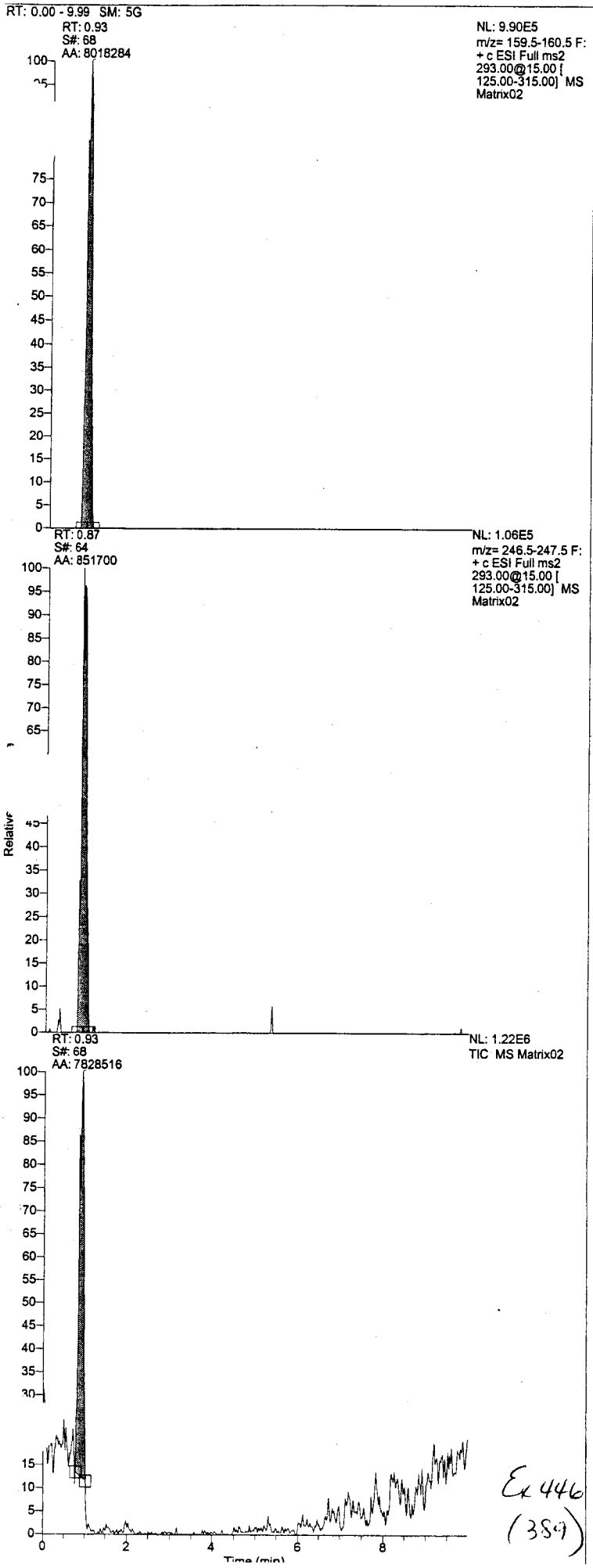
JOB
2/12/07

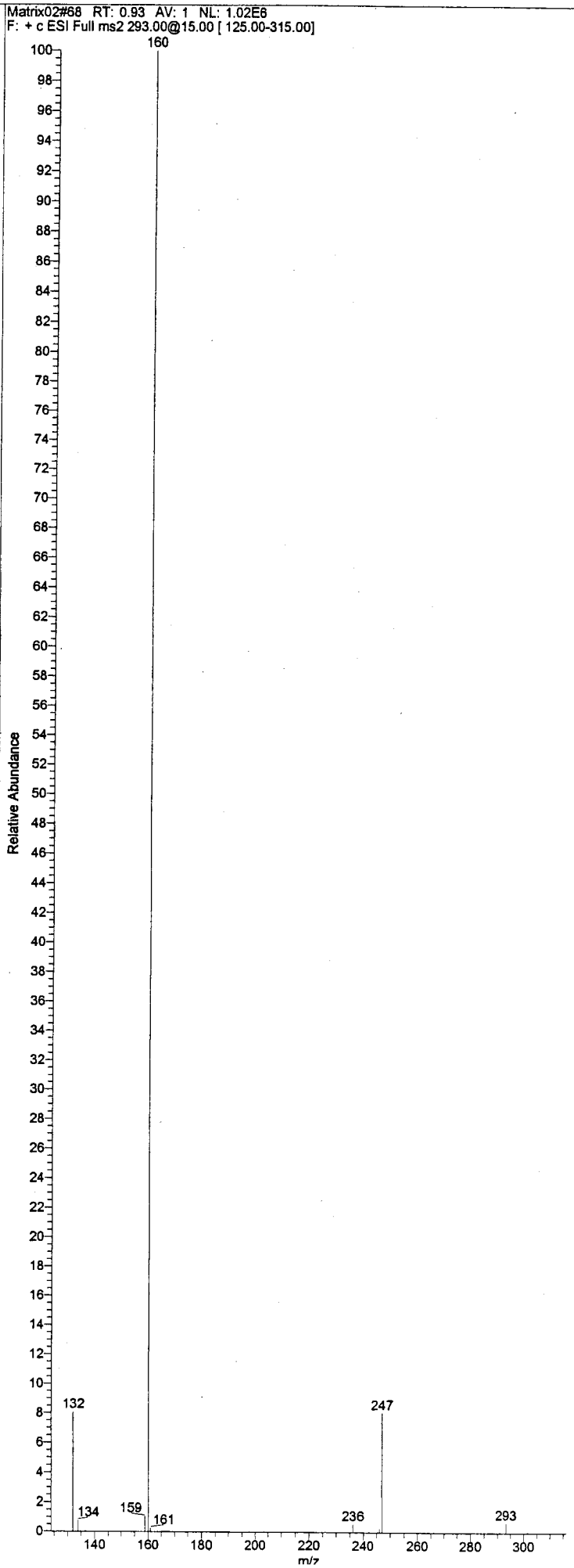

RT: 0.00 - 9.99 SM: 5G
RT: 0.95
S#: 70
AA: 4848899
NL: 5.53E5
m/z= 159.5-160.5 F: + c ESI Full ms2 293.00@15.00 [125.00-315.00] MS Matrix01
RT: 0.96
S#: 71
AA: 442270
NL: 7.58E4
m/z= 246.5-247.5 F: + c ESI Full ms2 293.00@15.00 [125.00-315.00] MS Matrix01
Relative
RT: 0.95
S#: 70
AA: 4419217
NL: 7.02E5
TIC MS Matrix01
Time (min)


Matrix01#73 RT: 0.99 AV: 1 NL: 4.80E5
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]
Relative Abundance
160
247
132
133
161
232
248
293
m/z

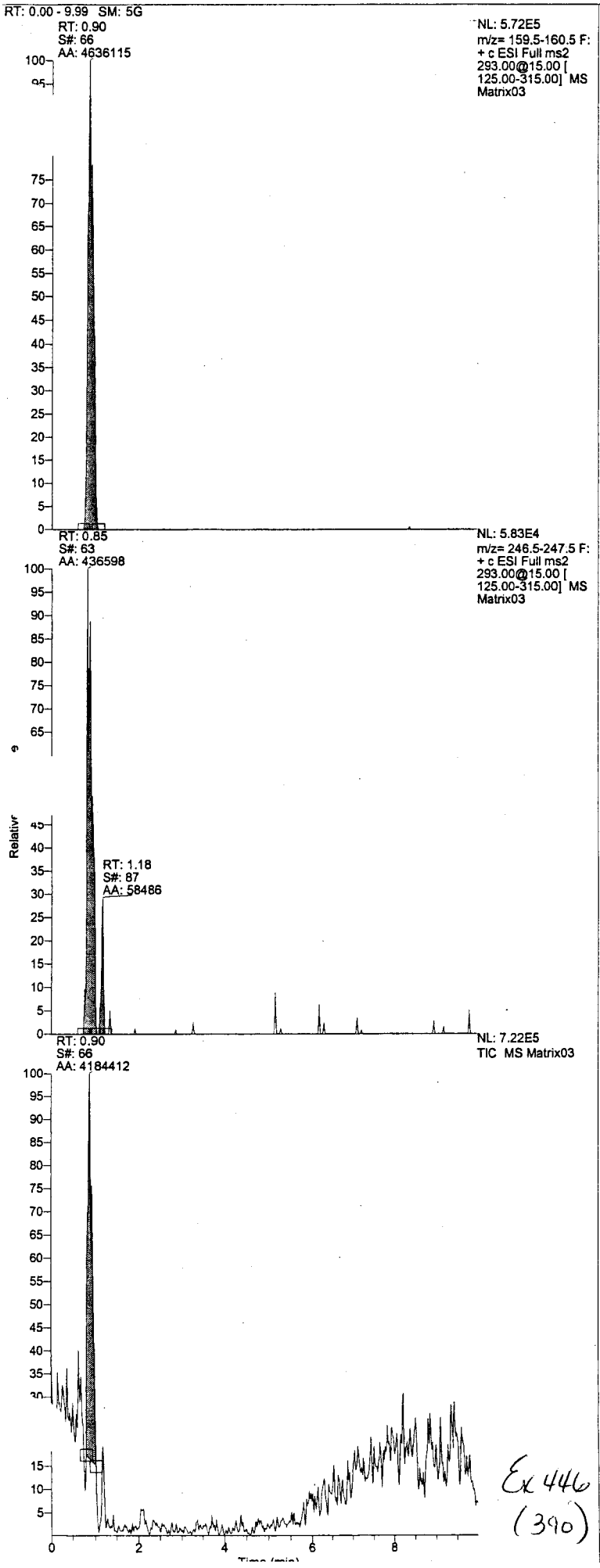
Ex 446
(358)

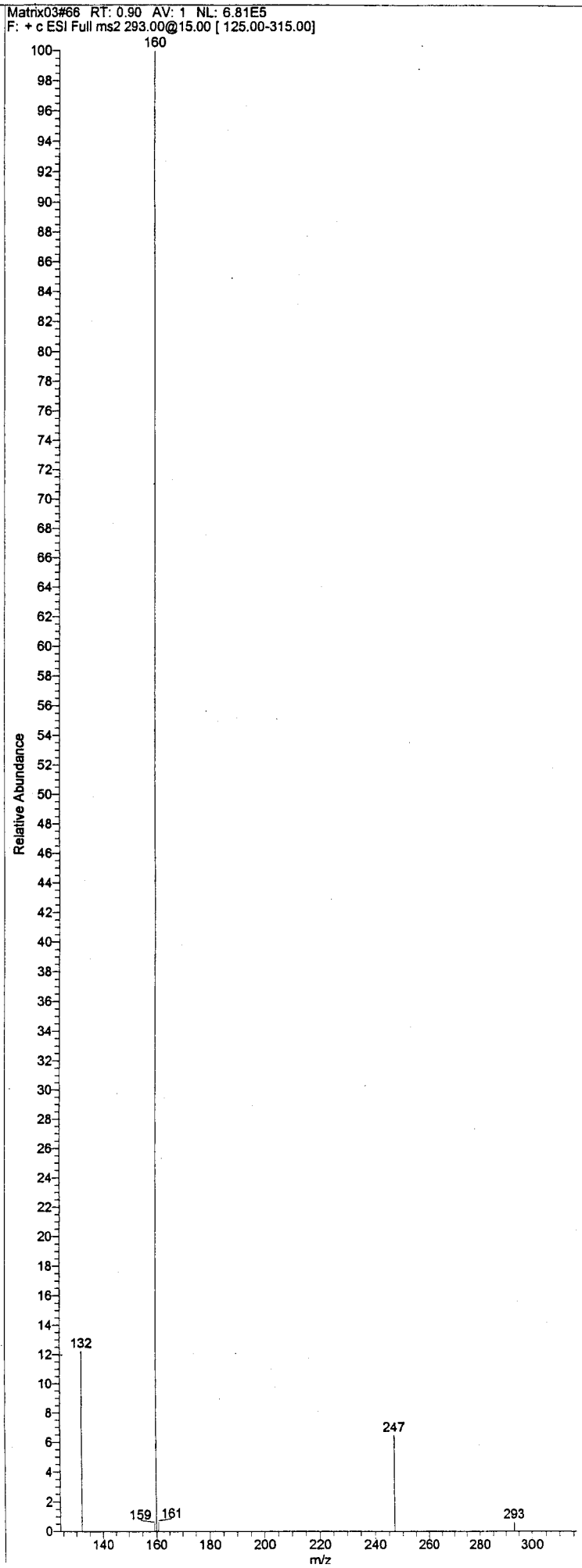
C:\Xcalibur\...\MAT_Positive\Matrix02 02/12/07 04:16:49 Matrix 7 ("50 ppm") JDB



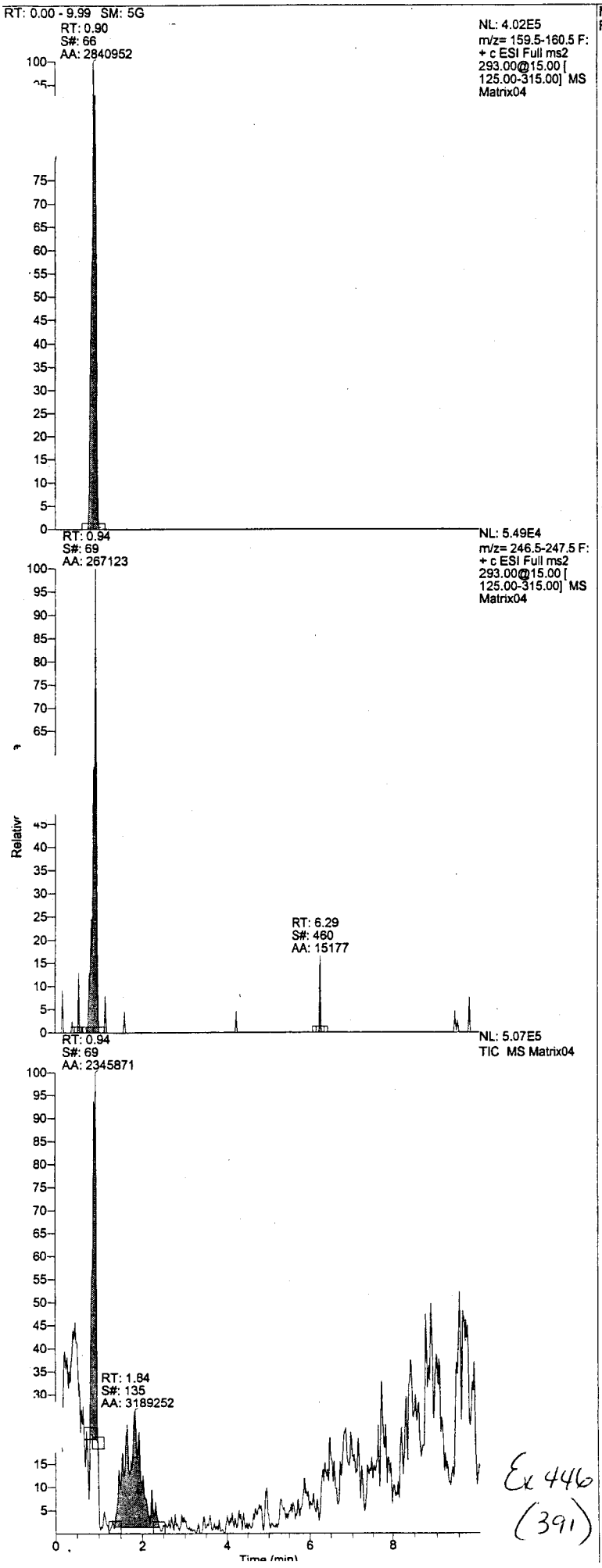


Ex 446
(389)

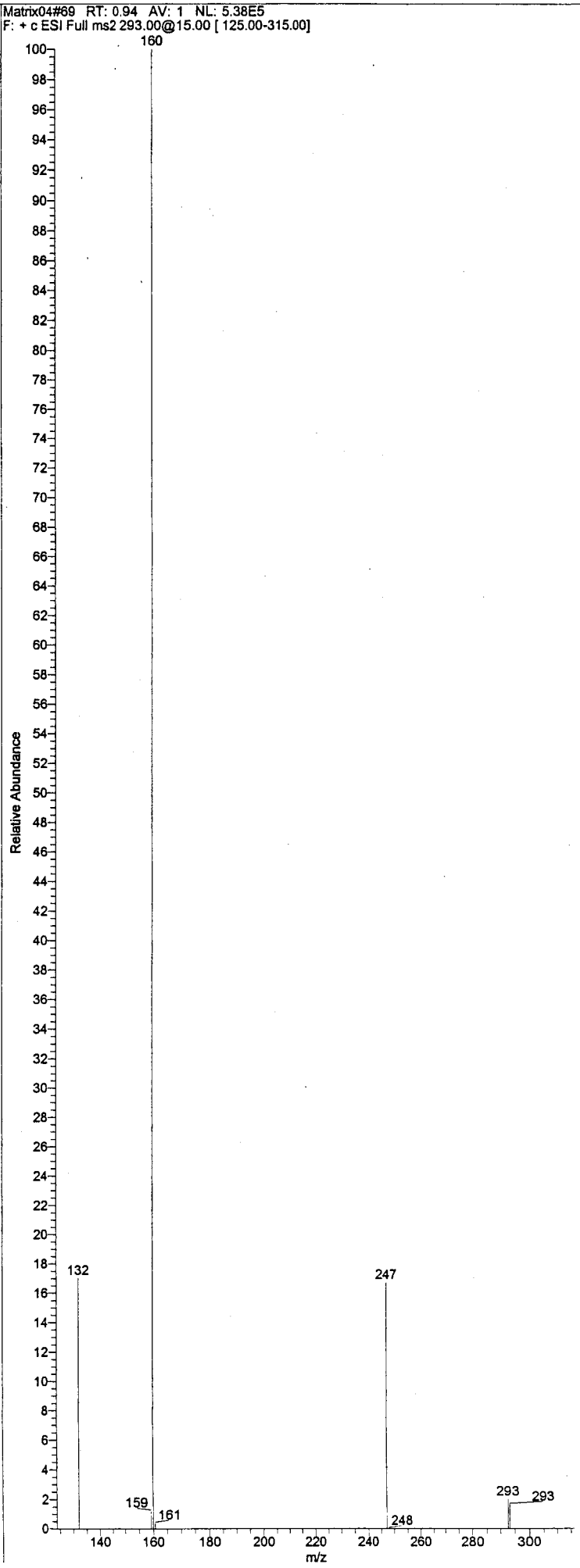

RT: 0.00 - 9.99 SM: 5G
RT: 0.90
S#: 66
AA: 4636115
NL: 5.72E5
m/z= 159.5-160.5 F: + c ESI Full ms2 293.00@15.00 [125.00-315.00] MS Matrix03
RT: 0.85
S#: 63
AA: 436598
NL: 5.83E4
m/z= 246.5-247.5 F: + c ESI Full ms2 293.00@15.00 [125.00-315.00] MS Matrix03
RT: 1.18
S#: 87
AA: 58486
RT: 0.90
S#: 66
AA: 4184412
NL: 7.22E5
TIC MS Matrix03
Relativ
Time (min)


Matrix03#66 RT: 0.90 AV: 1 NL: 6.81E5
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]
160
132
247
159
161
293
Relative Abundance
m/z

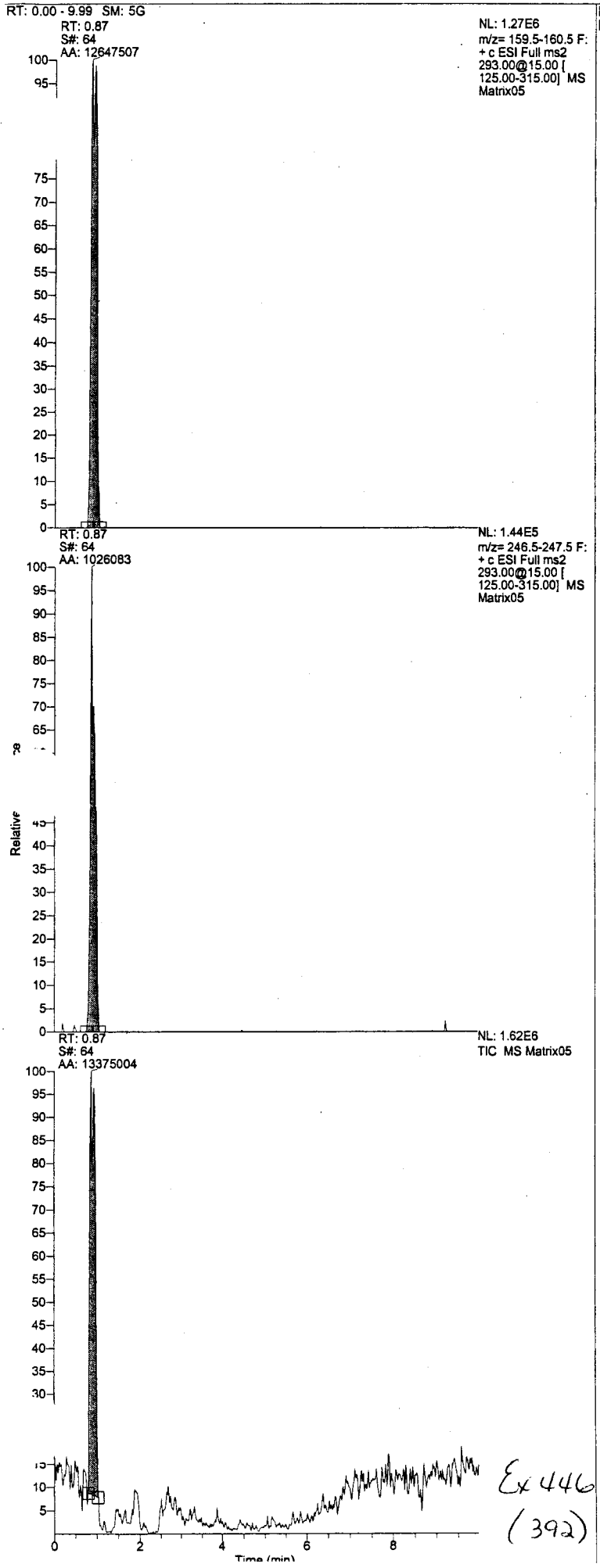
Ex 446
(390)

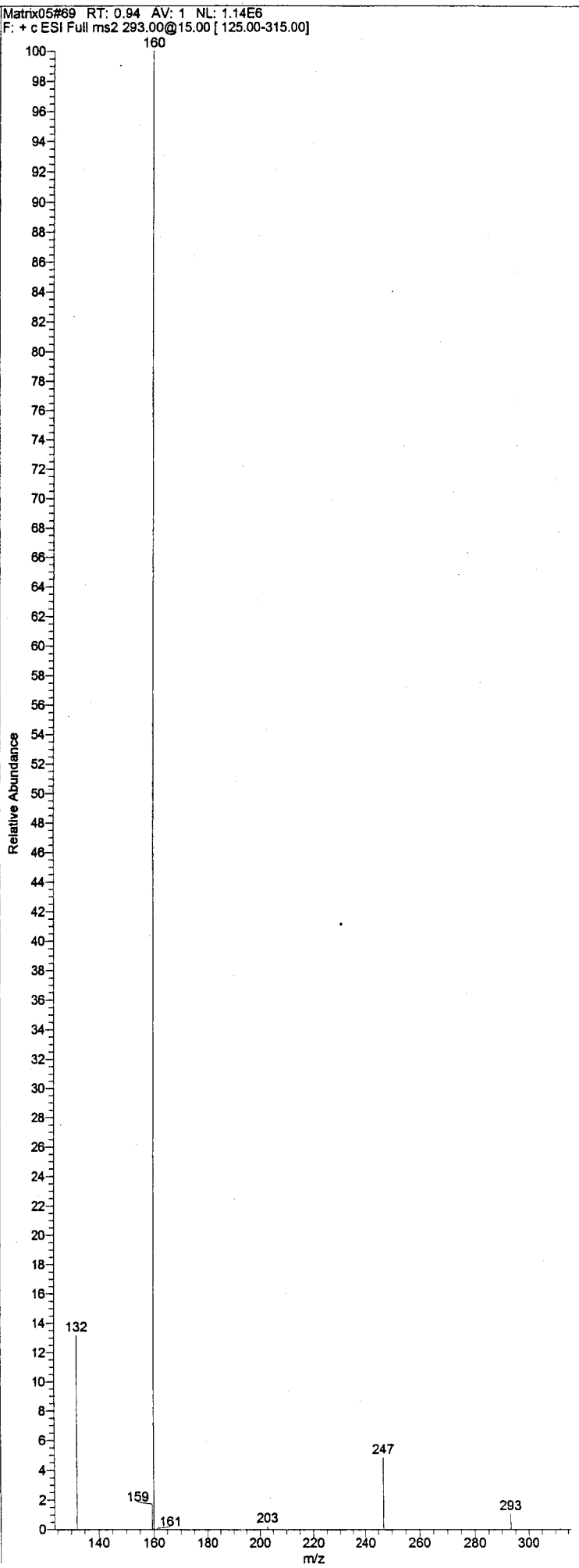

RT: 0.00 - 9.99 SM: 5G
RT: 0.90
S#: 66
AA: 2840952
NL: 4.02E5
m/z= 159.5-160.5 F:
+ c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Matrix04
RT: 0.94
S#: 69
AA: 267123
NL: 5.49E4
m/z= 246.5-247.5 F:
+ c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Matrix04
Relativ
RT: 6.29
S#: 460
AA: 15177
RT: 0.94
S#: 69
AA: 2345871
NL: 5.07E5
TIC MS Matrix04
RT: 1.84
S#: 135
AA: 3189252
Time (min)

Ex 446
(391)

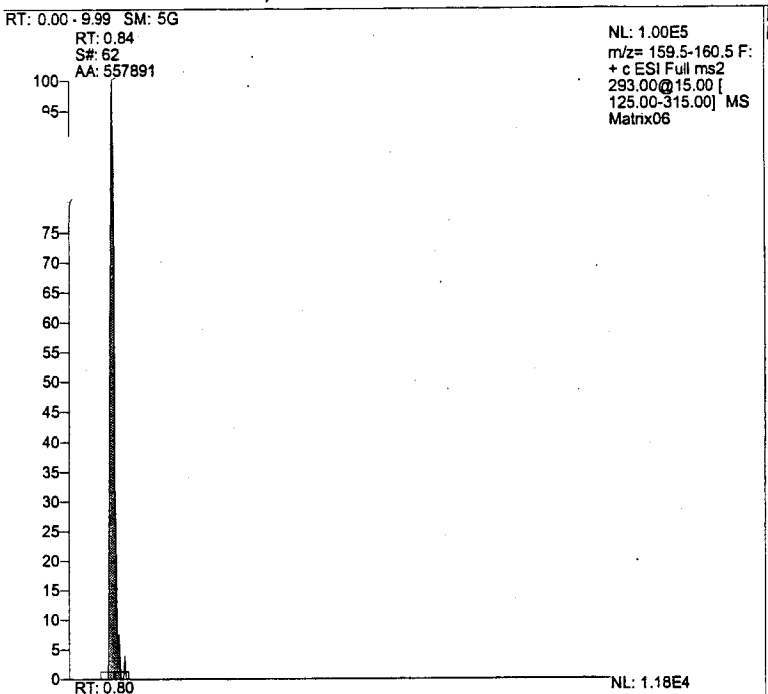

Matrix04#69 RT: 0.94 AV: 1 NL: 5.38E5
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]
160
Relative Abundance
132
247
159
161
248
293
293
m/z

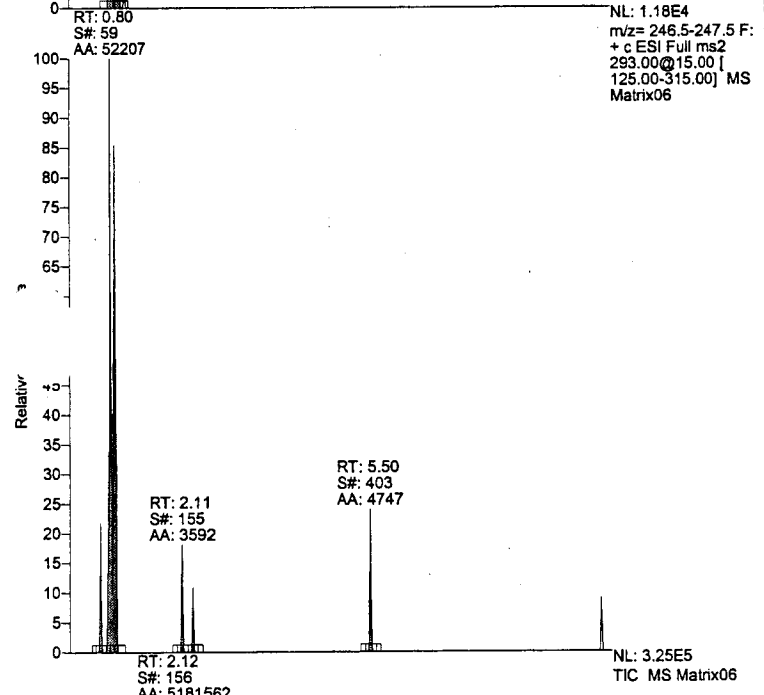
C:\Xcalibur\...\MAT_Positive\Matrix05 02/12/07 04:49:22 Matrix 10 ("50 ppm") TDP

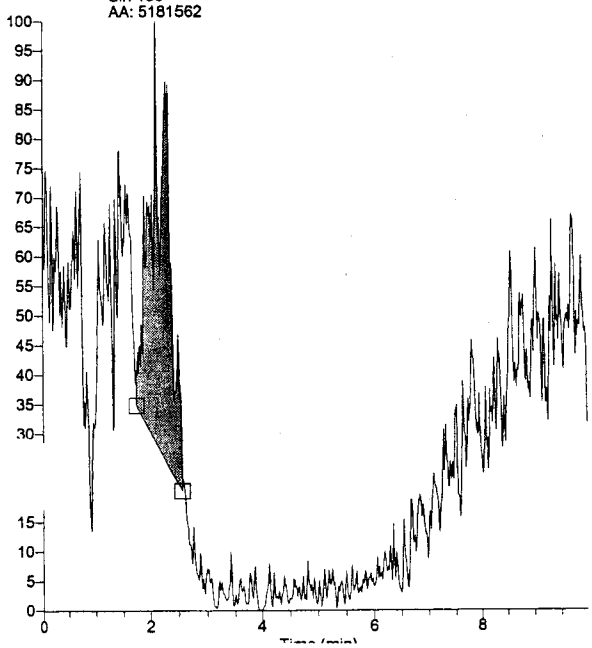




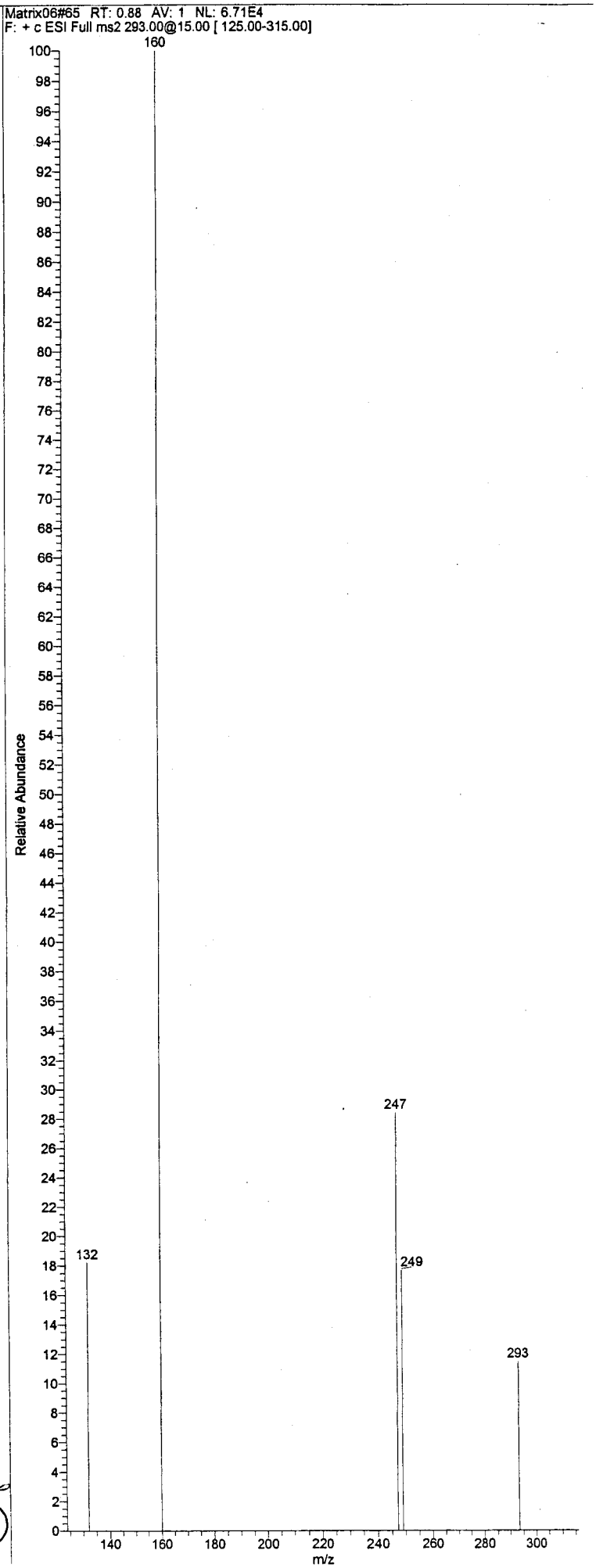
C:\Xcalibur\...\MAT_Positive\Matrix06 02/12/07 05:00:13 50 ppm EDTA JDB



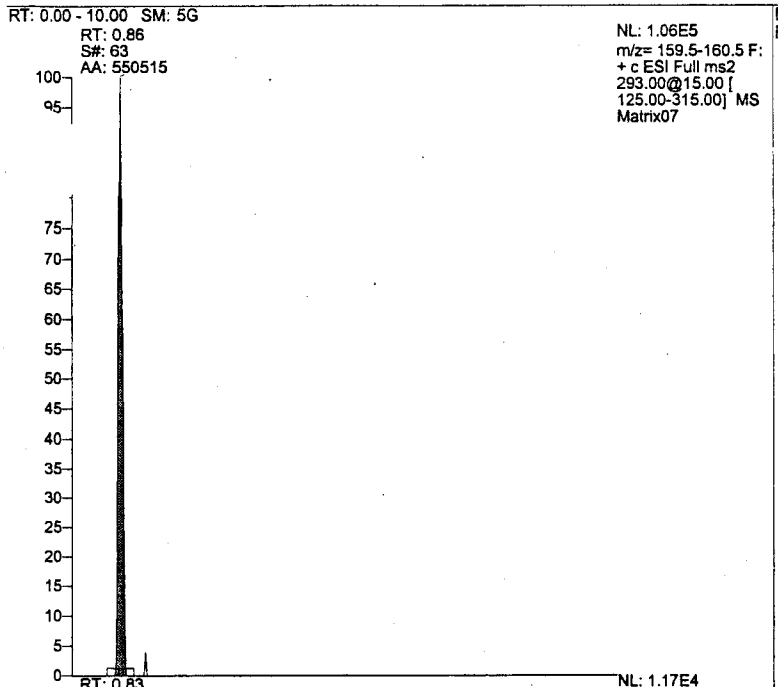


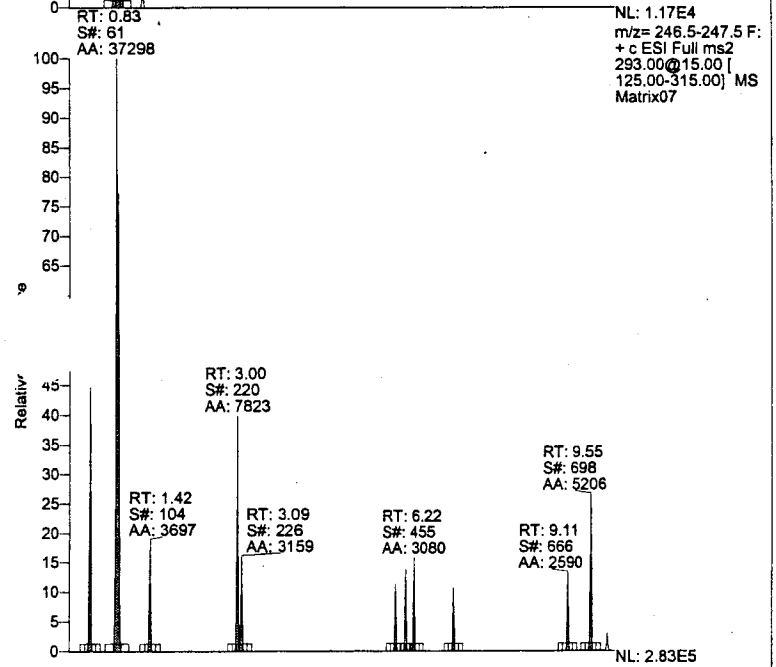


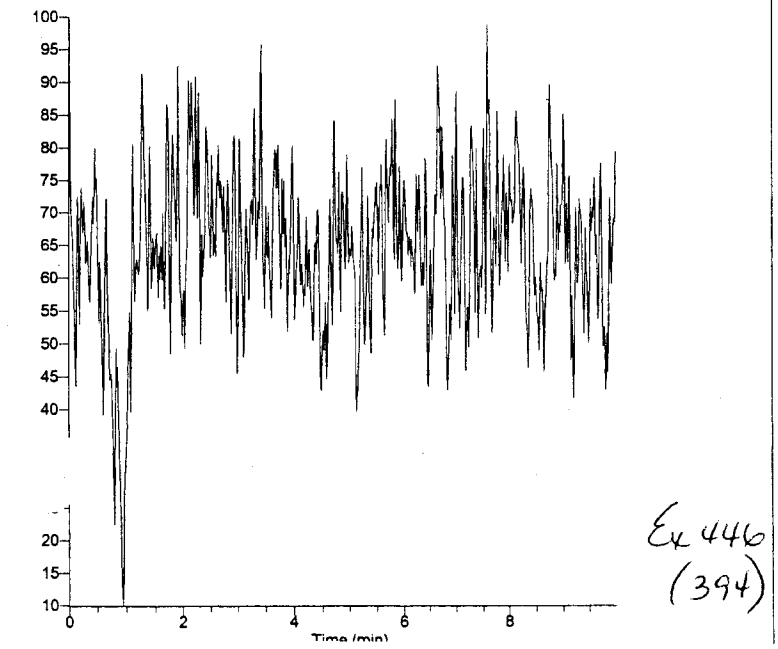
Ex 446
(393)

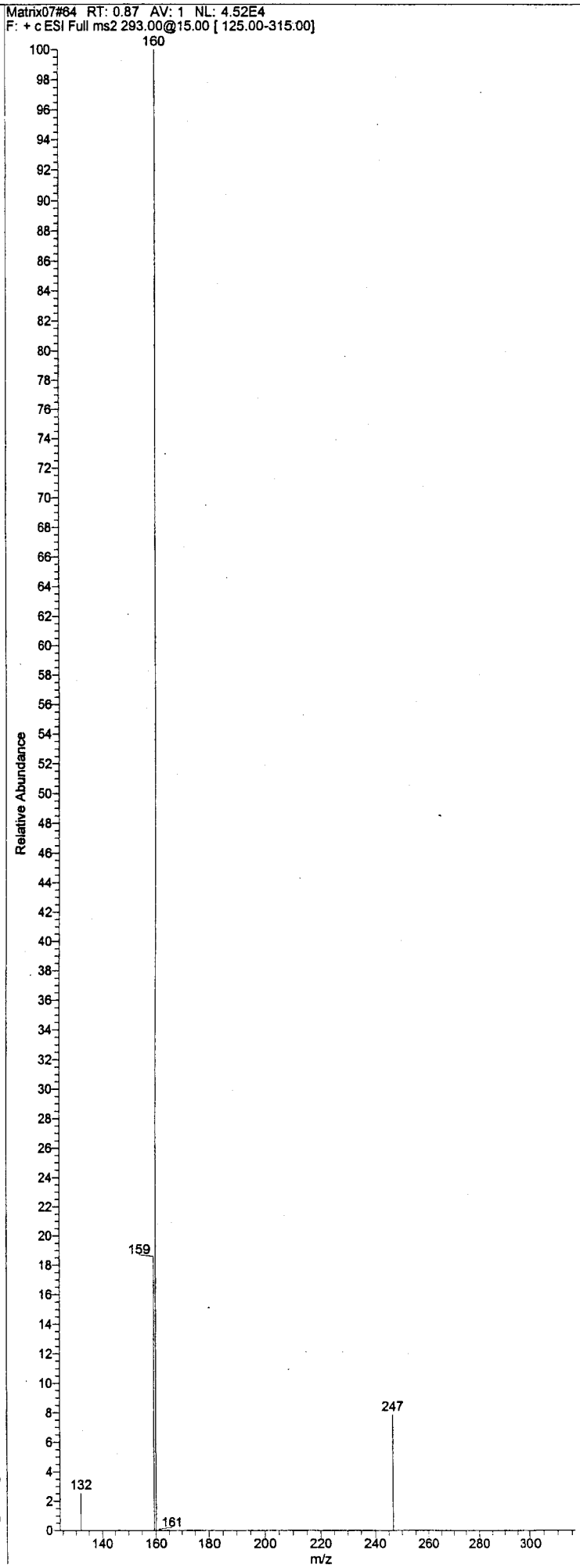


C:\Xcalibur\...\MAT_Positive\Matrix07 02/12/07 05:11:03 50 ppm EDTA JPB

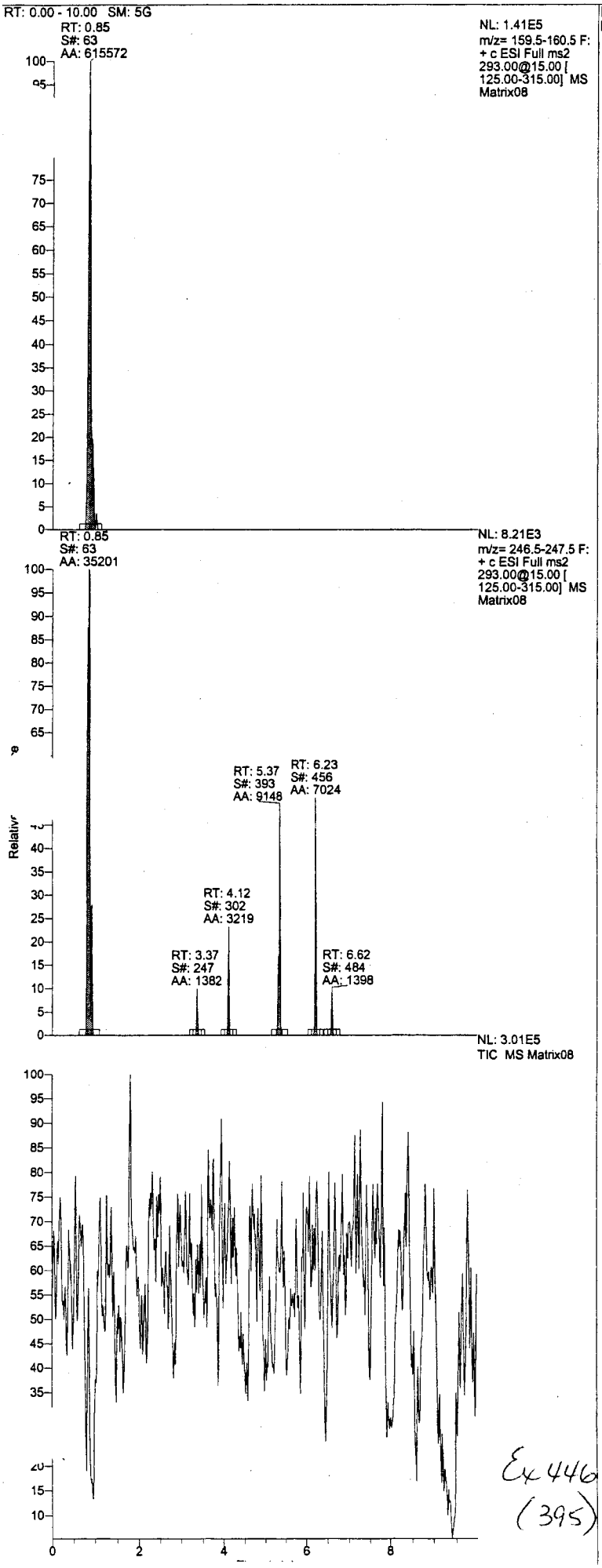


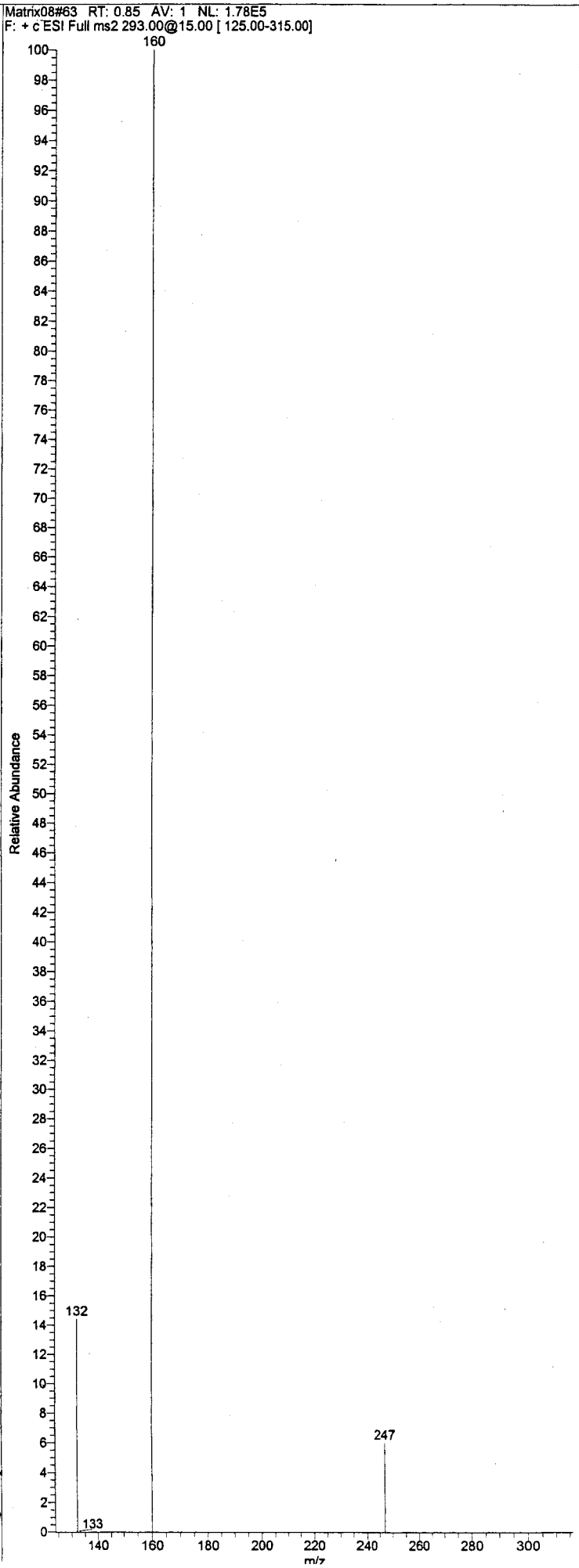






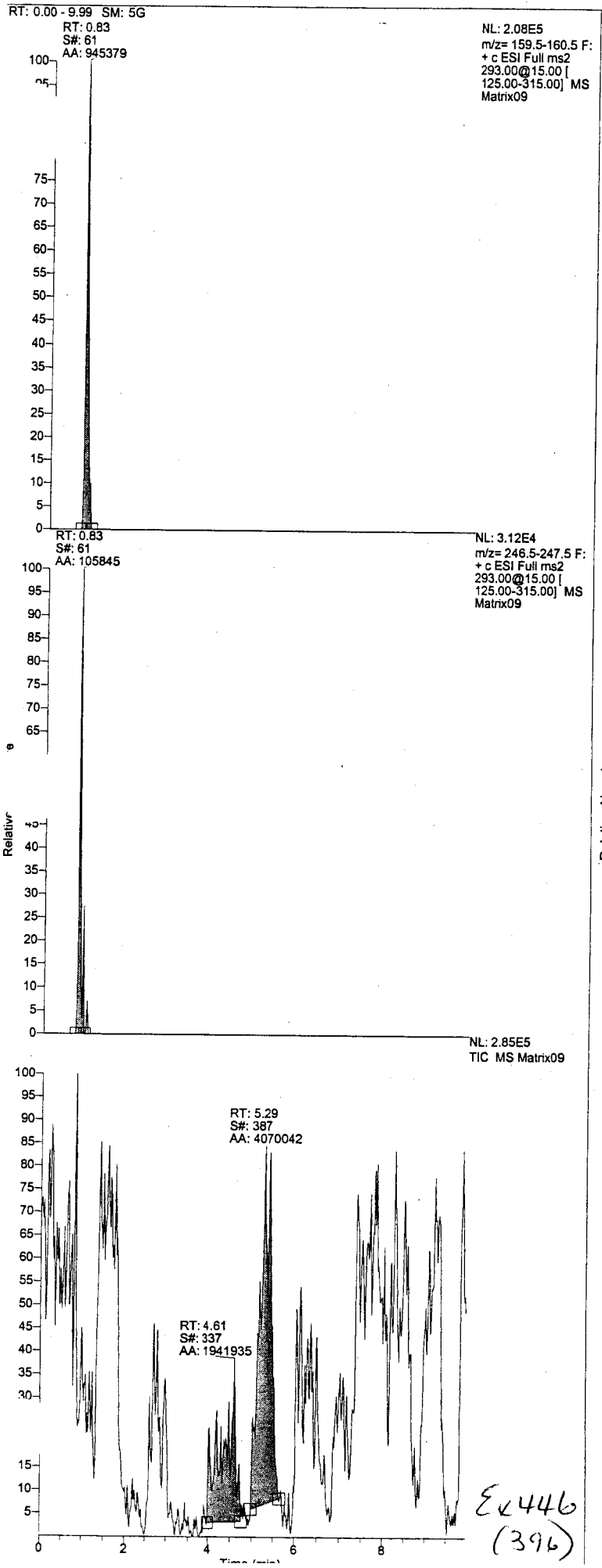
Ex 446
(394)

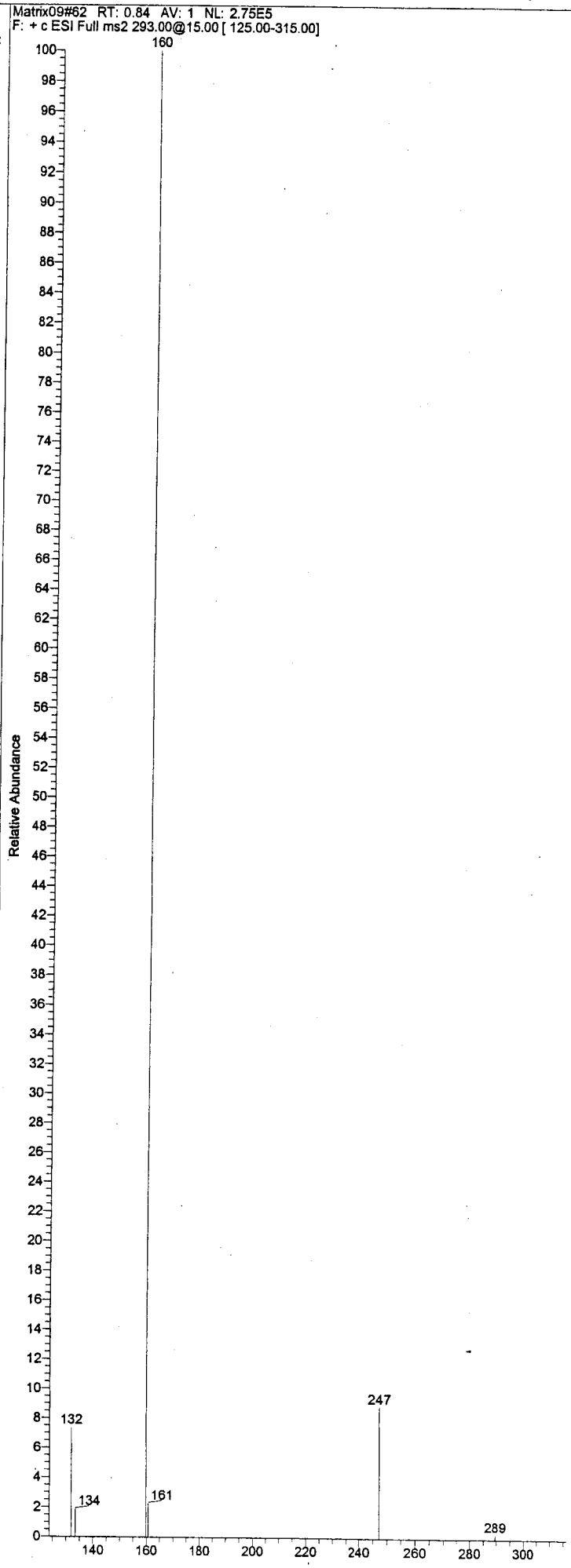

RT: 0.00 - 10.00 SM: 5G
RT: 0.85
S#: 63
AA: 615572
NL: 1.41E5
m/z= 159.5-160.5 F: + c ESI Full ms2 293.00@15.00 [125.00-315.00] MS Matrix08
RT: 0.85
S#: 63
AA: 35201
NL: 8.21E3
m/z= 246.5-247.5 F: + c ESI Full ms2 293.00@15.00 [125.00-315.00] MS Matrix08
RT: 5.37
S#: 393
AA: 9148
RT: 6.23
S#: 456
AA: 7024
RT: 4.12
S#: 302
AA: 3219
RT: 3.37
S#: 247
AA: 1382
RT: 6.62
S#: 484
AA: 1398
Relativ
NL: 3.01E5
TIC MS Matrix08


Matrix08#63 RT: 0.85 AV: 1 NL: 1.78E5
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]
160
132
133
247
Relative Abundance
m/z

Ex 446
(395)

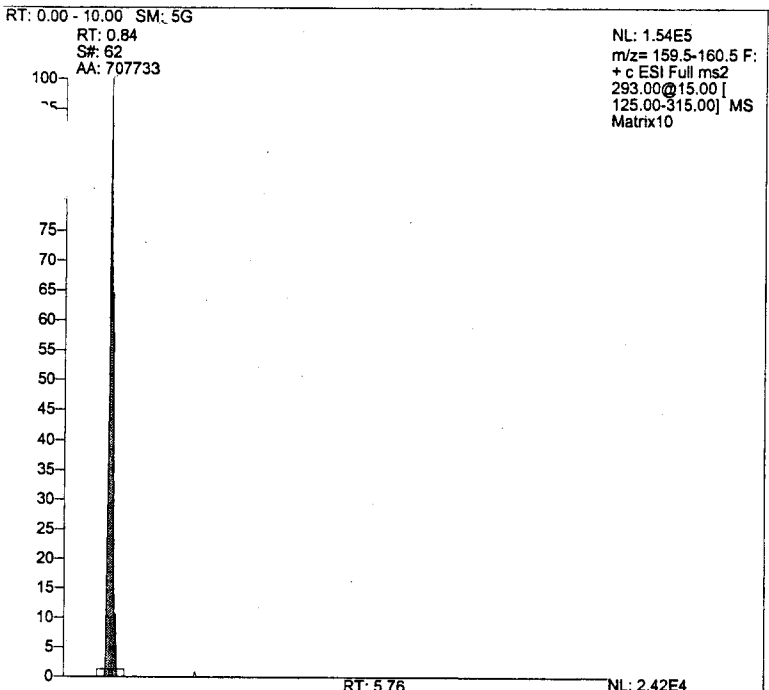
C:\Xcalibur\...\MAT_Positive\Matrix09 02/12/07 05:32:46 50 ppm EDTA JDB

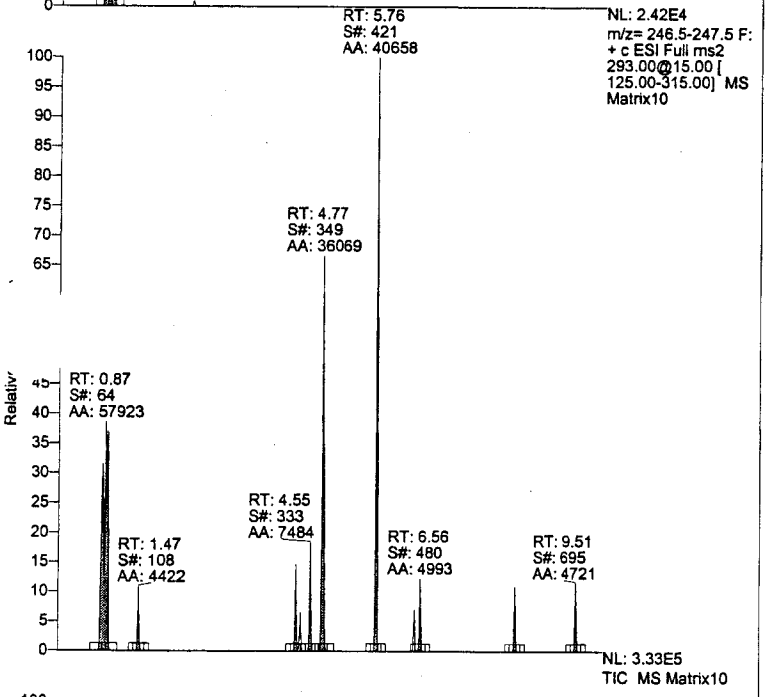


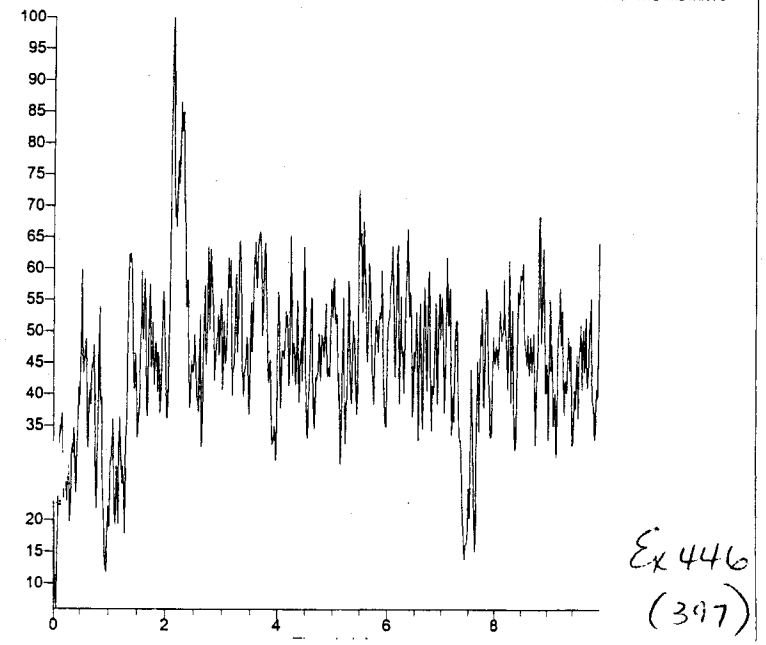


Ex446 (396)

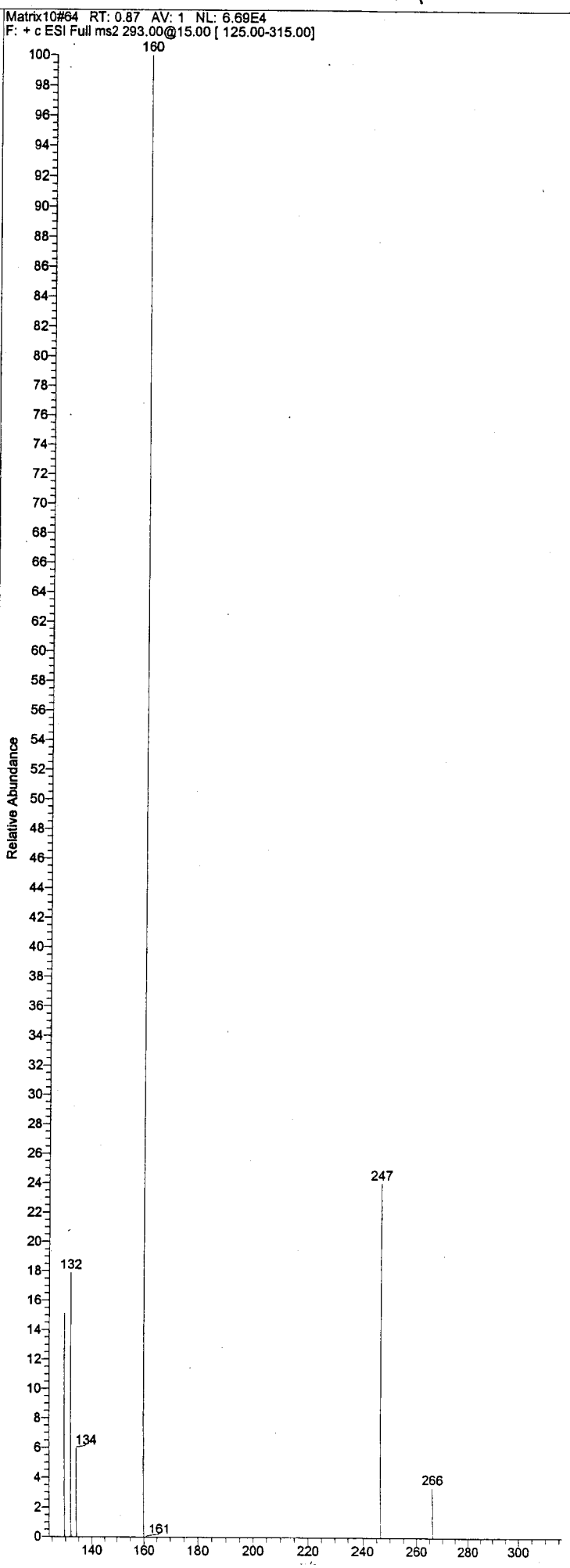
C:\Xcalibur\...\MAT_Positive\Matrix10 02/12/07 05:43:37 50 ppm EDTA JDB



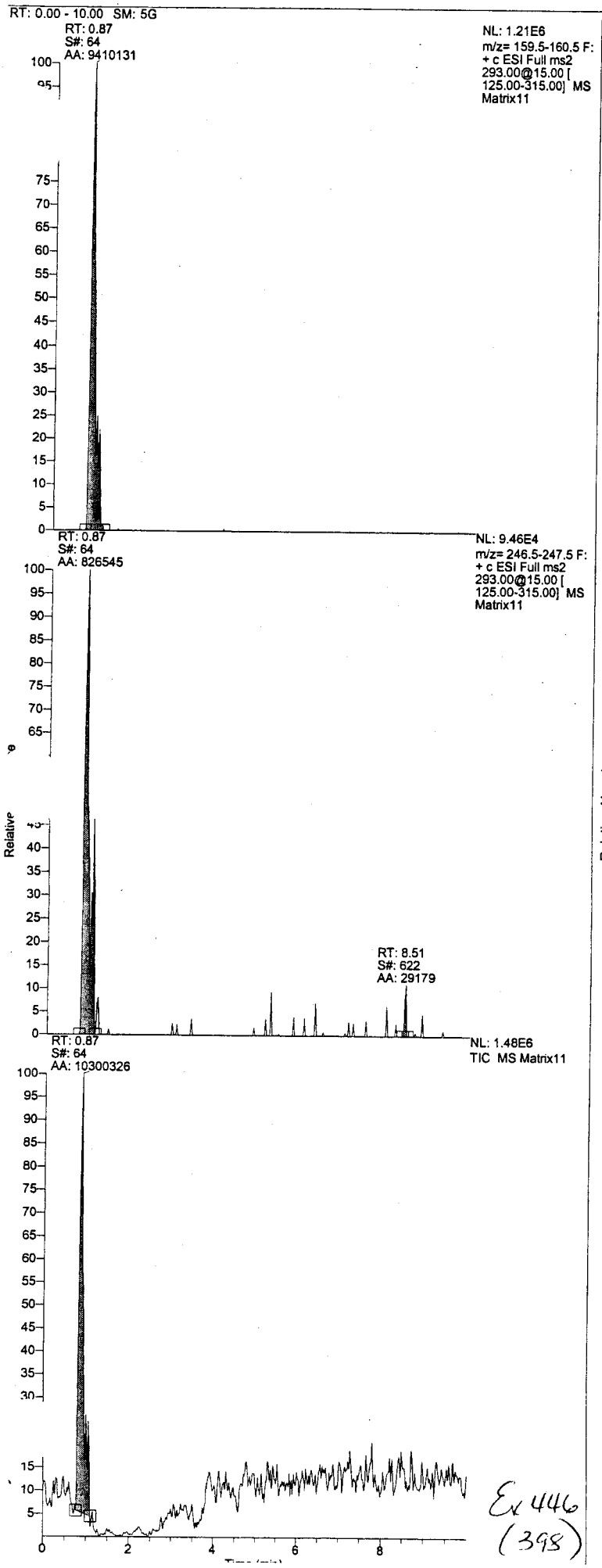


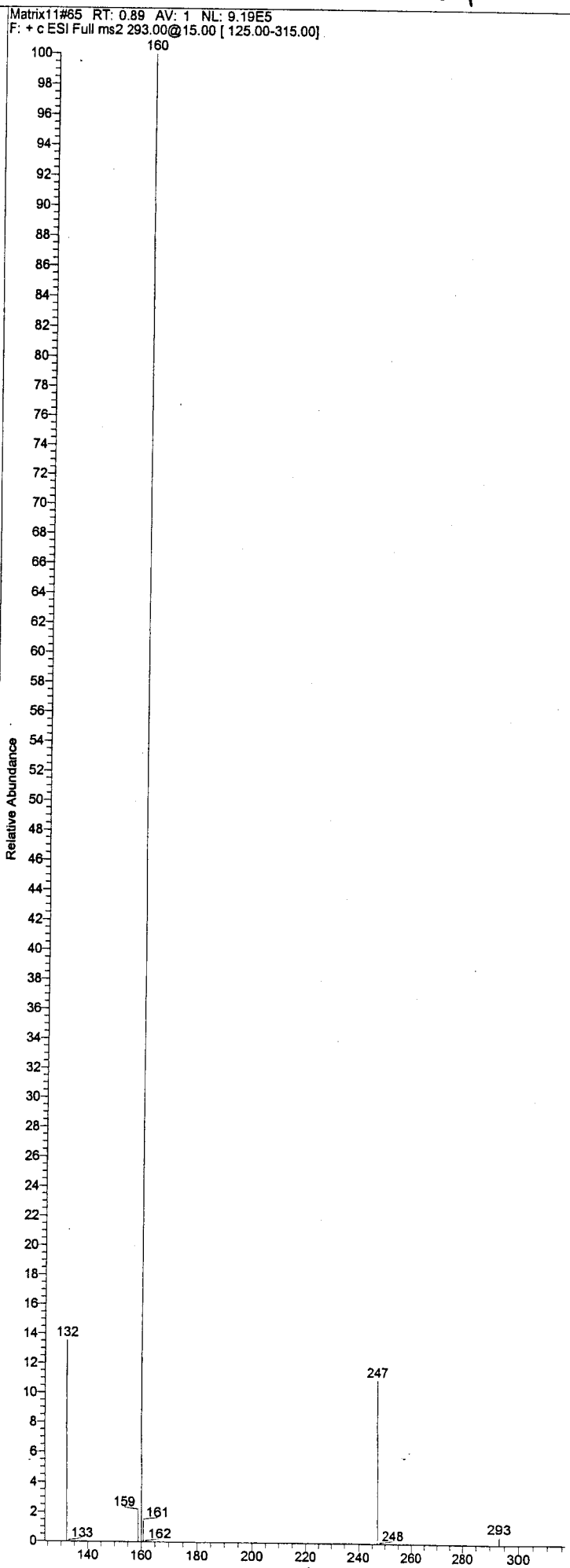


Ex 446
(307)



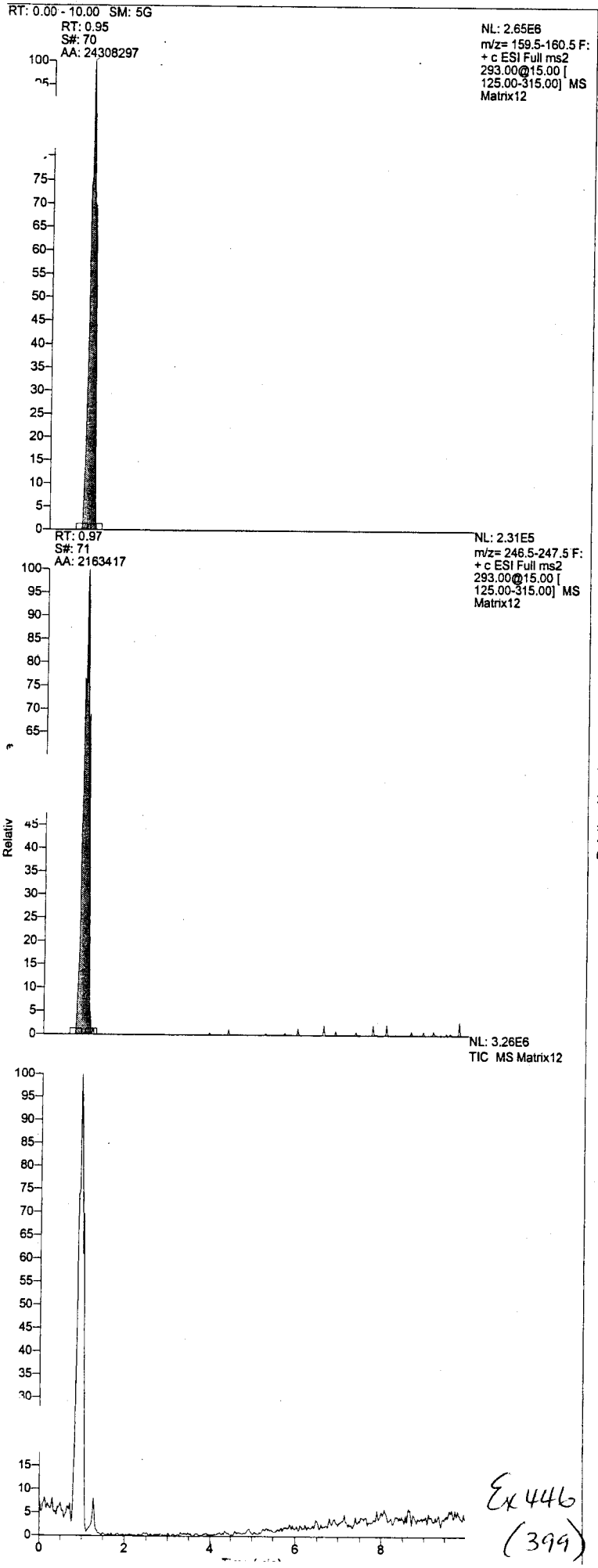
C:\Xcalibur\...\MAT_Positive\Matrix11 02/12/07 05:54:29 Matrix 1 ("500 ppm") JDP

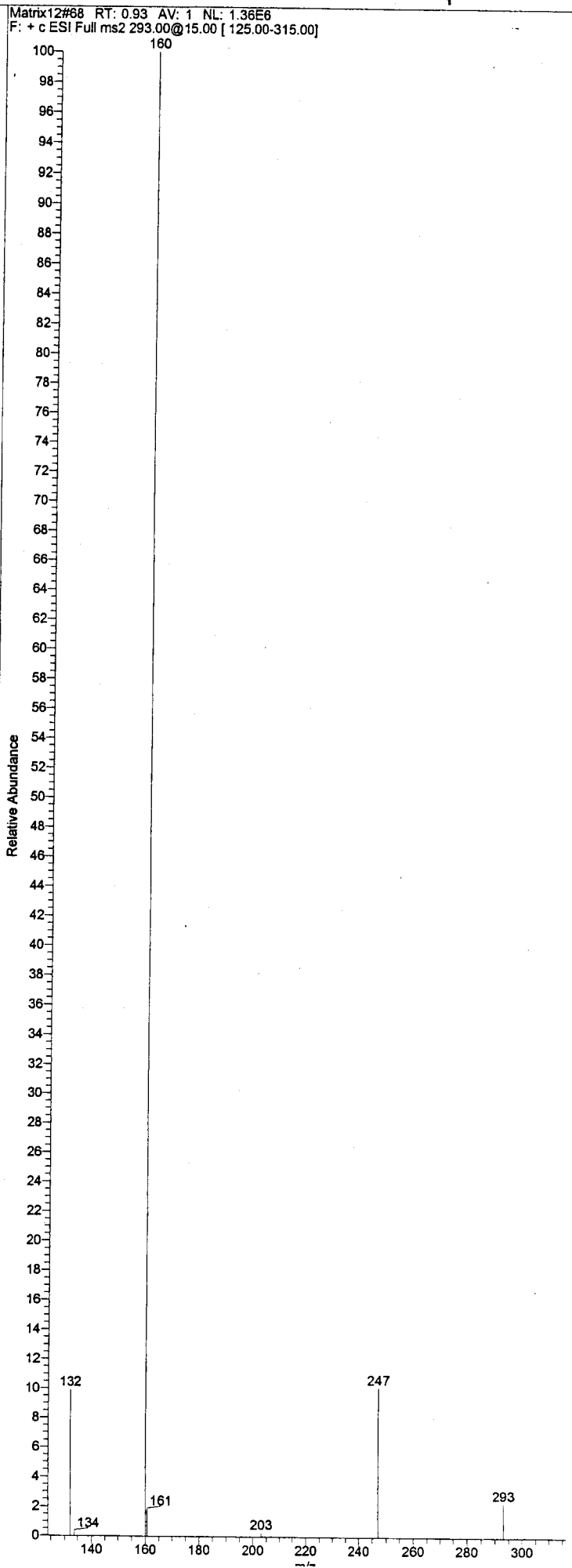




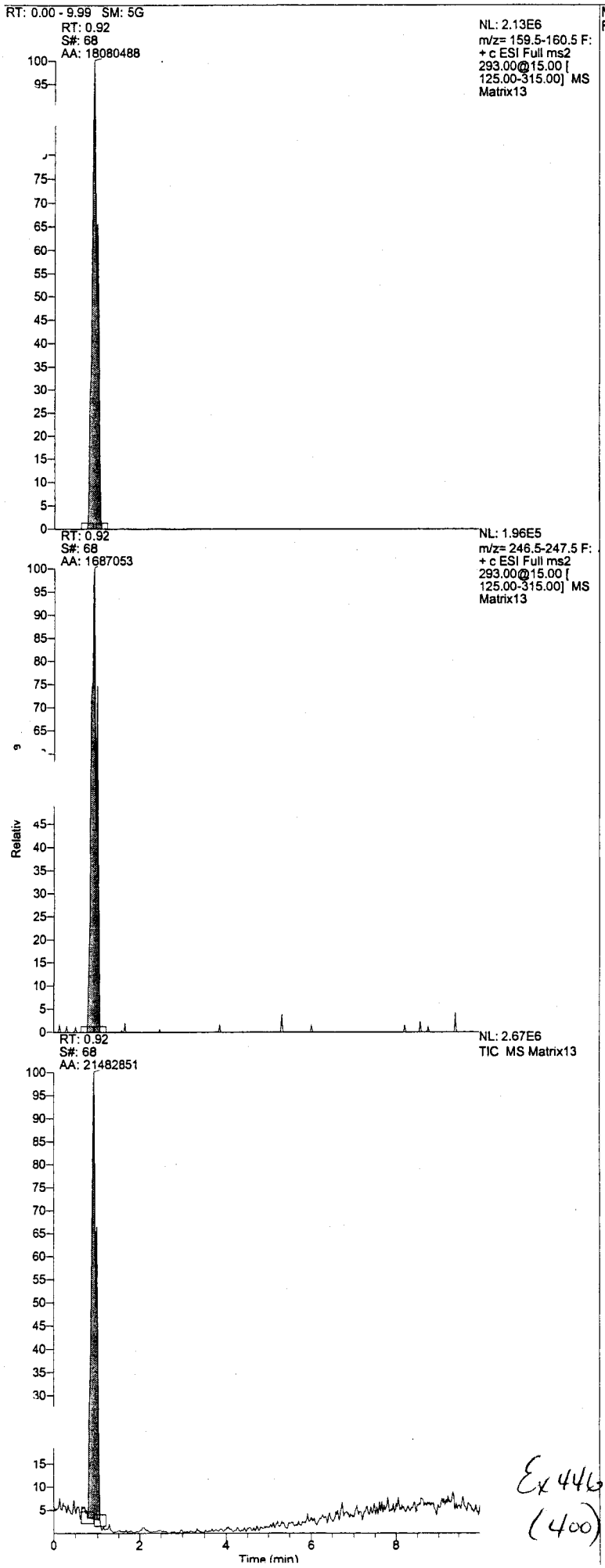
Ex 446
(398)

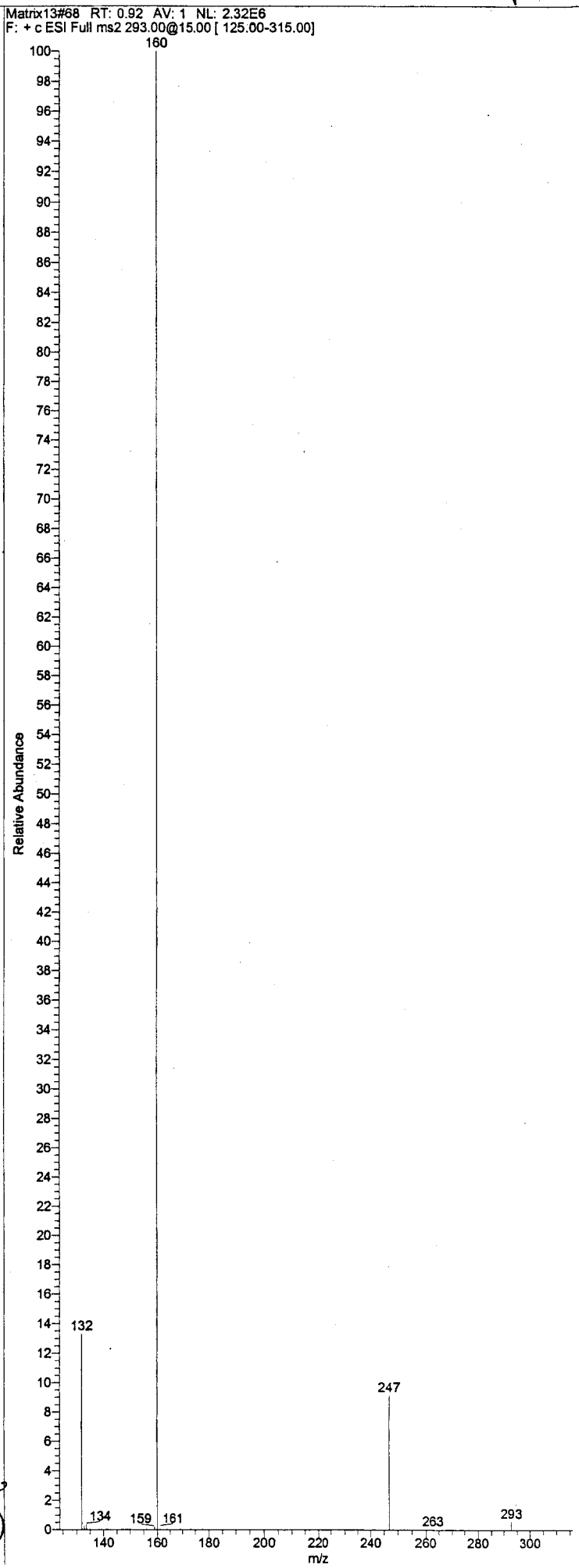
C:\Xcalibur\...\MAT_Positive\Matrix12 02/12/07 06:05:20 Matrix 2 ("500 ppm") JDB





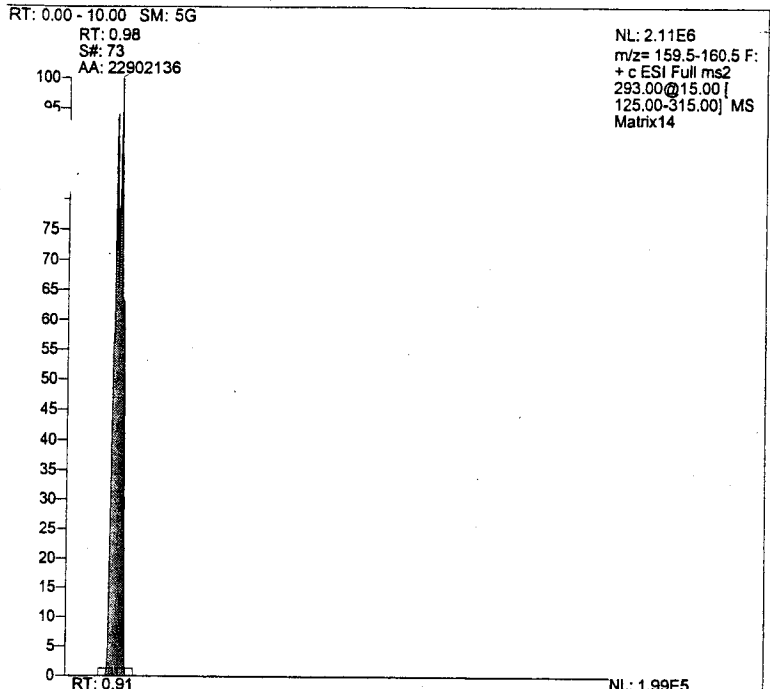
Ex 446
(399)

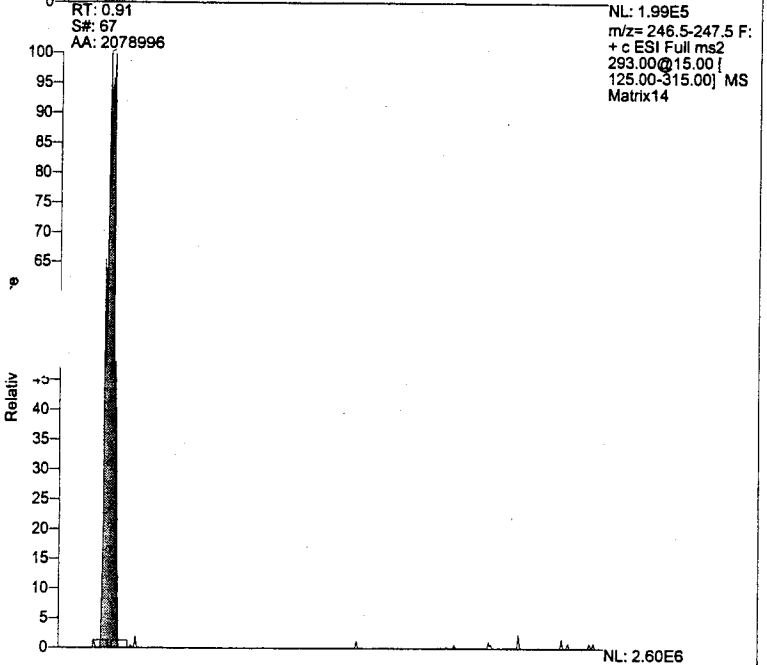

RT: 0.00 - 9.99 SM: 5G
RT: 0.92
S#: 68
AA: 18080488
NL: 2.13E6
m/z= 159.5-160.5 F: + c ESI Full ms2 293.00@15.00 [125.00-315.00] MS Matrix13
RT: 0.92
S#: 68
AA: 1687053
NL: 1.96E5
m/z= 246.5-247.5 F: + c ESI Full ms2 293.00@15.00 [125.00-315.00] MS Matrix13
Relativ
RT: 0.92
S#: 68
AA: 21482851
NL: 2.67E6
TIC MS Matrix13
Time (min)

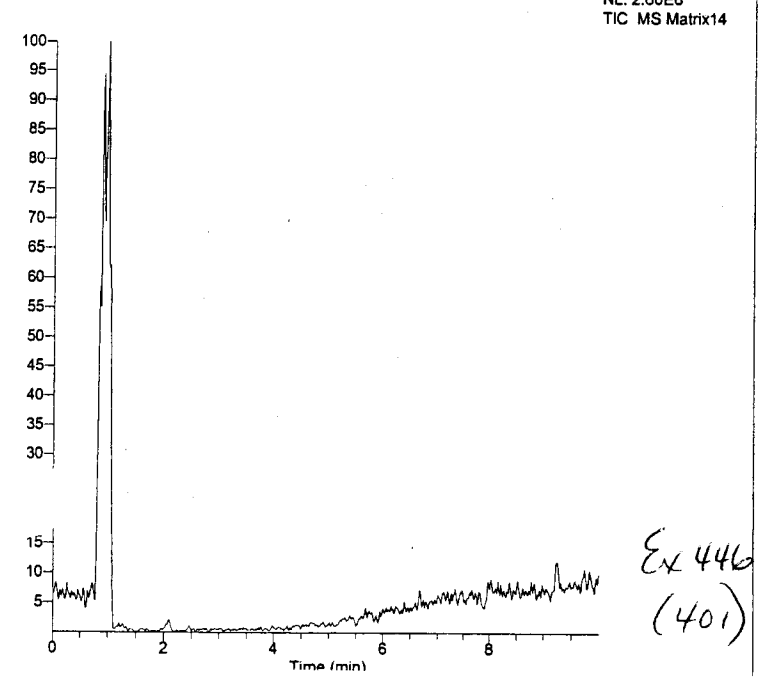

Matrix13#68 RT: 0.92 AV: 1 NL: 2.32E6
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]
160
132
247
134
159
161
263
293
Relative Abundance
m/z

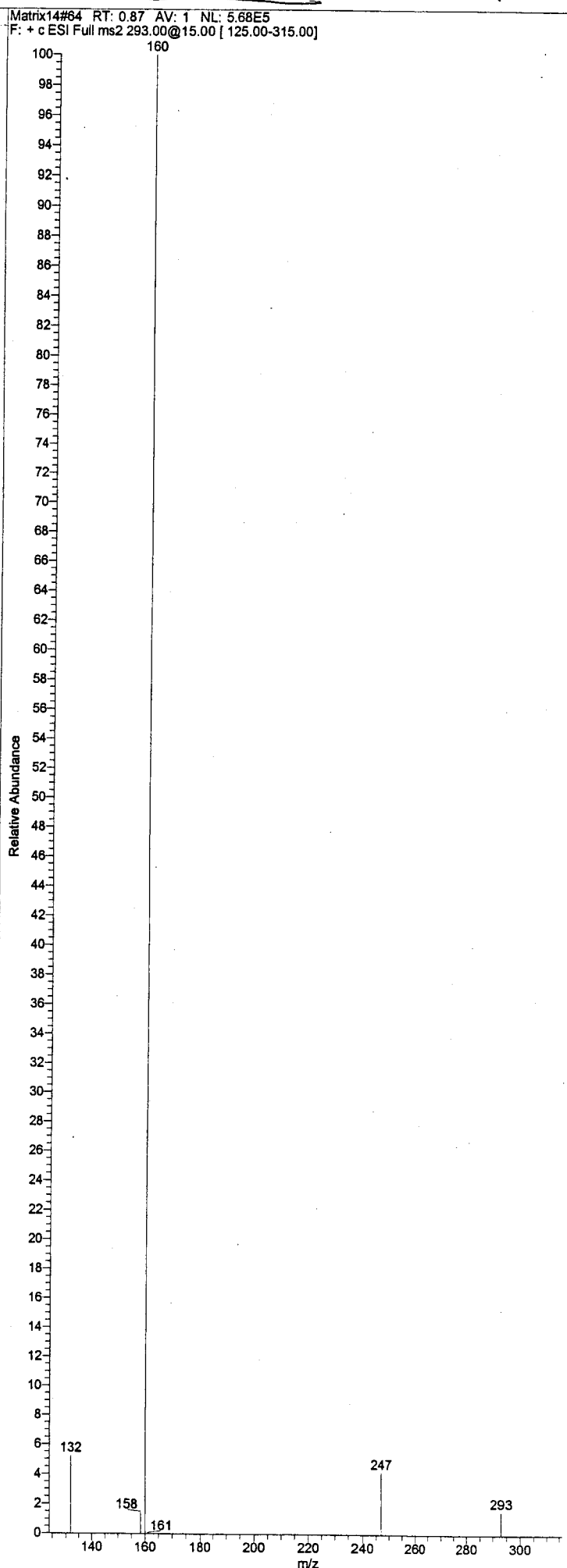
Ex 446
(400)

C:\Xcalibur\...\MAT_Positive\Matrix14 02/12/07 06:27:03 Matrix 4 ("500 ppm") JDB



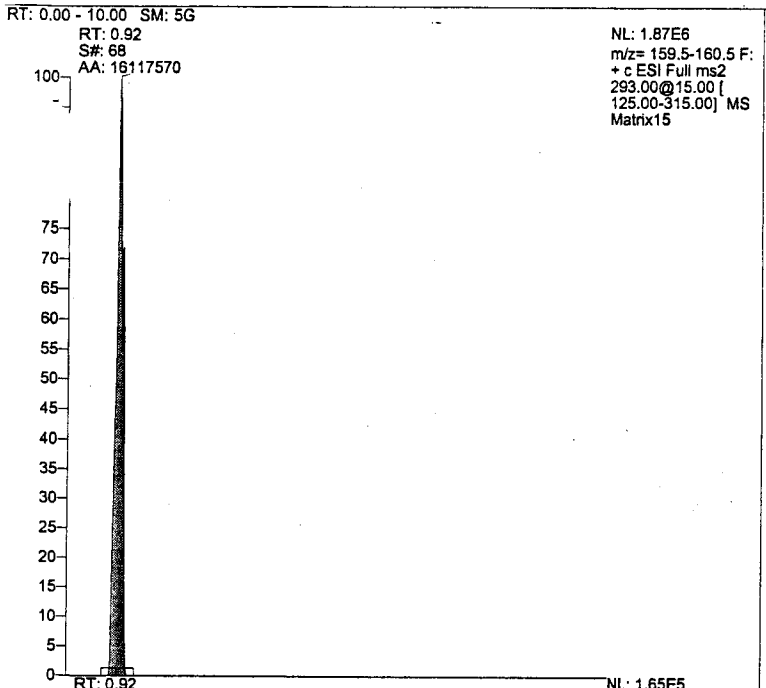


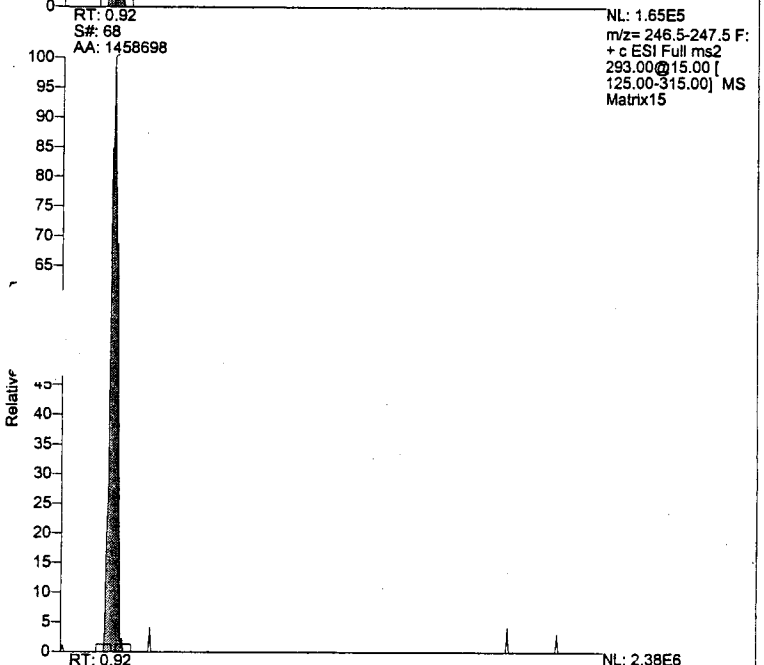


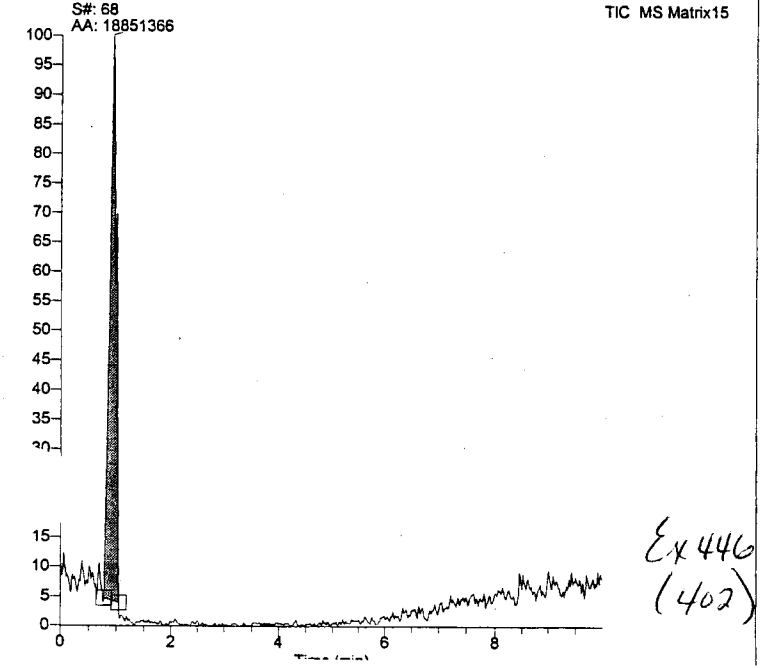


Ex 446
(401)

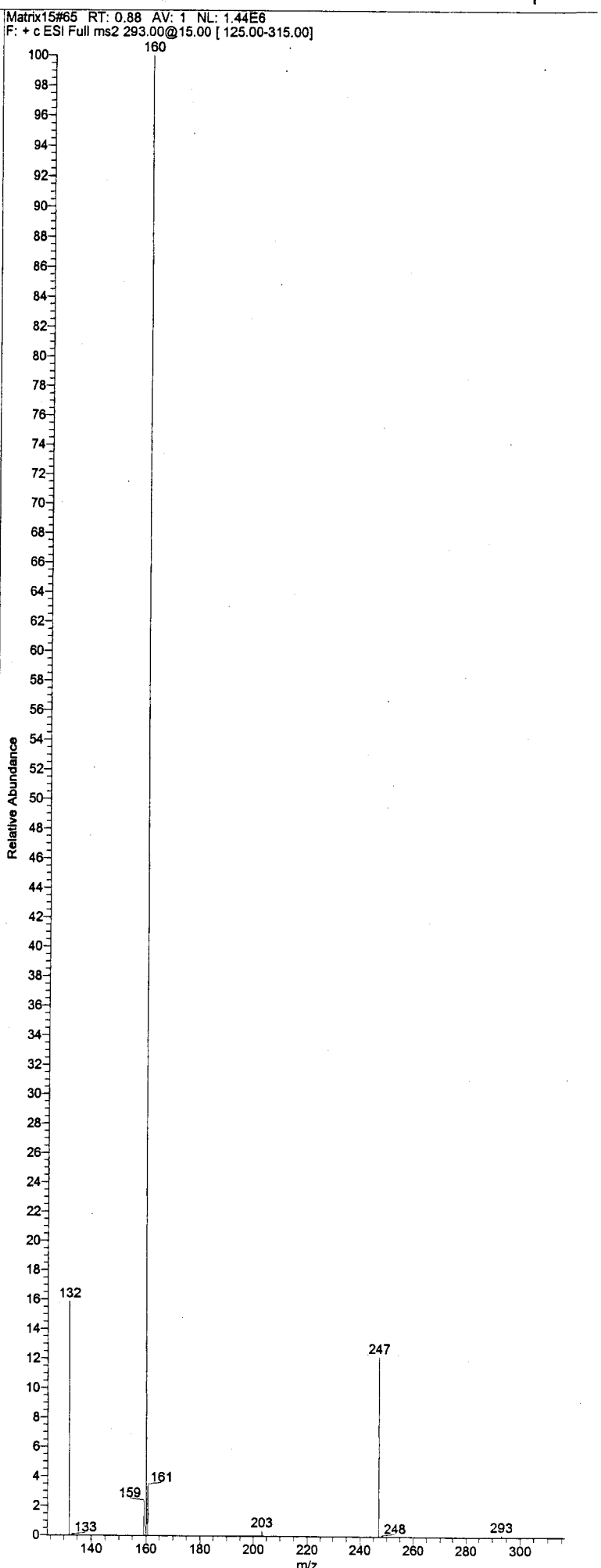
C:\Xcalibur\...\MAT_Positive\Matrix15 02/12/07 06:37:53 Matrix 5 ("500 ppm")

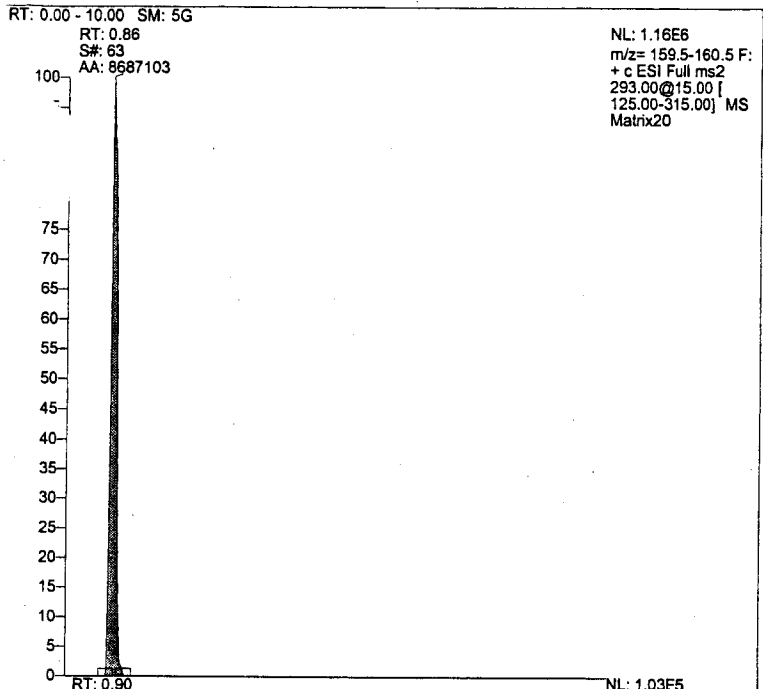


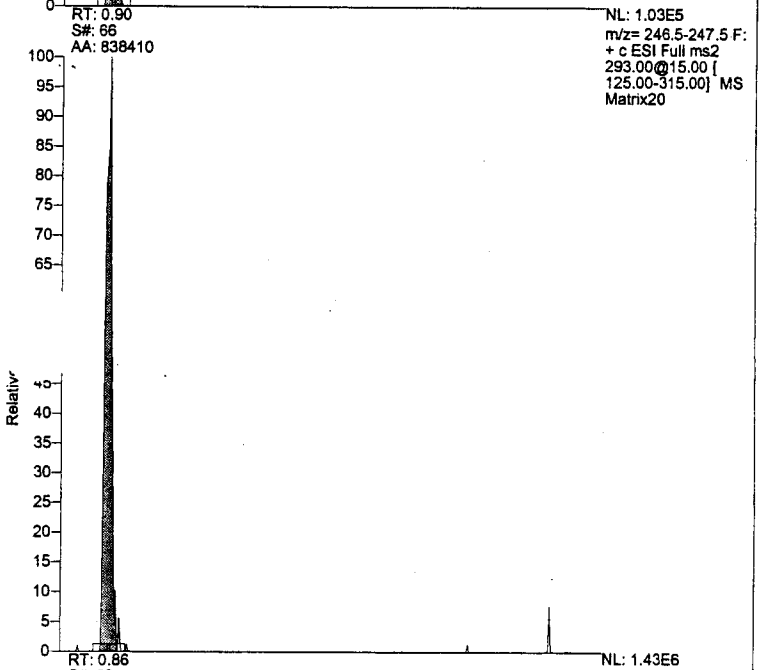


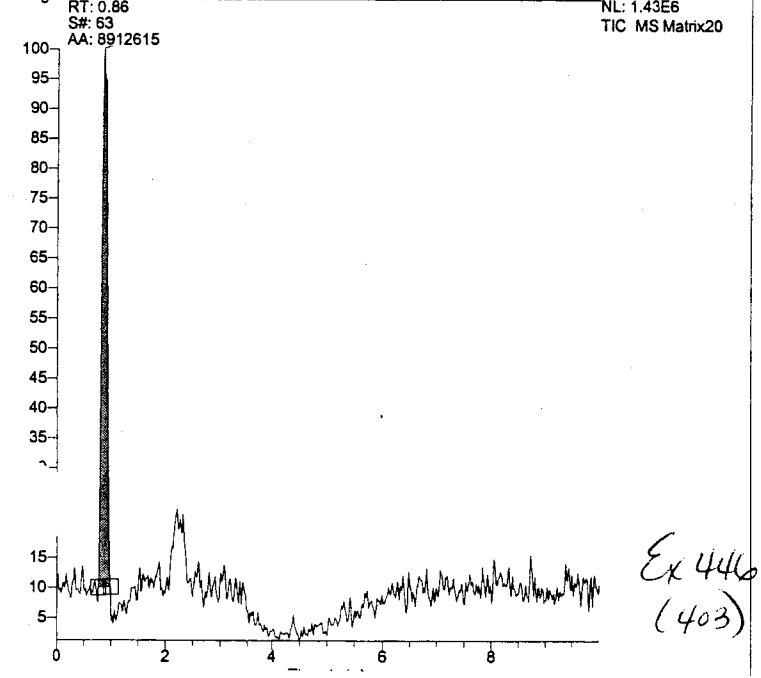


Ex 446
(402)

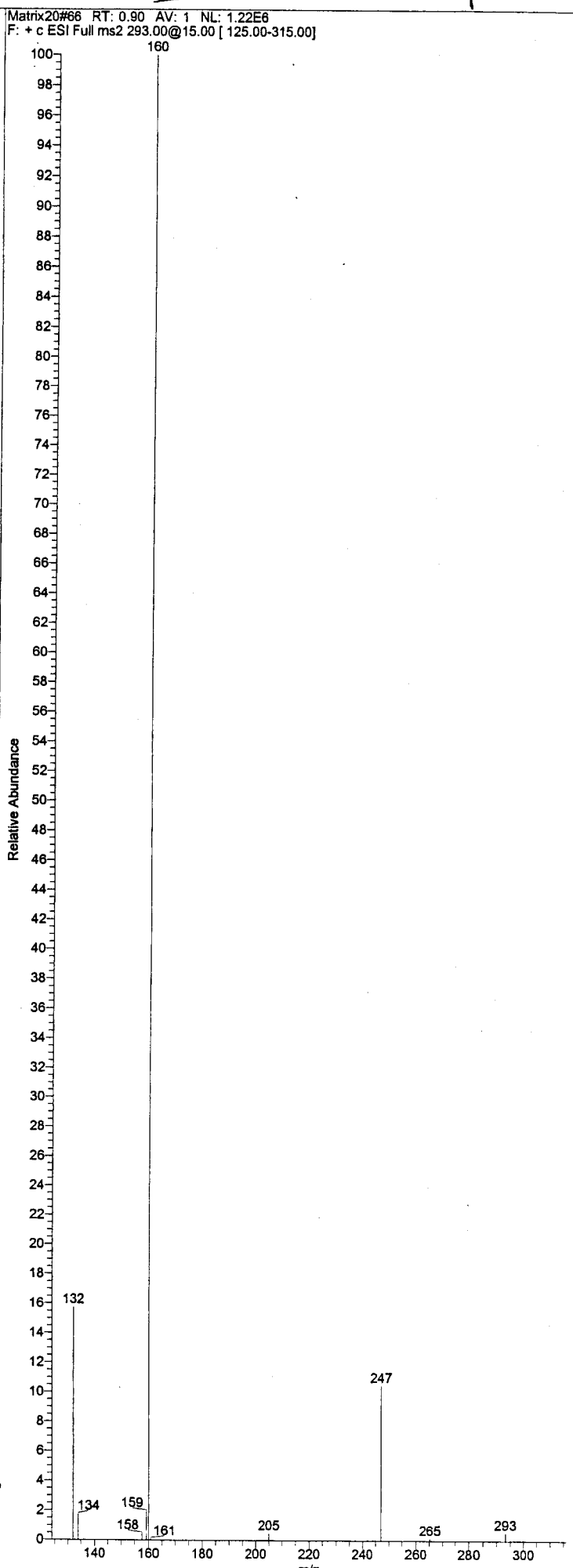



RT: 0.00 - 10.00 SM: 5G
RT: 0.86
S#: 63
AA: 8687103
NL: 1.16E6
m/z= 159.5-160.5 F: + c ESI Full ms2 293.00@15.00 [125.00-315.00] MS Matrix20

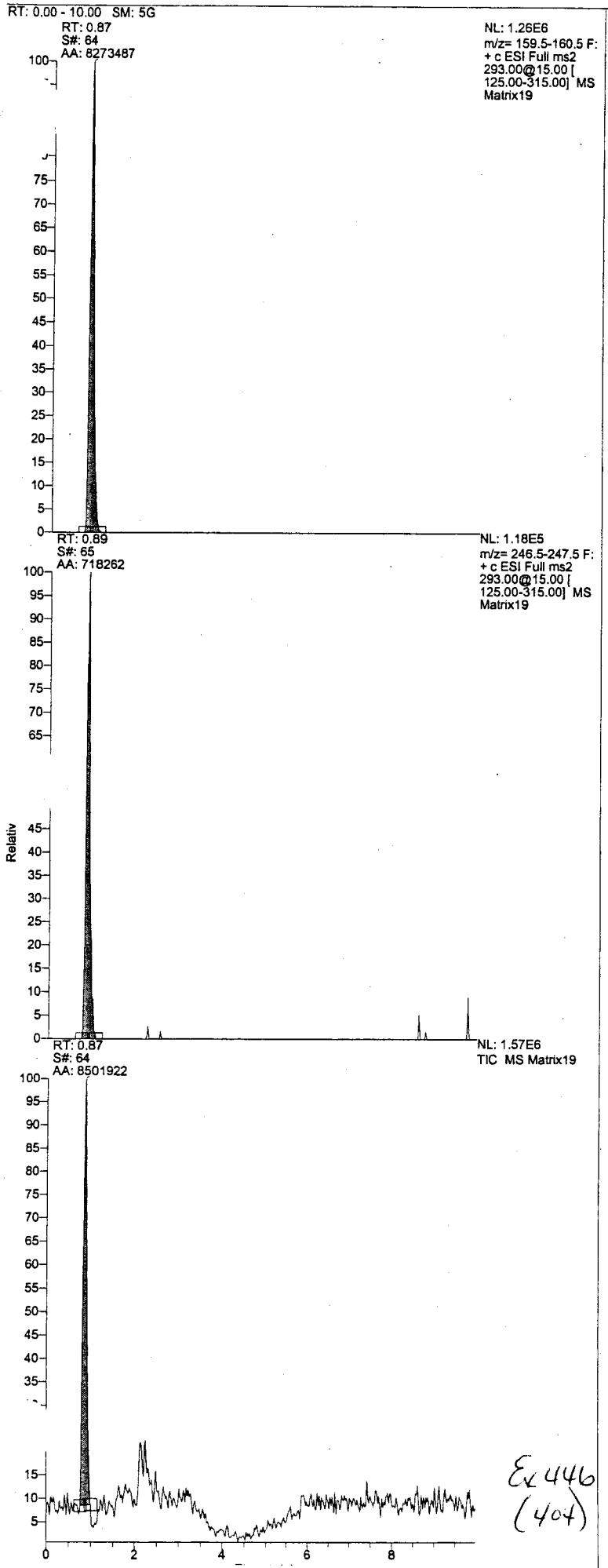

RT: 0.90
S#: 66
AA: 838410
NL: 1.03E5
m/z= 246.5-247.5 F: + c ESI Full ms2 293.00@15.00 [125.00-315.00] MS Matrix20
Relativ


RT: 0.86
S#: 63
AA: 8912615
NL: 1.43E6
TIC MS Matrix20

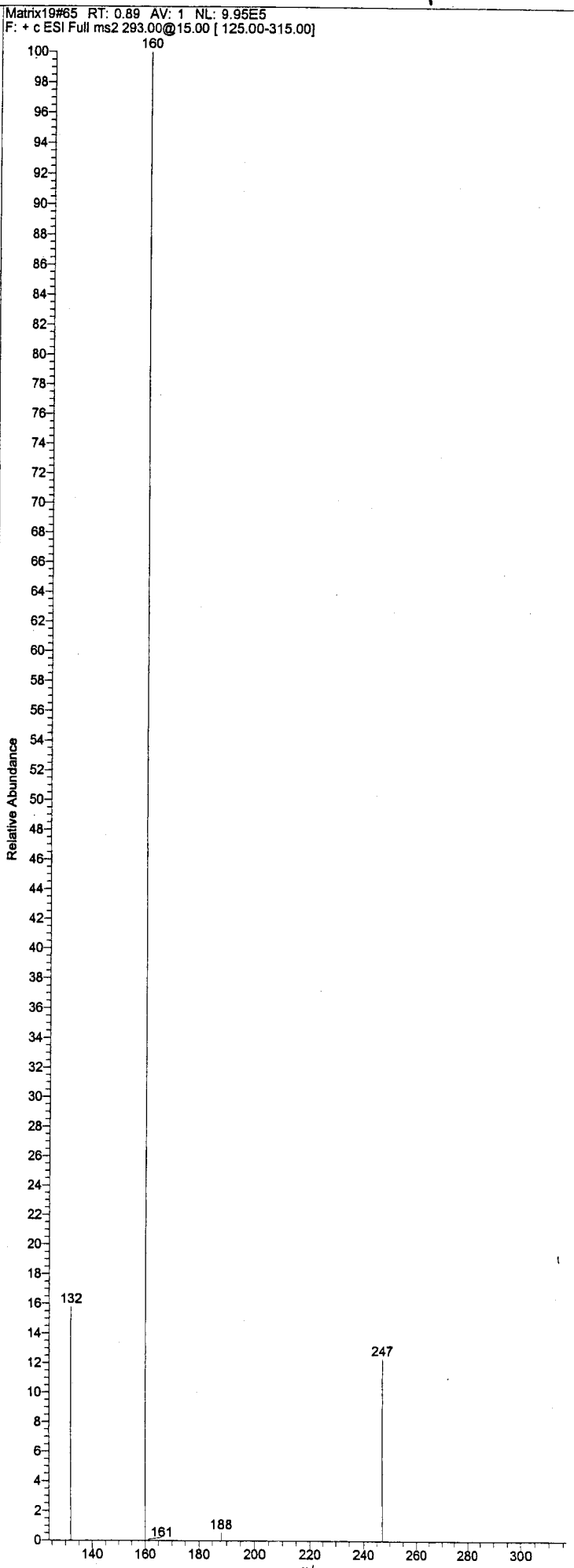
Ex 446
(403)


Matrix20#66 RT: 0.90 AV: 1 NL: 1.22E6
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]
Relative Abundance
160
132
247
134
159
158
161
205
265
293
m/z

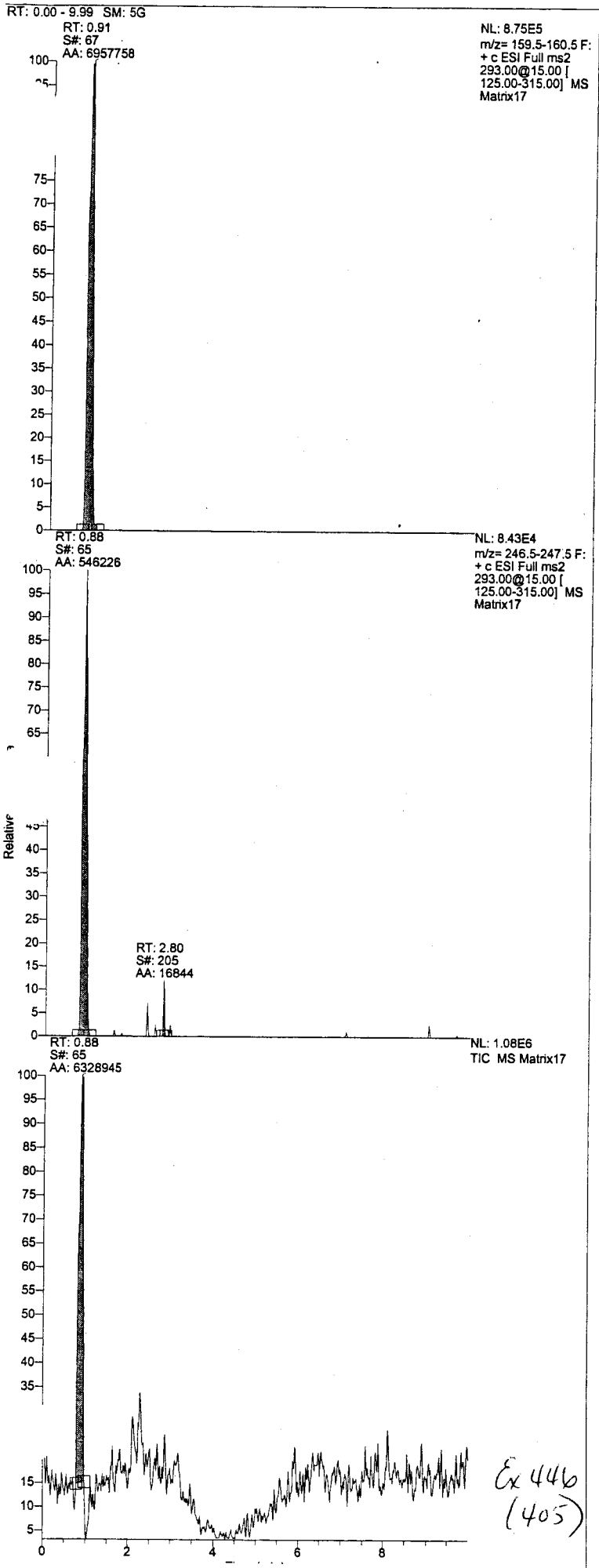
C:\Xcalibur\...\MAT_Positive\Matrix19 02/12/07 07:21:14 500 ppm EDTA JDB



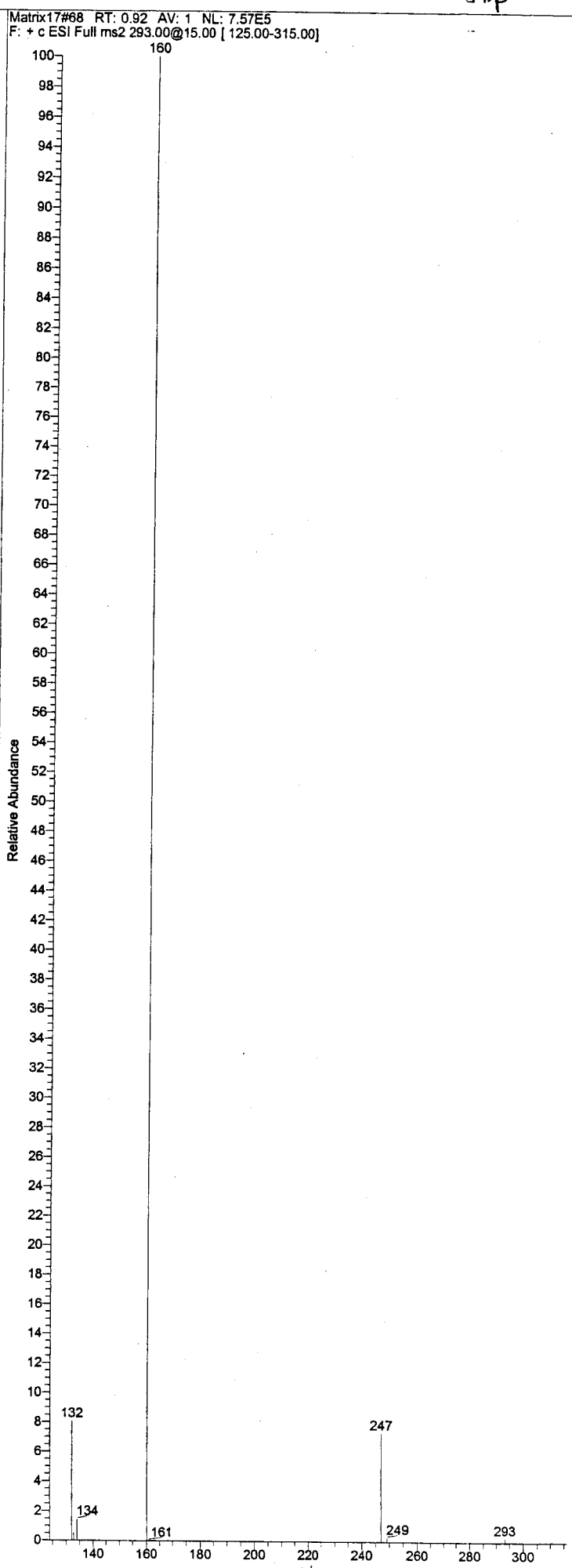
Ex 446 (404)



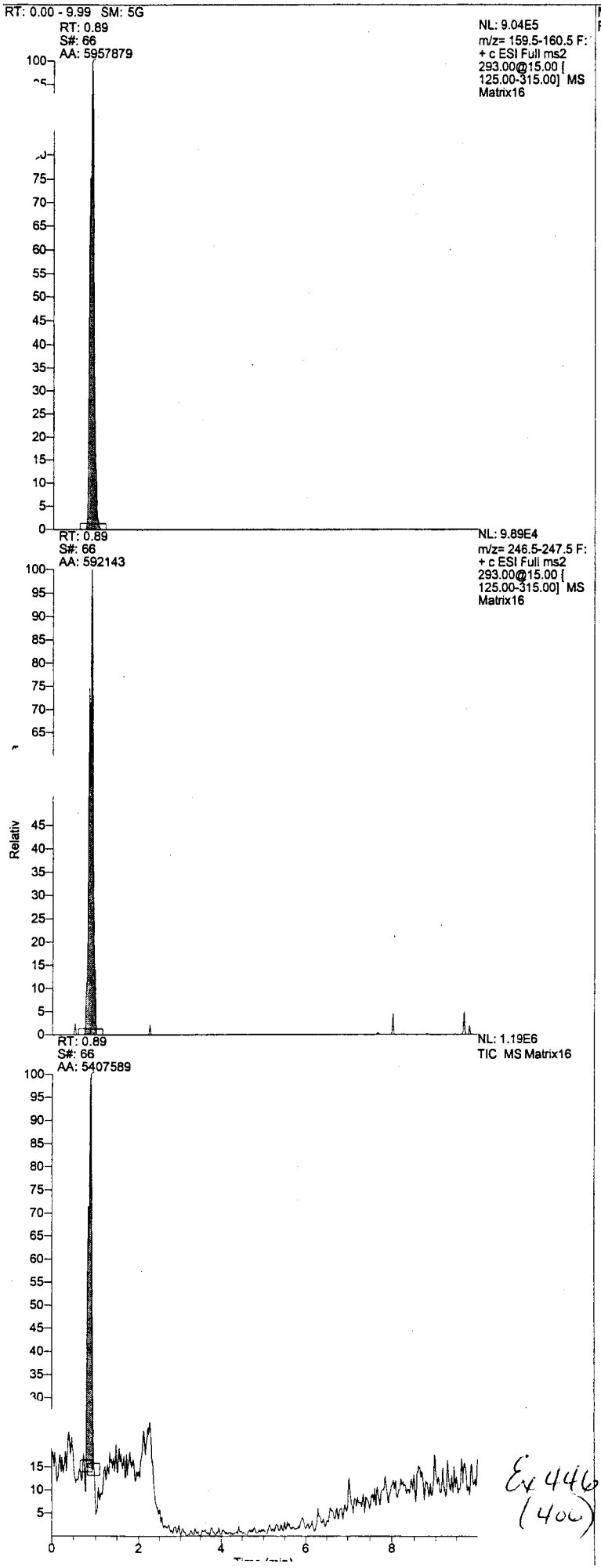
C:\Xcalibur\...\MAT_Positive\Matrix17 02/12/07 06:59:29 500 ppm EDTA JDB



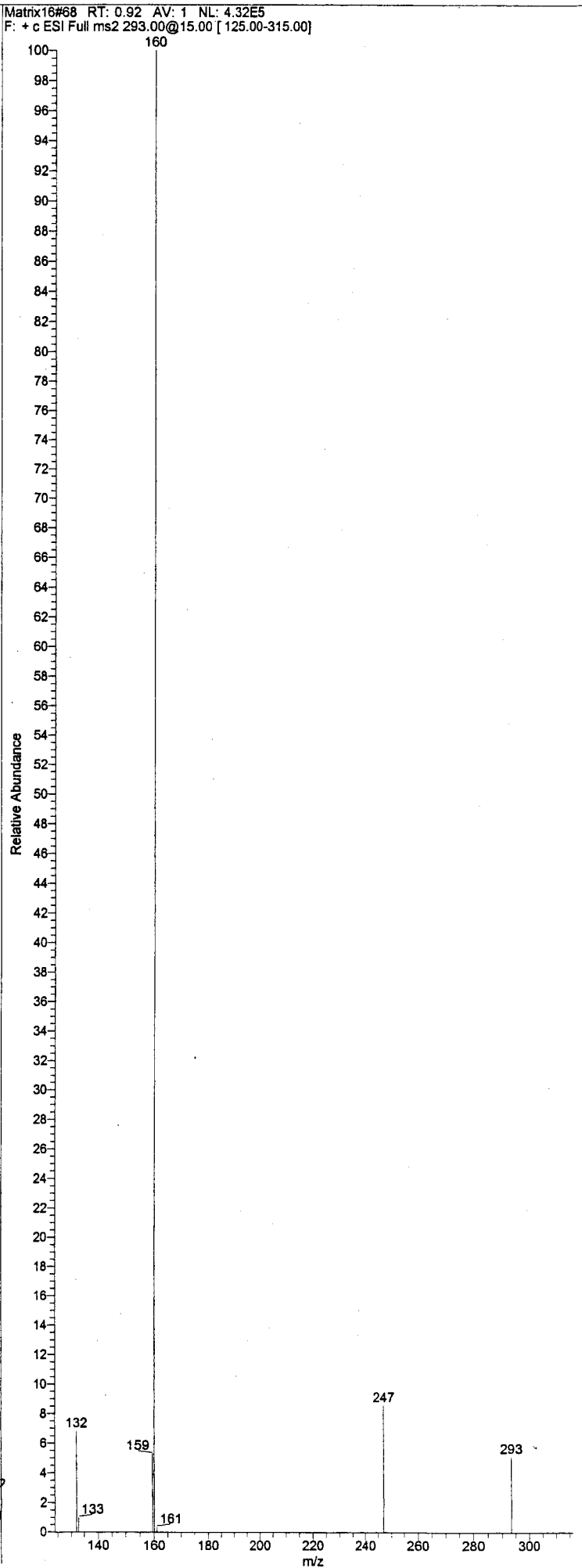
Ex 446 (405)



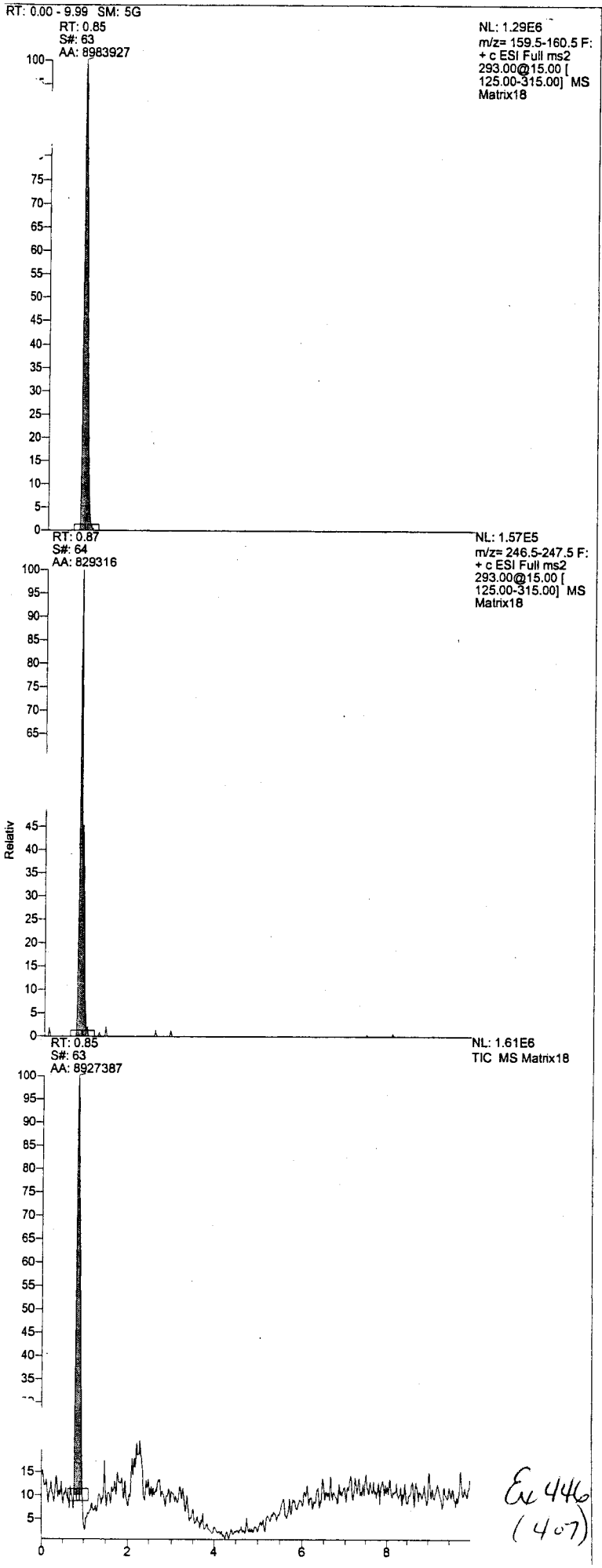
C:\Xcalibur\...\MAT_Positive\Matrix16 02/12/07 06:48:38 500 ppm EDTA JDB

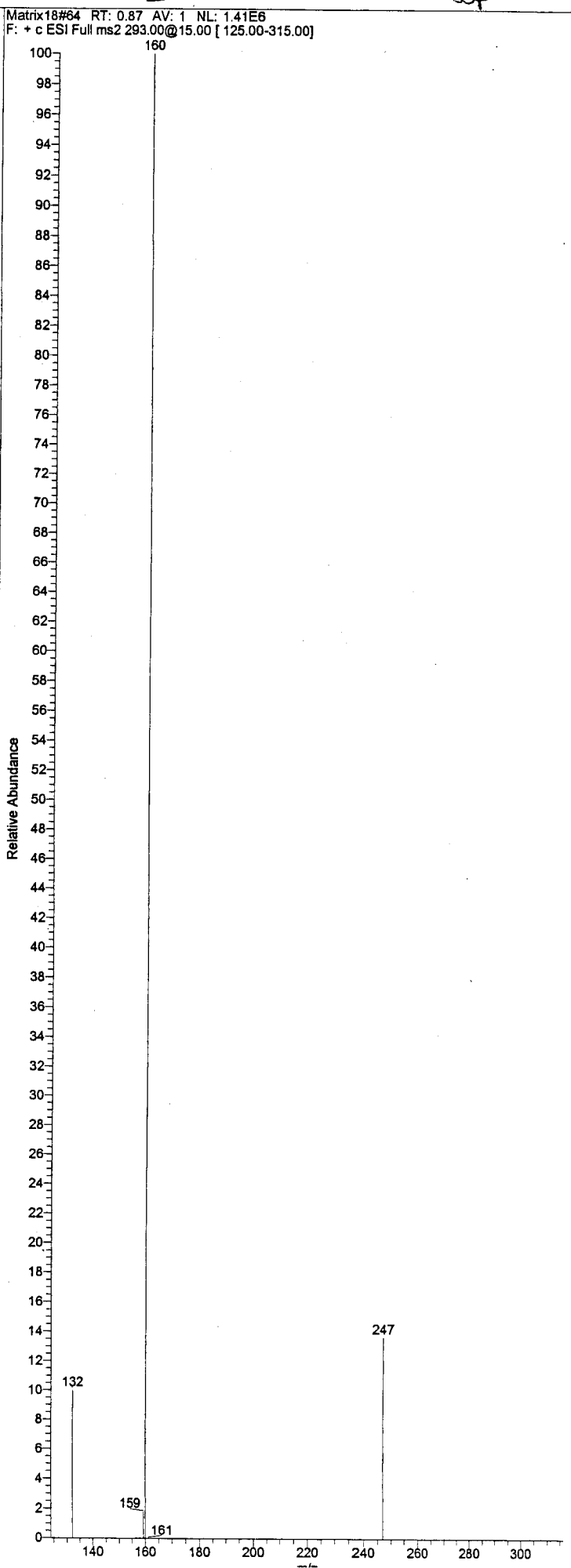


Ex 446 (400)



C:\Xcalibur\...\MAT_Positive\Matrix18 02/12/07 07:10:21 500 ppm EDTA





Sequence---MATRIX_neg.sld [Open]

Sample Name: Matrix 6 ("50 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX01	1	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			6	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Matrix 7 ("50 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX02	02	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			7	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JDP
2/13/07

Ex 446
(408)

Sequence---MATRIX_neg.sld [Open]

Sample Name: Matrix 8 ("50 ppm")
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX03	03	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			8	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Matrix 9 ("50 ppm")
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX04	04	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			9	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JoB
2/13/07

Ex 446
(409)

Sequence---MATRIX_neg.sld [Open]

Sample Name: Matrix 10 ("50 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX05	05	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			10	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX06	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JDB
2/17/07

Ex 446
(410)

Sequence---MATRIX_neg.sld [Open]

Sample Name: 50 ppm EDTA
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX07	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 50 ppm EDTA
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX08	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JOB
2/13/07

Ex 446
(411)

Sequence---MATRIX_neg.sld [Open]

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX09	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 50 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX10	12	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JDB
2/13/07

Ex 446
(412)

Sequence---MATRIX_neg.sld [Open]

Sample Name: Matrix 1 ("500 ppm")

C ment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX11	01	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Matrix 2 ("500 ppm")

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX12	02	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			2	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JvB
2/13/07

Ex 446
(413)

Sequence---MATRIX_neg.sld [Open]

Sample Name: Matrix 3 ("500 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX13	03	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			3	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Matrix 4 ("500 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX14	04	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			4	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JDB
2/12/07

Ex 446
(414)

Sequence---MATRIX_neg.sld [Open]

Sample Name: Matrix 5 ("500 ppm")

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX15	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			5	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 500 ppm EDTA

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX16	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JoP
2/13/07

Ex 446
(415)

Sequence---MATRIX_neg.sld [Open]

Sample Name: 500 ppm EDTA
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX17	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 500 ppm EDTA
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX18	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JbP
2/13/07

Ex 446
(416)

Sequence---MATRIX_neg.sld [Open]

Sample Name: 500 ppm EDTA

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX19	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 500 ppm EDTA

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	MATRIX20	06	C:\Xcalibur\Data\EDTA\Brewer\MAT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

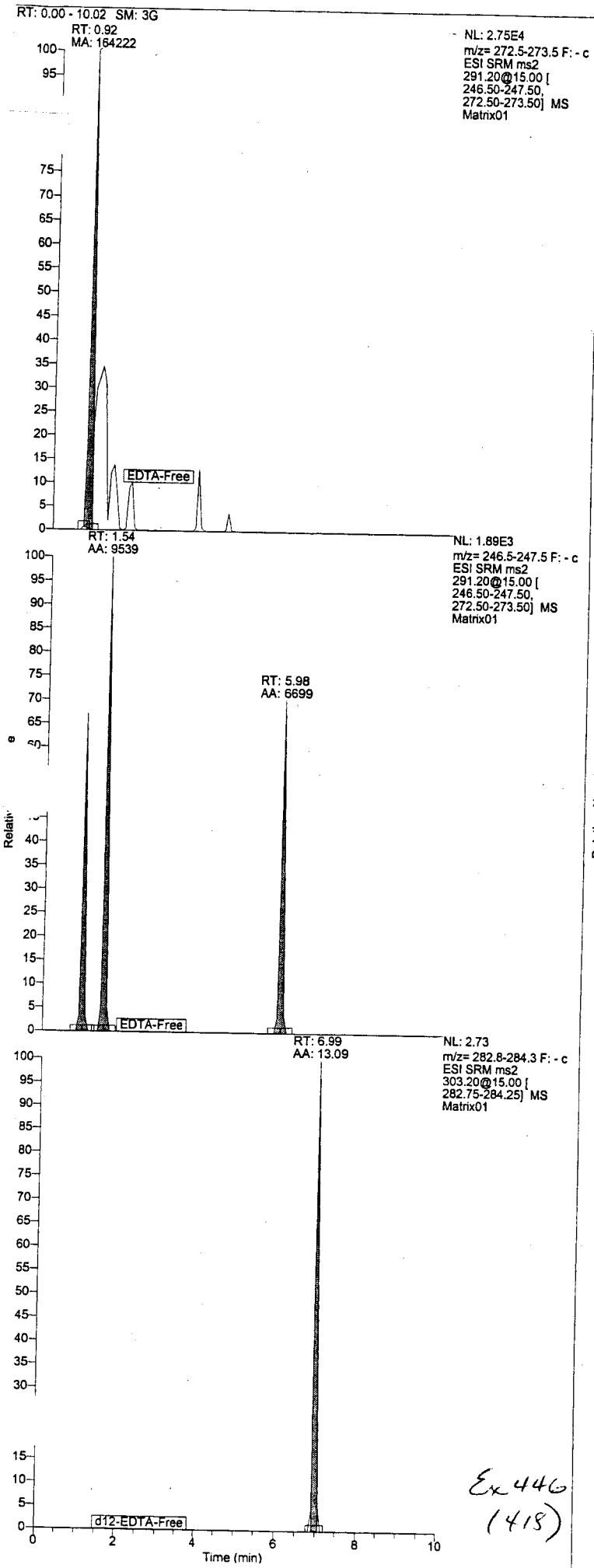
Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

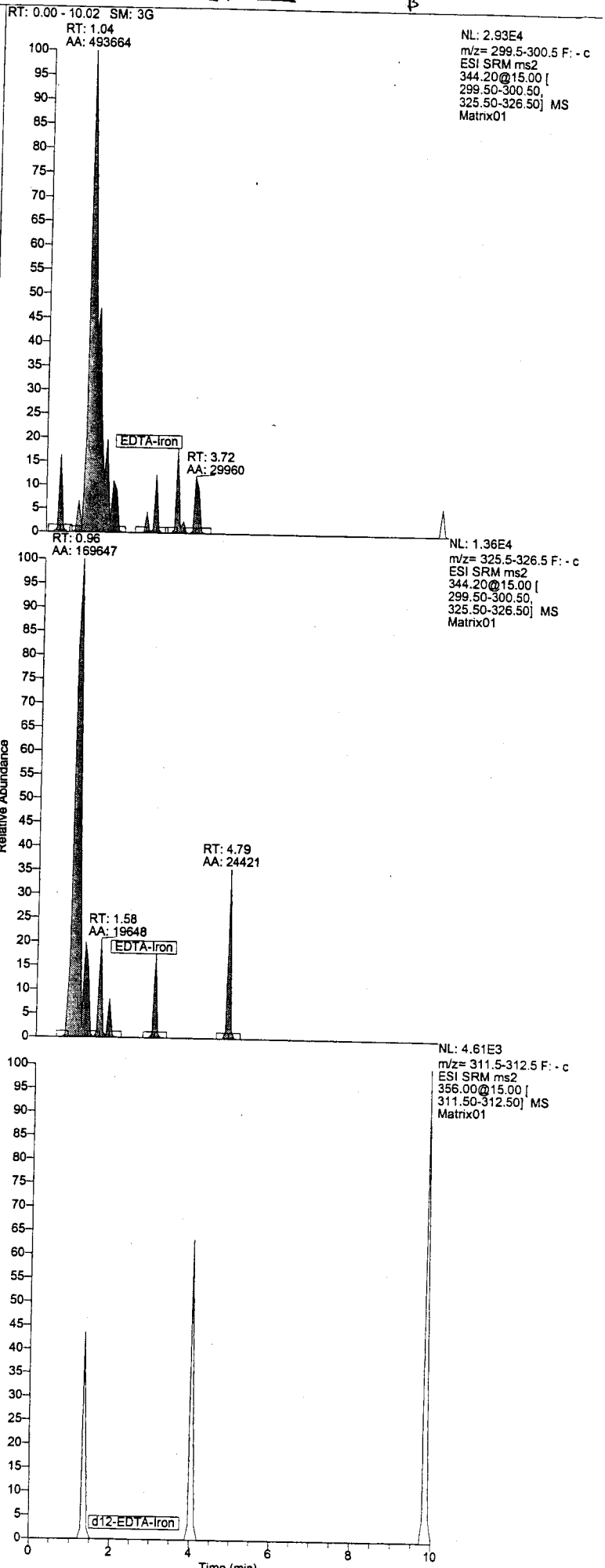
****

JvP
2/13/07

Ex 446
(417)

C:\Xcalibur\...\MAT_Negative\Matrix01 02/13/07 09:05:10 Matrix 6 ("50 ppm") JbB





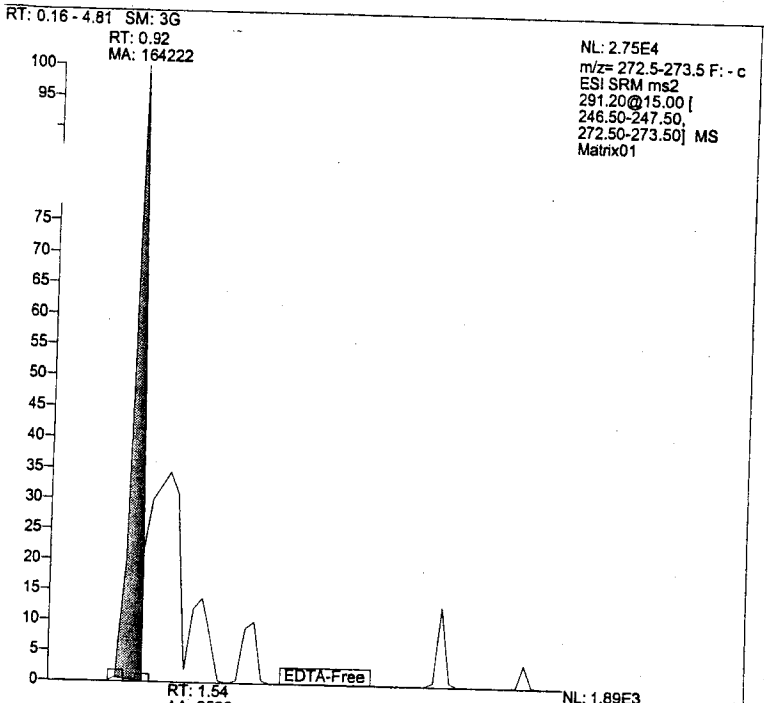
Ex 446
(418)

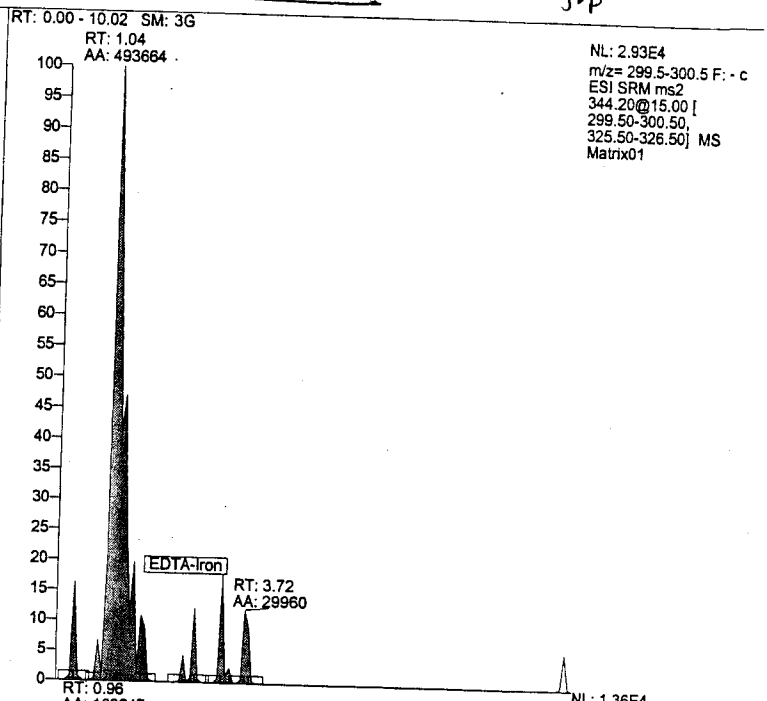
C:\Xcalibur\...\MAT_Negative\Matrix01 200M

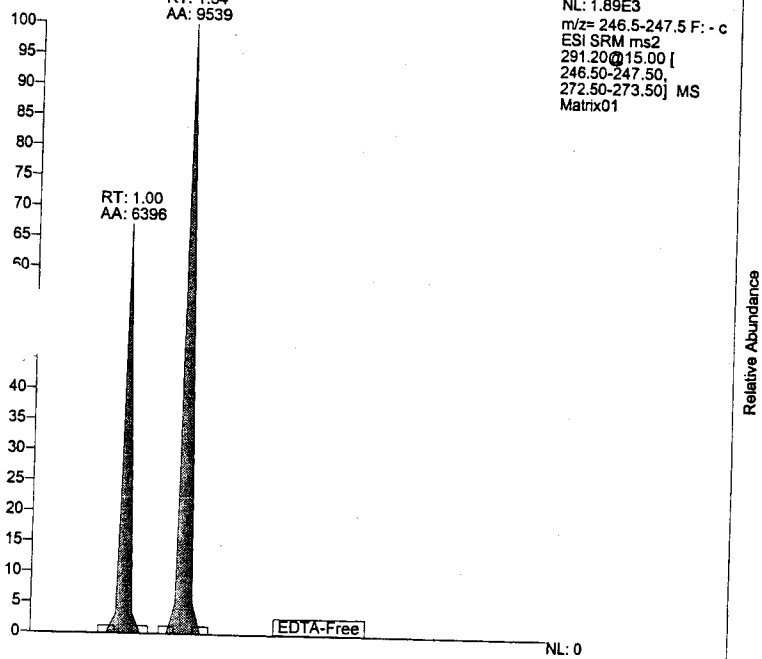
02/13/07 09:05:10

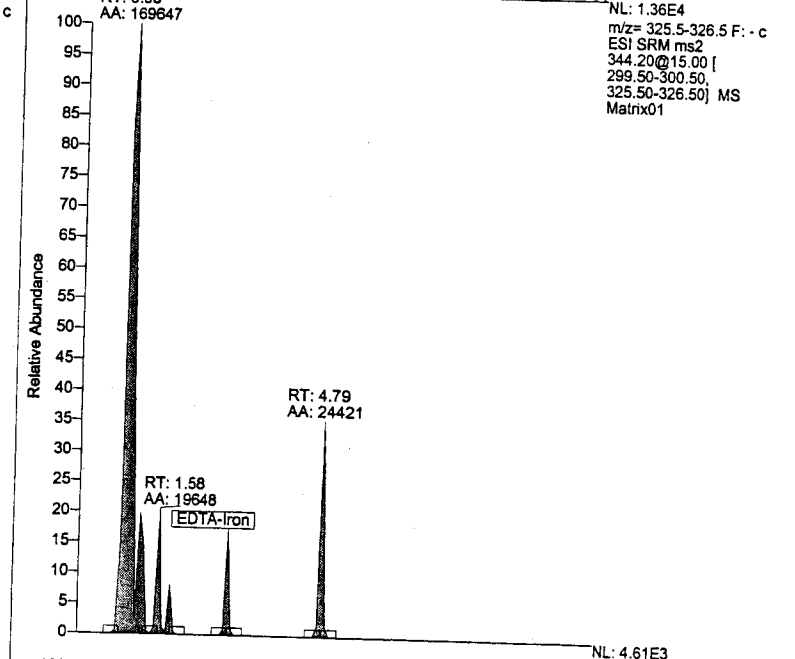
Matrix 6 ("50 ppm")

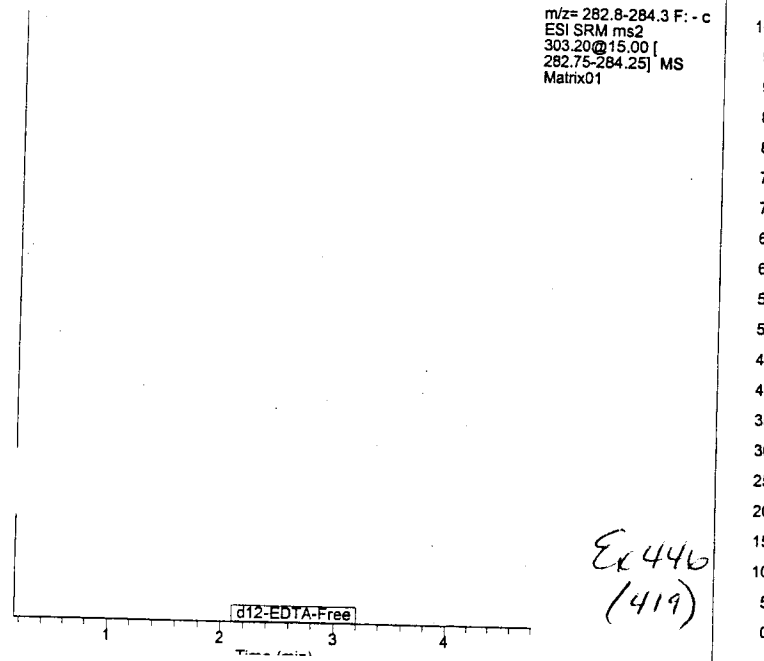
JDB

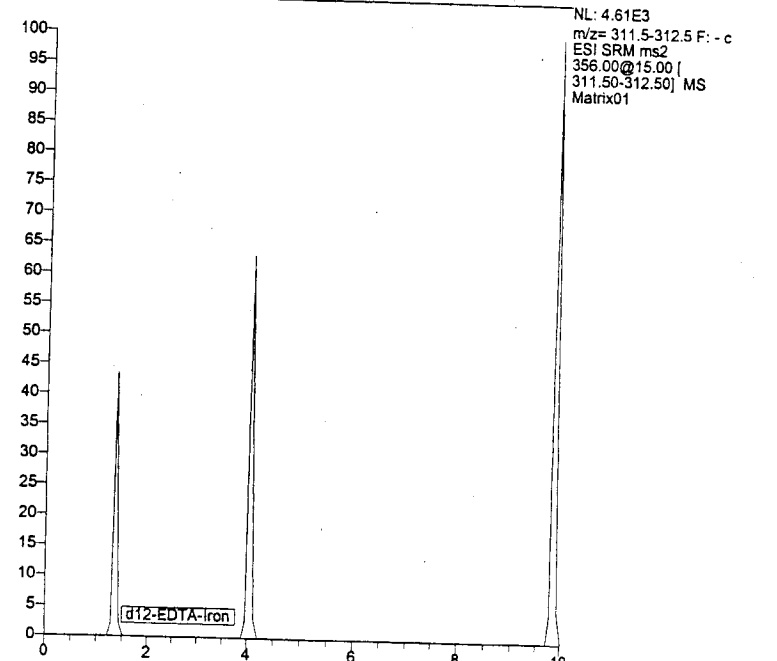






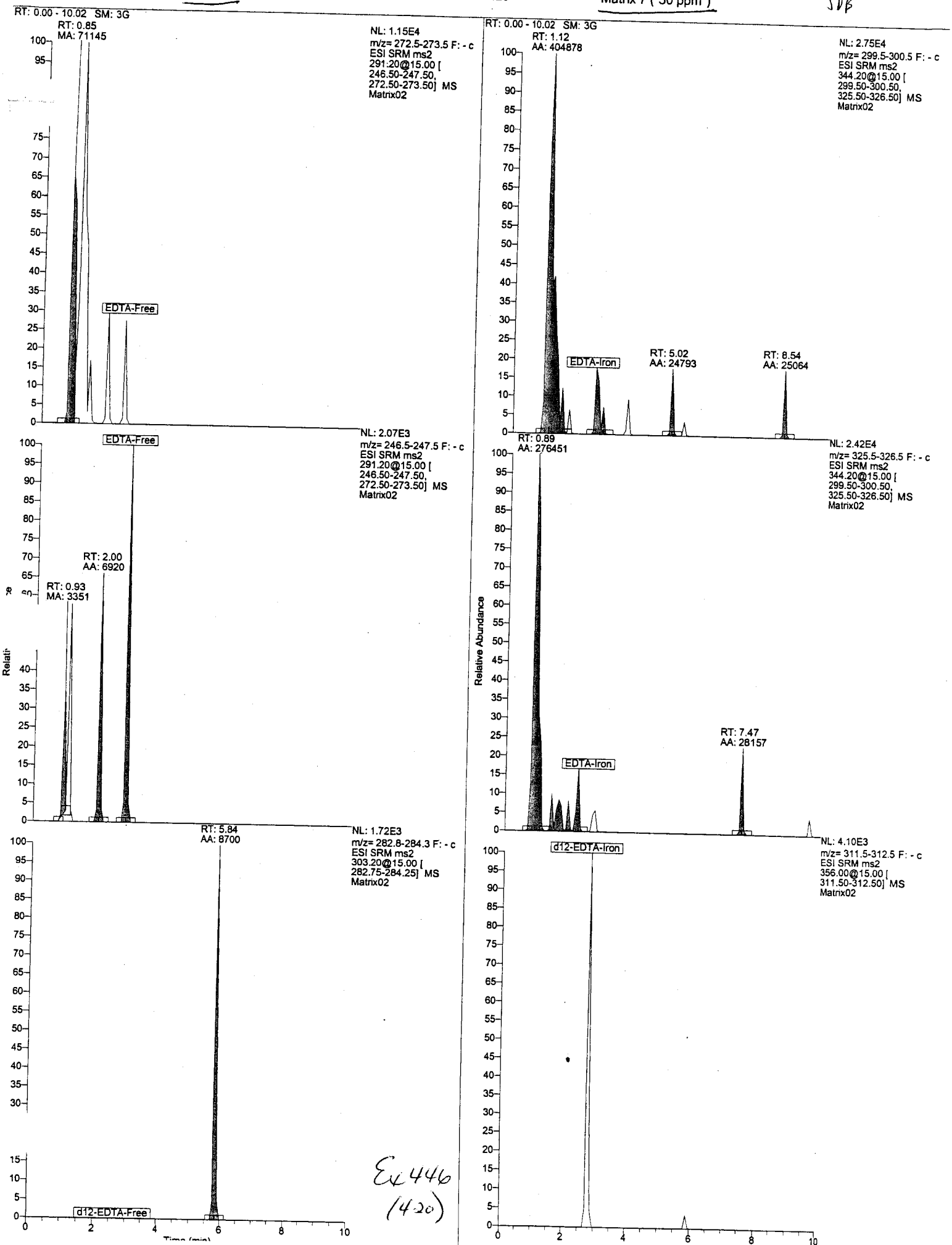






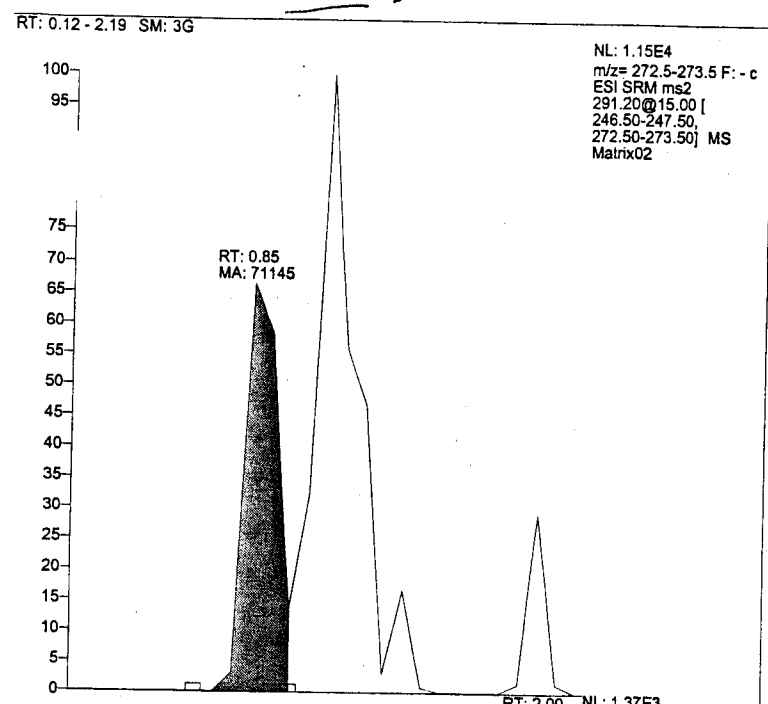
Ex 446
(419)

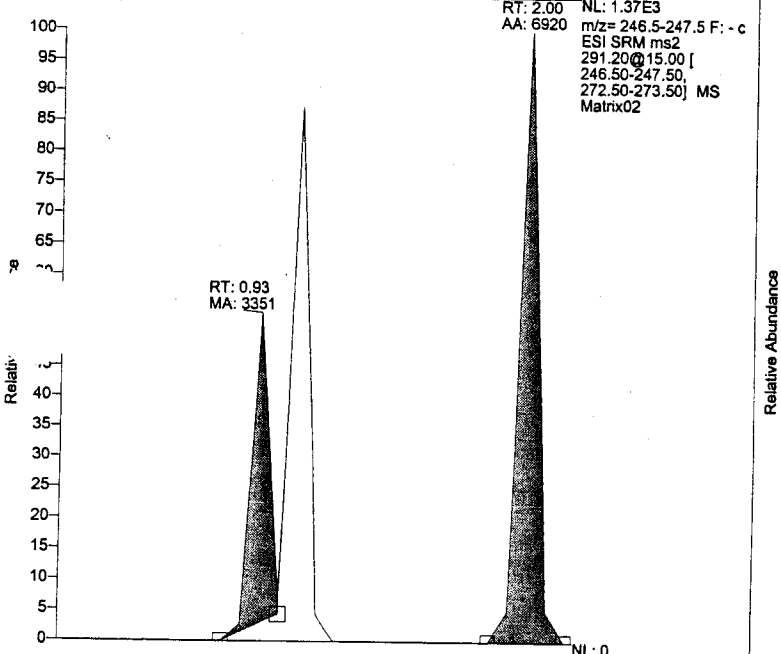
C:\Xcalibur\...\MAT_Negative\Matrix02 02/13/07 09:16:25 Matrix 7 ("50 ppm") JDB

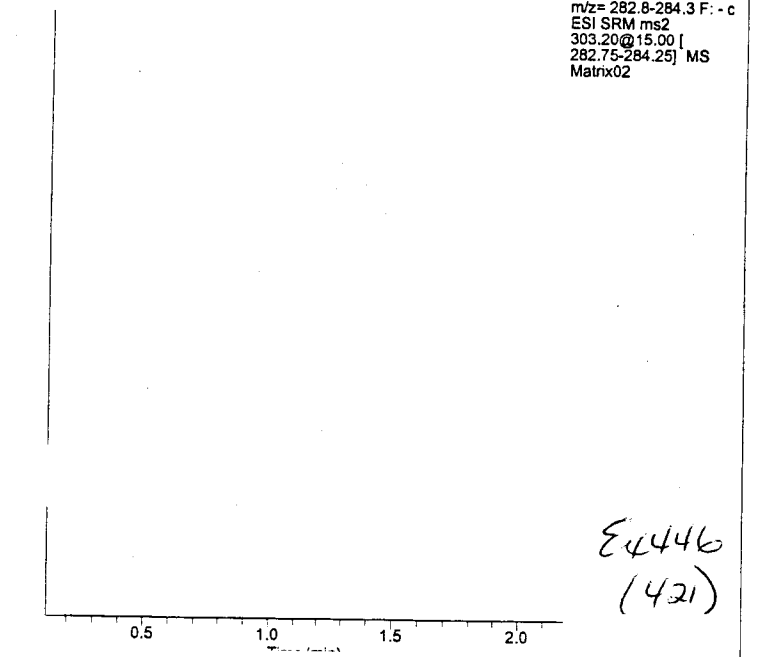


Ex 446
(420)

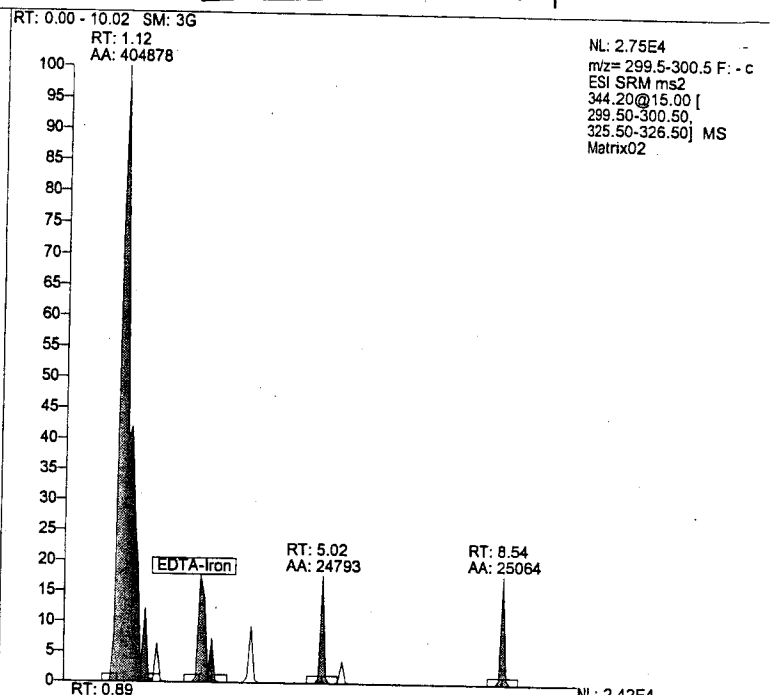
C:\Xcalibur\...\MAT_Negative\Matrix02 200M 02/13/07 09:16:25 Matrix 7 ("50 ppm") JDB

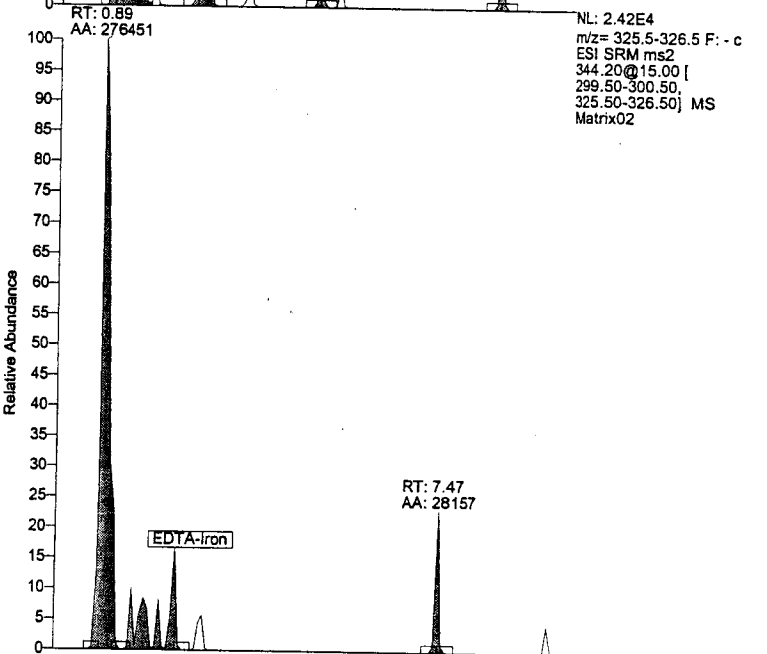


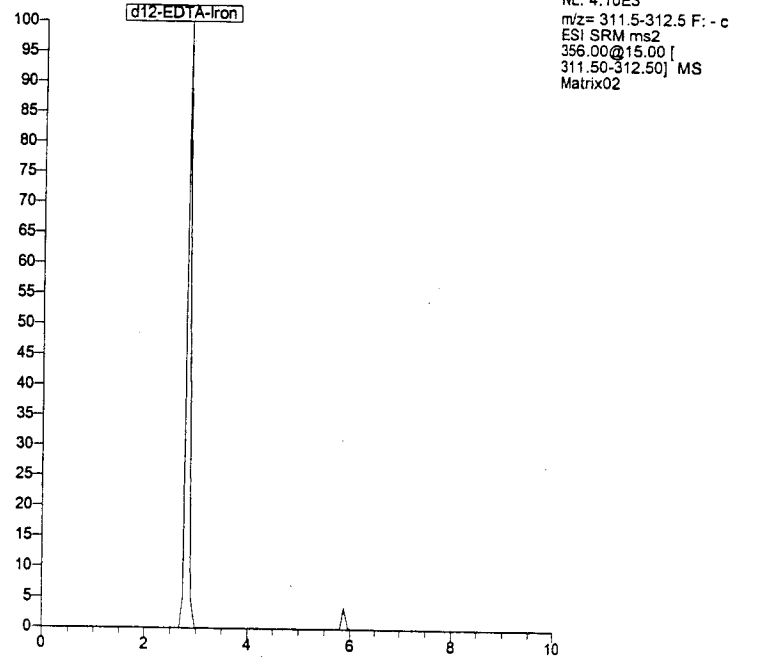


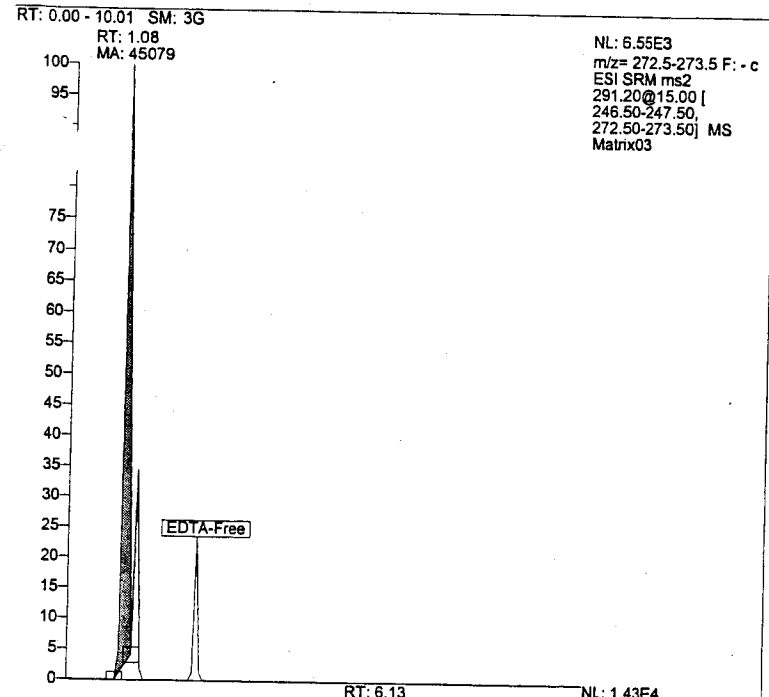


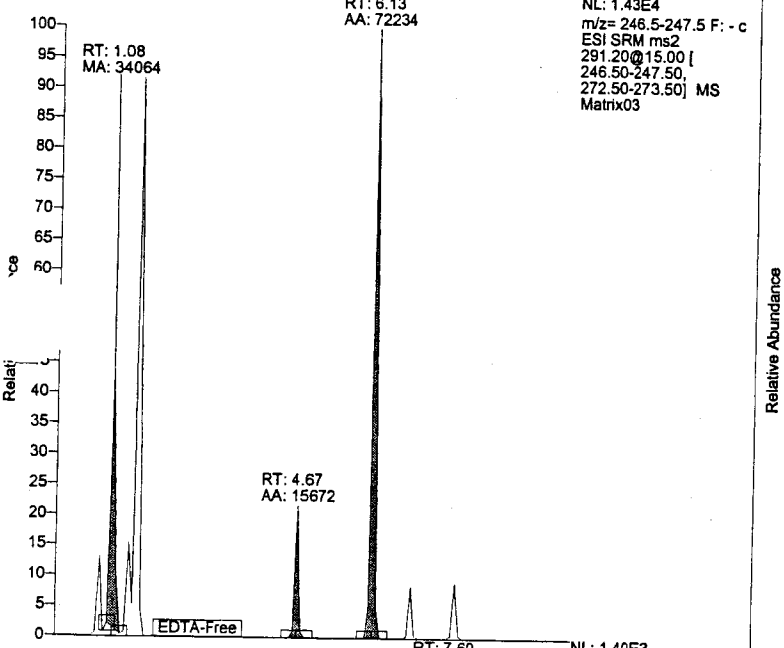
E4446
(421)

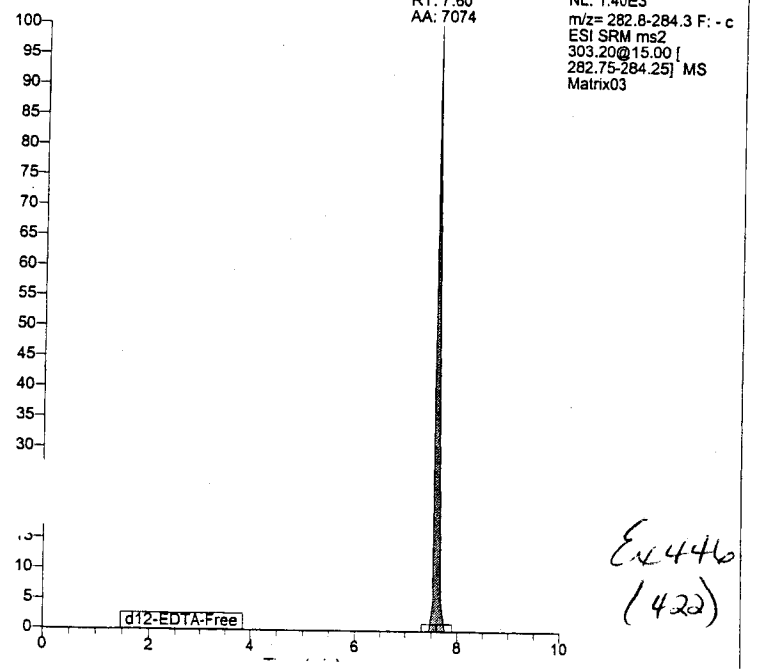




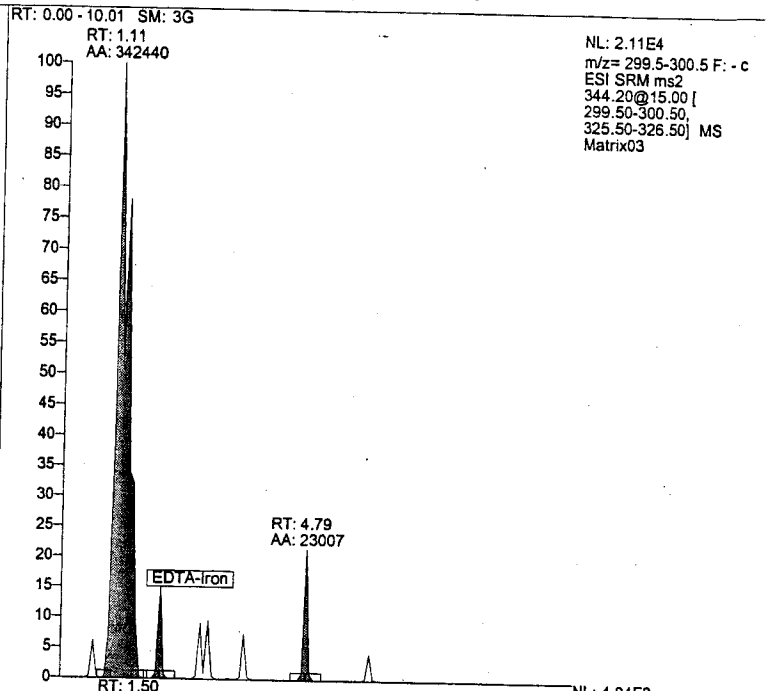


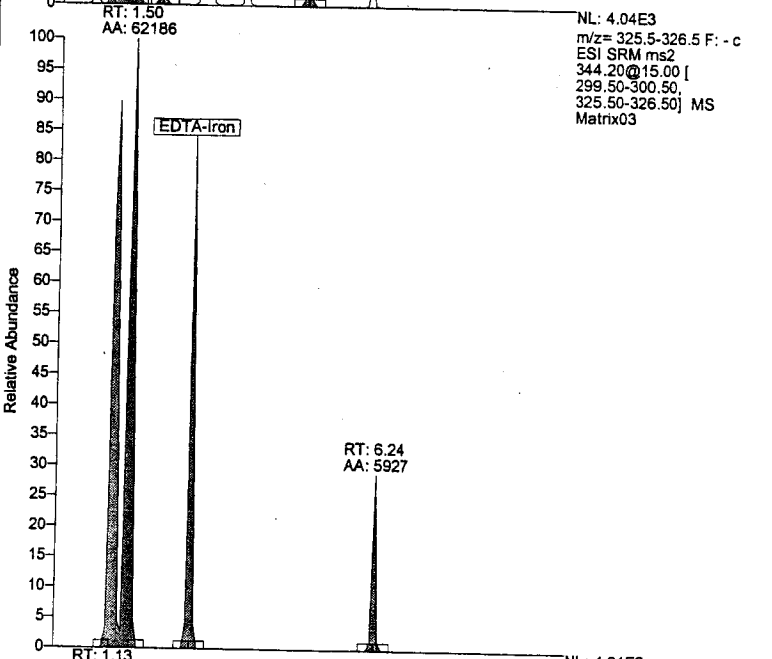

RT: 0.00 - 10.01 SM: 3G
RT: 1.08
MA: 45079
NL: 6.55E3
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Matrix03
EDTA-Free

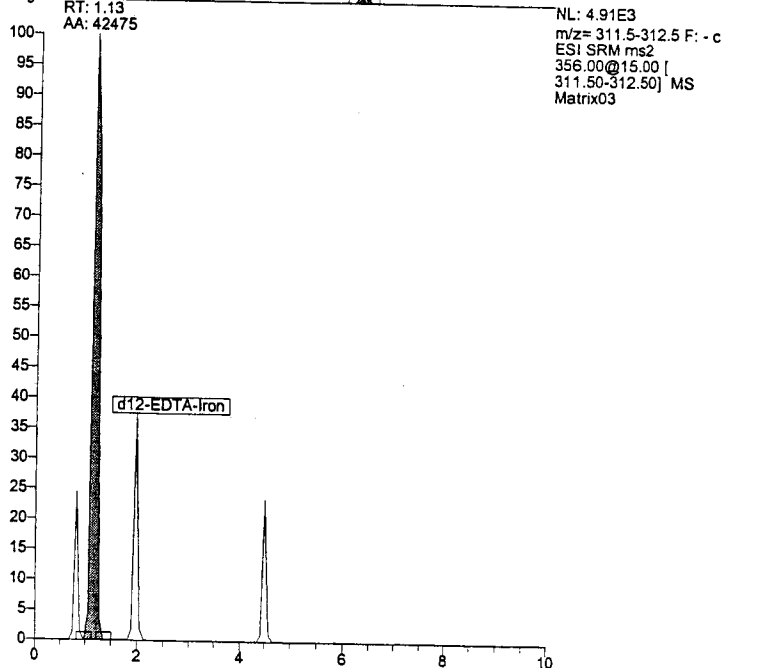

RT: 1.08
MA: 34064
RT: 6.13
AA: 72234
NL: 1.43E4
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Matrix03
RT: 4.67
AA: 15672
EDTA-Free


RT: 7.60
AA: 7074
NL: 1.40E3
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Matrix03
d12-EDTA-Free

Ex446 (422)


RT: 0.00 - 10.01 SM: 3G
RT: 1.11
AA: 342440
NL: 2.11E4
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Matrix03
RT: 4.79
AA: 23007
EDTA-Iron


RT: 1.50
AA: 62186
NL: 4.04E3
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Matrix03
EDTA-Iron
Relative Abundance
RT: 6.24
AA: 5927

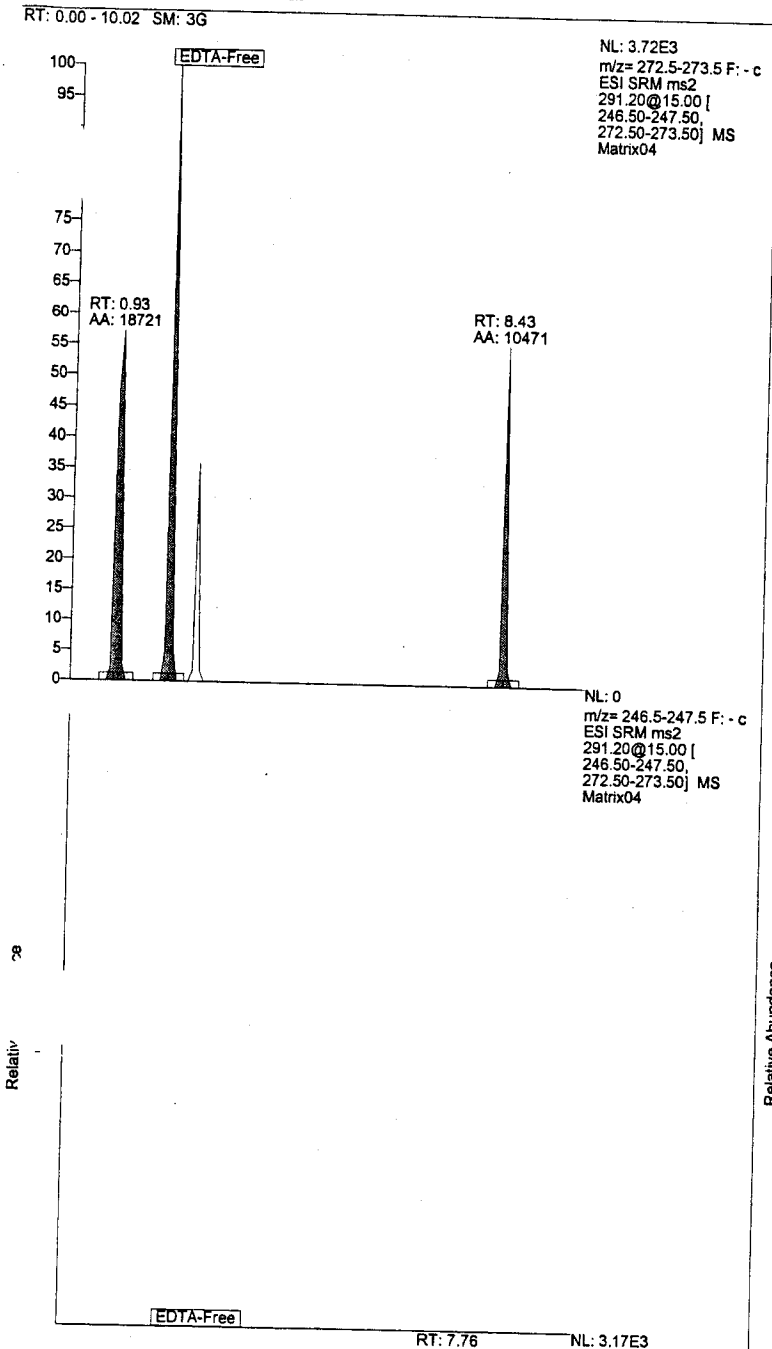

RT: 1.13
AA: 42475
NL: 4.91E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Matrix03
d12-EDTA-Iron

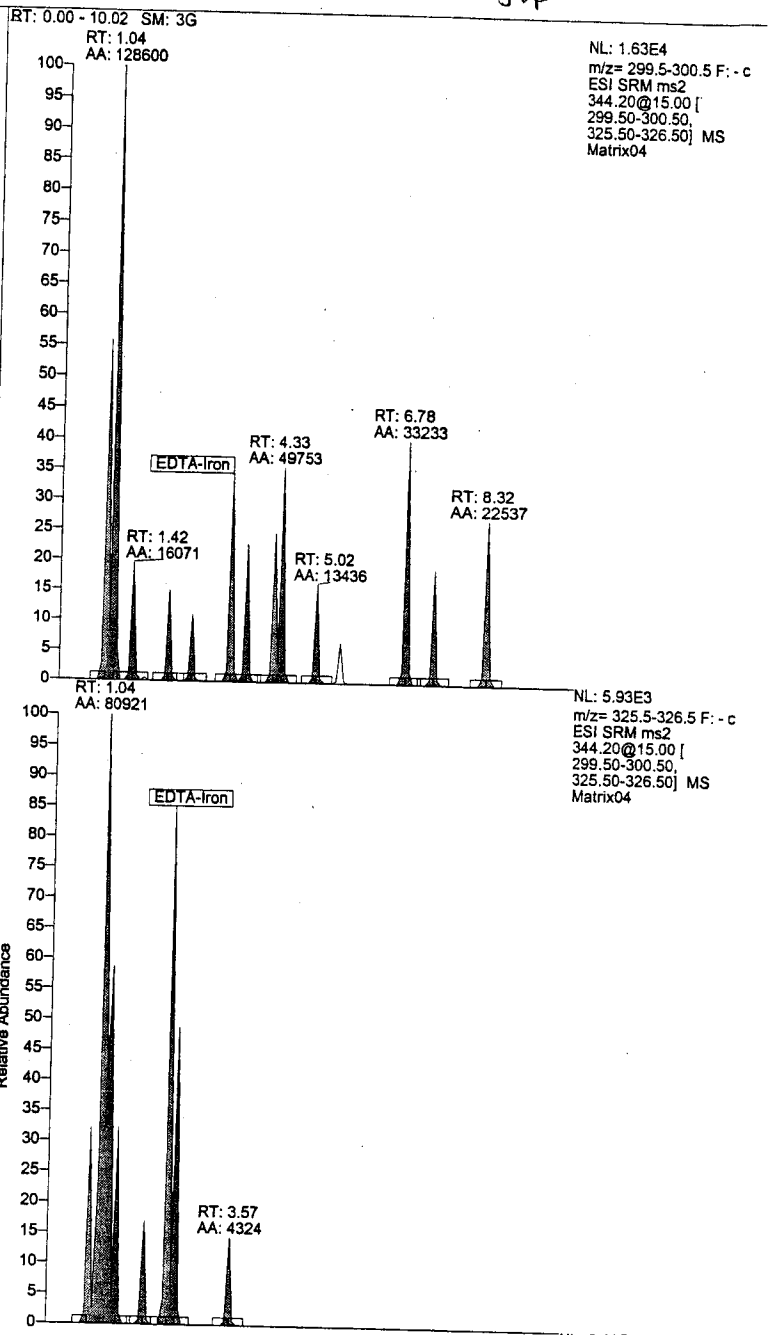
C:\Xcalibur\...\MAT_Negative\Matrix04

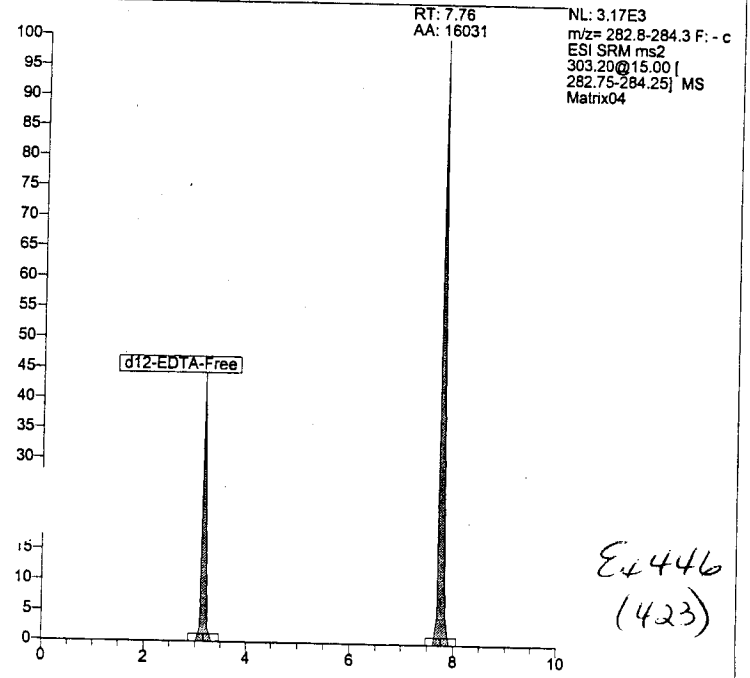
02/13/07 09:38:15

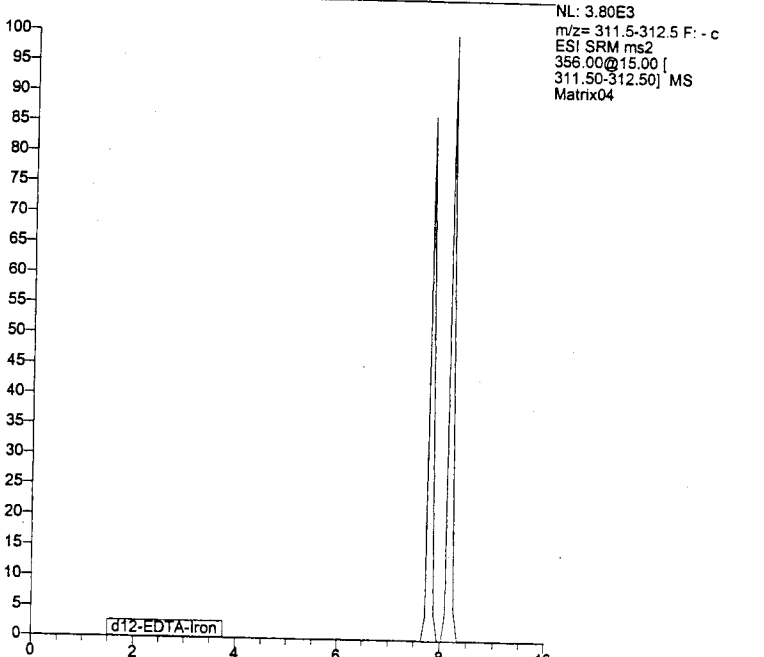
Matrix 9 ("50 ppm")

JDB



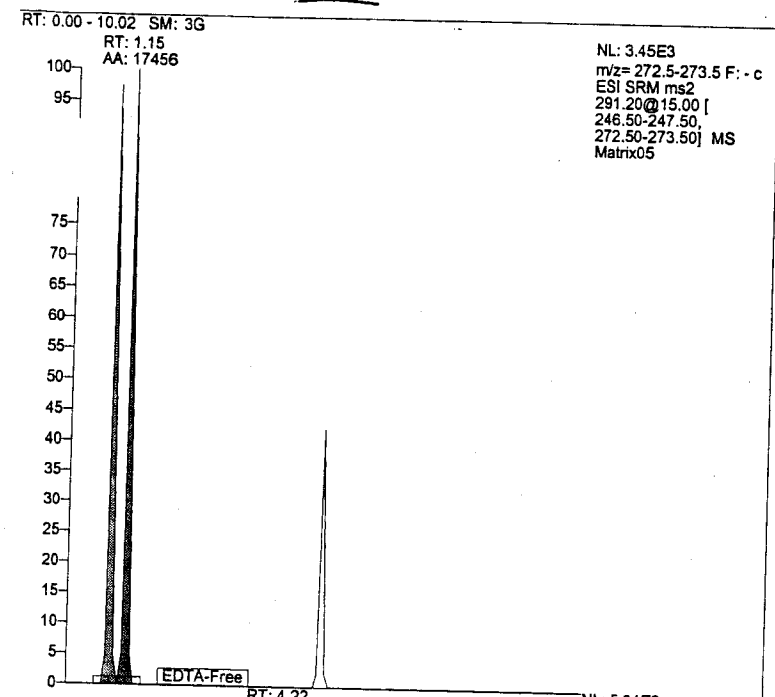


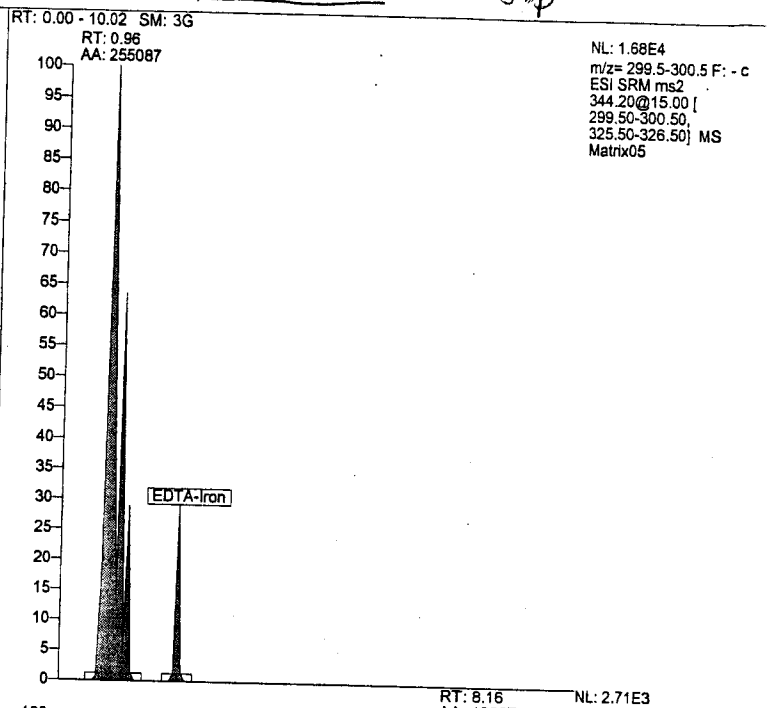


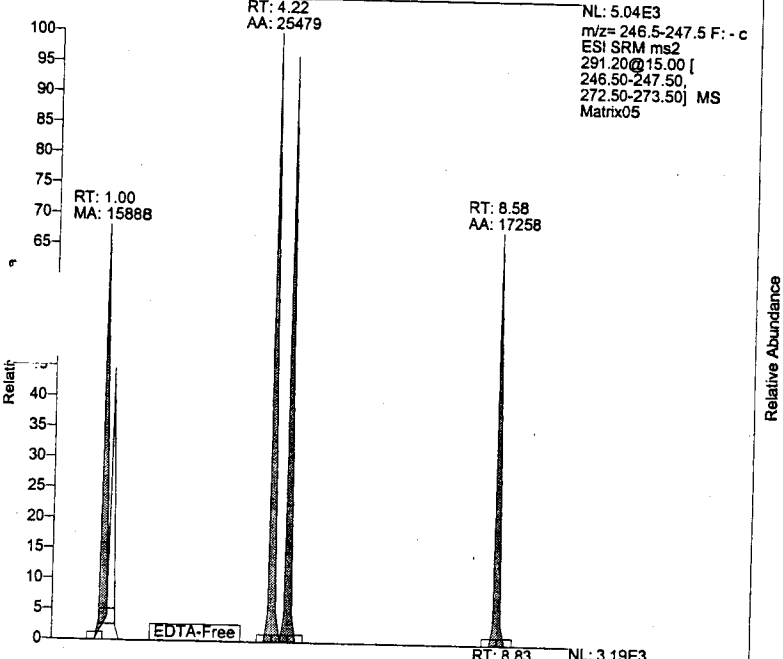


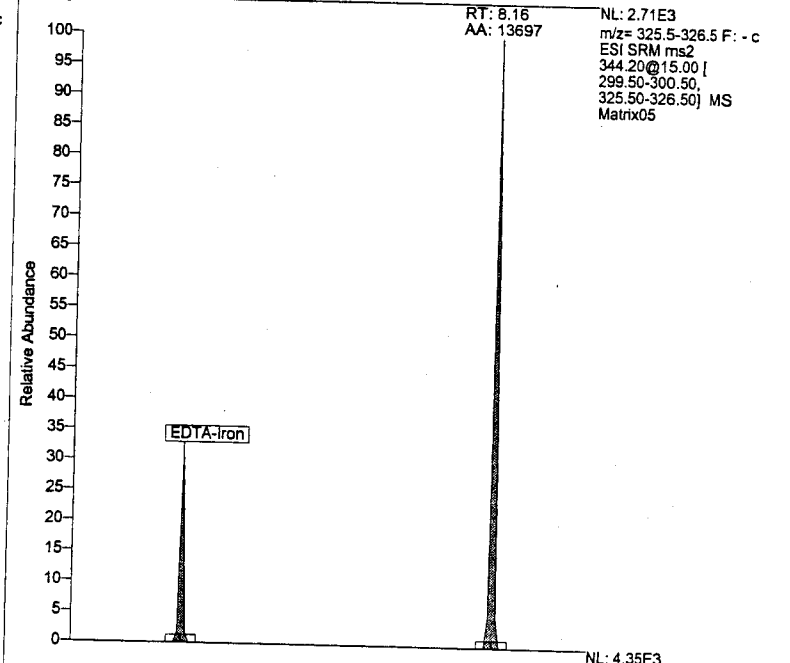
E.446
(423)

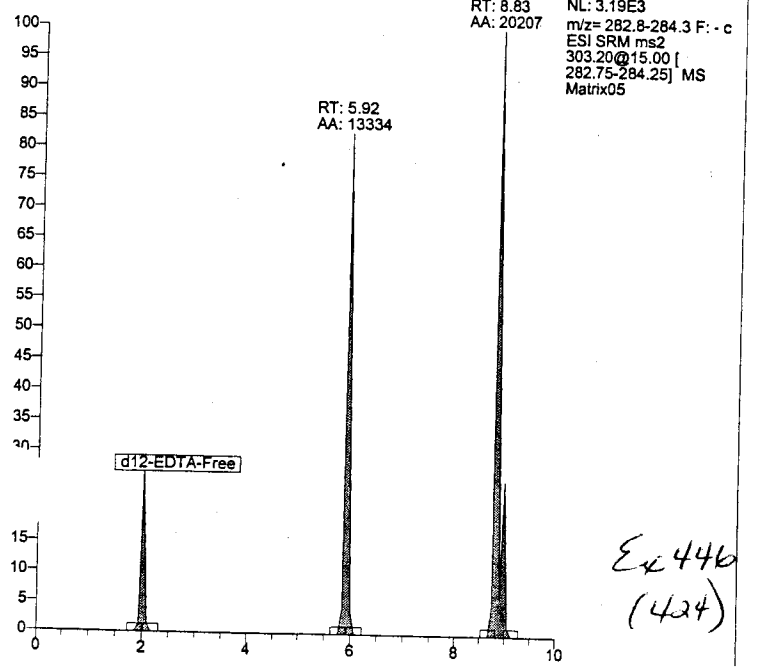
C:\Xcalibur\...\MAT_Negative\Matrix05 02/13/07 09:49:09 Matrix 10 ("50 ppm") JDB

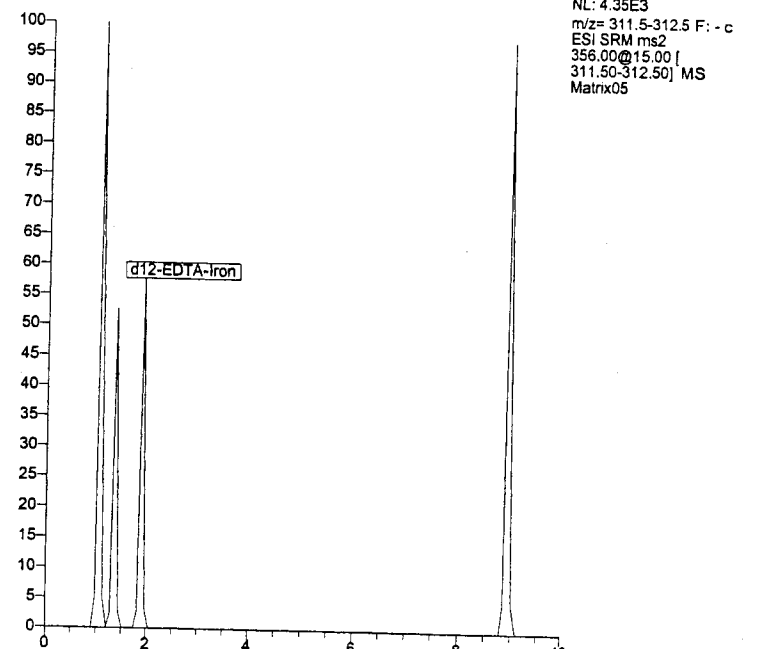






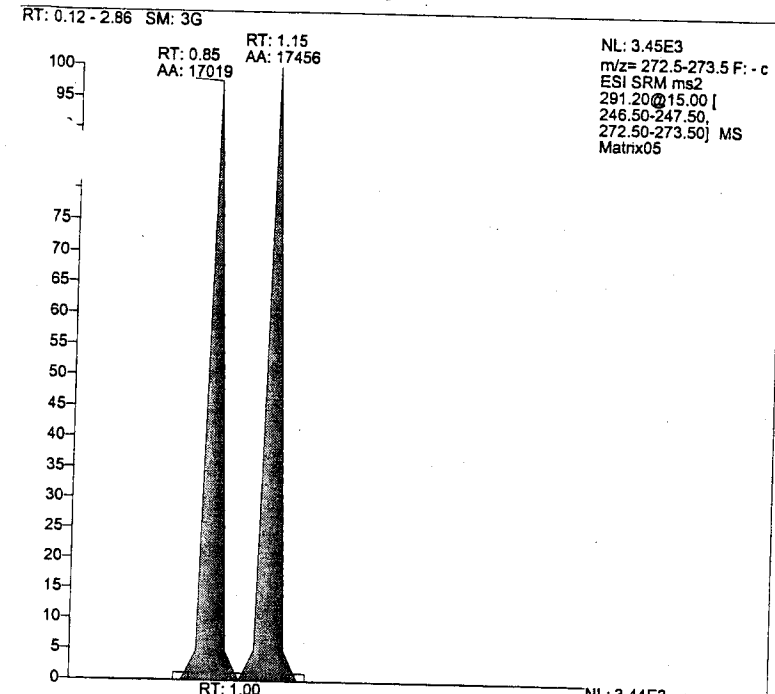


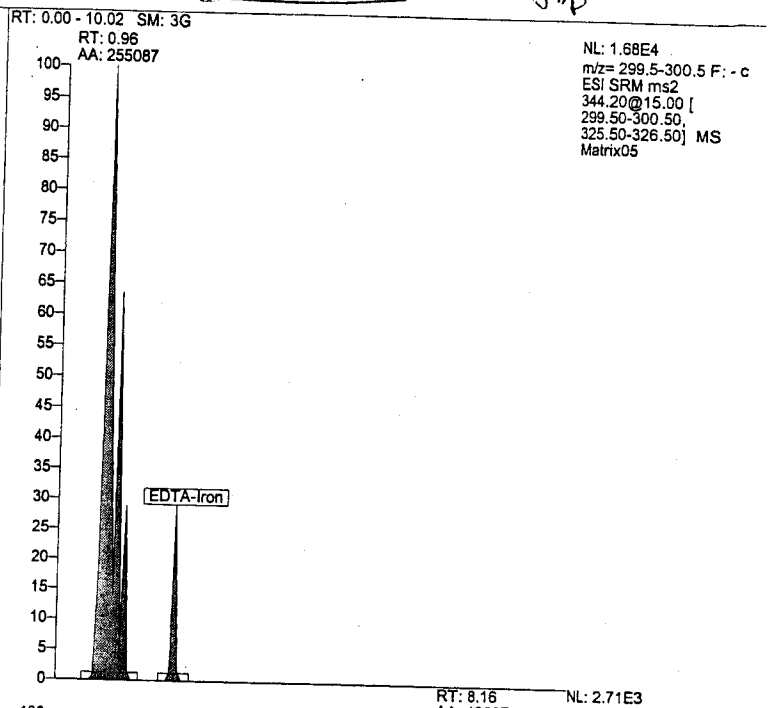


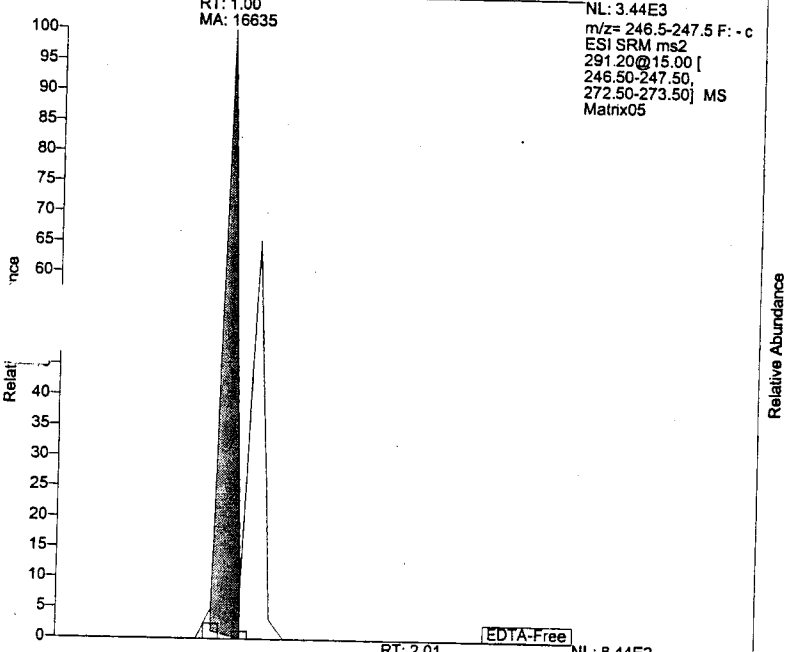


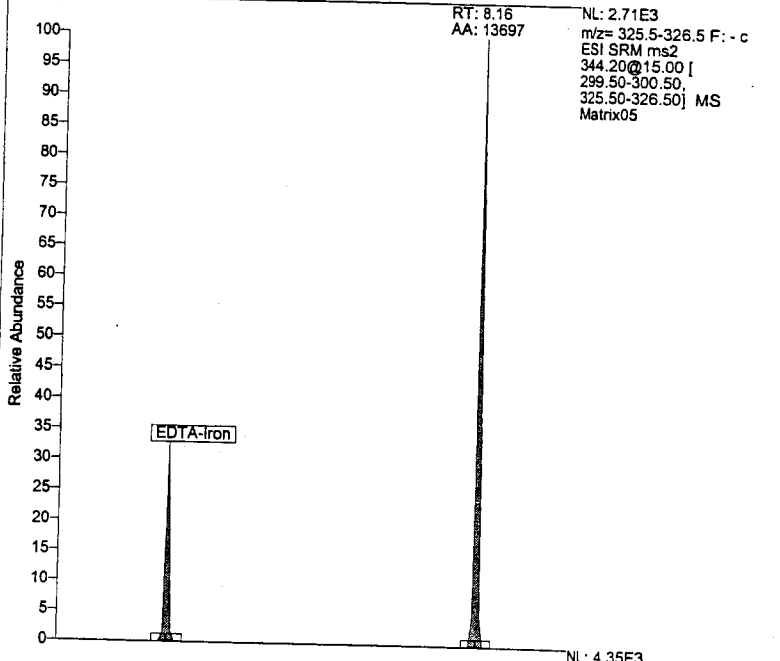
Ex 446
(424)

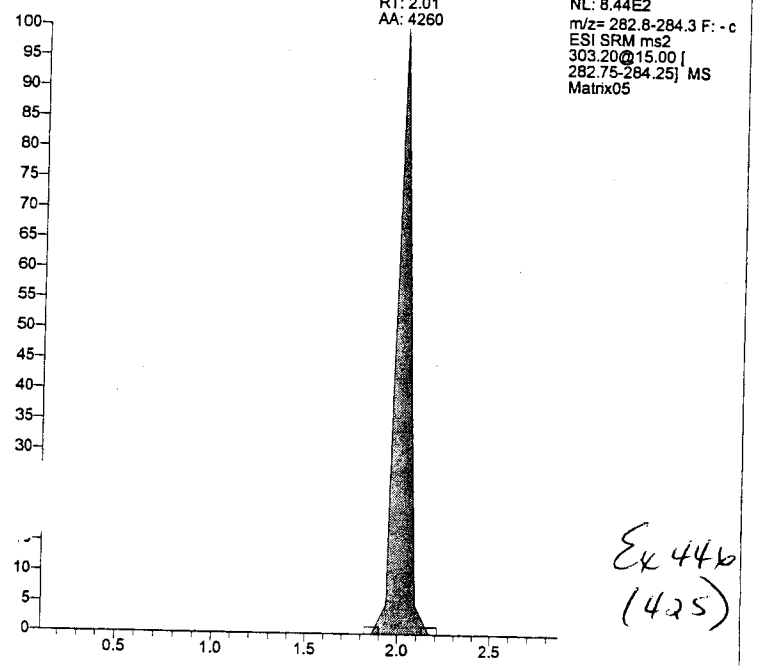
C:\Xcalibur\...\MAT_Negative\Matrix05 200M 02/13/07 09:49:09 Matrix 10 ("50 ppm") JMB

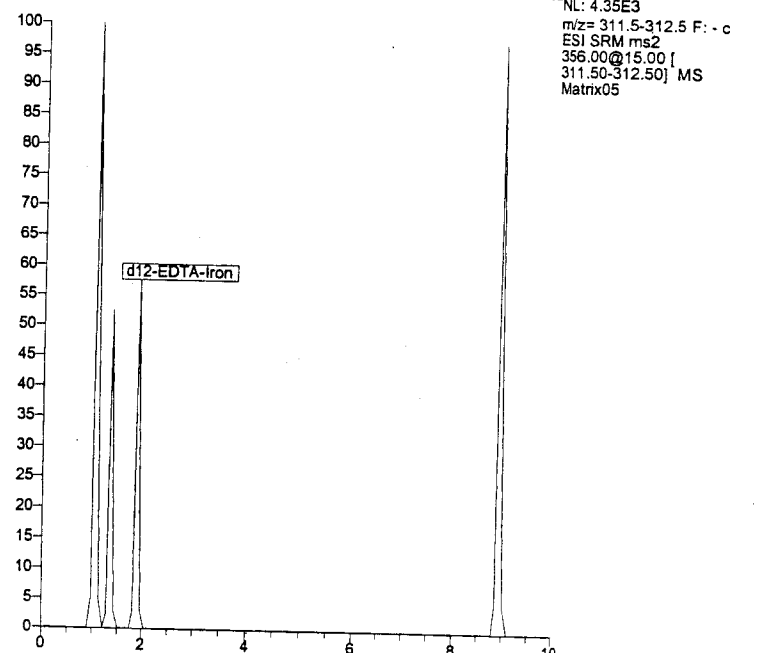












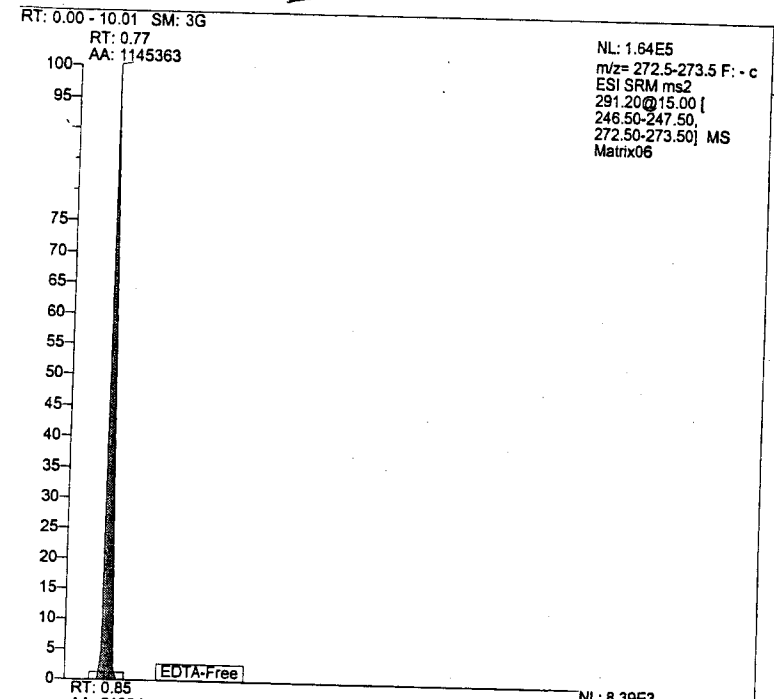
Ex 446
(425)

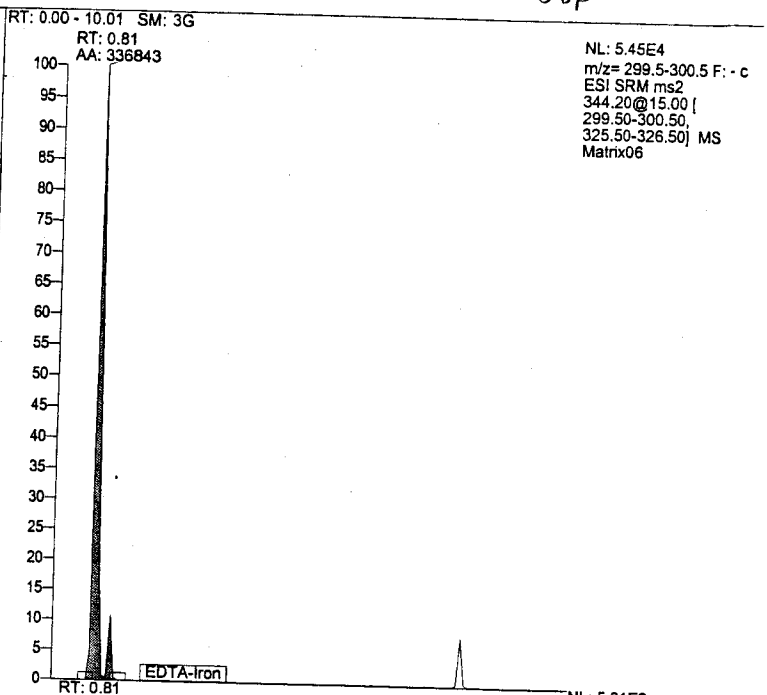
C:\Xcalibur\...\MAT_Negative\Matrix06

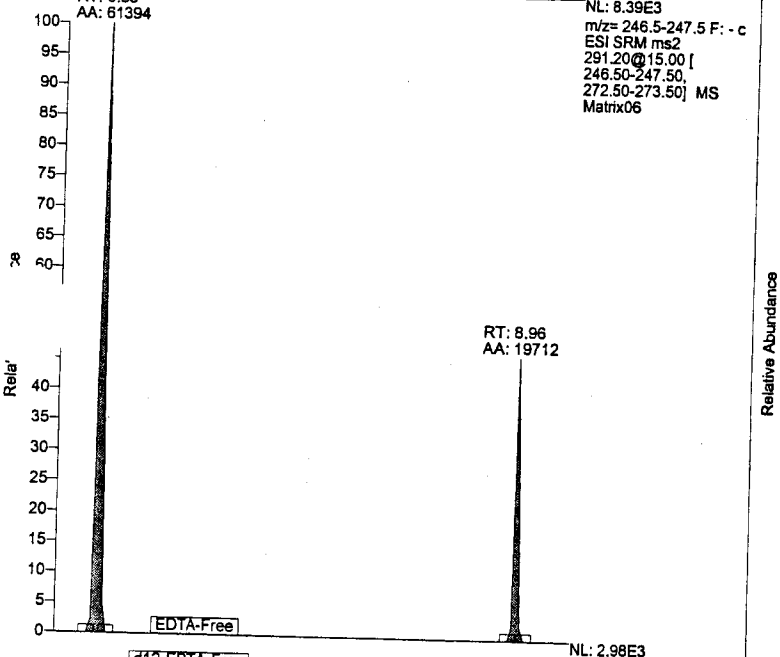
02/13/07 10:00:00

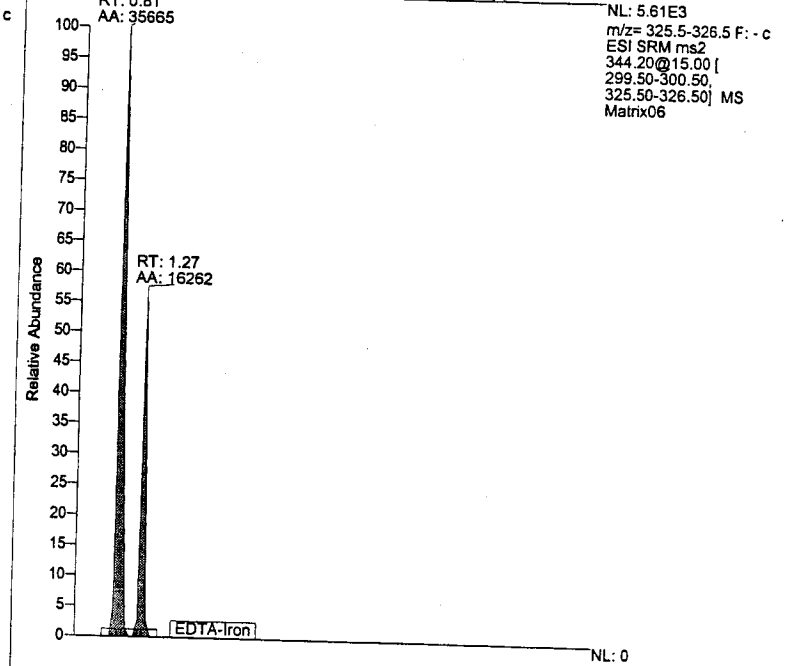
50 ppm EDTA

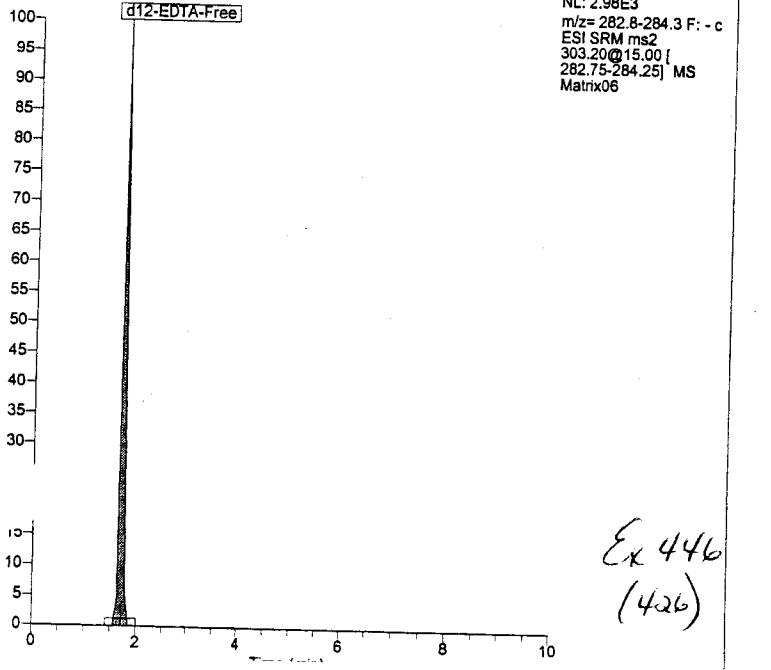
JDB

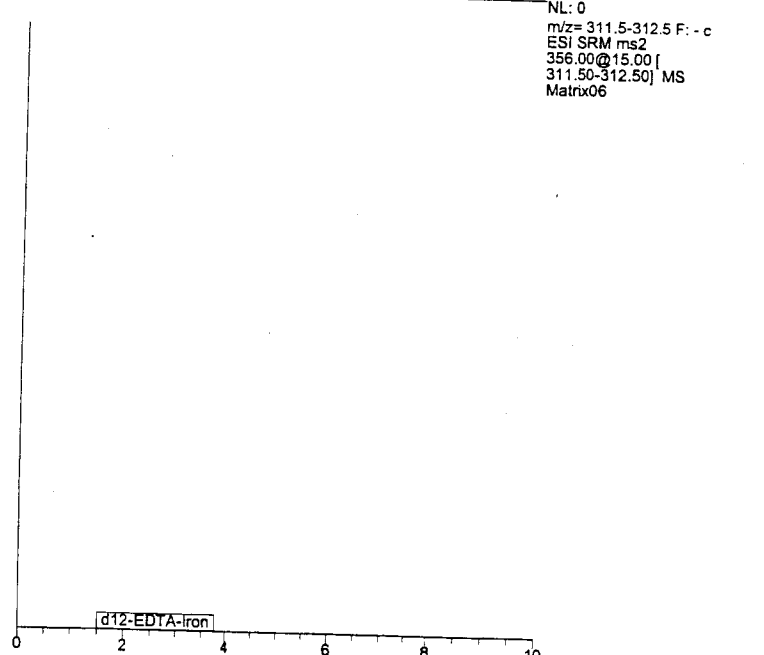












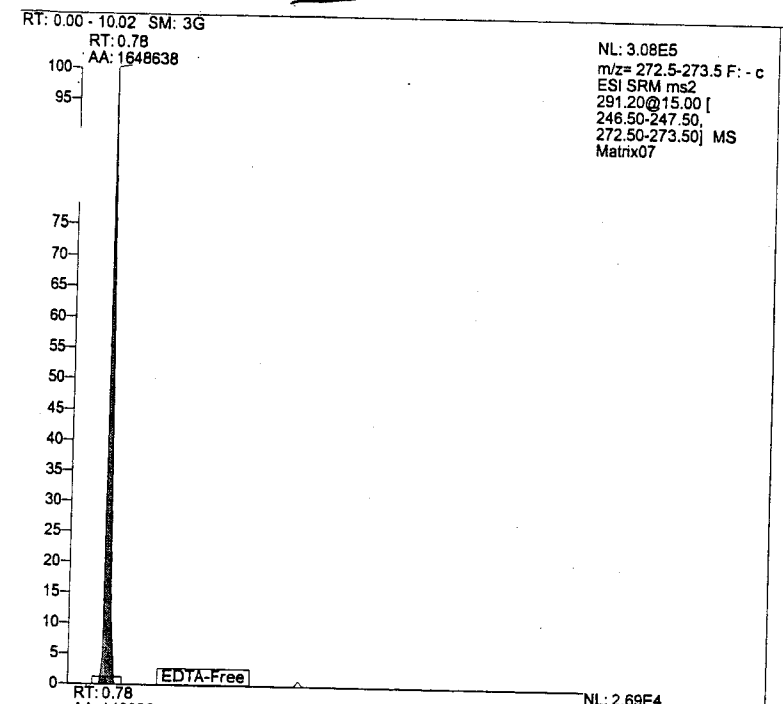
Ex 446
(426)

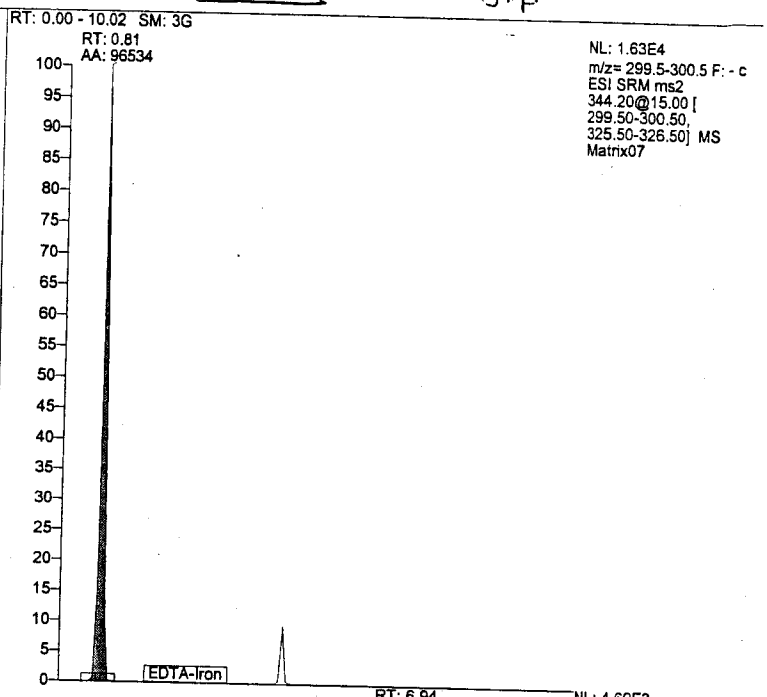
C:\Xcalibur\...\MAT_Negative\Matrix07

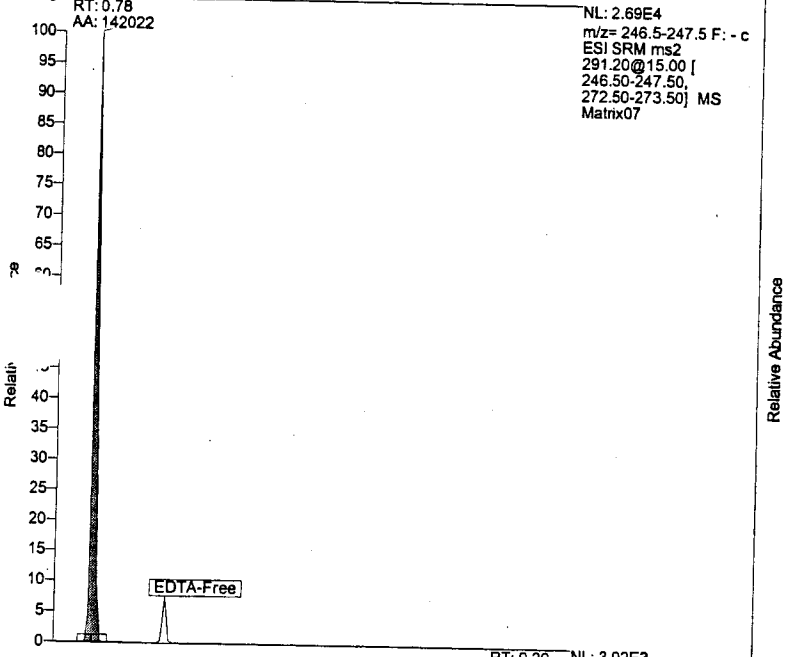
02/13/07 10:10:54

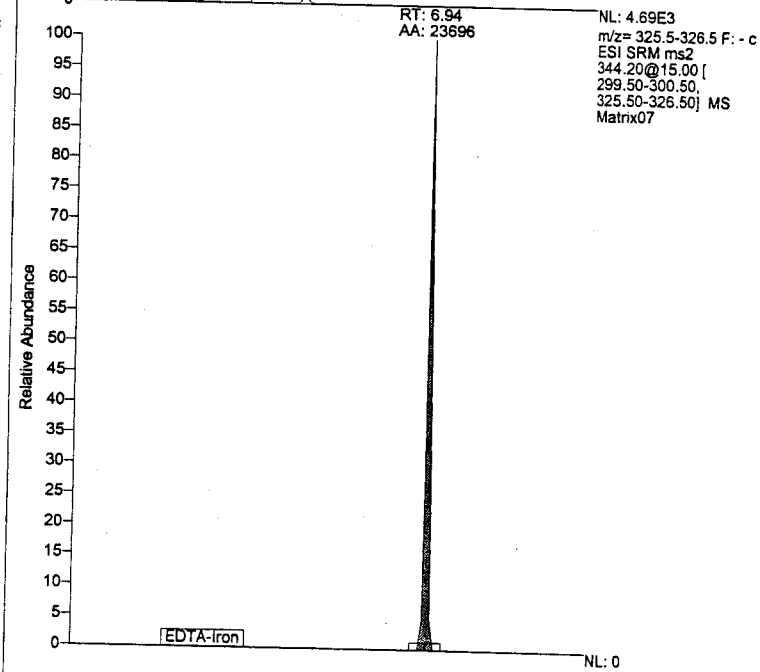
50 ppm EDTA

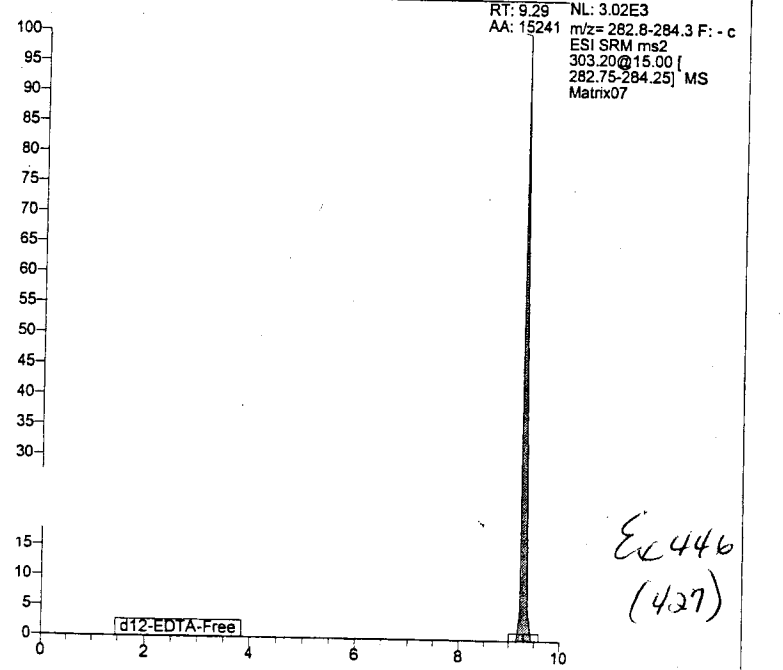
JMB

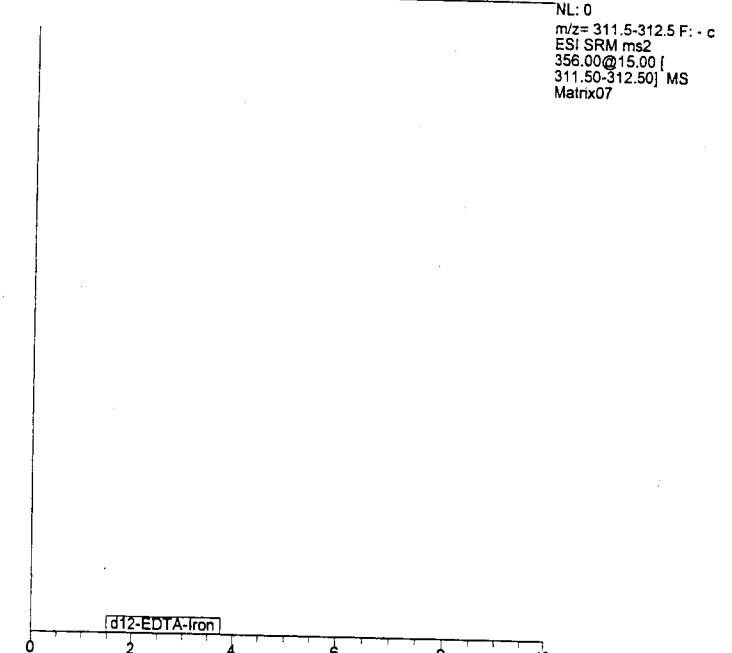






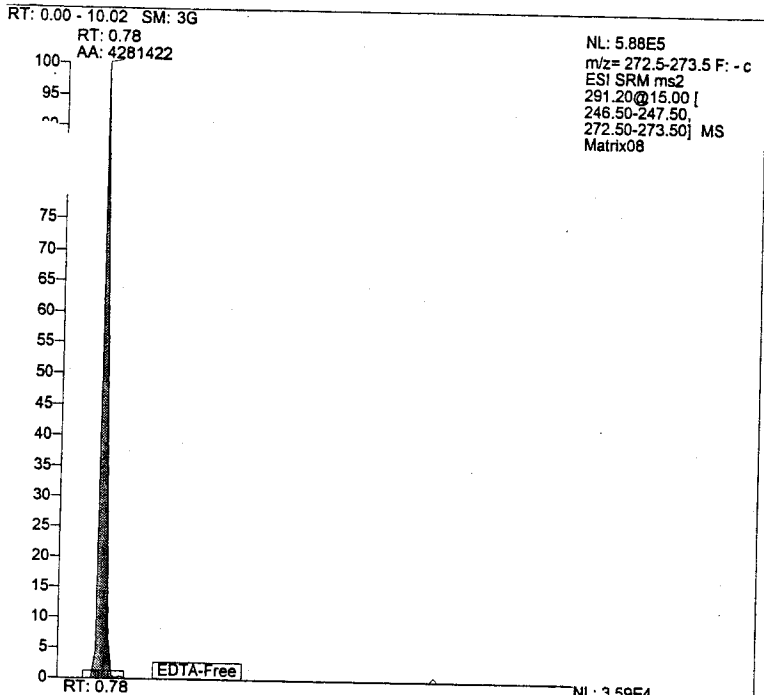


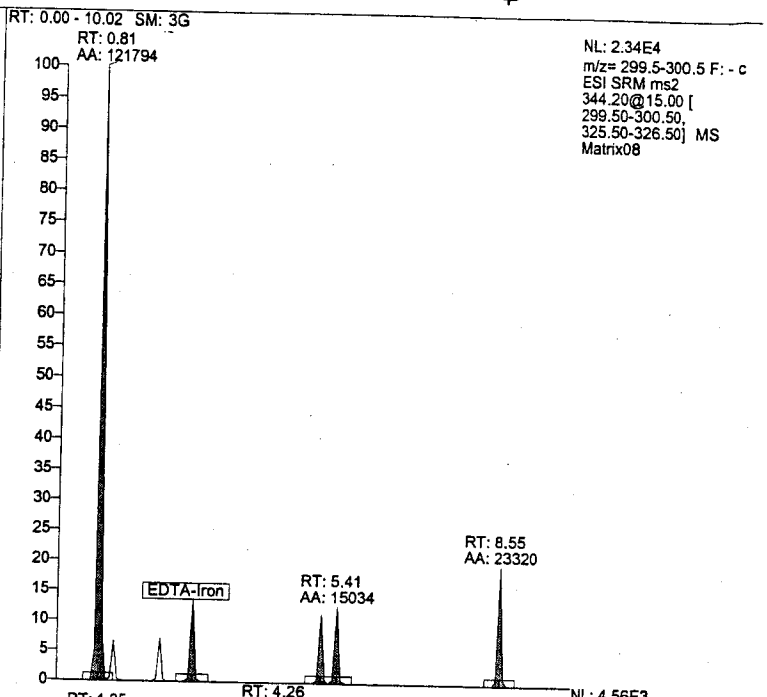


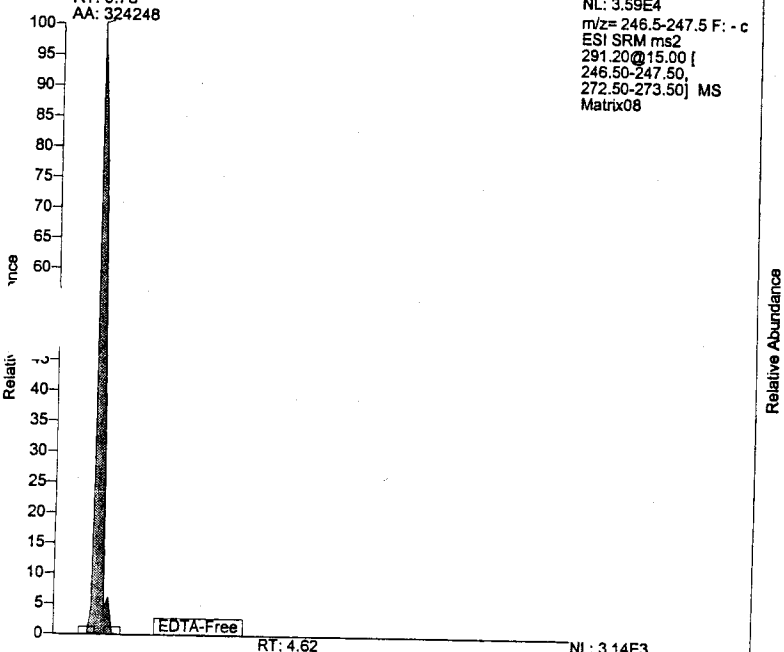


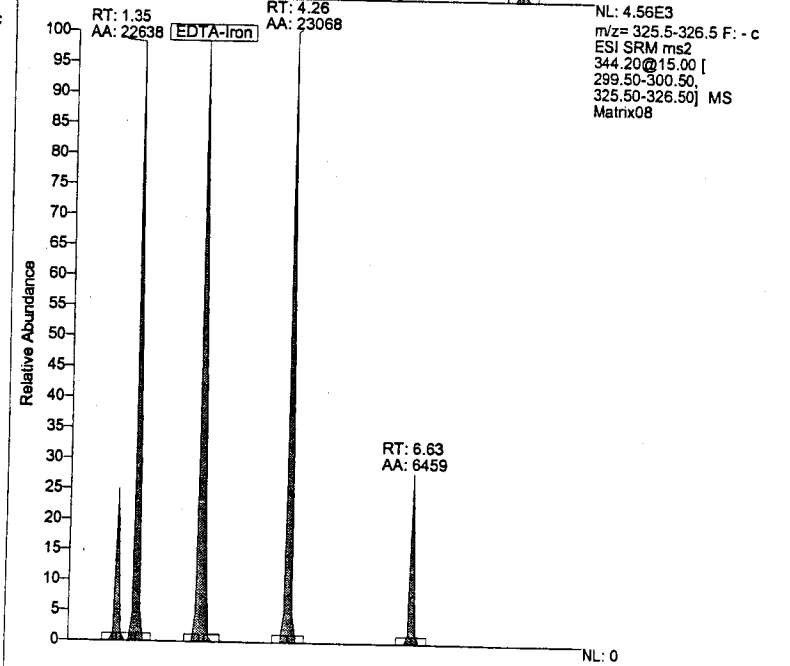
Ec446
(427)

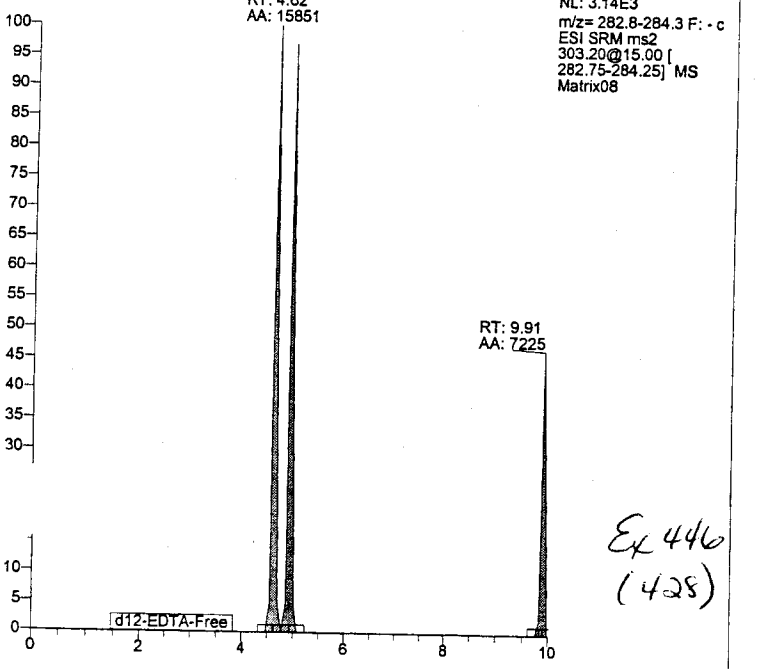
C:\Xcalibur\...\MAT_Negative\Matrix08 02/13/07 10:21:43 50 ppm EDTA JDB

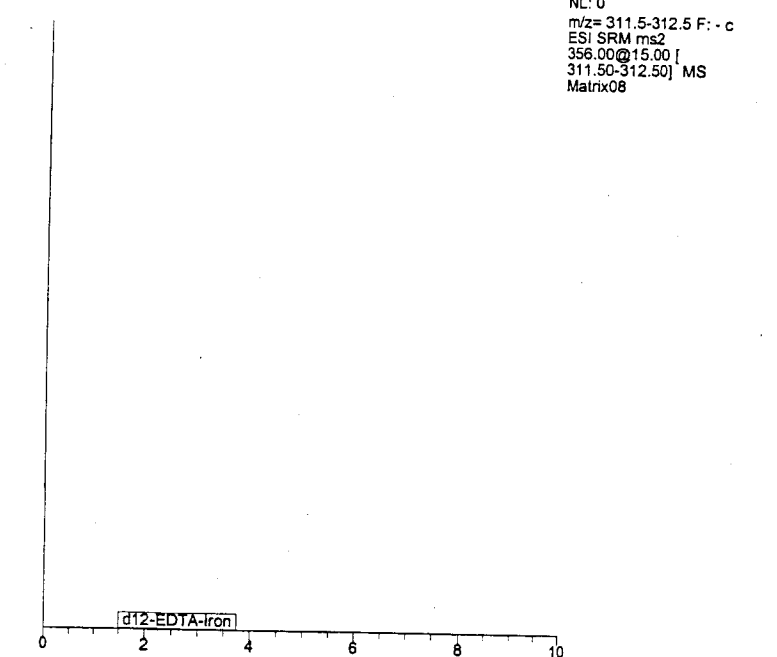






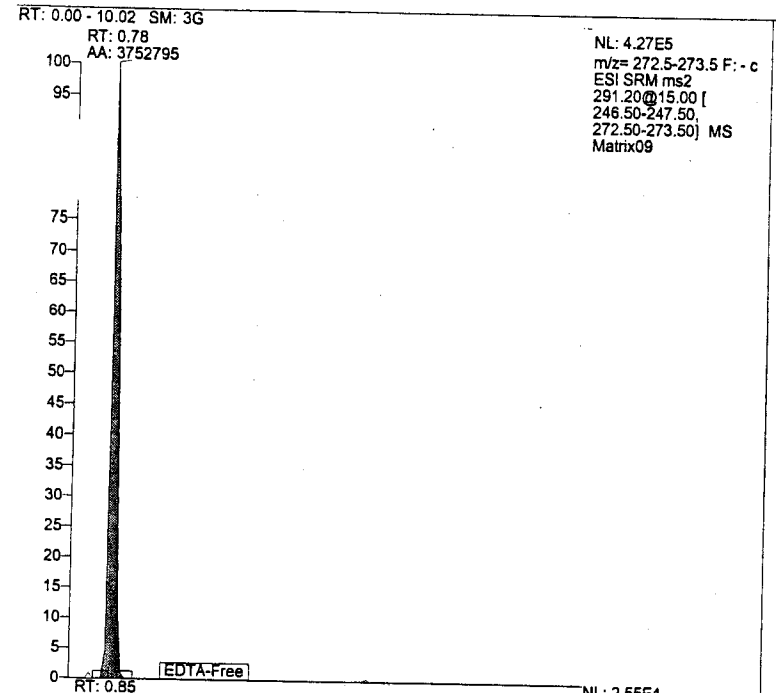


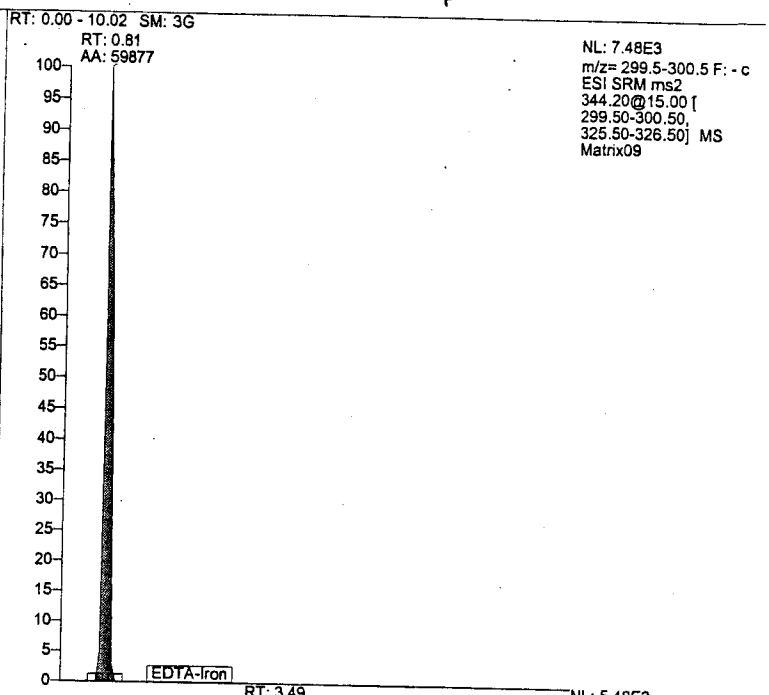


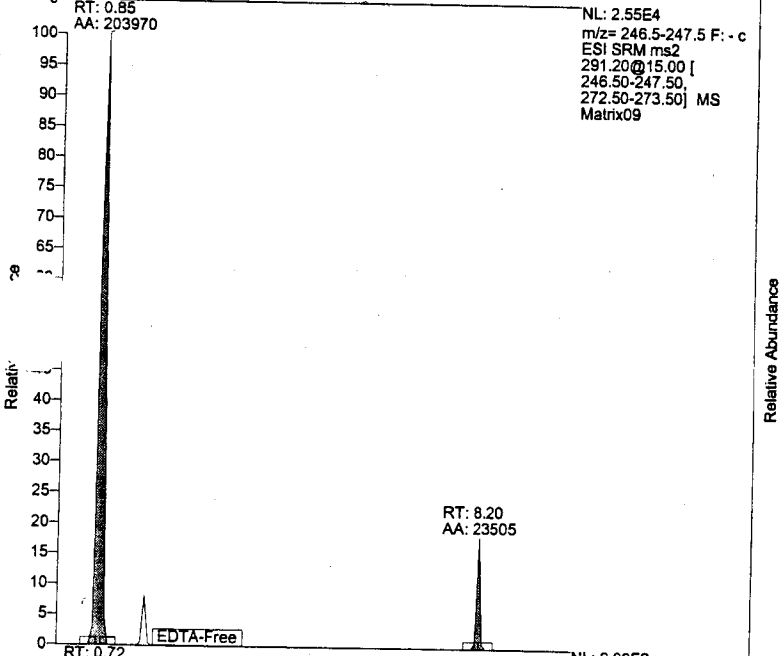


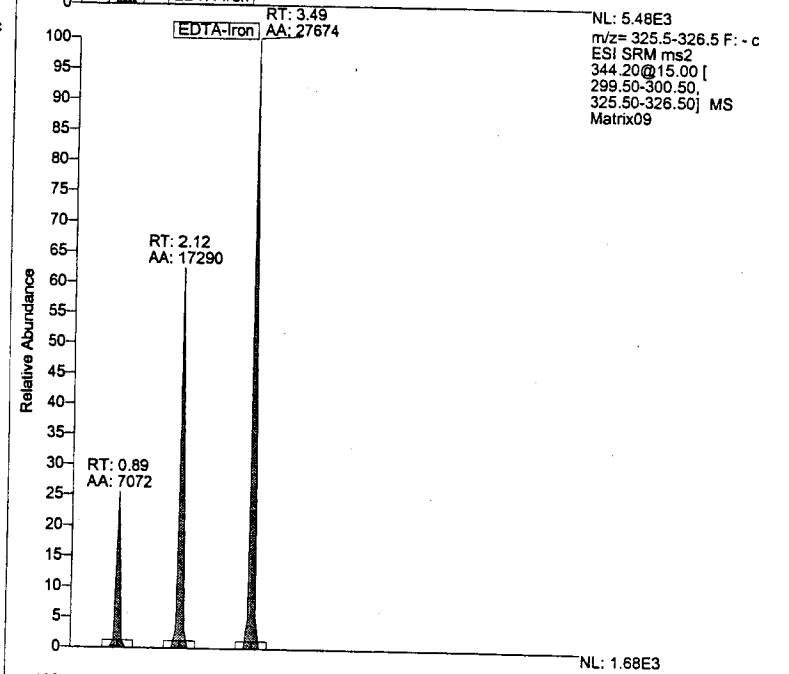
Ex 446
(428)

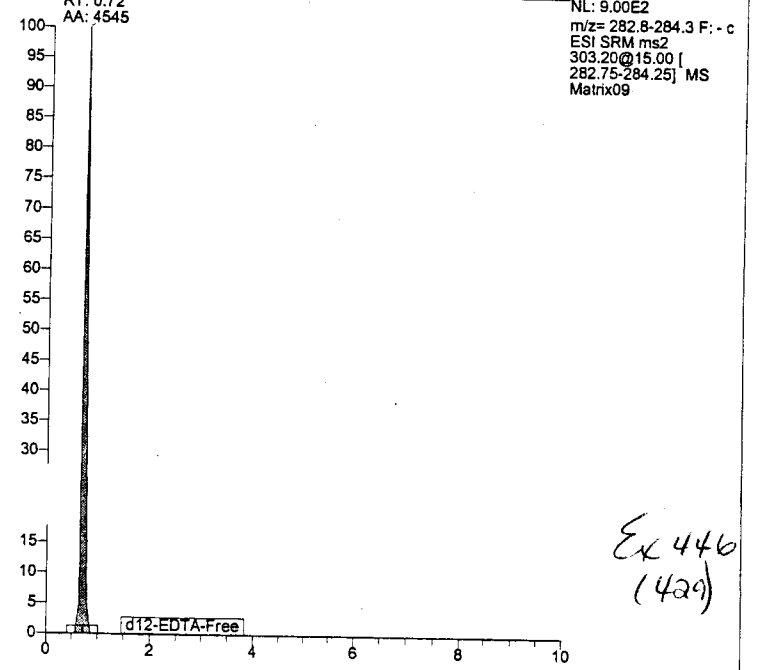
C:\Xcalibur\...\MAT_Negative\Matrix09 02/13/07 10:32:34 50 ppm EDTA JDB

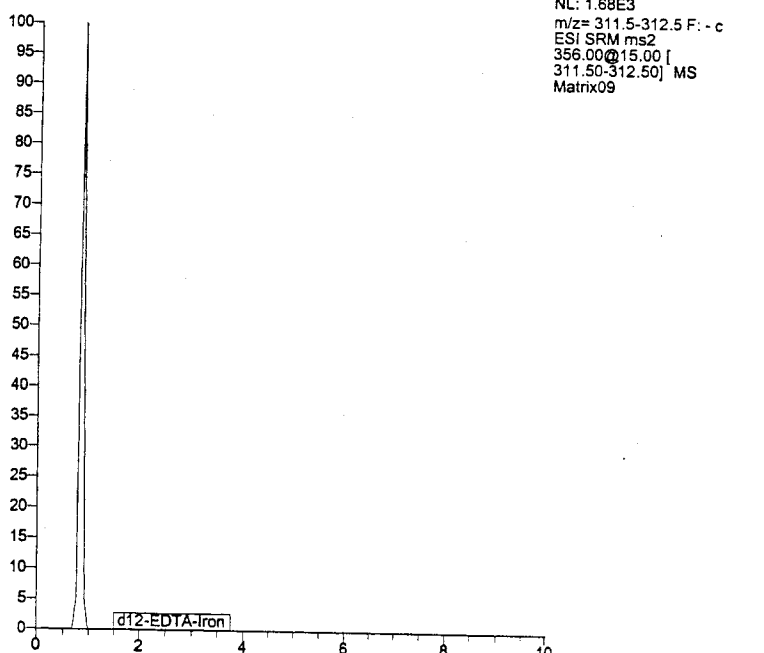






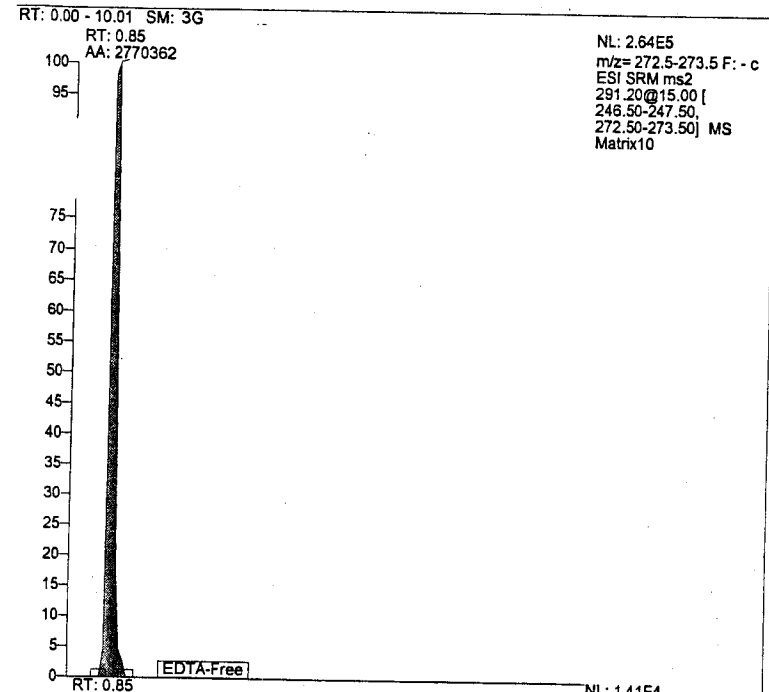


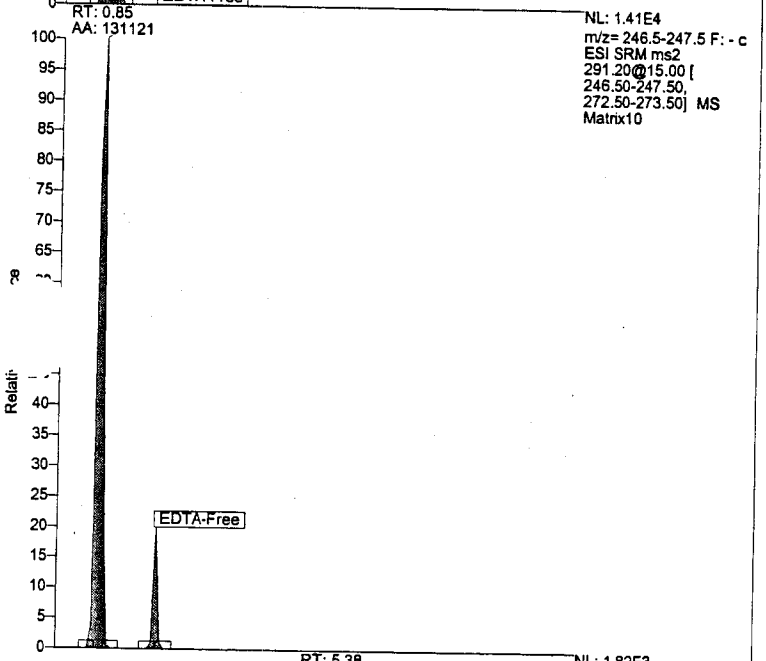


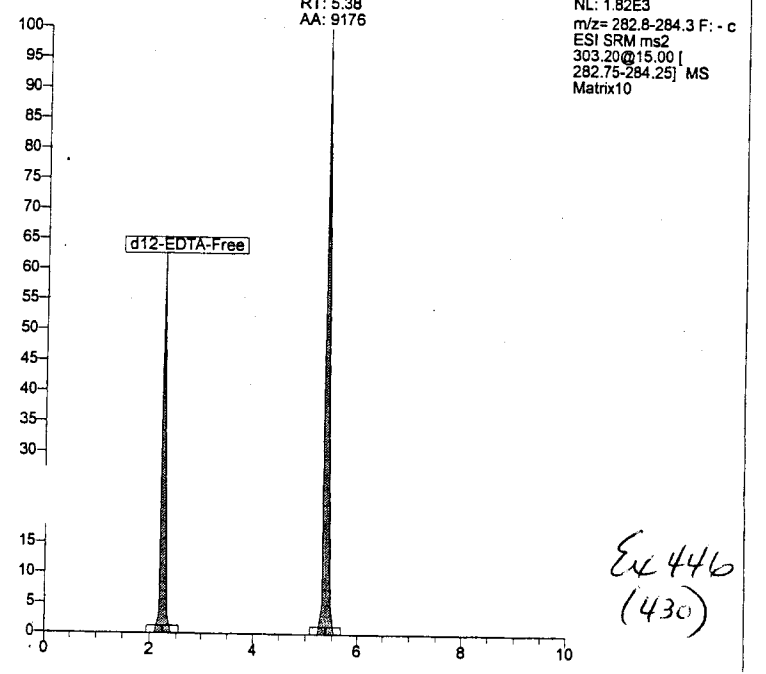


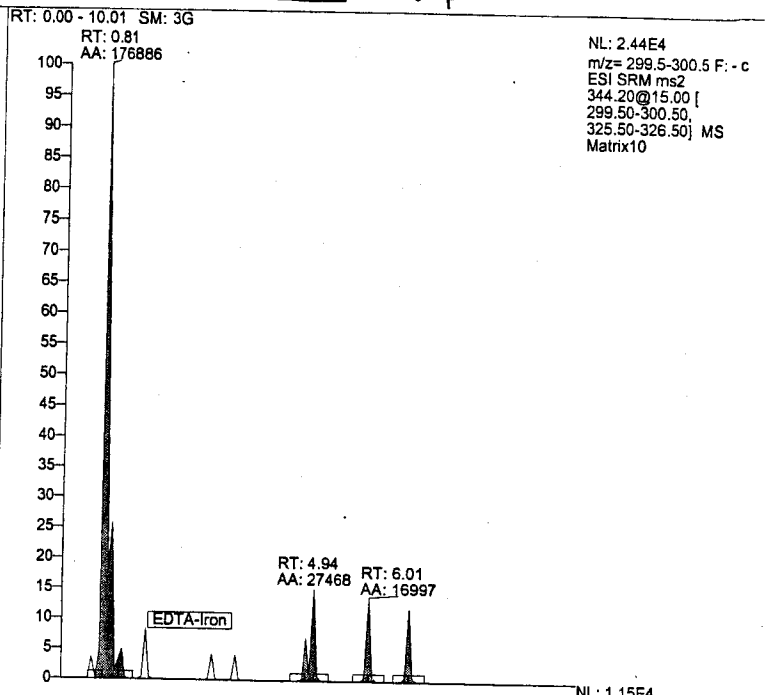
Ex 446
(429)

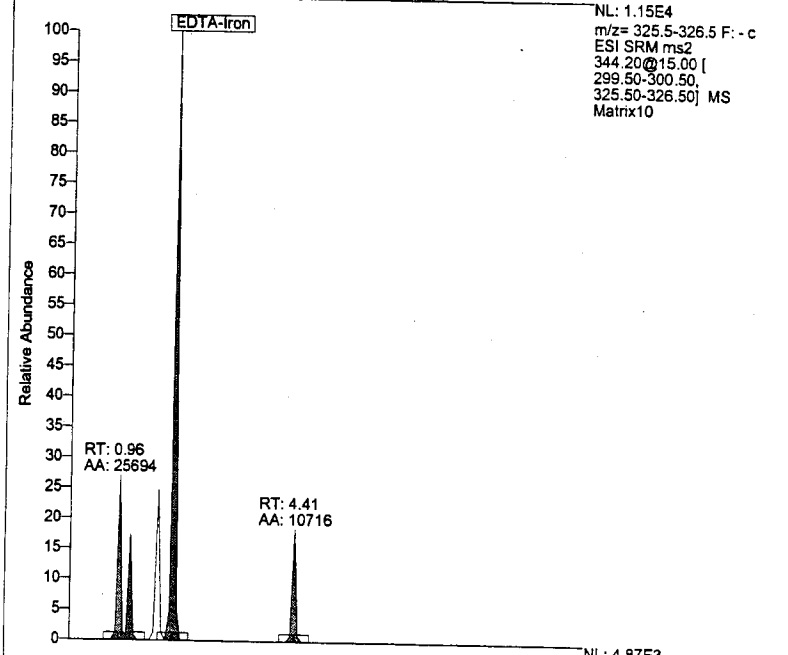
C:\Xcalibur\...\MAT_Negative\Matrix10 02/13/07 10:43:28 50 ppm EDTA JDR

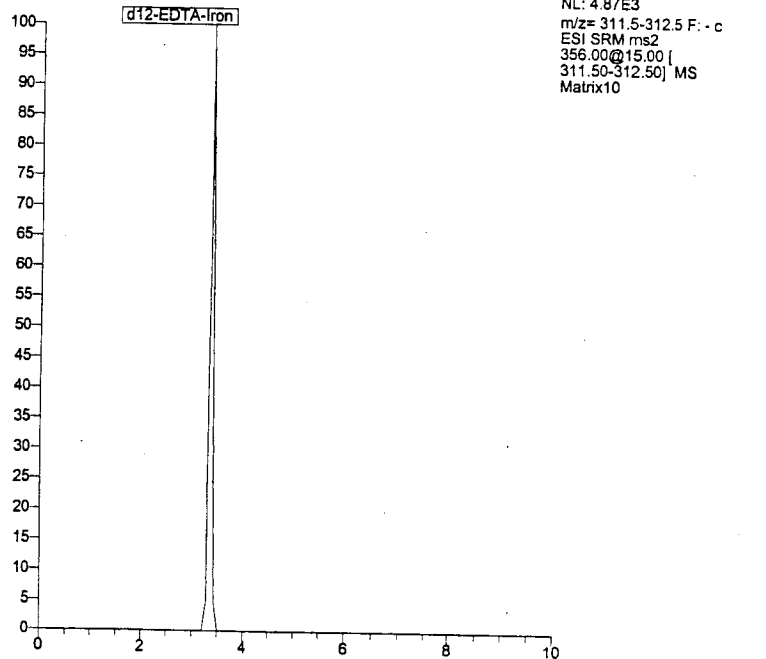






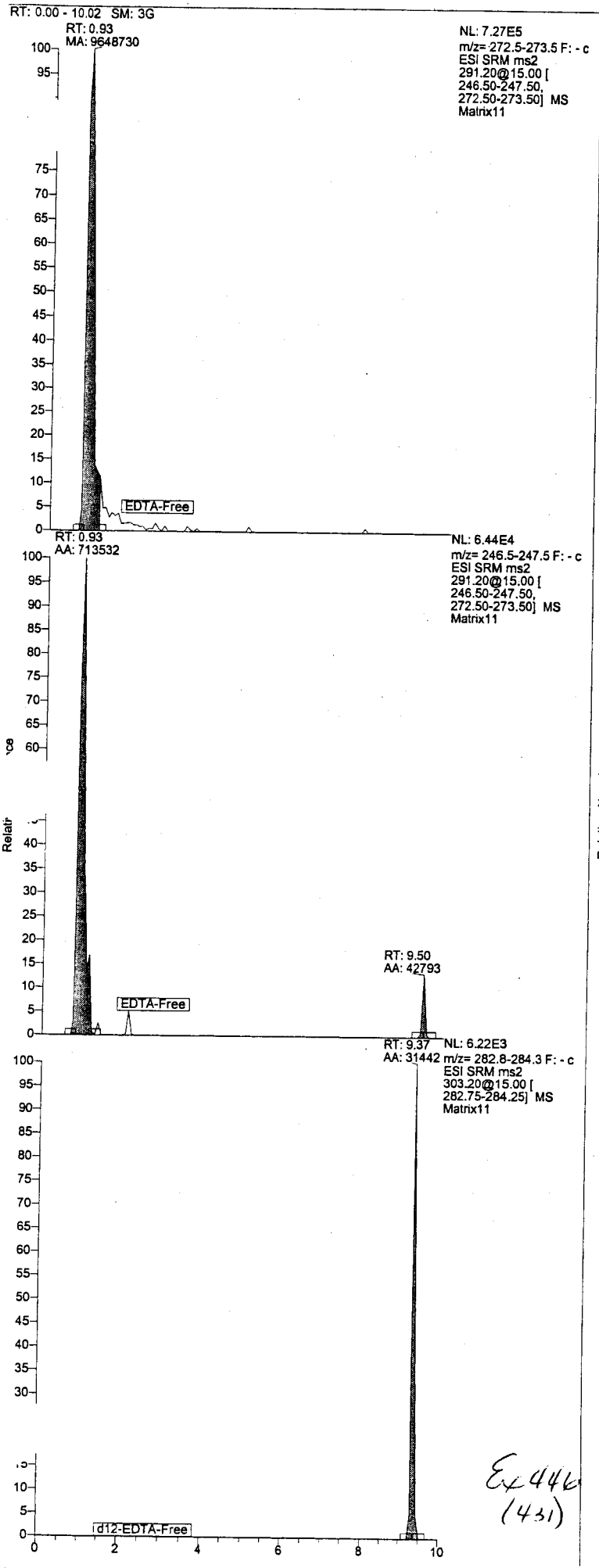


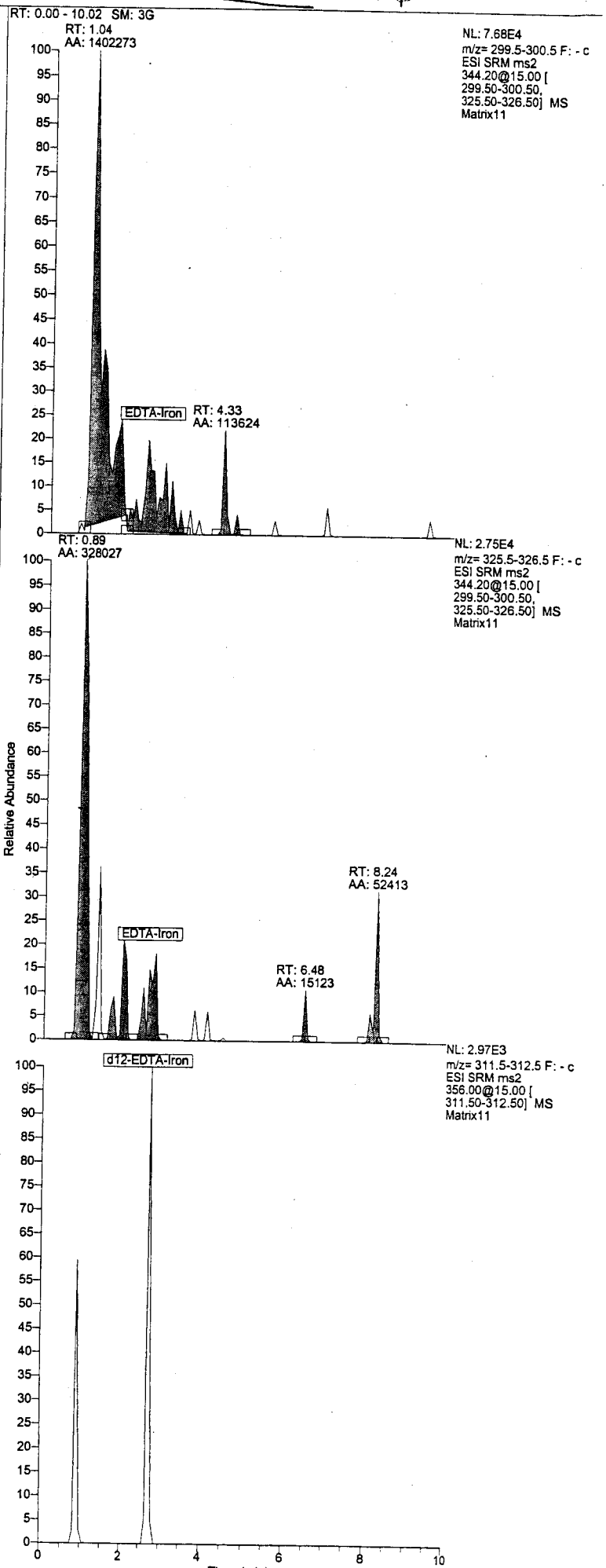




Ex 446
(430)

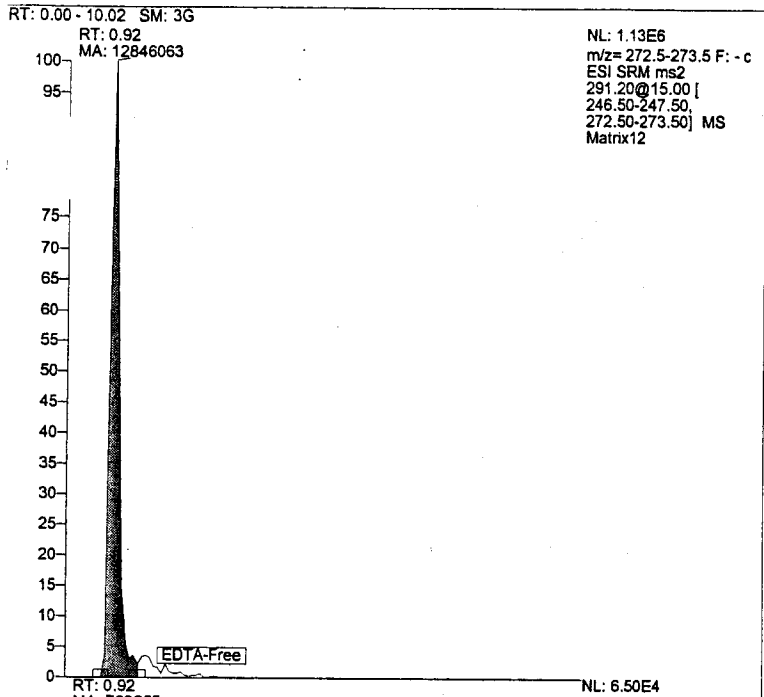
C:\Xcalibur\...\MAT_Negative\Matrix11 02/13/07 10:54:17 Matrix 1 ("500 ppm") JDB

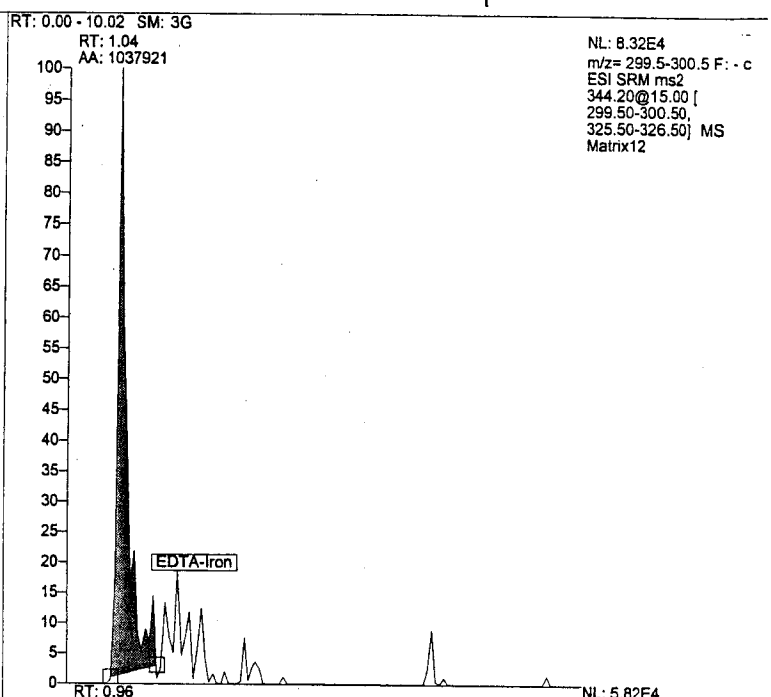


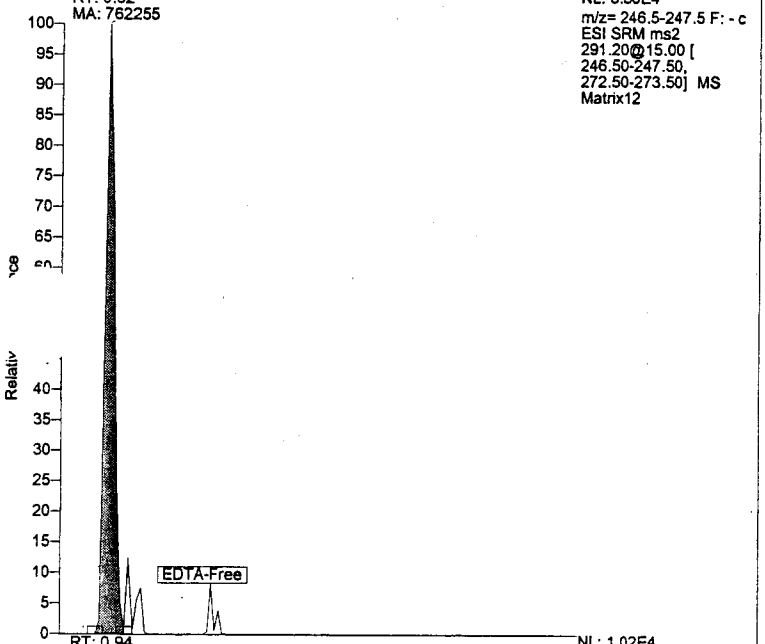


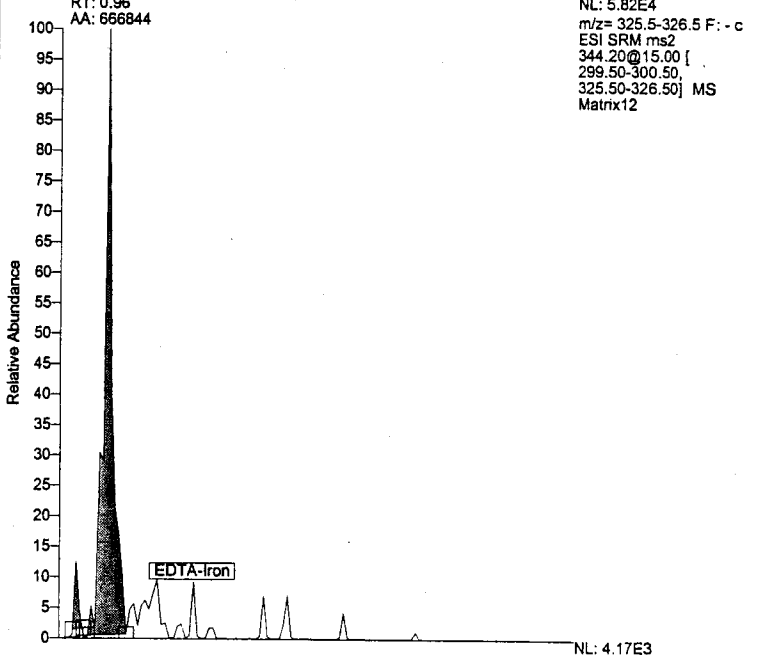
Ex 446
(431)

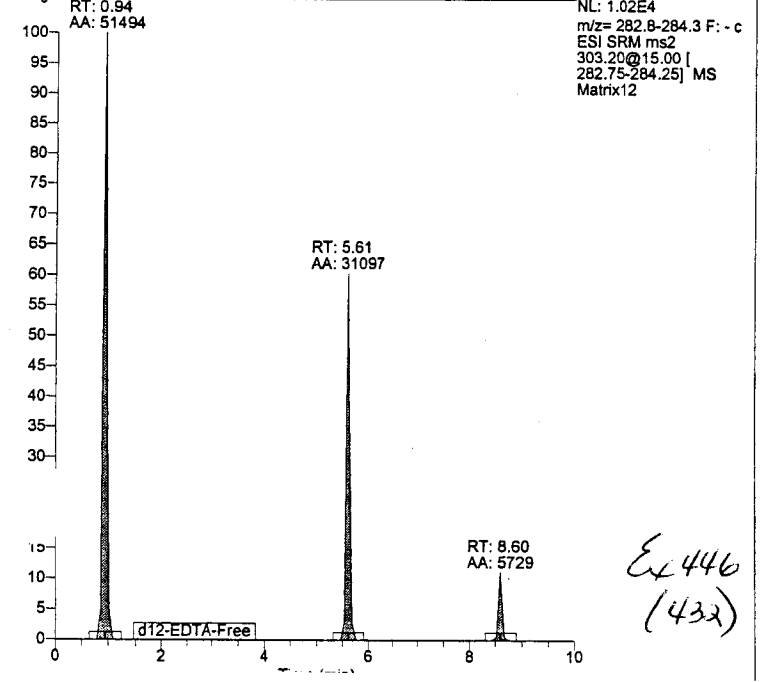
C:\Xcalibur\...\MAT_Negative\Matrix12 02/13/07 11:05:13 Matrix 2 ("500 ppm") JMP

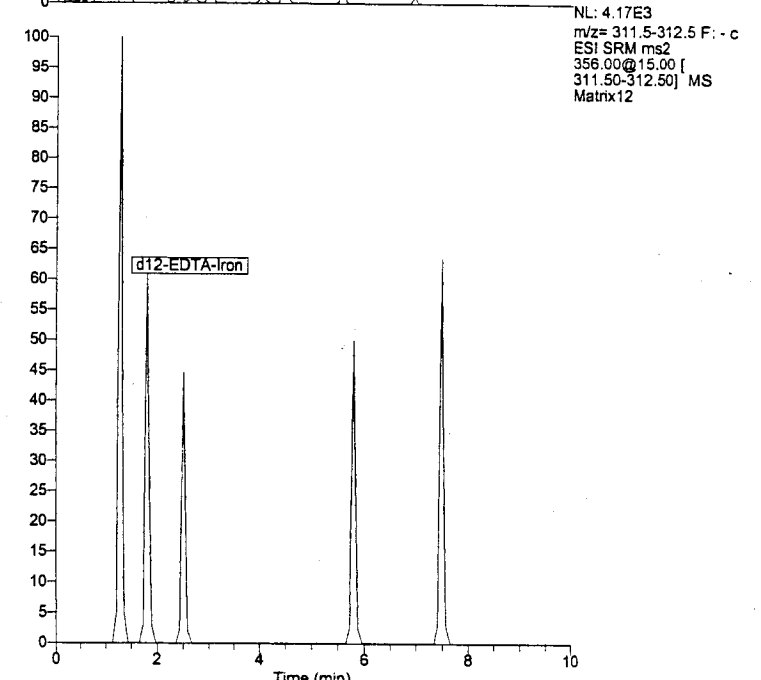






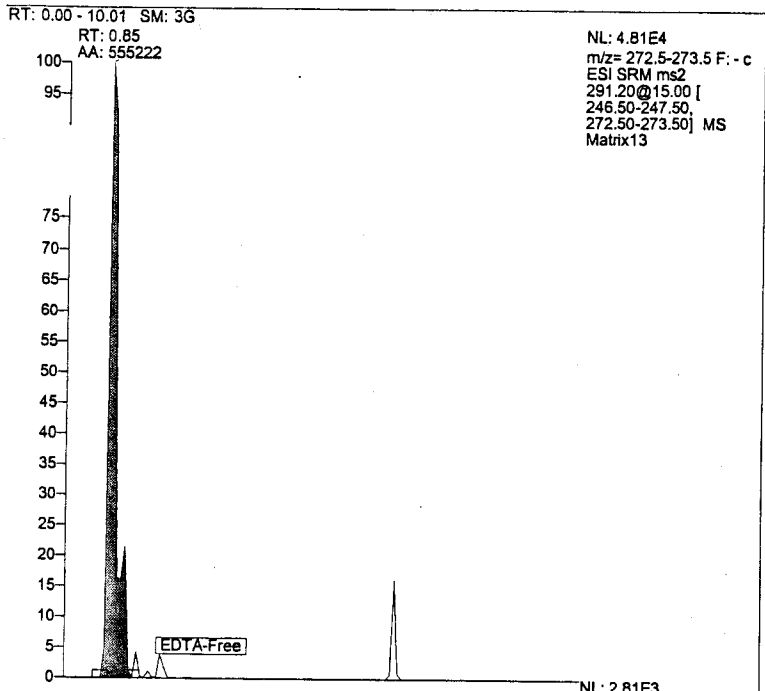


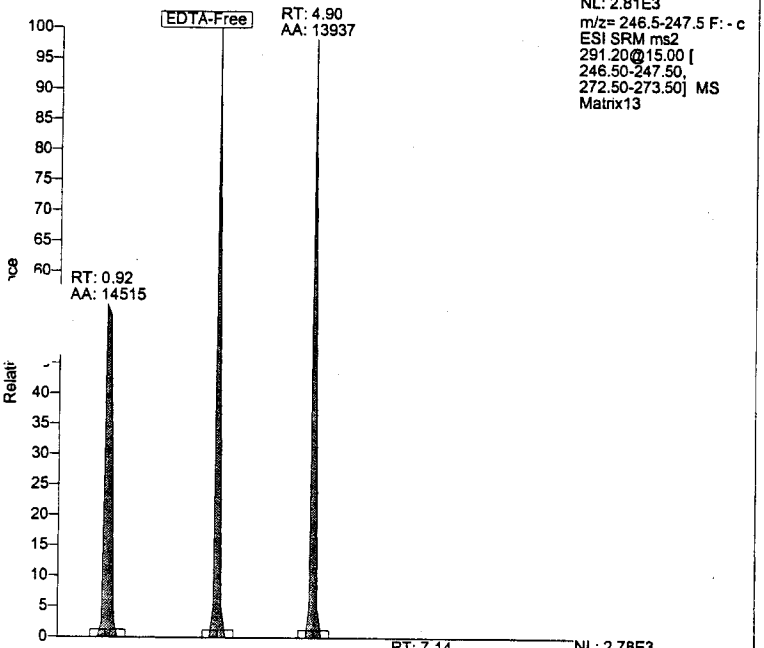


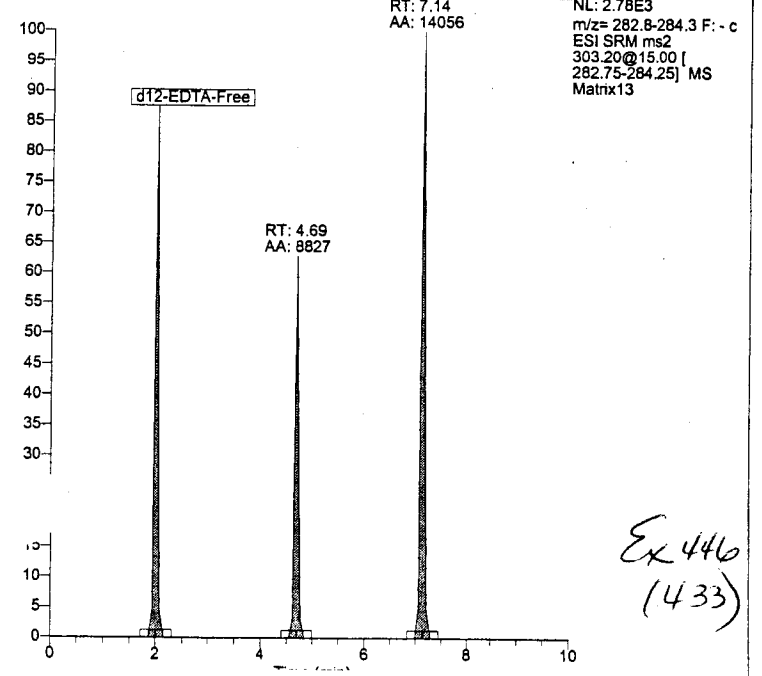


Ex 446
(432)

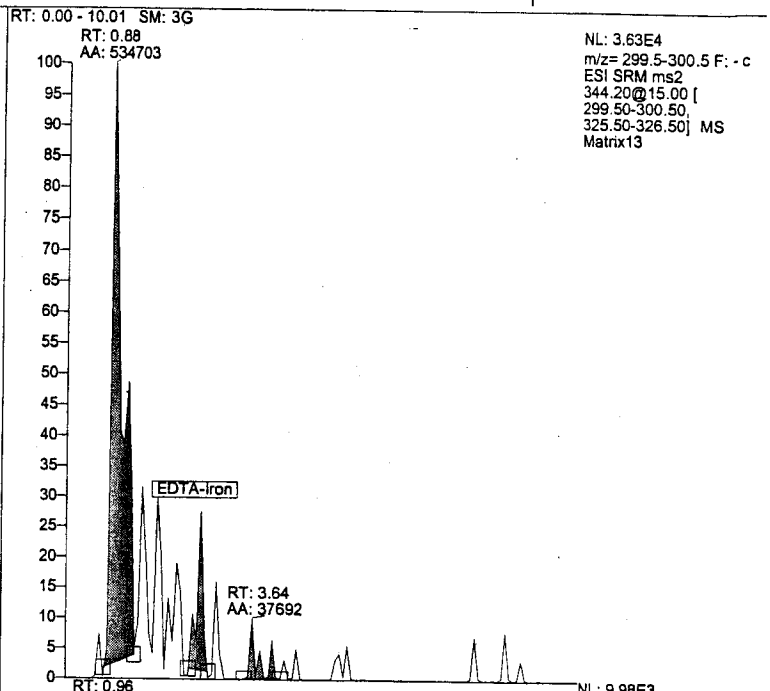
C:\Xcalibur\...\MAT_Negative\Matrix13 02/13/07 11:16:02 Matrix 3 ("500 ppm") DB

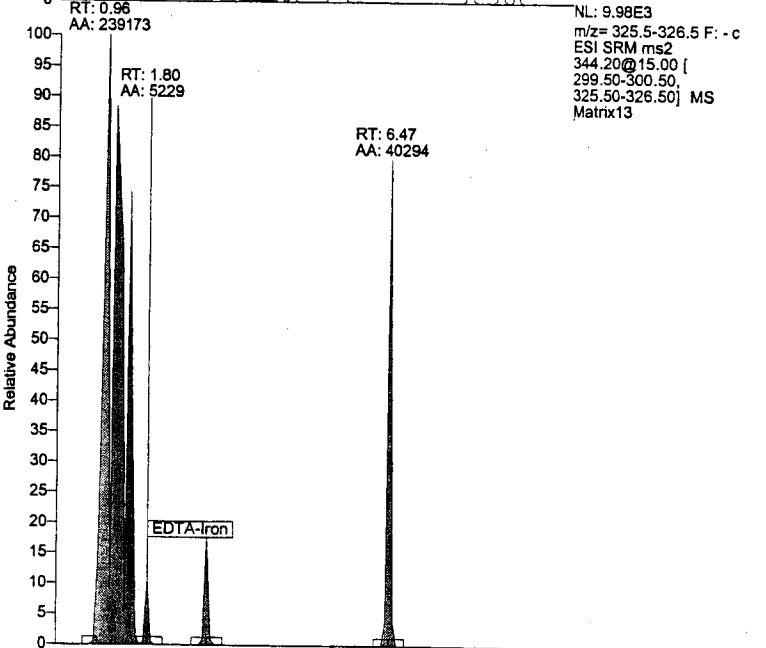


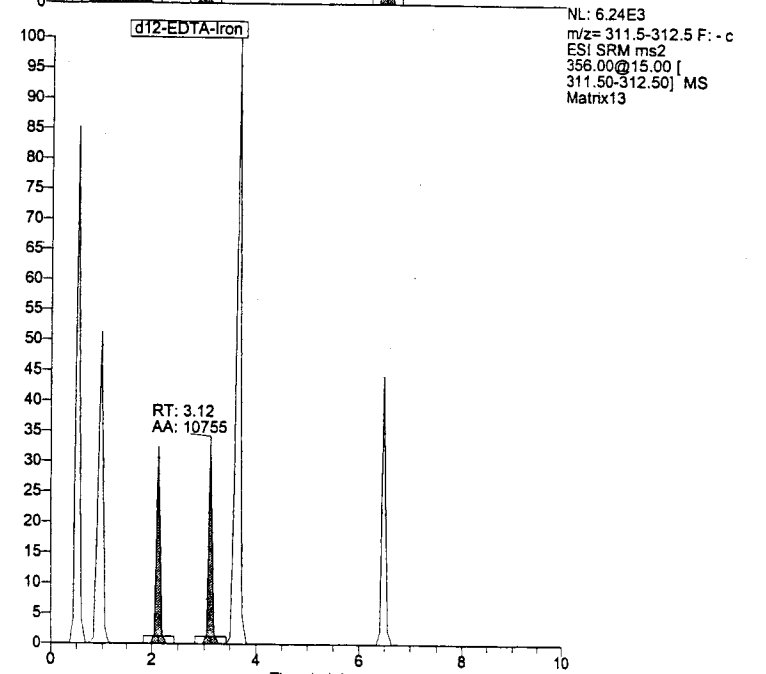




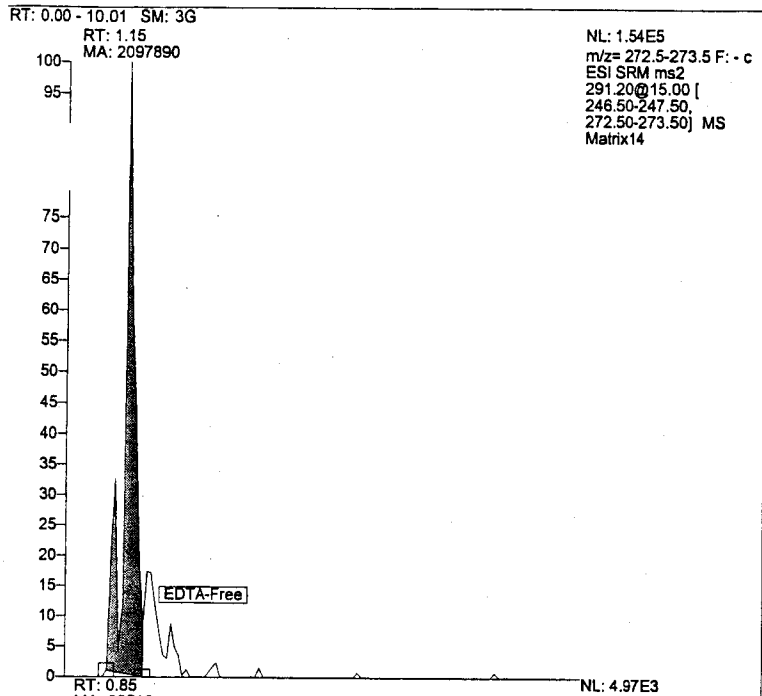
Ex 446
(433)

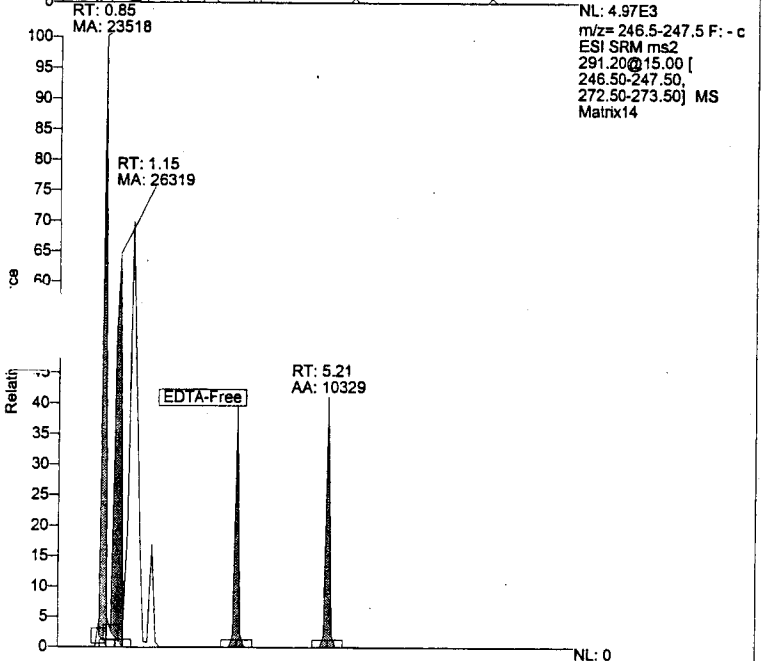


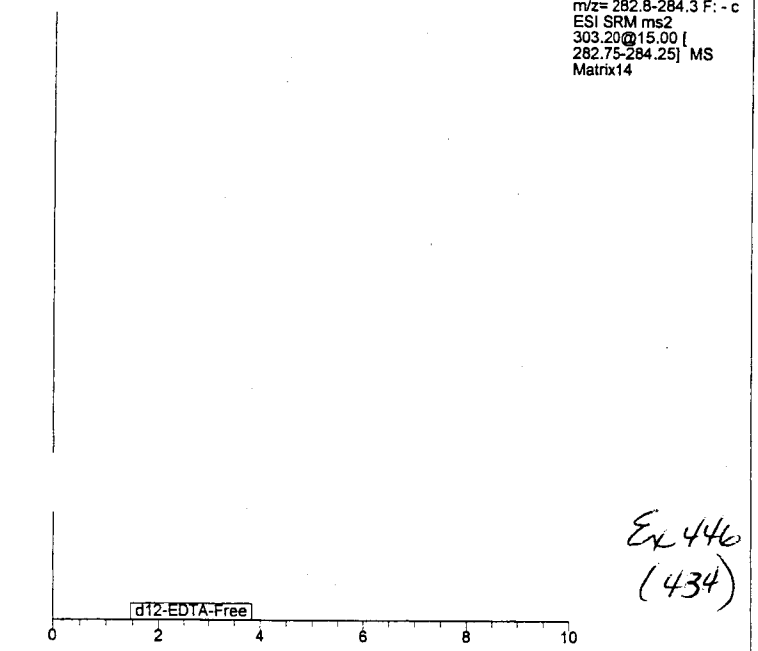




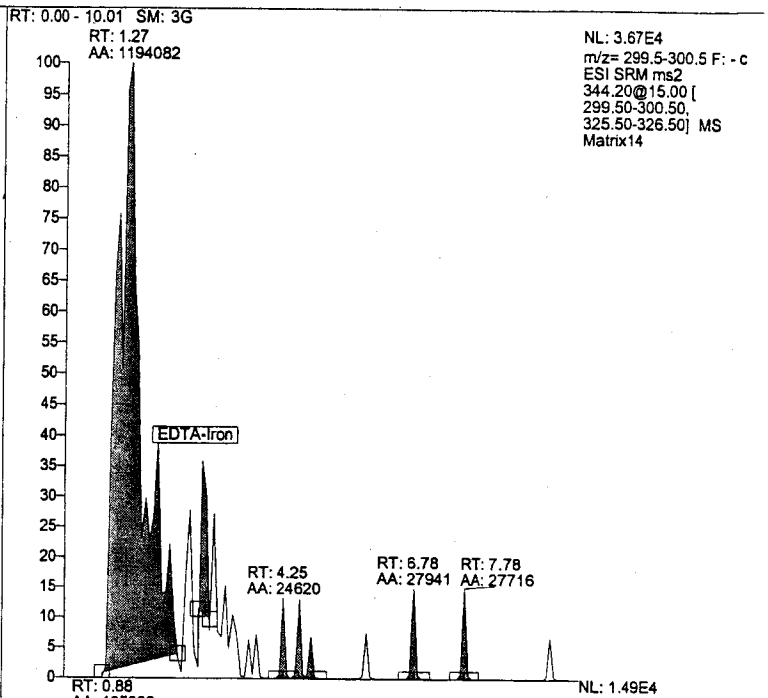
C:\Xcalibur\...\MAT_Negative\Matrix14 02/13/07 11:26:52 Matrix 4 ("500 ppm") JMB

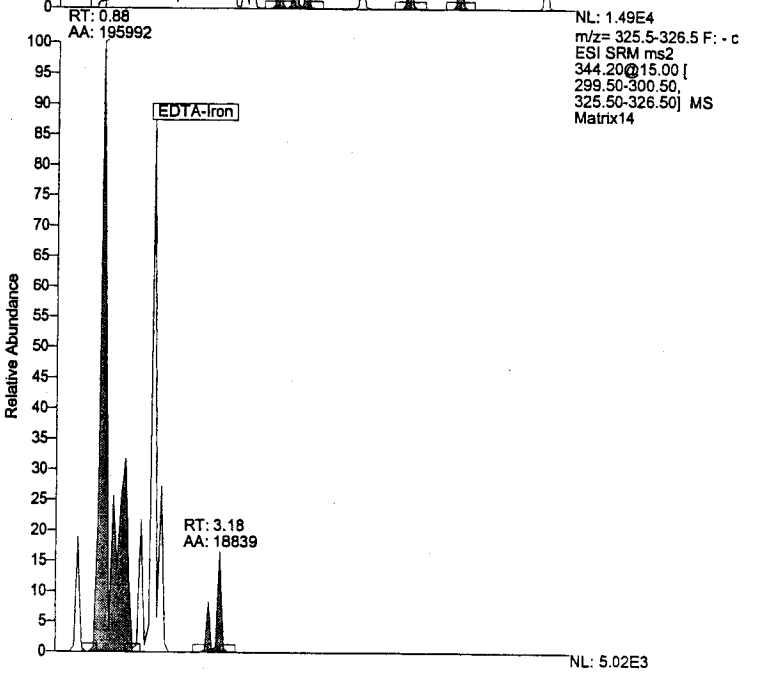


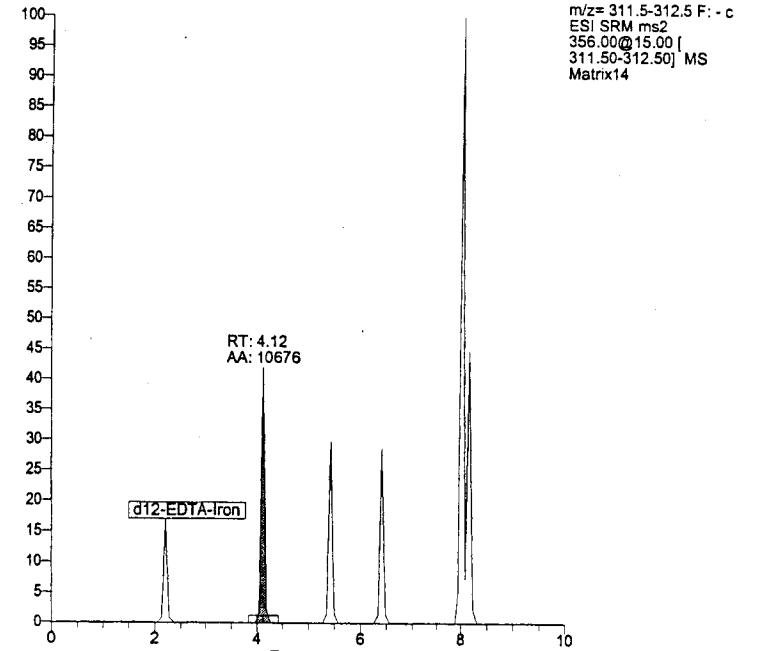




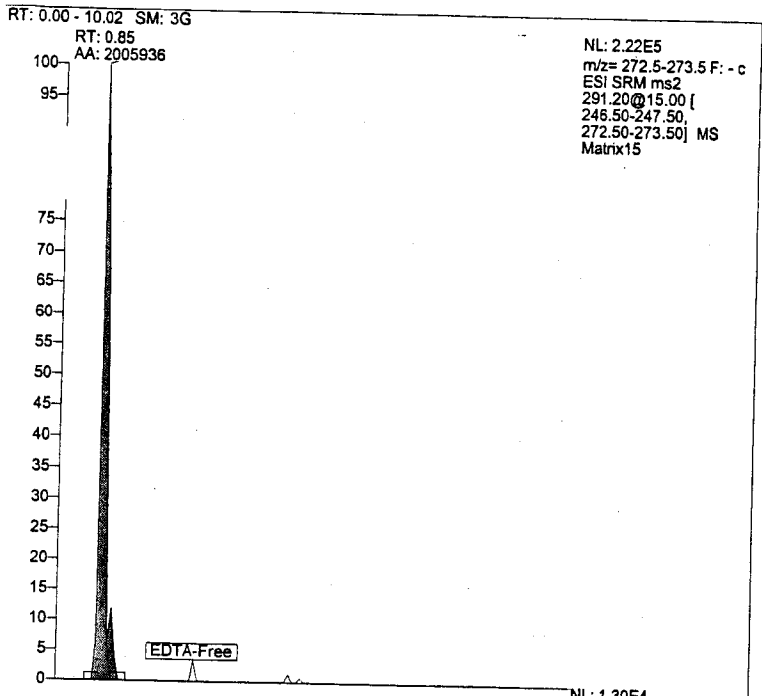
Ex 446
(434)

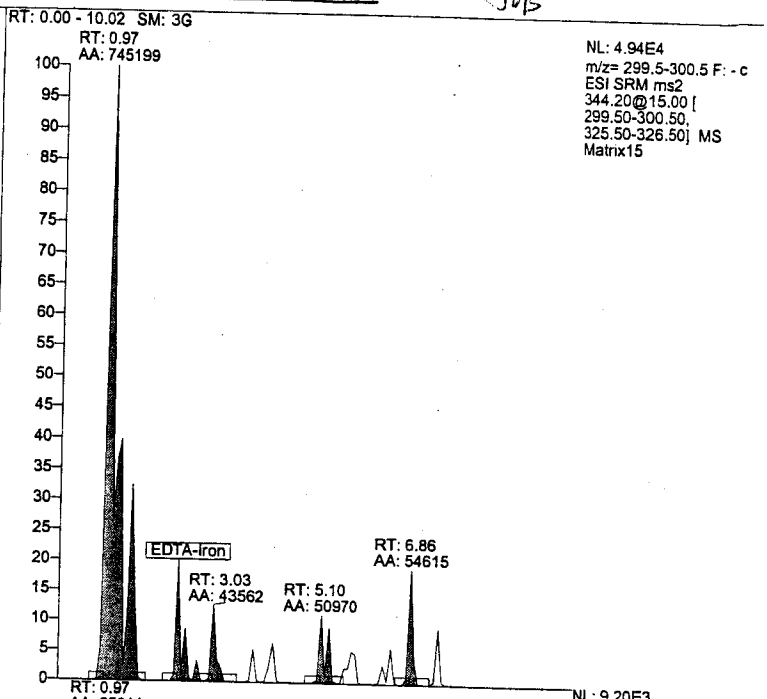


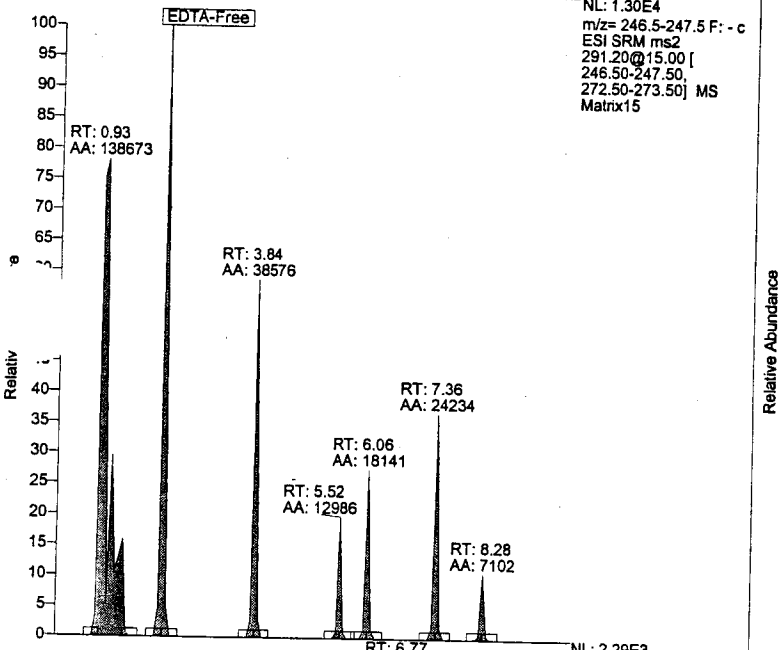


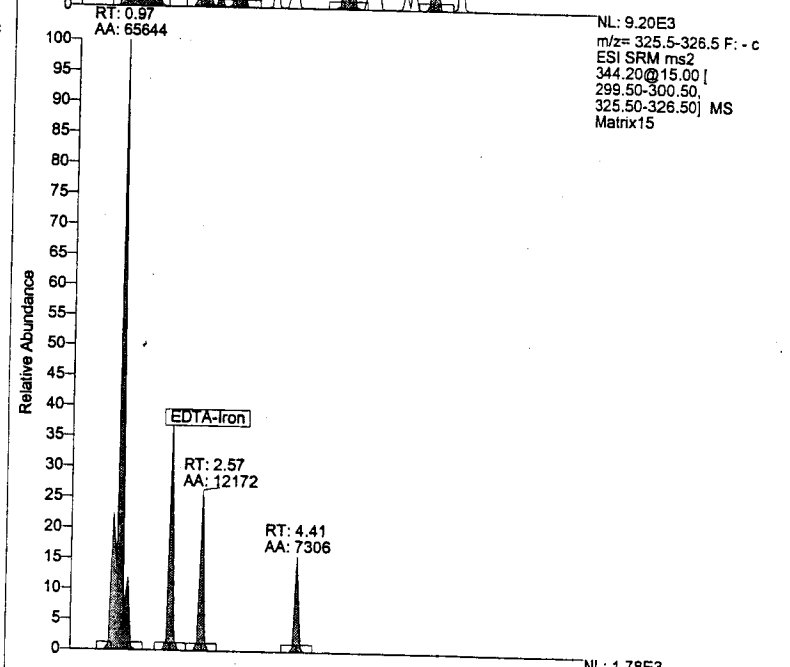


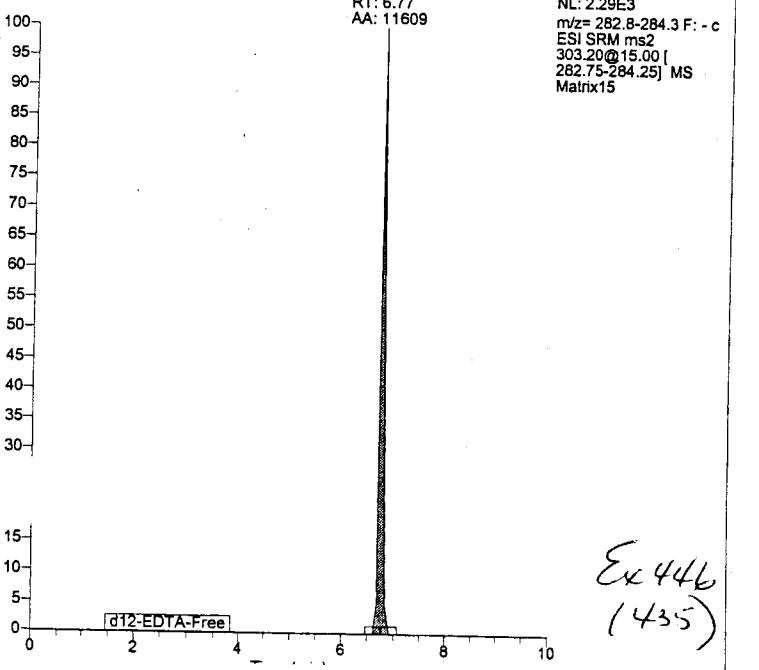
C:\Xcalibur\...\MAT_Negative\Matrix15 02/13/07 11:37:47 Matrix 5 ("500 ppm") JDB

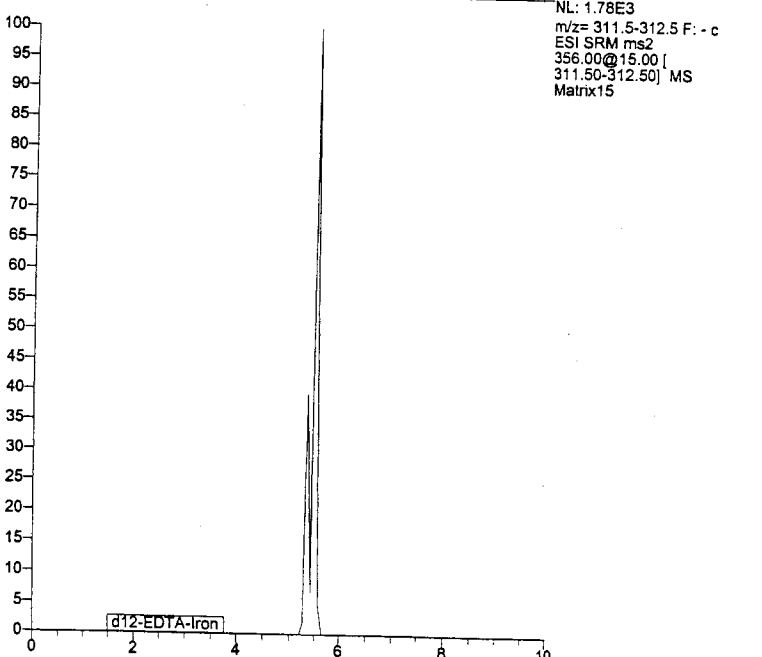






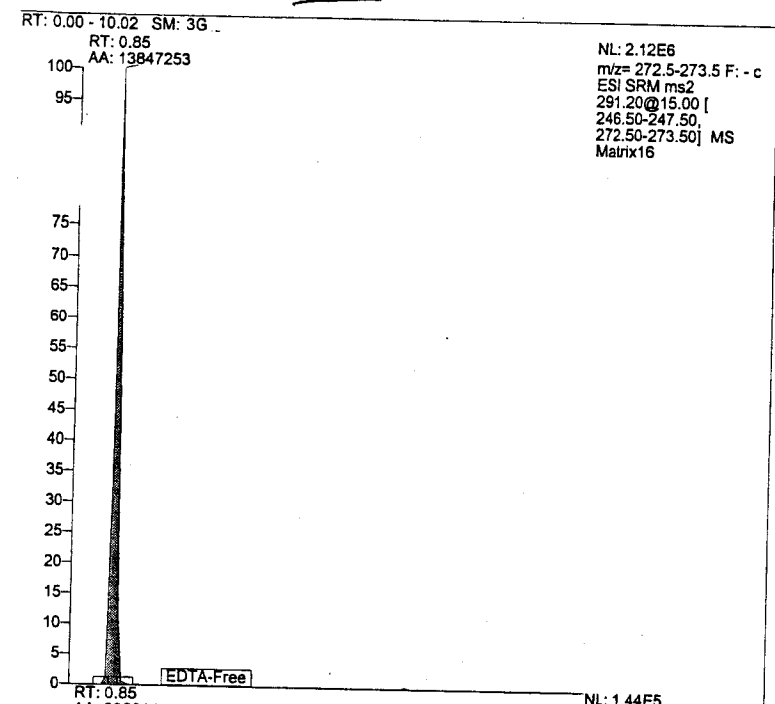


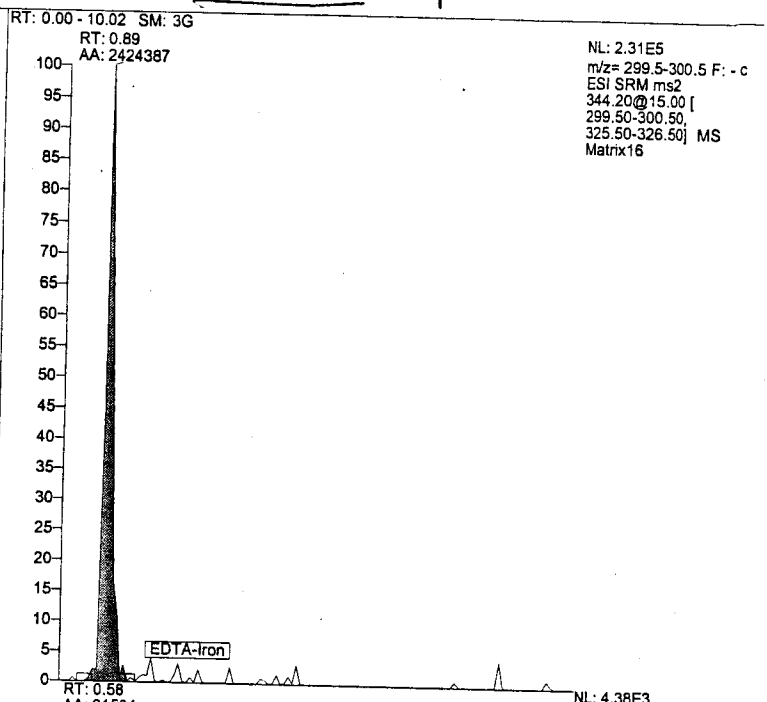


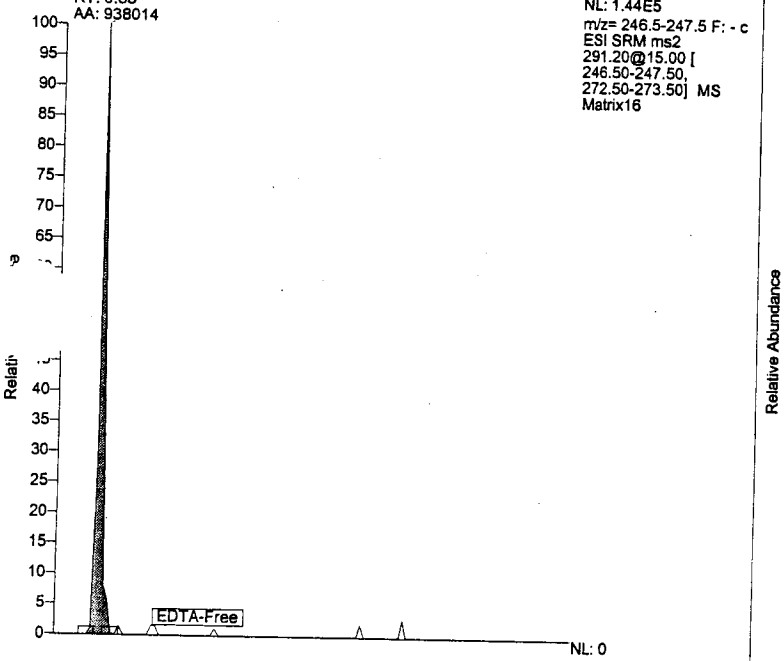


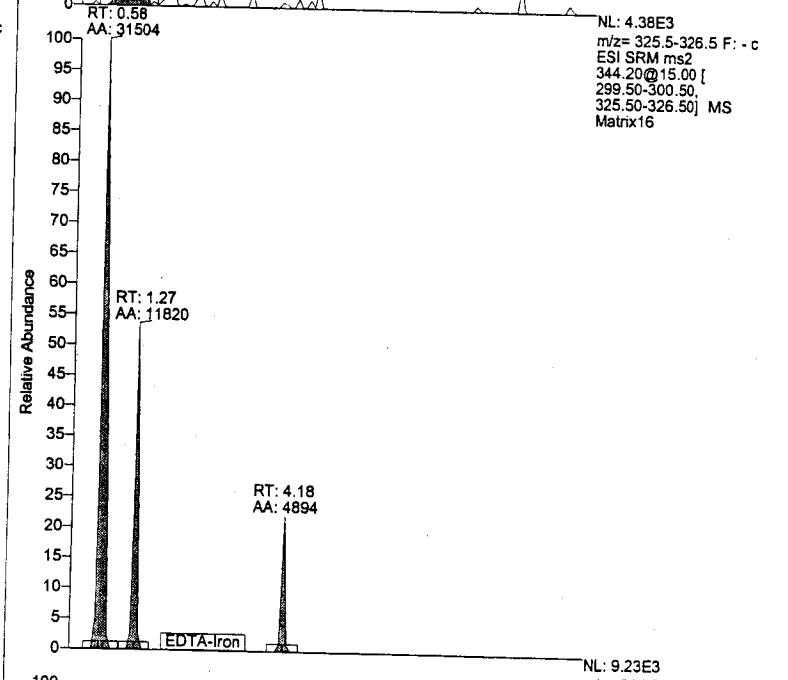
Ex 446 (435)

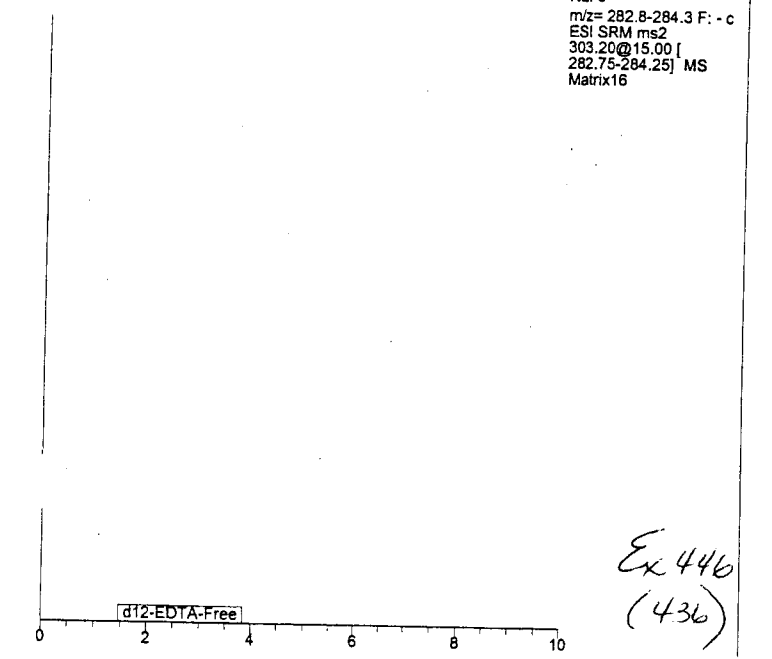
C:\Xcalibur\...\MAT_Negative\Matrix16 02/13/07 11:48:42 500 ppm EDTA

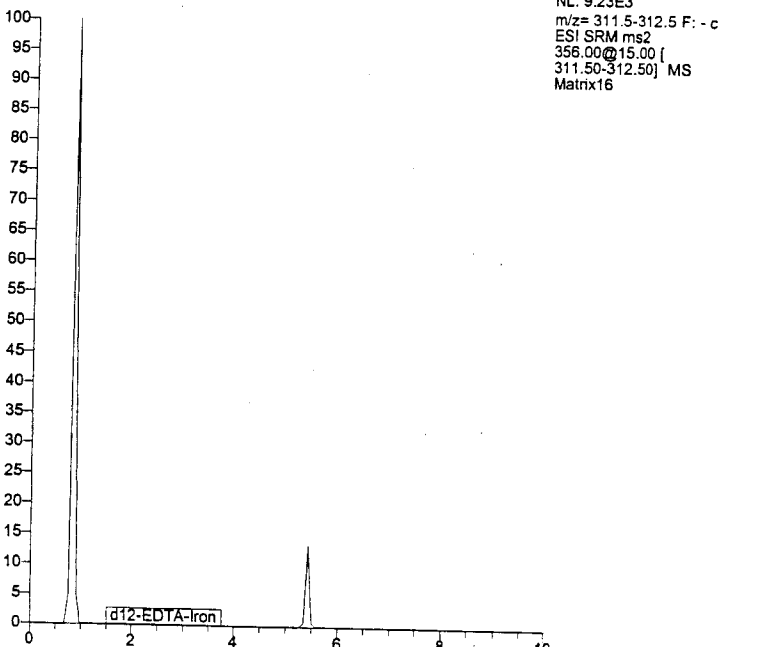












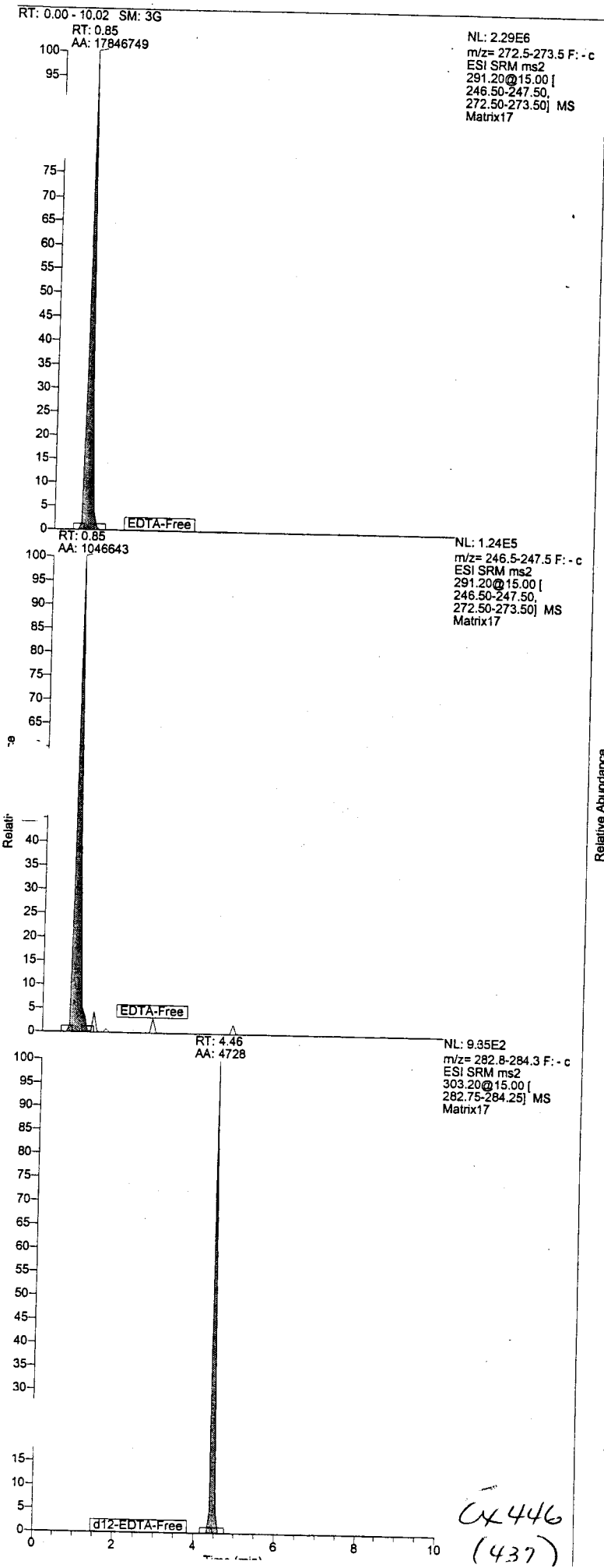
Ex 446
(436)

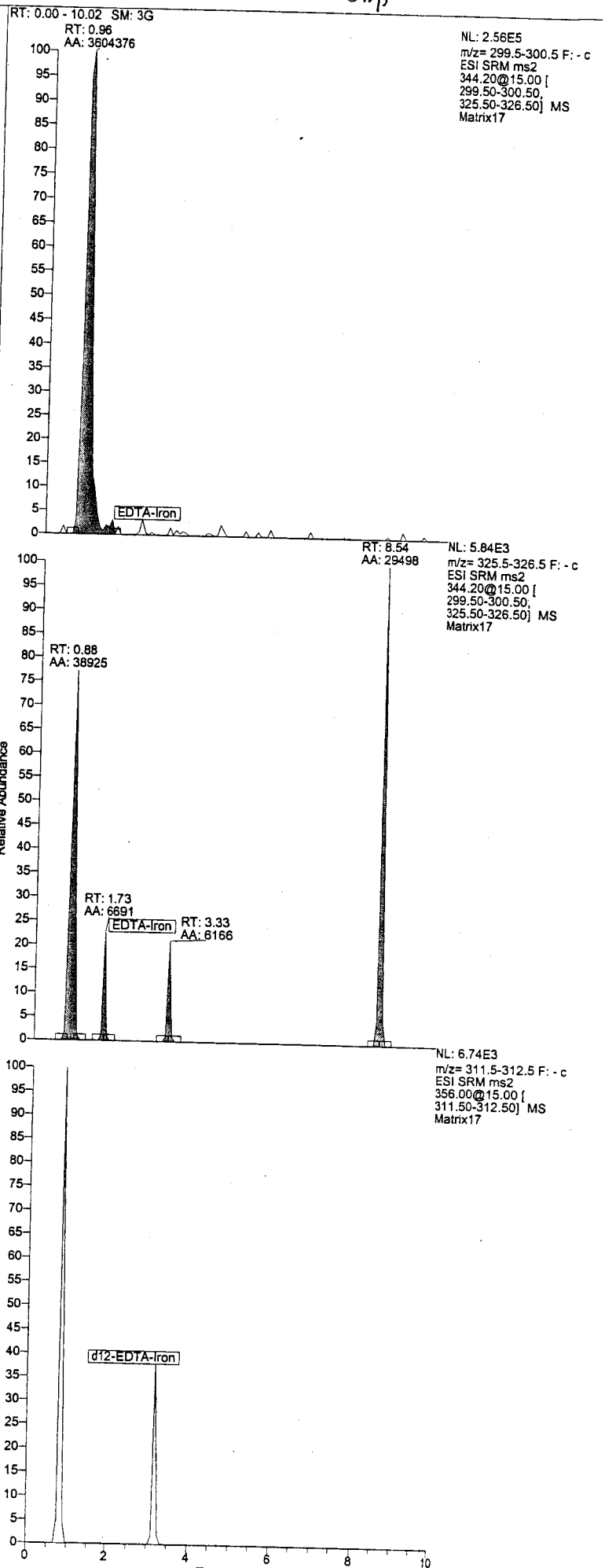
C:\Xcalibur\...\MAT_Negative\Matrix17

02/13/07 11:59:36

500 ppm EDTA

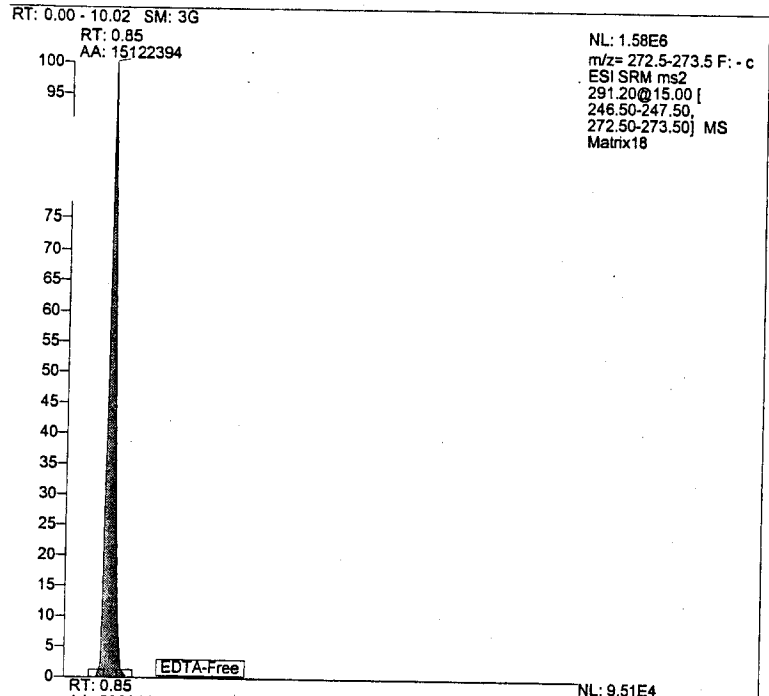
MB

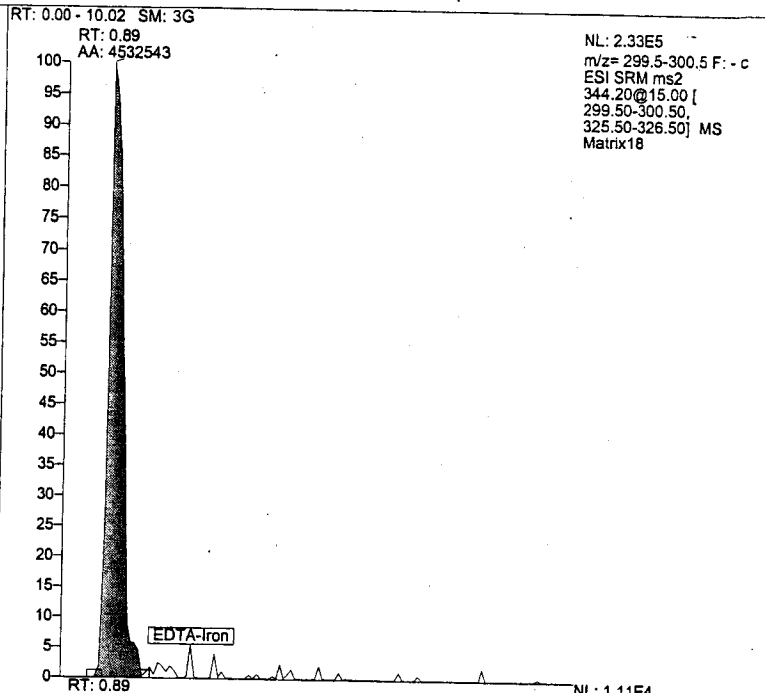


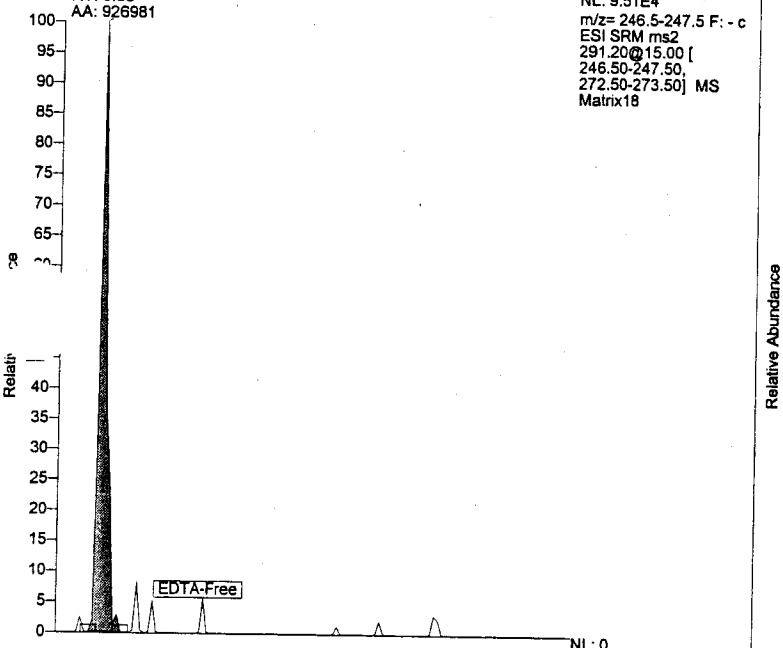


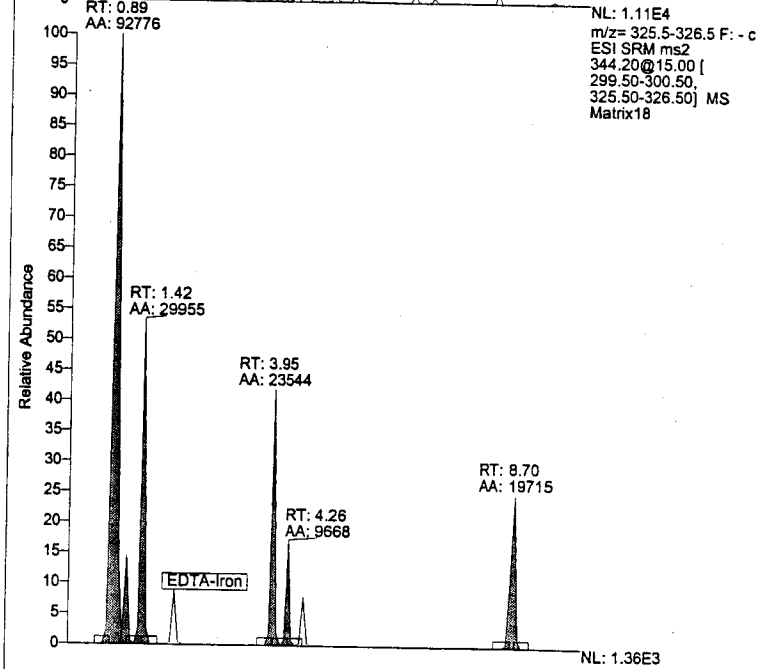
CX446
(437)

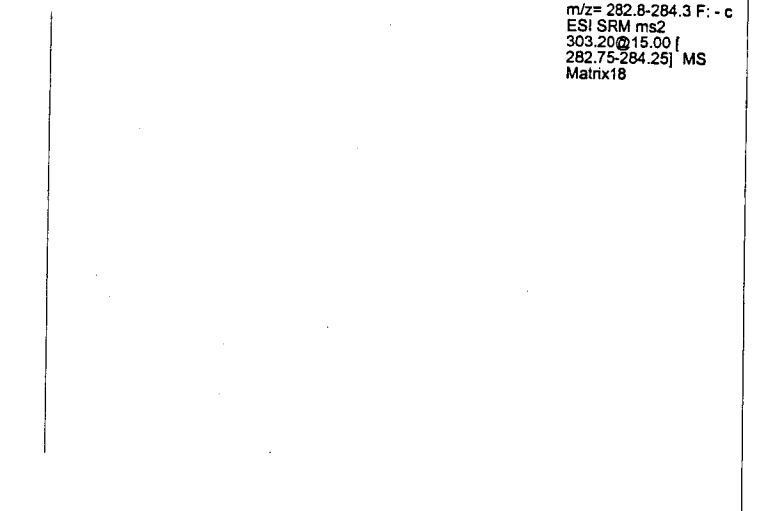
C:\Xcalibur\...\MAT_Negative\Matrix18 02/13/07 12:10:25 500 ppm EDTA JDB

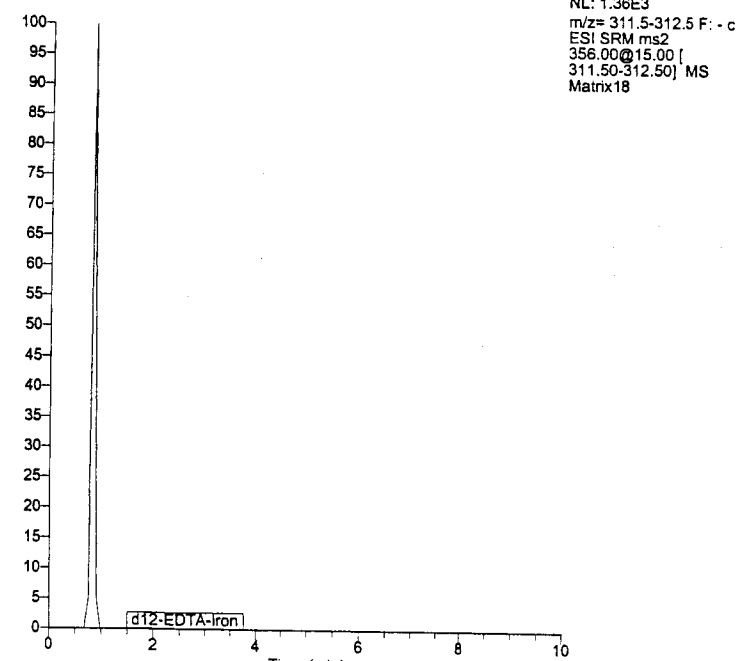






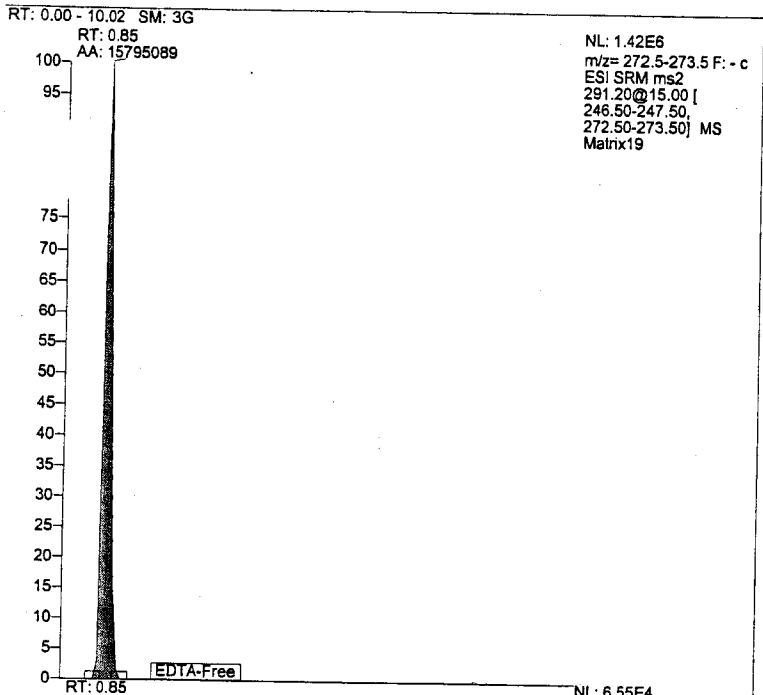


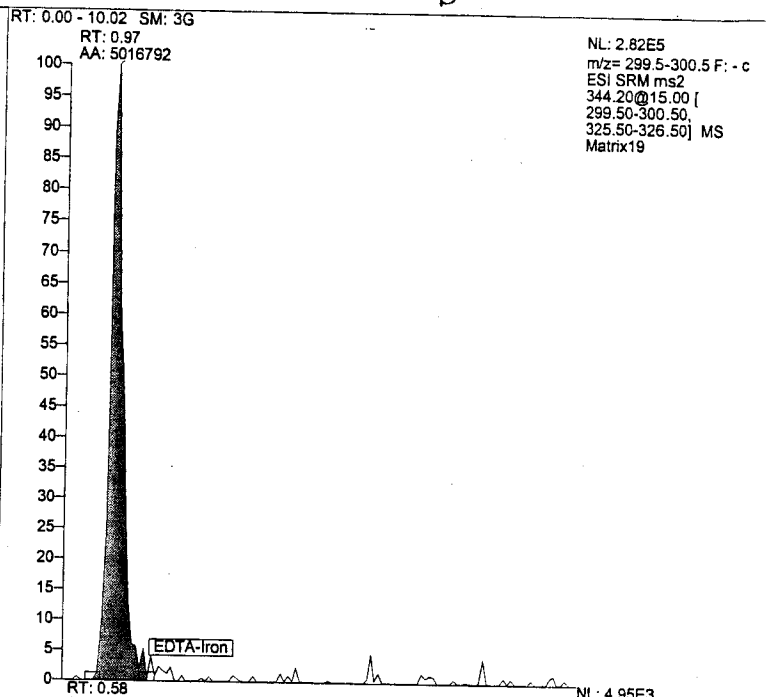


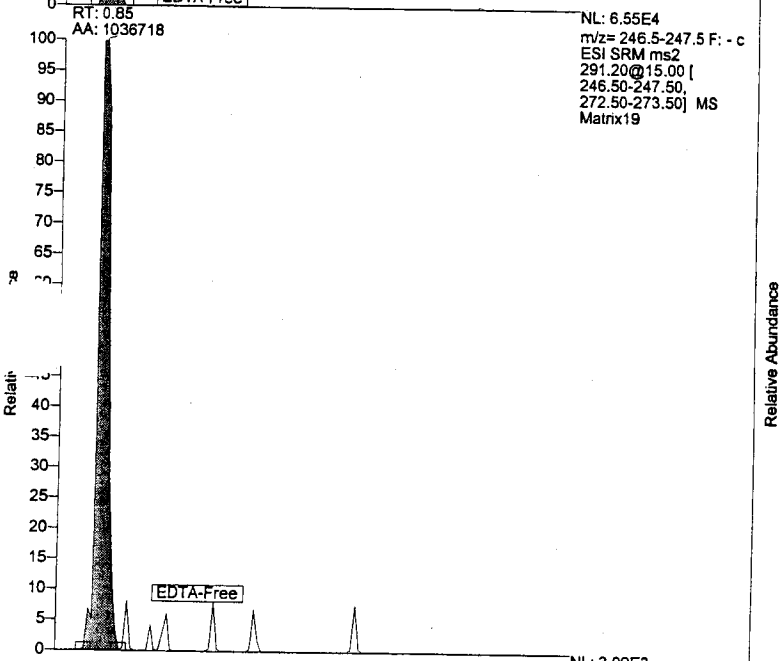


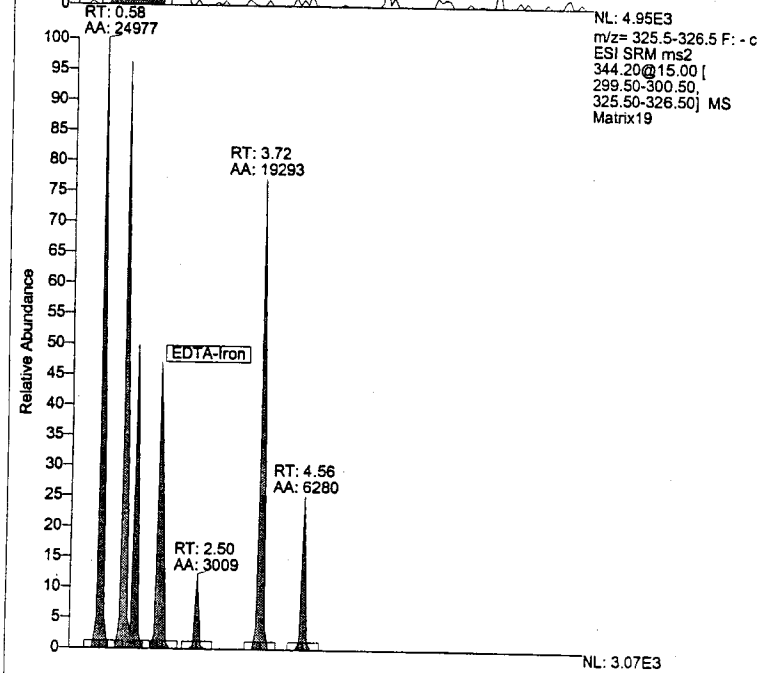
Ex 446
(435)

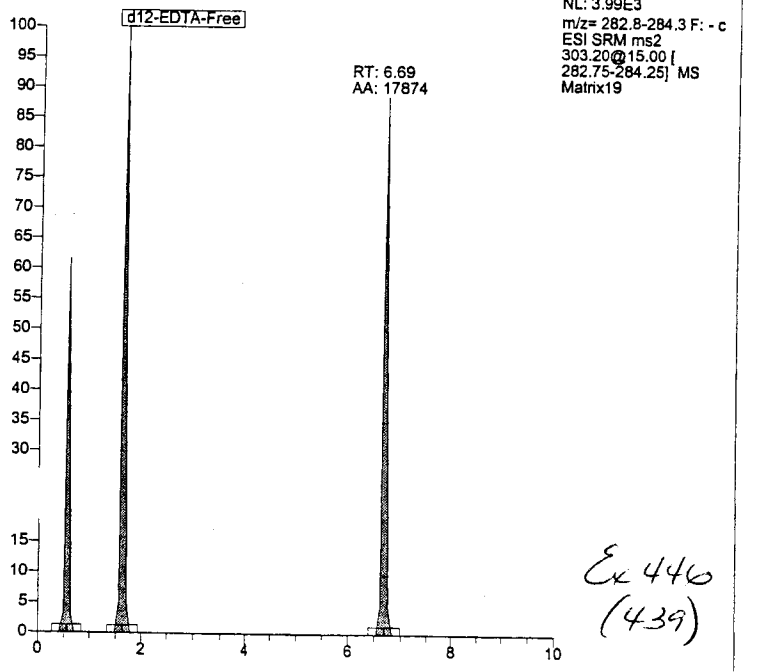
C:\Xcalibur\...\MAT_Negative\Matrix19 02/13/07 12:21:15 500 ppm EDTA JJB

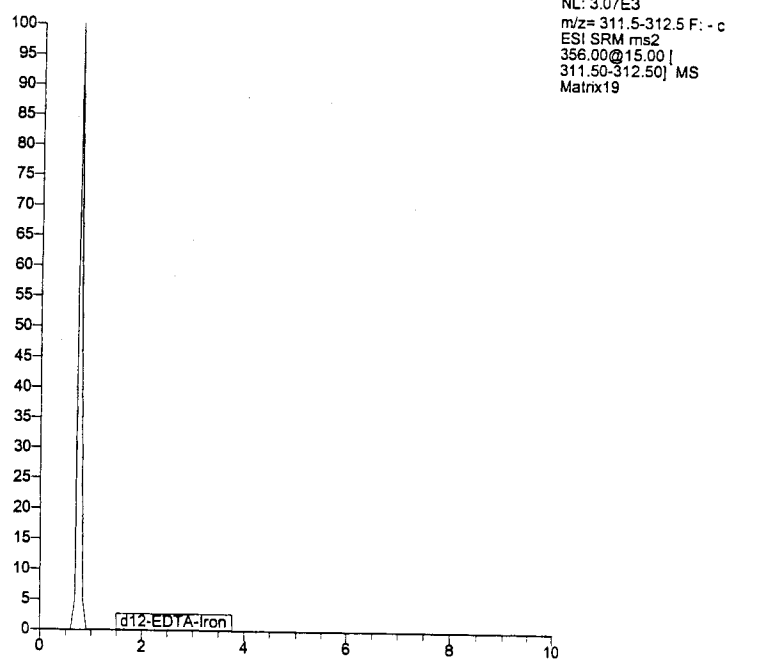






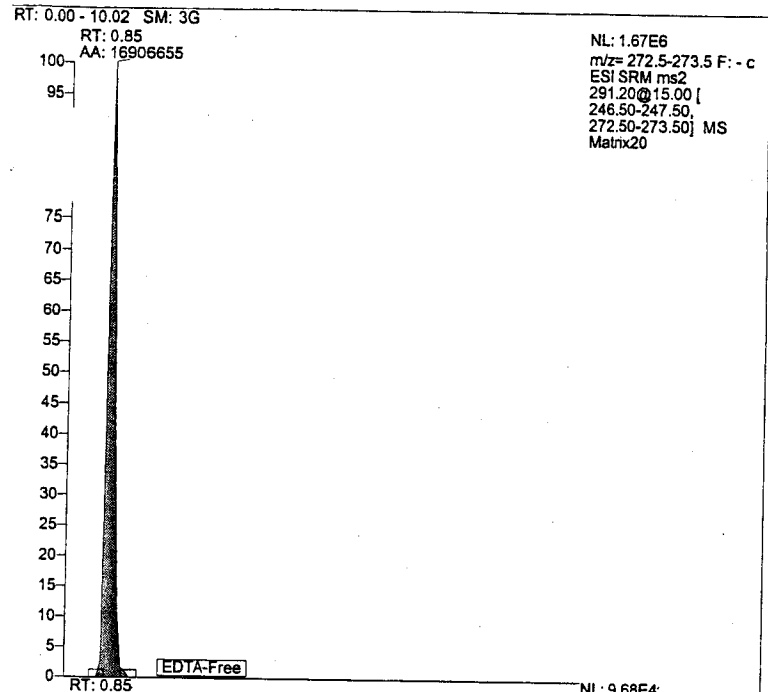


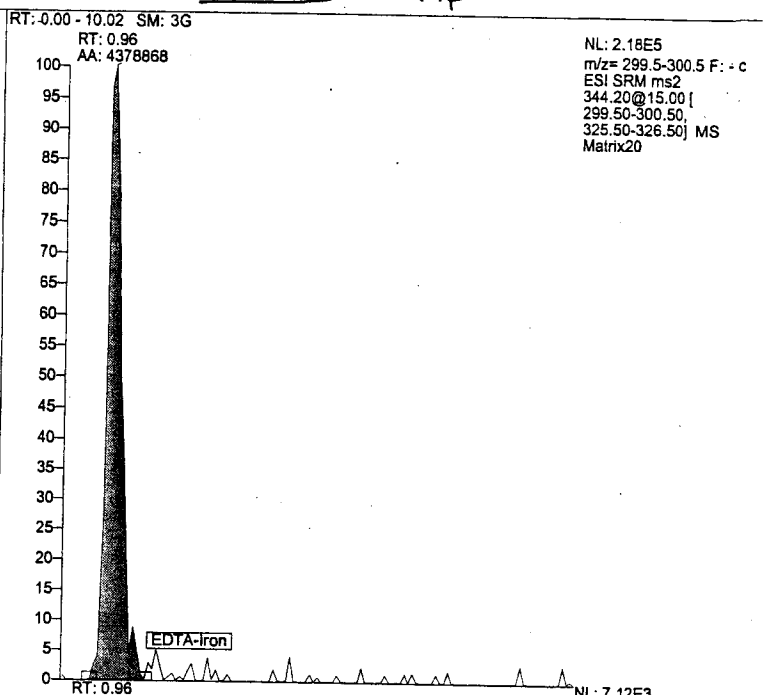


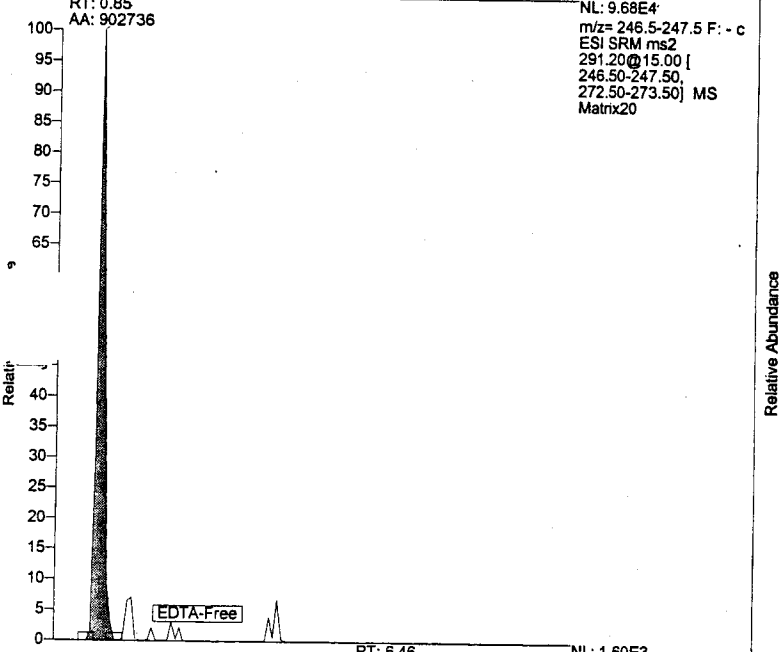


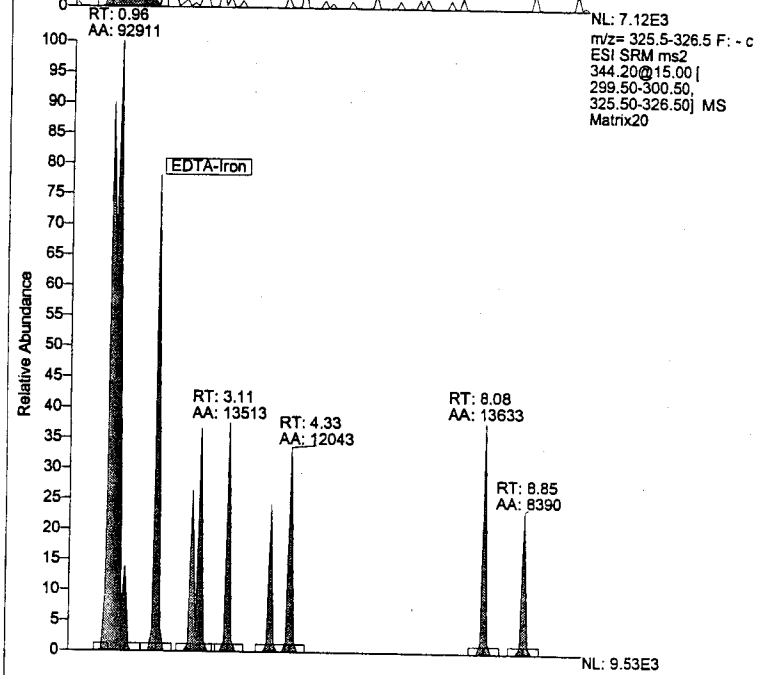
Ex 446
(439)

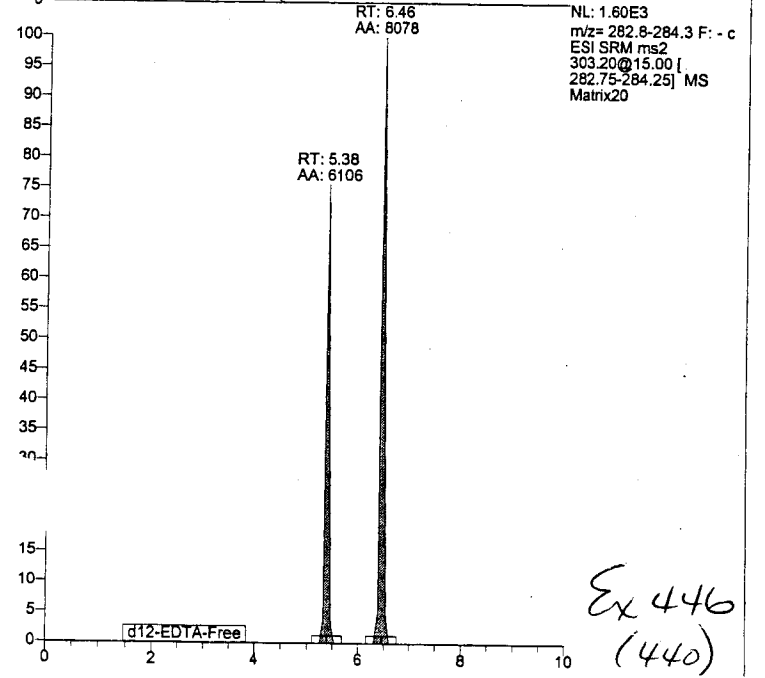
C:\Xcalibur\...\MAT_Negative\Matrix20 02/13/07 12:32:06 500 ppm EDTA TDB

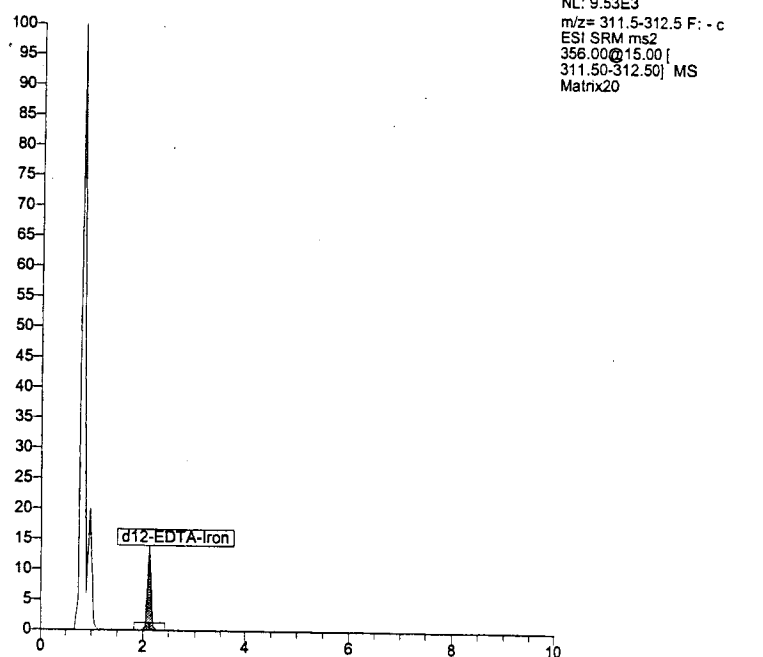












Ex 446
(440)

Positive Ionization Mode- Matrix Effect

EDTA free blood swabs- extracted then spiked to be 50 ppm EDTA

Sample	Area of RIC for base peak (160 m/z)
1	4848899
2	8018284
3	4636115
4	2840952
5	12647507
AVERAGE	6598351.4

Neat EDTA solution- 50 ppm

Injection	Area of RIC for base peak (160 m/z)
1	557891
2	550515
3	615572
4	945379
5	707733
AVERAGE	675418

Matrix Effect (50 ppm), % **976.9285687**

EDTA free blood swabs- extracted then spiked to be 500 ppm EDTA

Sample	Area of RIC for base peak (160 m/z)
1	9410131
2	24308297
3	18080488
4	22902136
5	16117570
AVERAGE	18163724.4

Neat EDTA solution- 500 ppm

Injection	Area of RIC for base peak (160 m/z)
1	5957879
2	6957758
3	8983927
4	8273487
5	8687103
AVERAGE	7772030.8

Matrix Effect (500 ppm), % **233.7062843**

Ex 446
(441)

2/10/07
JDB

NEGATIVE Ionization Mode- Matrix Effect

EDTA free blood swabs- extracted then spiked to be 50 ppm EDTA

Sample	Sum of Areas of free EDTA peaks (m/z 273 + 247)
1	170618
2	74496
3	79143
4	18721
5	17019
AVERAGE	87945

Neat EDTA solution- 50 ppm

Injection	Sum of Areas of free EDTA peaks (m/z 273 + 247)
1	1206757
2	1790660
3	4605670
4	3956765
5	2901483
AVERAGE	2892267

Matrix Effect (50 ppm), % **3.040694376**

EDTA free blood swabs- extracted then spiked to be 500 ppm EDTA

Sample	Sum of Areas of free EDTA peaks (m/z 273 + 247)
1	10362262
2	13608318
3	569737
4	2147727
5	2144609
AVERAGE	5766530.6

Neat EDTA solution- 500 ppm

Injection	Sum of Areas of free EDTA peaks (m/z 273 + 247)
1	14785267
2	18893392
3	16049375
4	16831807
5	17809391
AVERAGE	16873846.4

Matrix Effect (500 ppm), % **34.1743694**

Ex 446
(442)

2/13/07
JDB

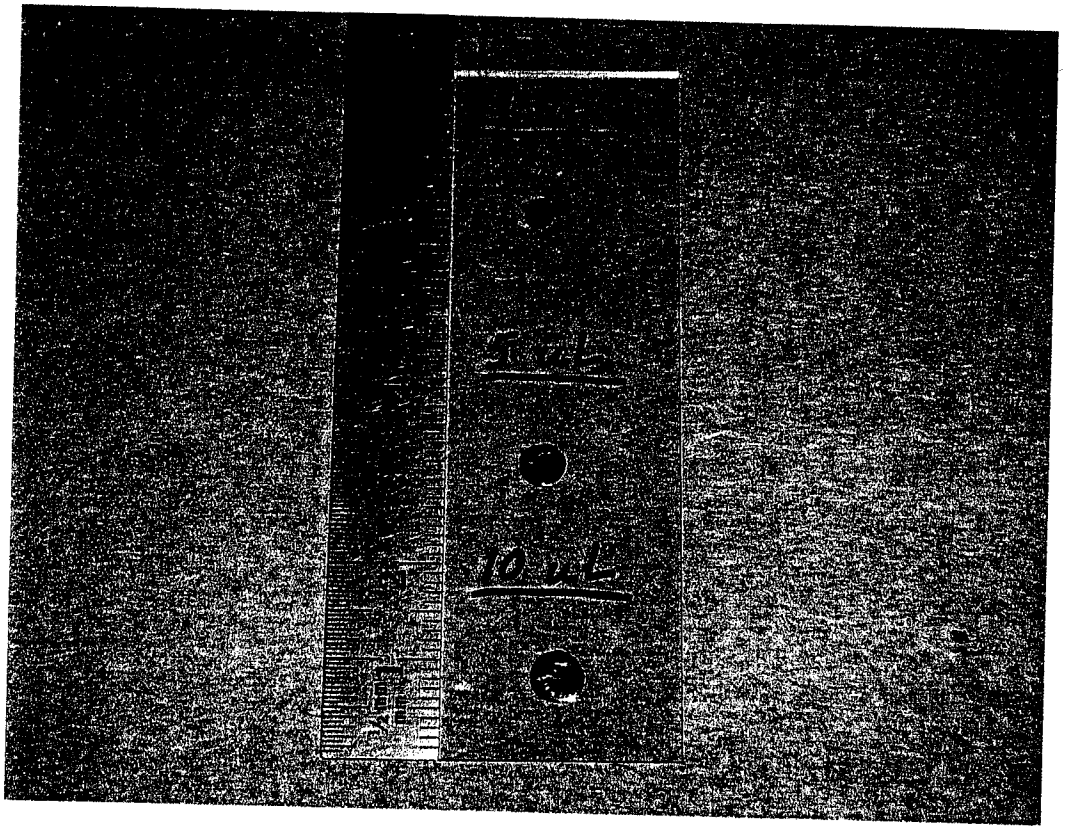
SPOT SIZE L.O.D.

Ex 446
(443)

JDP

02/15/2007

Blood spot sizes (1 uL, 5 uL, 10 uL)



Ex 446
(444)

Sequence---Spot Size_pos.sld [Open]

Sample Name: Neg blood swab extract

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	1	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 1 uL EDTA + A

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot02	02	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			2	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(445)

2/15/07
JDB

Sequence---Spot Size_pos.sld [Open]

Sample Name: Neg blood swab extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot03	01	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 1 uL EDTA + B

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot04	03	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			3	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(446)

2/15/07
JnP

Sequence---Spot Size_pos.sld [Open]

Sample Name: Neg blood swab extract
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot05	01	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 1 uL EDTA + C
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot06	04	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			4	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(447)

2/15/07
JMB

Sequence---Spot Size_pos.sld [Open]

Sample Name: Neg blood swab extract
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot07	01	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 5 uL EDTA + A
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot08	05	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			5	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(448)

2/15/07
JNP

Sequence---Spot Size_pos.sld [Open]

Sample Name: Neg blood swab extract
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot09	01	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 5 uL EDTA + B
Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot10	06	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			6	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(449)

2/15/07
JNB

Sequence---Spot Size_pos.sld [Open]

Sample Name: Neg blood swab extract

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot11	01	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 5 uL EDTA + C

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot12	07	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			7	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(450)

2/15/07
JMP

Sequence---Spot Size_pos.sld [Open]

JDB

Sample Name: Neg blood swab extract

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot13	01	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JDB

Sample Name: 10 uL EDTA + A

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot14	08	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			8	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(451)

2/15/07
JNB

Sequence---Spot Size_pos.sld [Open]

Sample Name: Neg blood swab extract

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

JMB

Sample Type	File Name	Sample ID	Path
Unknown	Spot15	01	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 10 uL EDTA + B

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

JMB

Sample Type	File Name	Sample ID	Path
Unknown	Spot16	09	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			9	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(452)

2/15/07
JMB

Sequence---Spot Size_pos.sld [Open]

Sample Name: Neg blood swab extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

JDB

Sample Type	File Name	Sample ID	Path
Unknown	Spot17	01	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 10 uL EDTA + C

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

JDB

Sample Type	File Name	Sample ID	Path
Unknown	Spot18	10	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			10	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(453)

2/15/07
JDB

Sequence---Spot Size_pos.sld [Open]

Sample Name: Neg blood swab extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

JMB

Sample Type	File Name	Sample ID	Path
Unknown	Spot19	01	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Positive

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Pos_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

* STOPPED SEQUENCE AFTER 5uL RUN, SINCE 5 uL SAMPLES WERE ADEQUATELY DETECTED. JMP

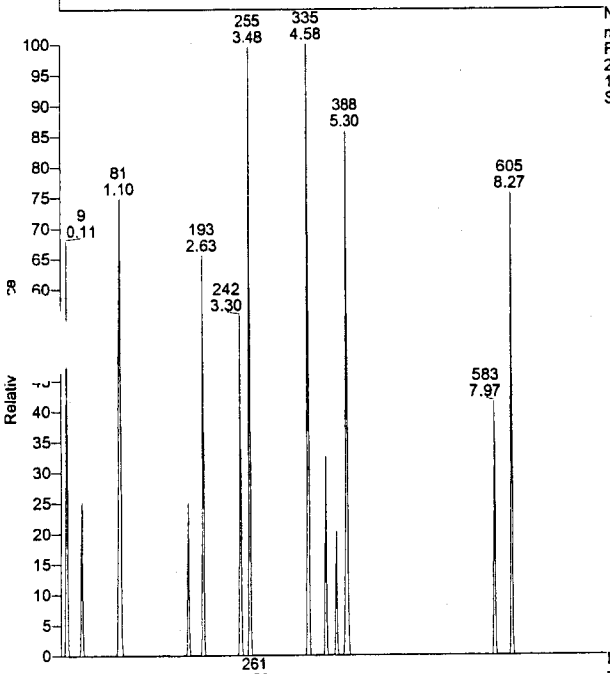
Ex 446
(454)

2/15/07
JMB

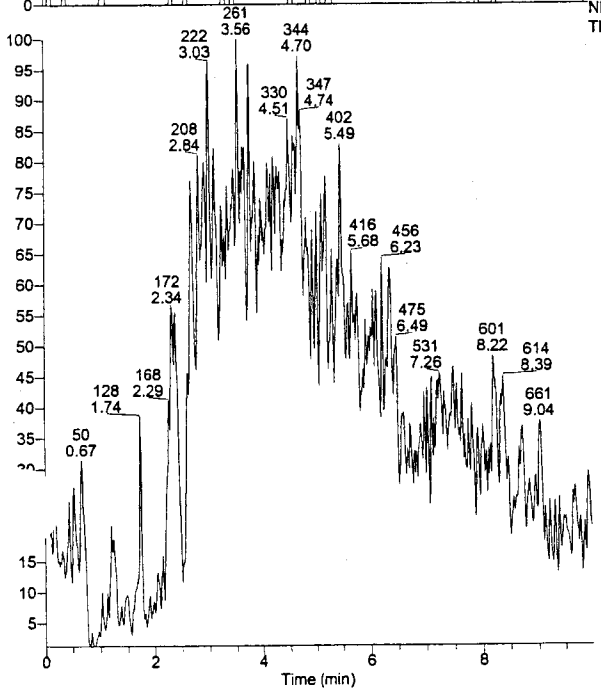
RT; 0.00 - 10.00 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot01

NL: 5.81E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot01

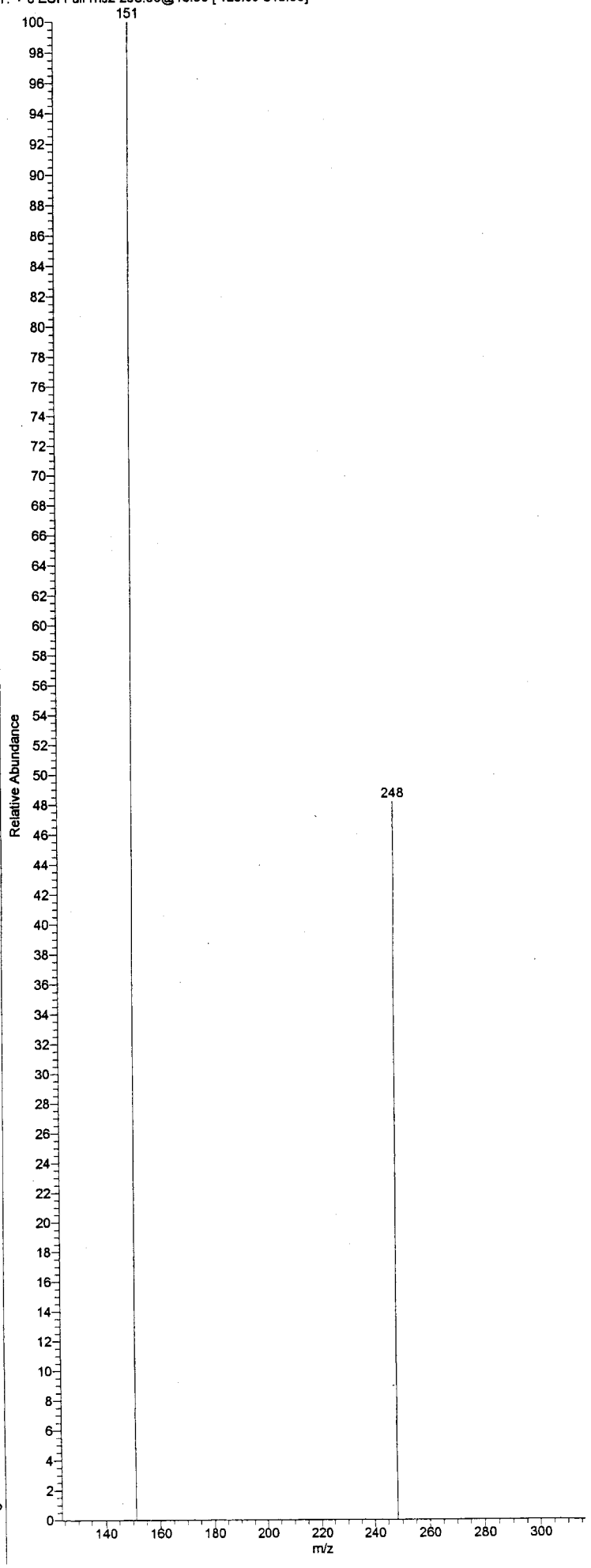


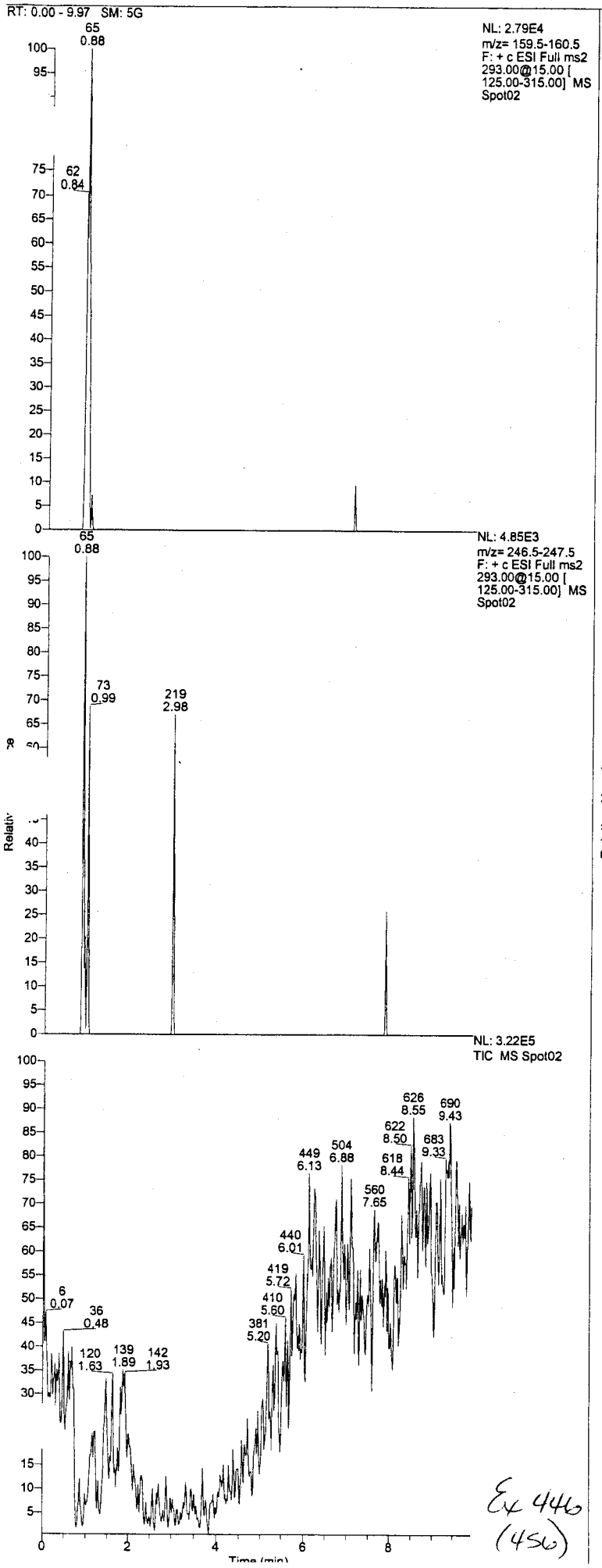
NL: 5.29E5
TIC MS Spot01

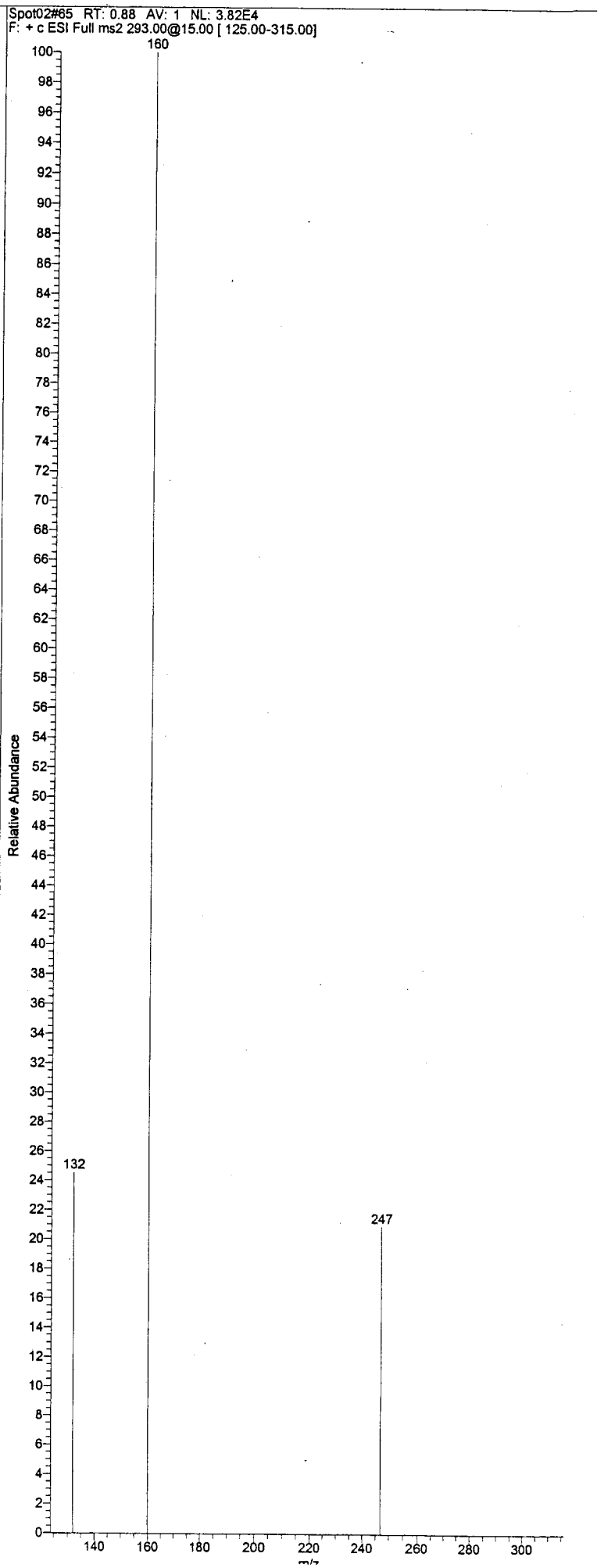


Ex 446
(455)

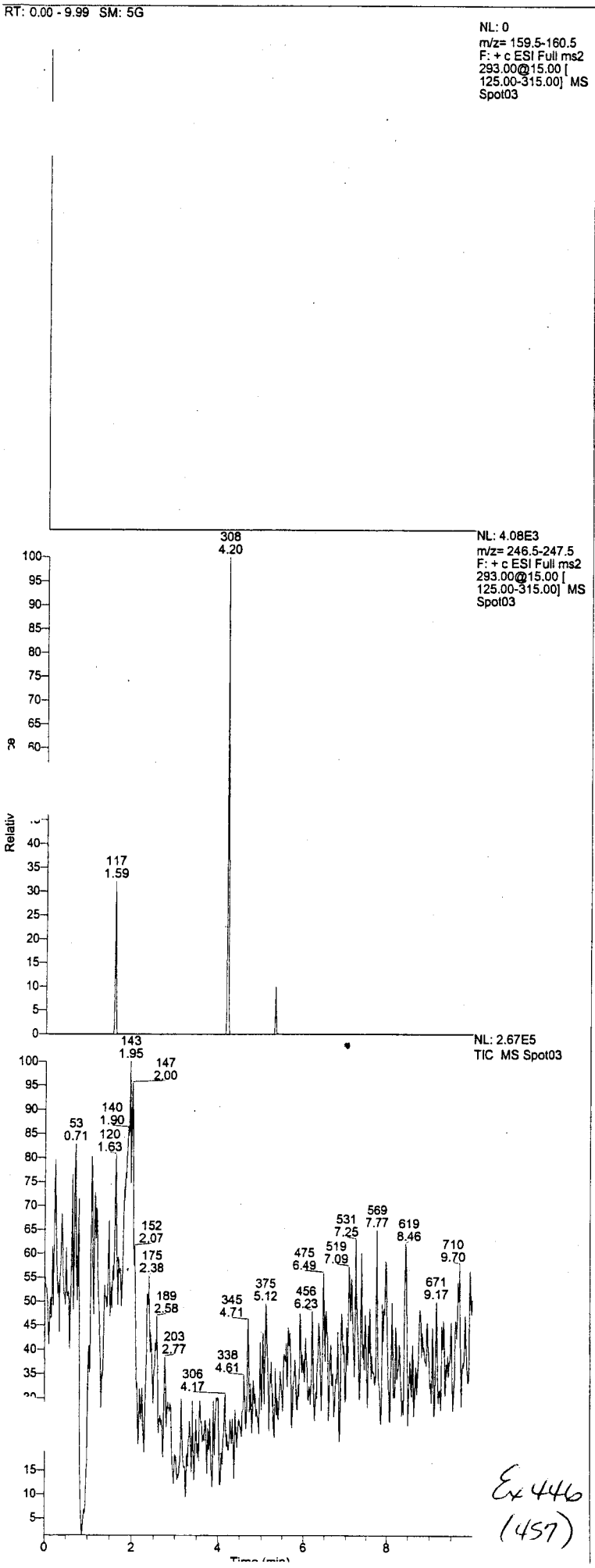
Spot01#65 RT: 0.88 AV: 1 NL: 6.14E3
T: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

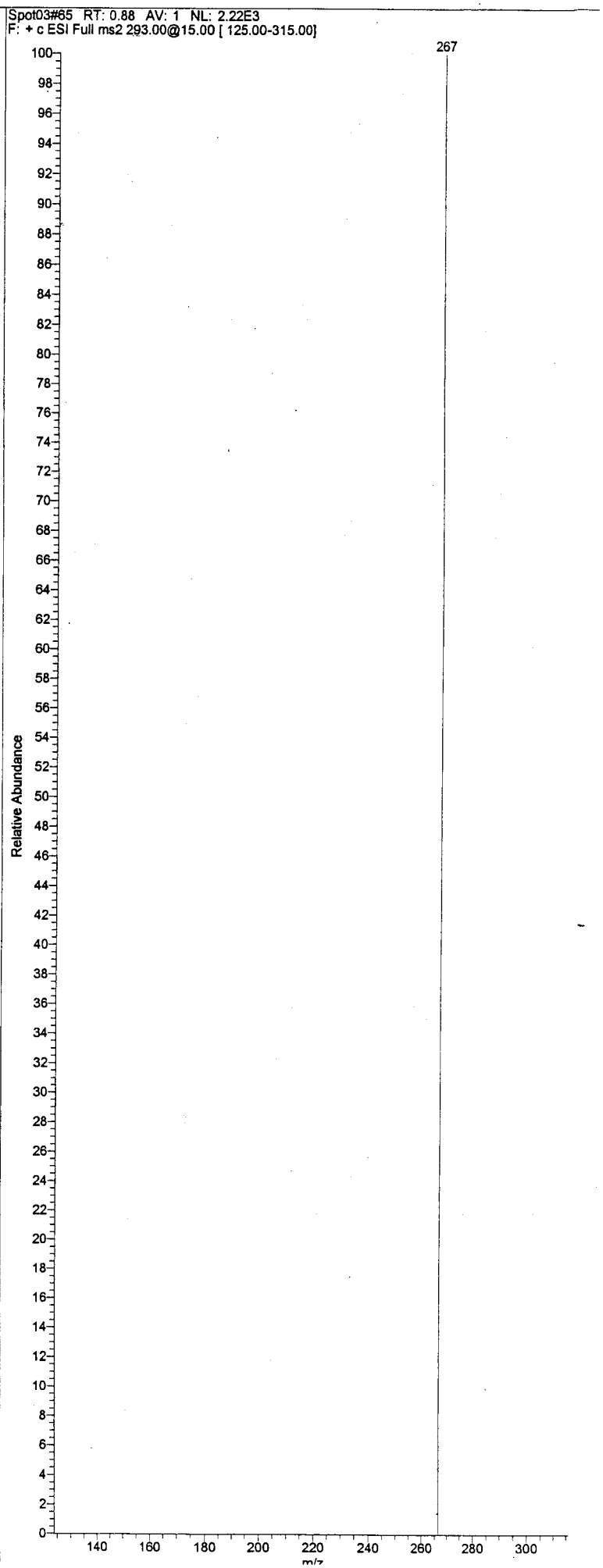



RT: 0.00 - 9.97 SM: 5G
NL: 2.79E4
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot02
65
0.88
62
0.84
NL: 4.85E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot02
65
0.88
73
0.99
219
2.98
NL: 3.22E5
TIC MS Spot02
626
8.55
690
9.43
622
8.50
683
9.33
504
6.88
449
6.13
618
8.44
560
7.65
440
6.01
419
5.72
410
5.60
381
5.20
6
0.07
36
0.48
120
1.63
139
1.89
142
1.93
Relative Abundance
Time (min)


Spot02#65 RT: 0.88 AV: 1 NL: 3.82E4
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]
160
132
247
Relative Abundance
m/z

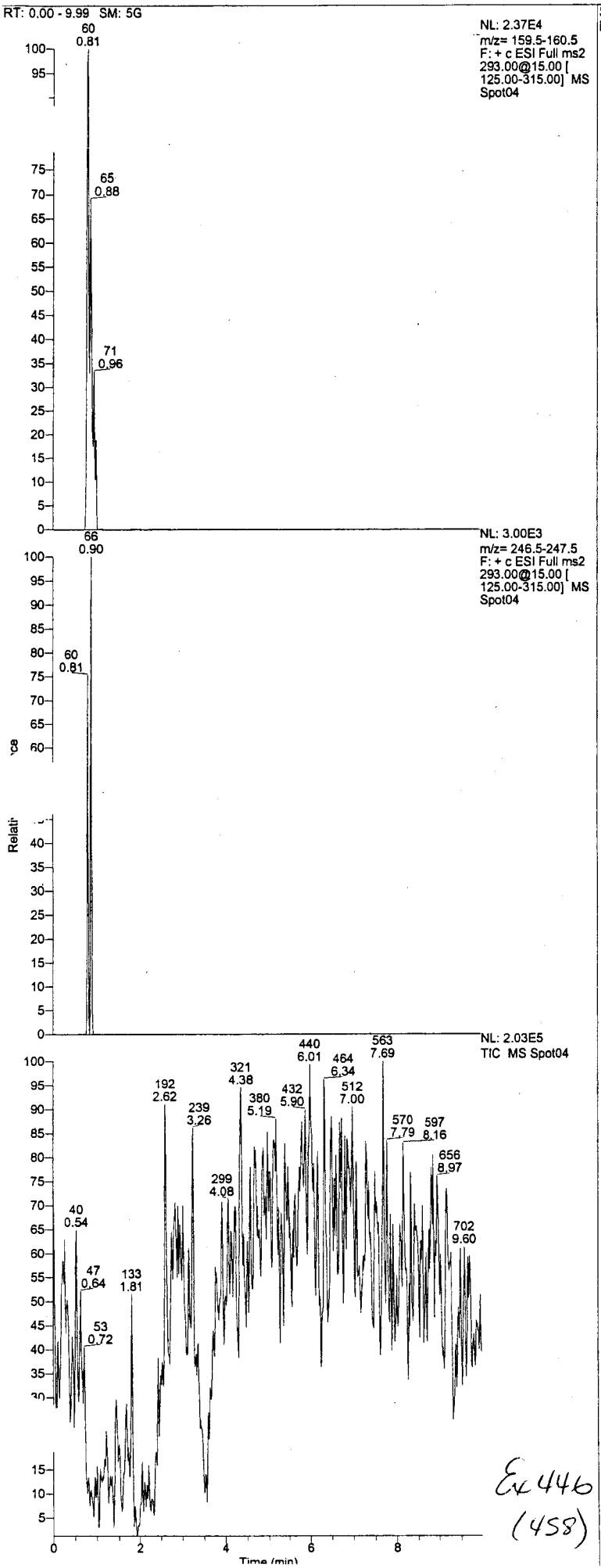
Ex 446
(456)

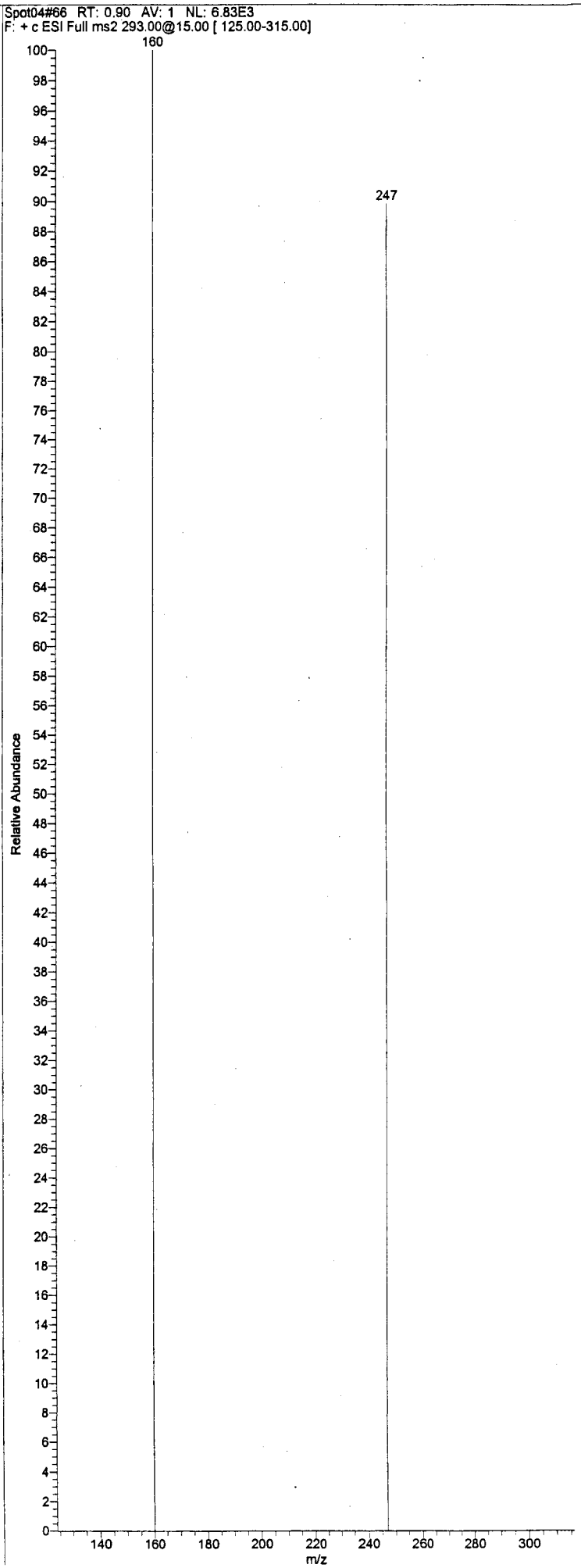

RT: 0.00 - 9.99 SM: 5G
NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot03
NL: 4.08E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot03
308
4.20
117
1.59
NL: 2.67E5
TIC MS Spot03
143
1.95
147
2.00
140
1.90
53
0.71
120
1.63
152
2.07
175
2.38
189
2.58
203
2.77
306
4.17
338
4.61
345
4.71
375
5.12
456
6.23
475
6.49
519
7.09
531
7.25
569
7.77
619
8.46
671
9.17
710
9.70
Relativ
Time (min)


Spot03#65 RT: 0.88 AV: 1 NL: 2.22E3
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]
267
Relative Abundance
m/z

Ex 446
(457)

C:\Xcalibur\...\Spot04 02/15/07 12:19:30 1 uL EDTA + B





Ex 446
(458)

RT: 0.00 - 9.98 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot05

665
9.09

NL: 4.58E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot05

485
6.63

Relati

NL: 2.93E5
TIC MS Spot05

302
4.12

26
0.35
47
0.64
138
1.88
183
2.50
189
2.58
440
6.01
465
6.35
479
6.54
552
7.54
615
8.40
685
9.36

Time (min)

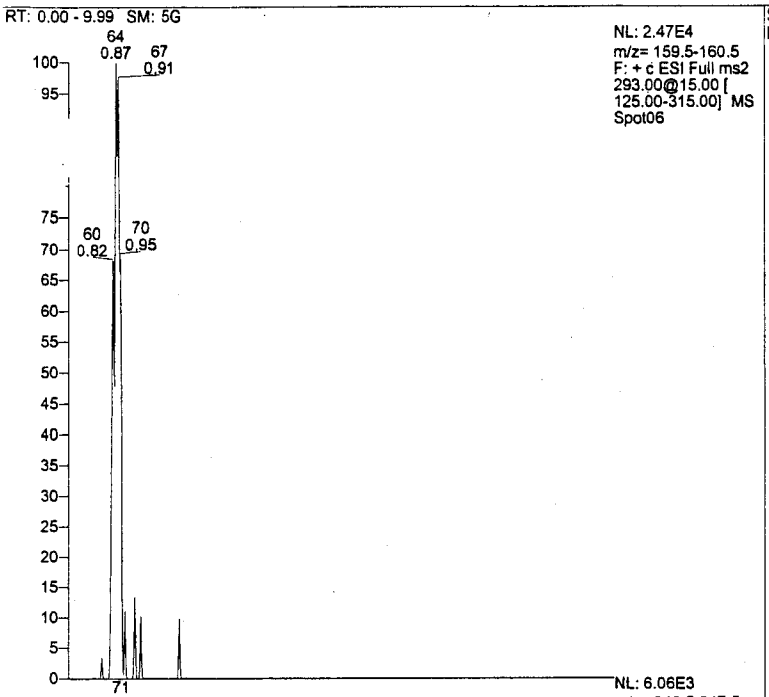
Ex 446
(459)

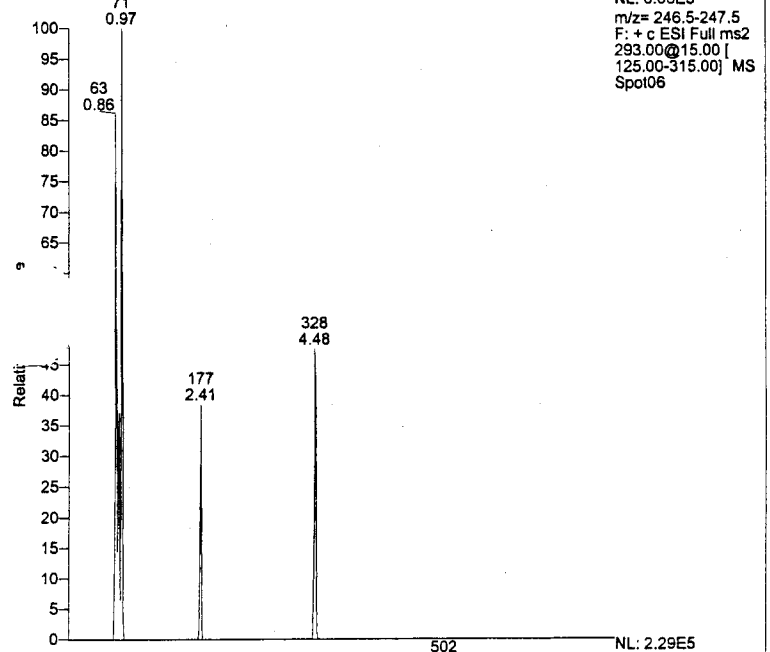
Spot05#65 RT: 0.88 AV: 1 NL: 2.85E3
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

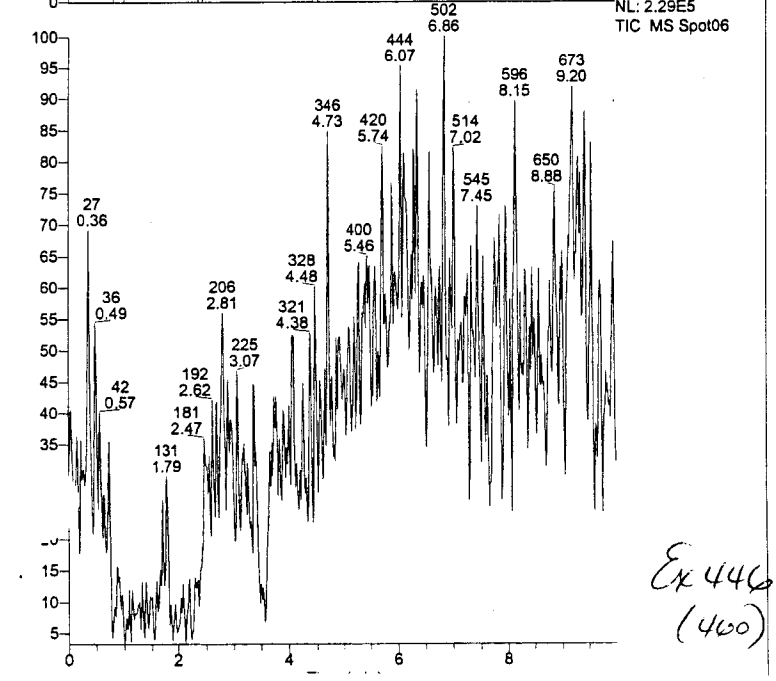
293

Relative Abundance

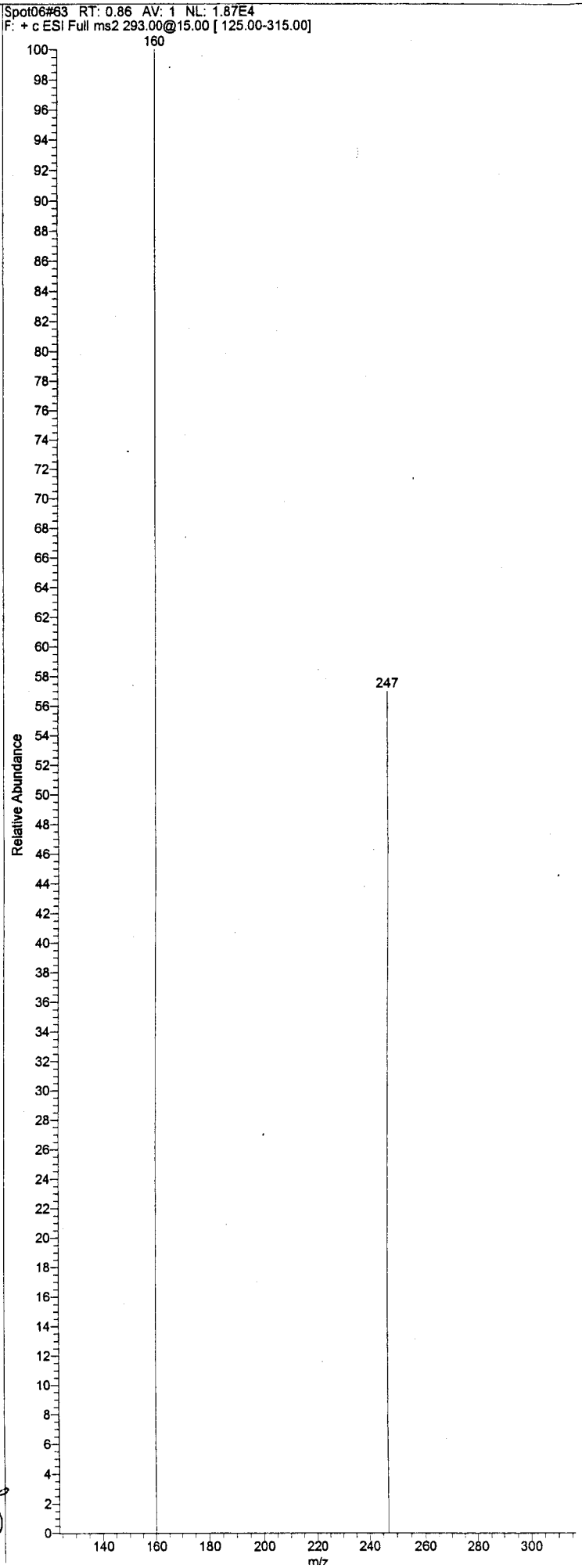
m/z


RT: 0.00 - 9.99 SM: 5G
NL: 2.47E4
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot06
64
0.87
67
0.91
60
0.82
70
0.95


NL: 6.06E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot06
71
0.97
63
0.86
328
4.48
177
2.41
Relati


NL: 2.29E5
TIC MS Spot06
502
6.86
444
6.07
673
9.20
596
8.15
346
4.73
420
5.74
514
7.02
650
8.88
545
7.45
400
5.46
328
4.48
321
4.38
206
2.81
225
3.07
192
2.62
181
2.47
131
1.79
27
0.36
36
0.49
42
0.57

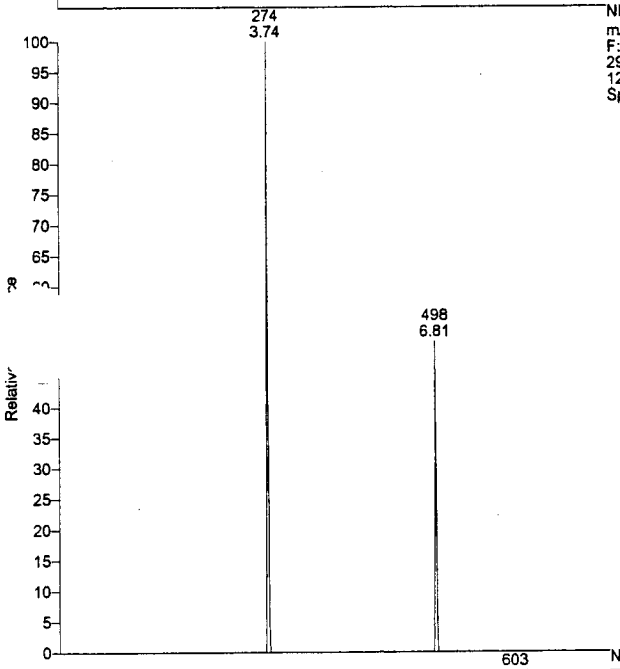
Ex 446
(460)


Spot06#63 RT: 0.86 AV: 1 NL: 1.87E4
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]
160
247
Relative Abundance
m/z

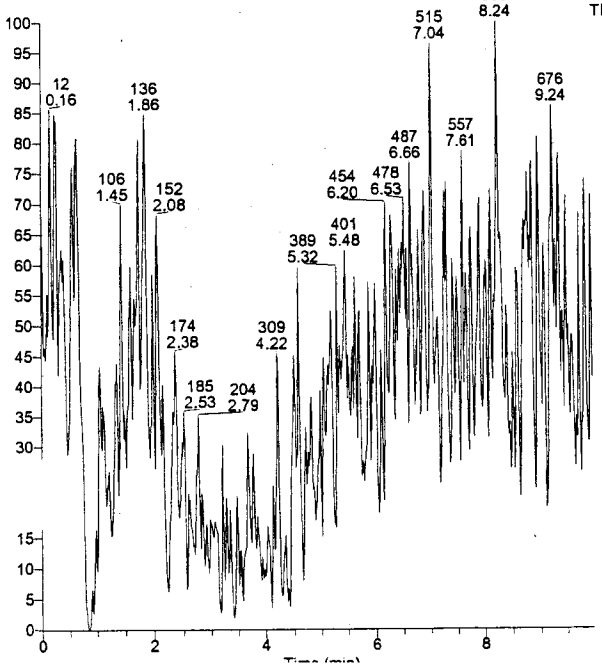
RT: 0.00 - 9.98 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot07

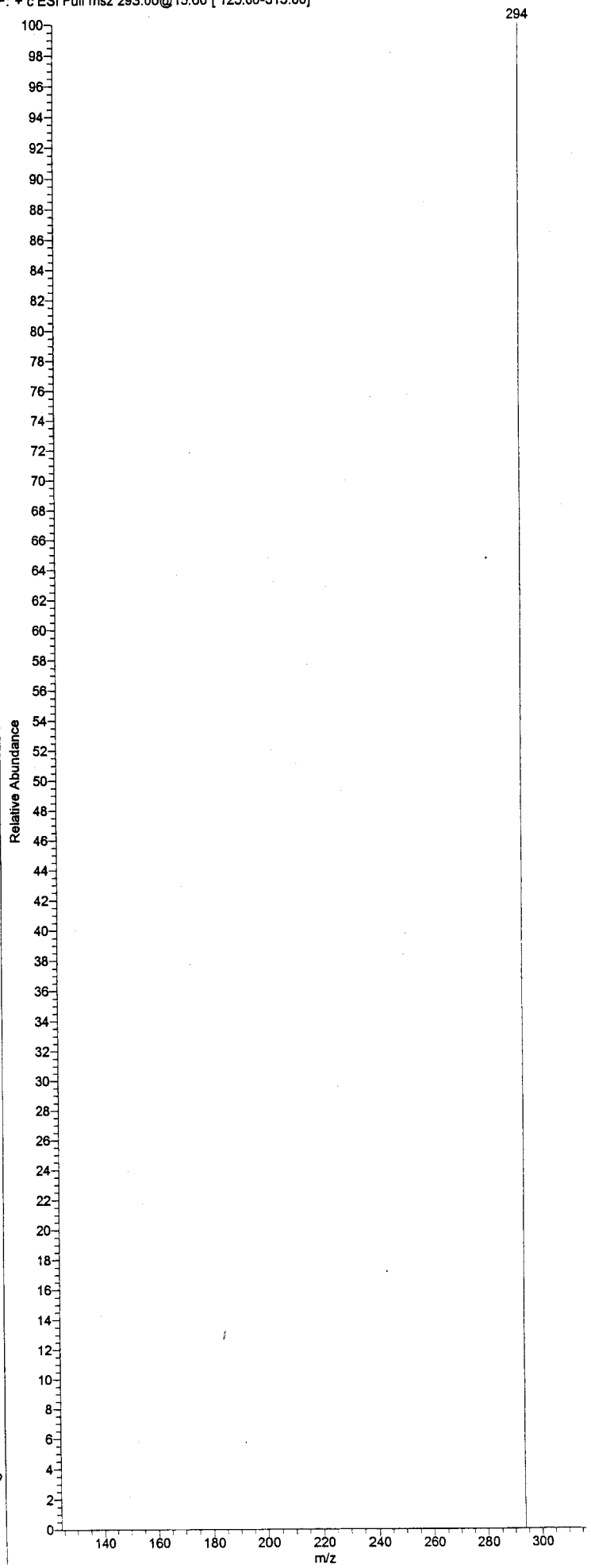
NL: 2.02E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot07



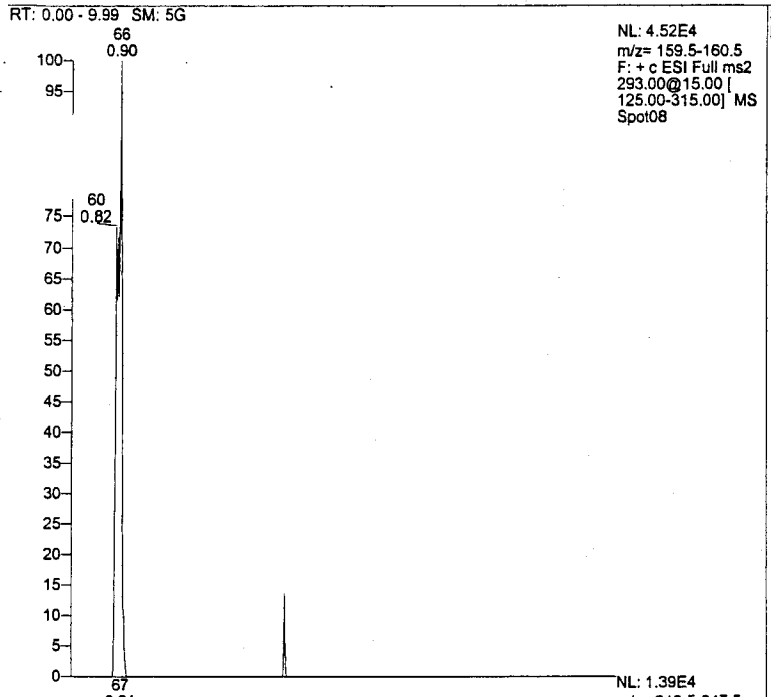
NL: 1.75E5
TIC MS Spot07

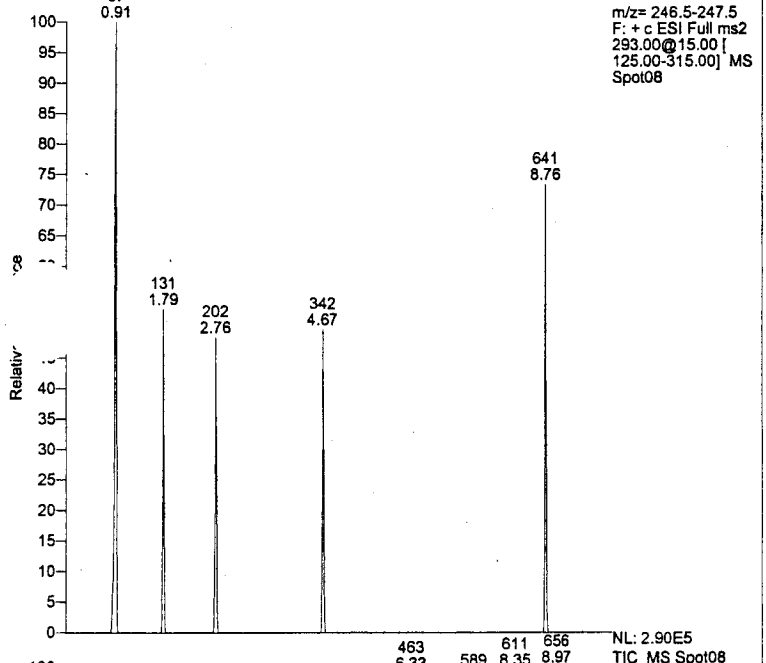


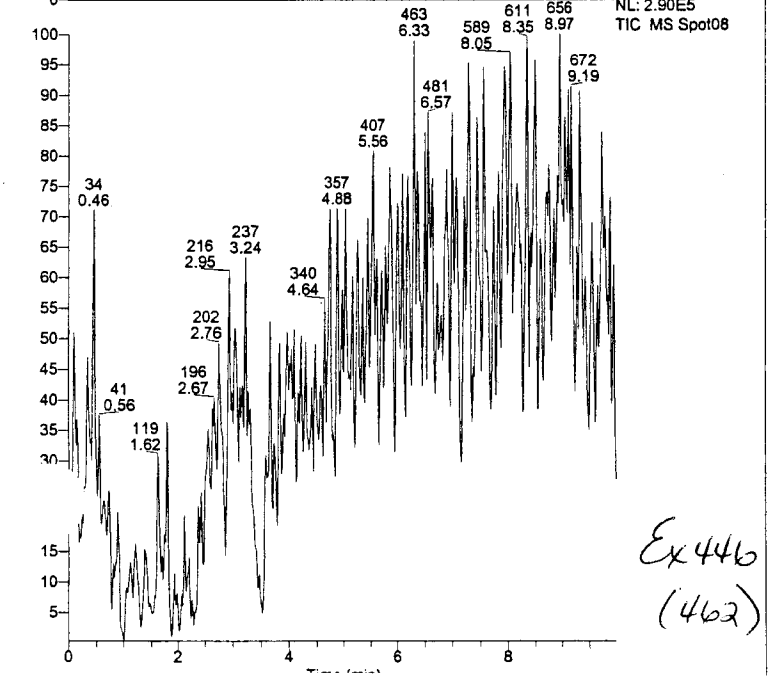
Spot07#65 RT: 0.89 AV: 1 NL: 4.76E3
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



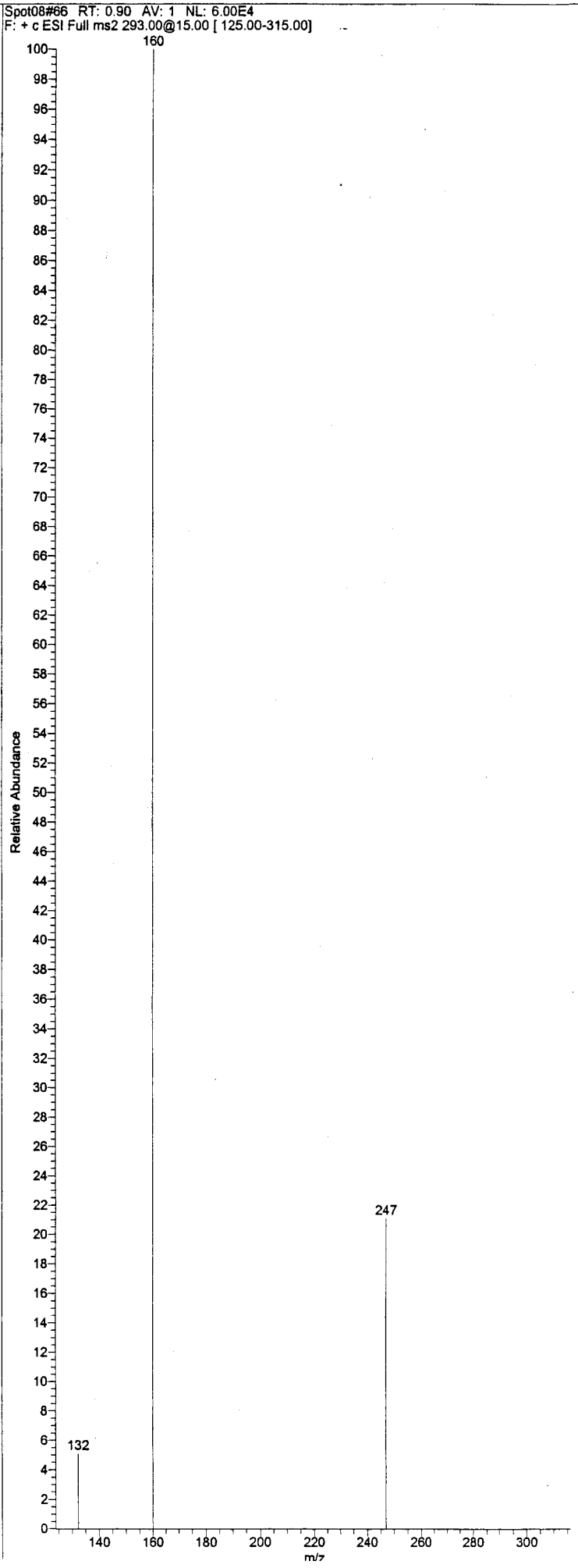
Ex 446
(461)


RT: 0.00 - 9.99 SM: 5G
NL: 4.52E4
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot08
66
0.90
60
0.82


NL: 1.39E4
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot08
67
0.91
641
8.76
131
1.79
202
2.76
342
4.67
Relativ


NL: 2.90E5
TIC MS Spot08
463
6.33
589
8.05
611
8.35
656
8.97
672
9.19
481
6.57
407
5.56
357
4.88
340
4.64
237
3.24
216
2.95
202
2.76
196
2.67
34
0.46
41
0.56
119
1.62

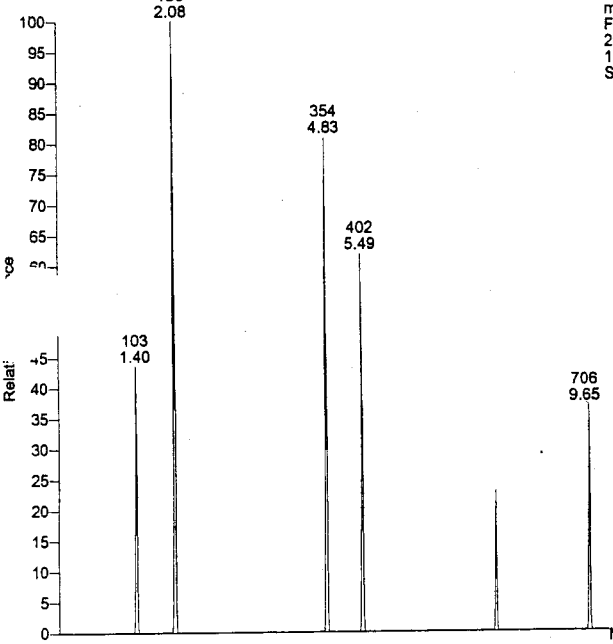
Ex 446
(462)


Spot08#66 RT: 0.90 AV: 1 NL: 6.00E4
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]
160
247
132
Relative Abundance
m/z

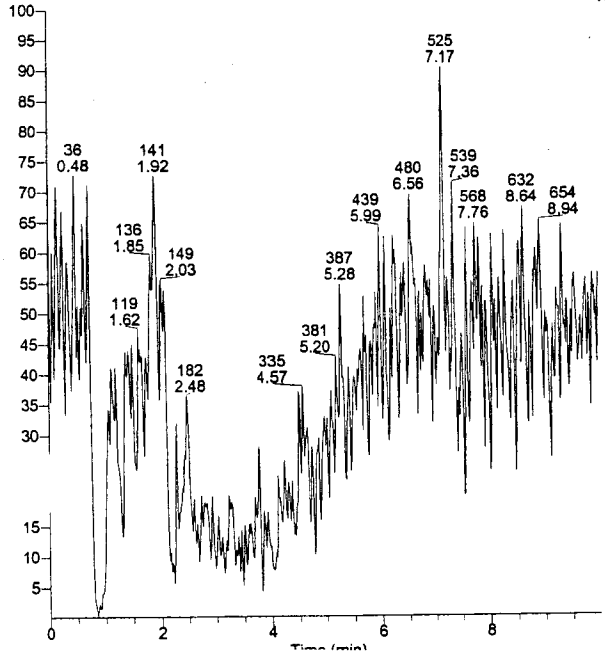
RT: 0.00 - 9.99 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot09

NL: 6.45E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot09

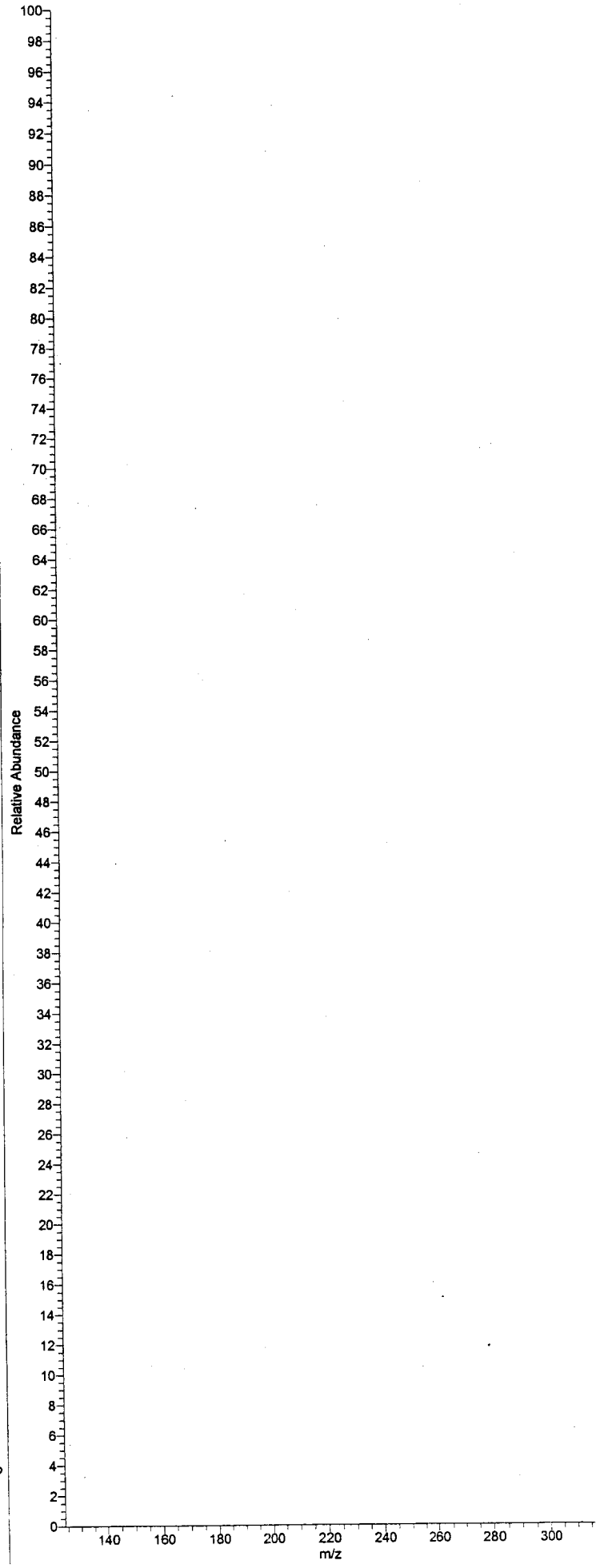


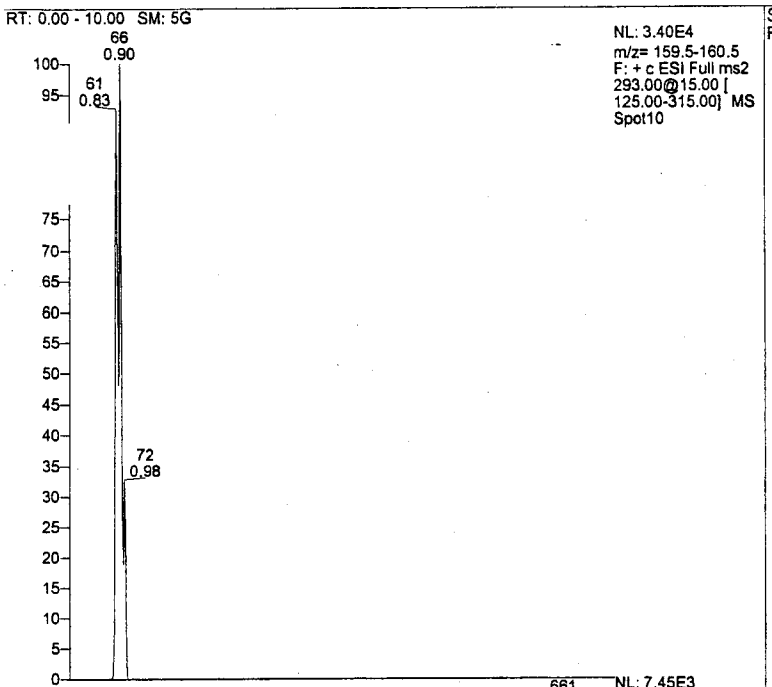
NL: 3.20E5
TIC MS Spot09

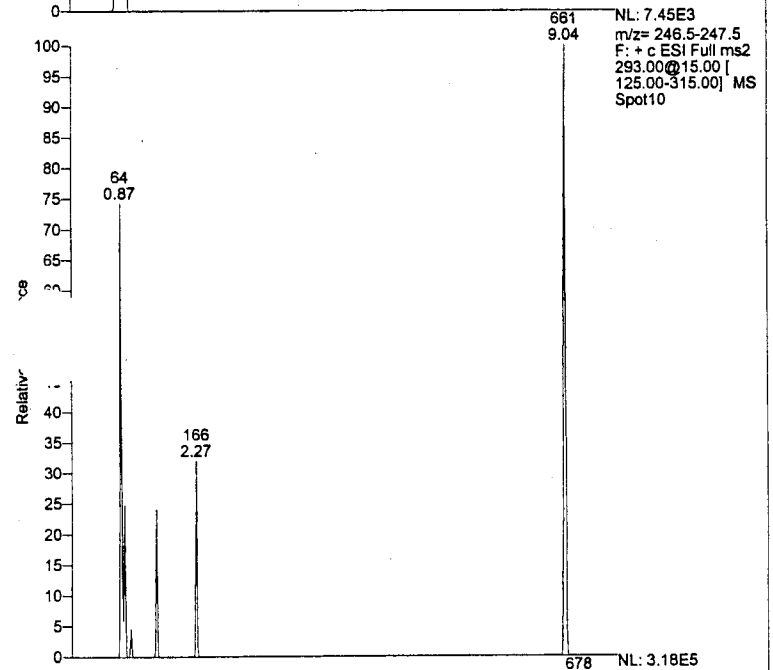


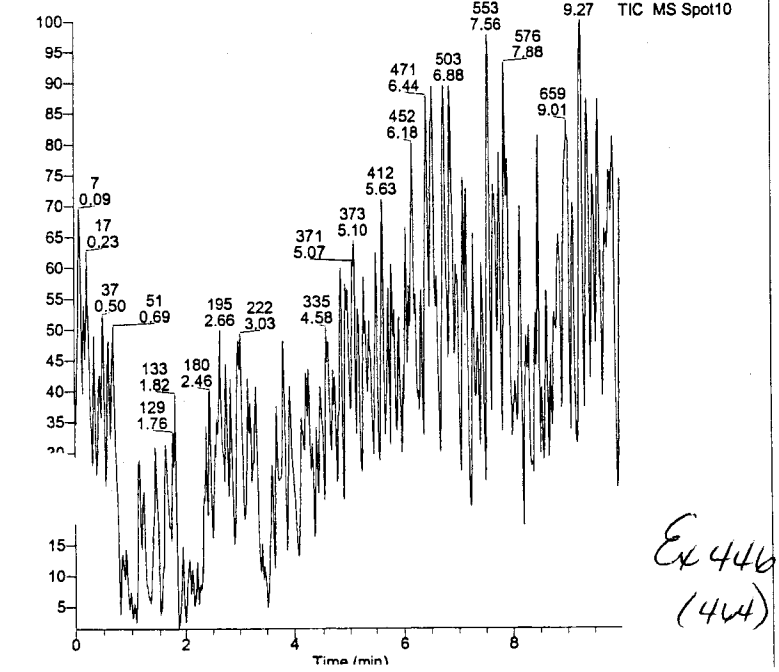
Ex 446
(463)

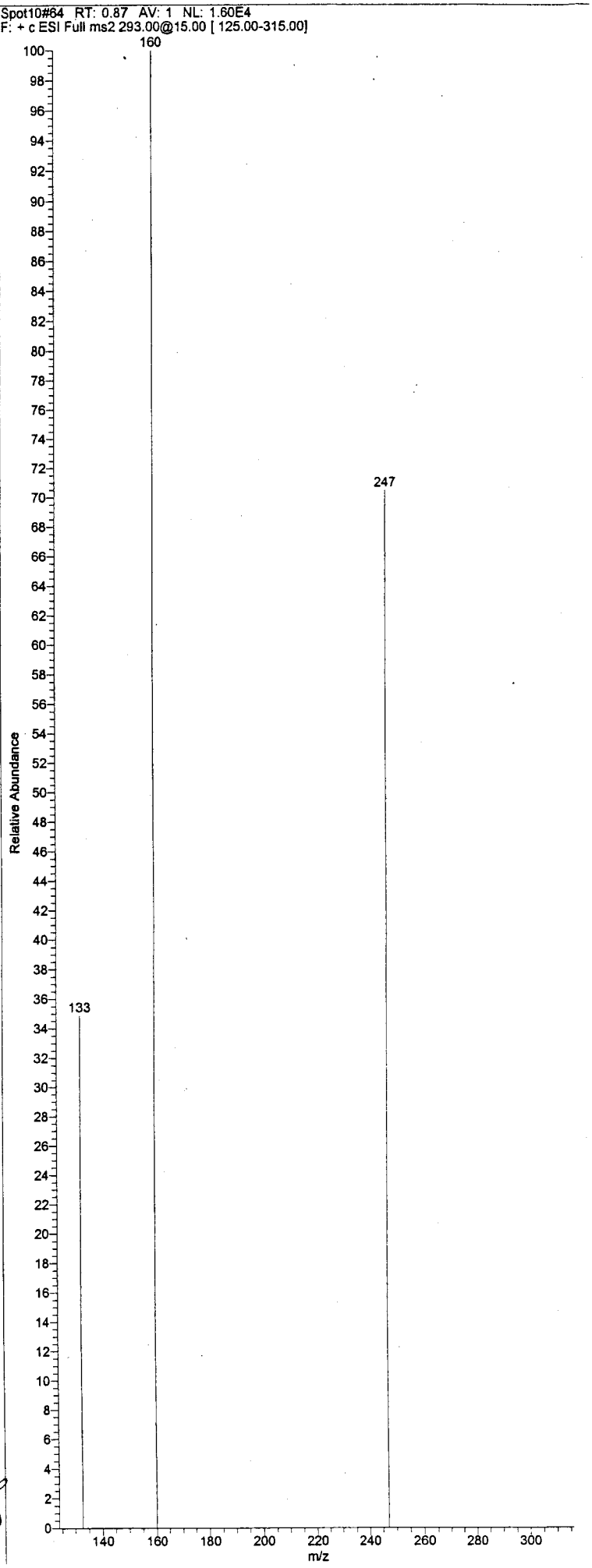
Spot09#66 RT: 0.89 AV: 1 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]




RT: 0.00 - 10.00 SM: 5G
NL: 3.40E4
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot10
66
0.90
61
0.83
72
0.98


NL: 7.45E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot10
661
9.04
64
0.87
166
2.27
Relativ


NL: 3.18E5
TIC MS Spot10
678
9.27
553
7.56
576
7.88
503
6.88
471
6.44
659
9.01
452
6.18
412
5.63
373
5.10
371
5.07
7
0.09
17
0.23
37
0.50
51
0.69
195
2.66
222
3.03
335
4.58
133
1.82
180
2.46
129
1.76
Time (min)

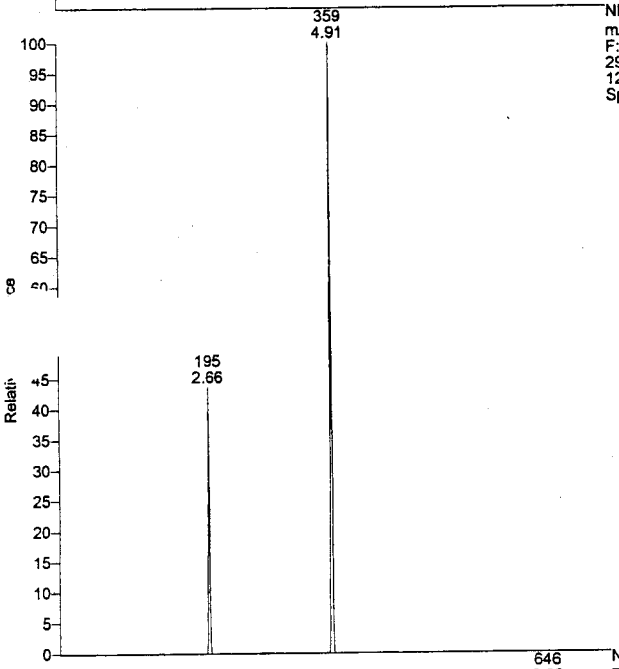

Spot10#64 RT: 0.87 AV: 1 NL: 1.60E4
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]
160
247
133
Relative Abundance
m/z

Ex 446
(444)

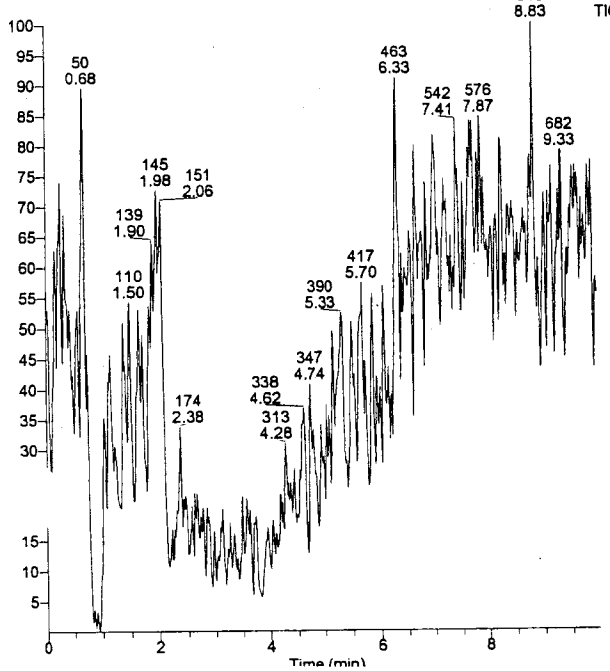
RT: 0.00 - 9.98 SM: 5G

NL: 0
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot11

NL: 2.54E3
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
Spot11

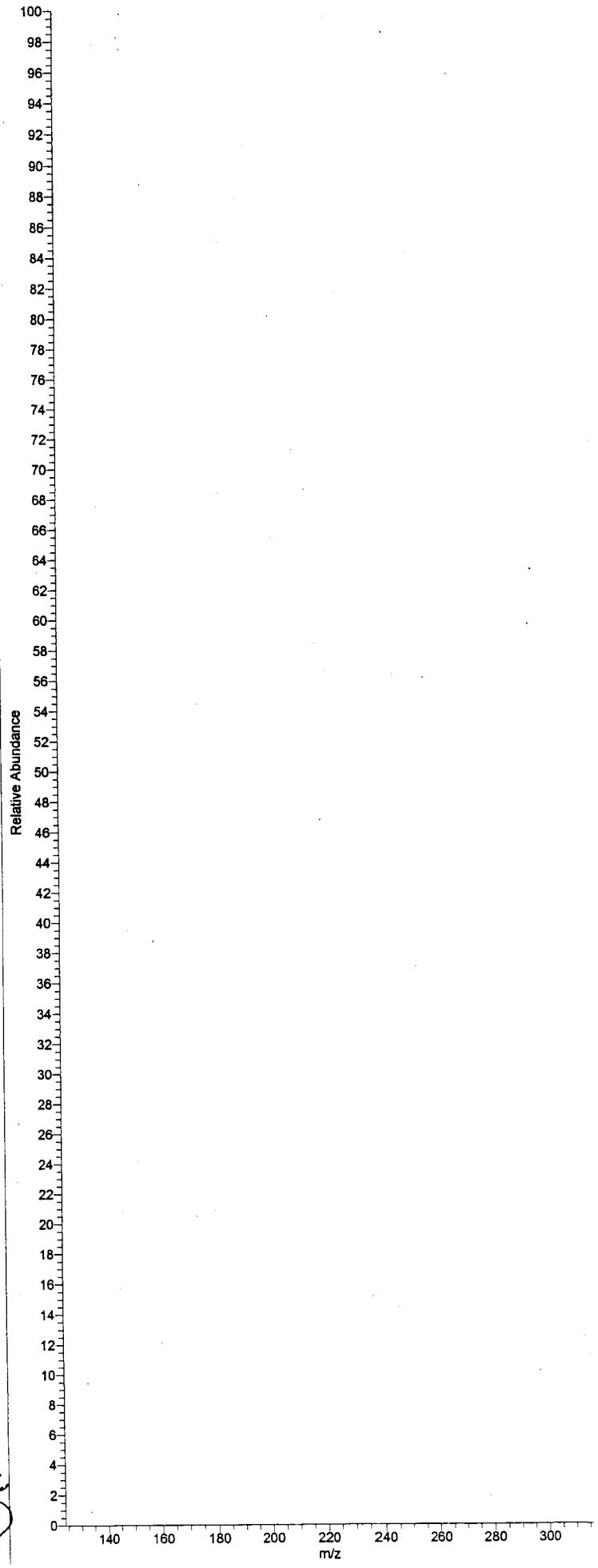


NL: 3.30E5
TIC MS Spot11

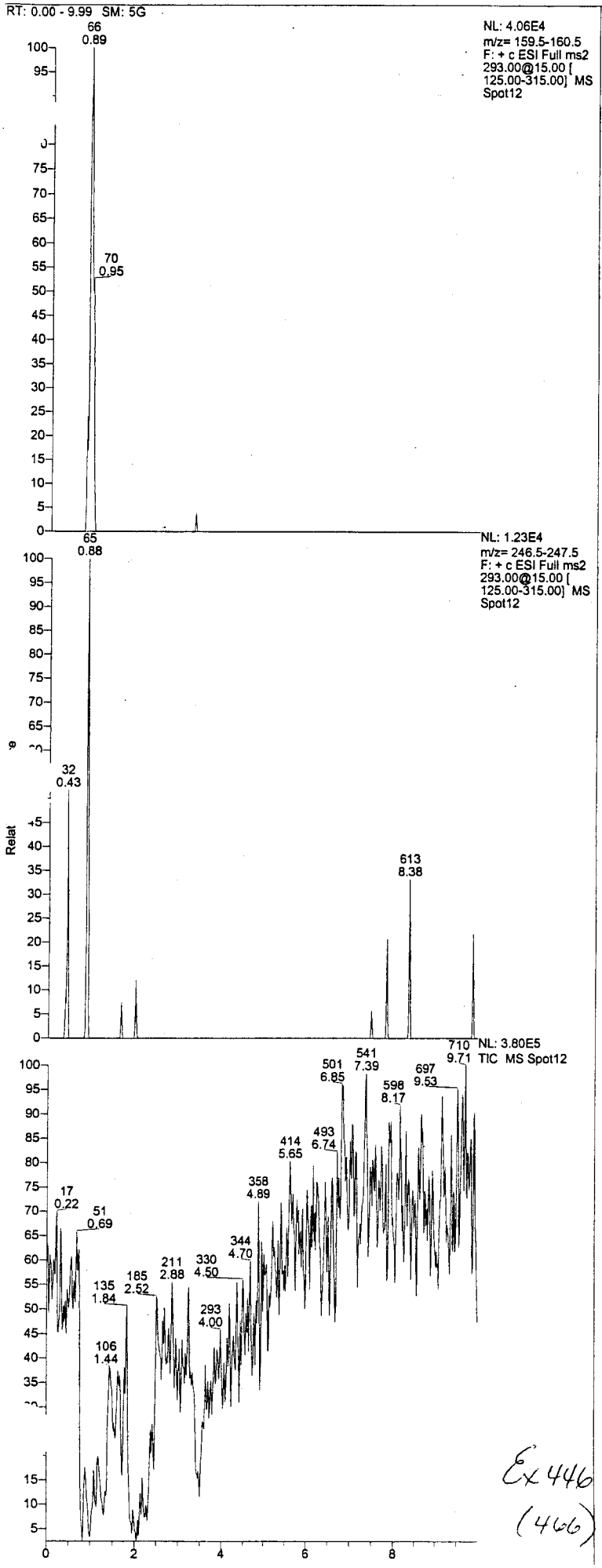


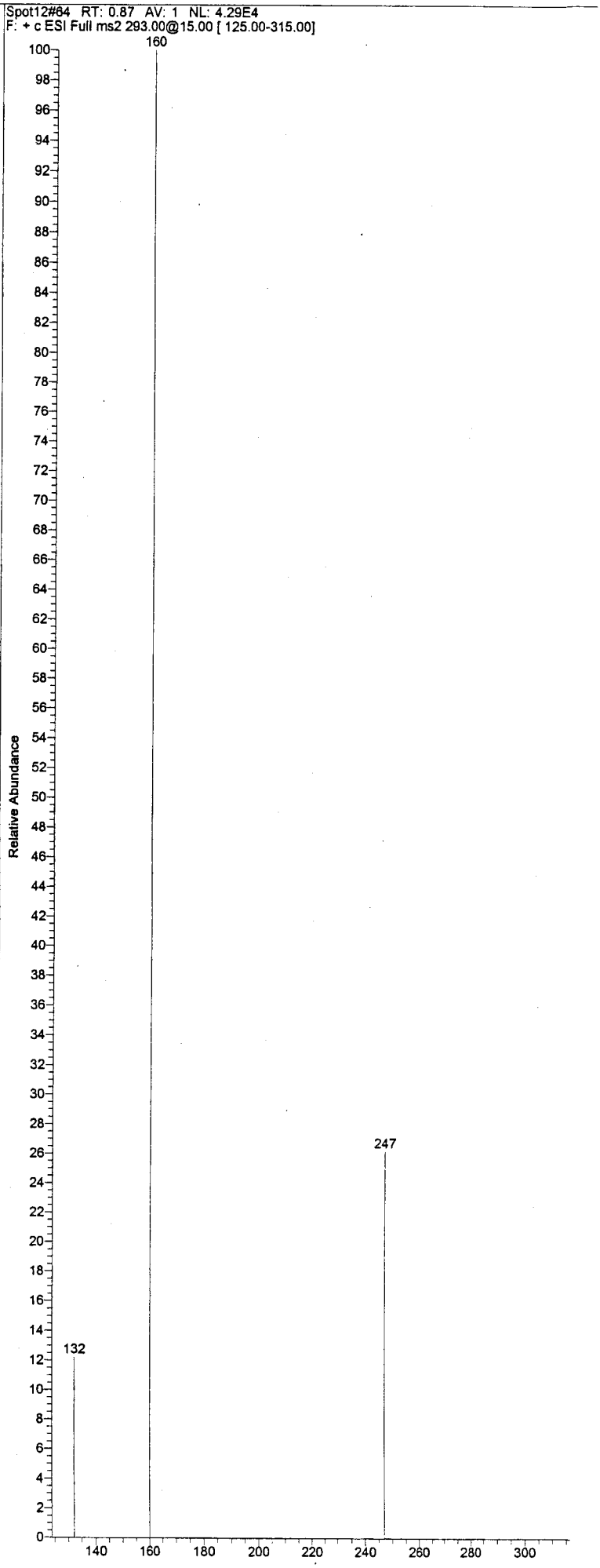
Ex 446
(465)

Spot11#64 RT: 0.87 AV: 1 NL: 0
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



C:\Xcalibur\...\Spot12 02/15/07 01:46:44 5 uL EDTA + C





Ex 446
(466)

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

ment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT01	1	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: JC1neg

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT02	01	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			1	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(467)

JDP
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

ment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT03	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT04	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JhB
2/15/07

Ex 446
(468)

Sequence---CT_Spot_neg.sld [Open]

Sample Name: JC2neg

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT05	02	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			2	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Negative Blood extract

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT06	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(469)

JWP
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT07	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: JC3neg

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT08	03	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			3	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JBB
2/15/07

Ex 446
(470)

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT13	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: JC5neg

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT14	05	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			5	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(471)

JMB
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: JC4neg

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT11	04	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			4	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT12	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(472)

JbB
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT09	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Negative Blood extract

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT10	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JDP
2/15/07

Ex 446
(473)

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT15	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT16	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(474)

JDP
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: JC6neg

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT17	06	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			6	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT18	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(475)

JDB
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT19	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: JC7neg

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT20	07	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			7	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JDP 2/15/07

Ex 446 (476)

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT21	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT22	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JDB 2/15/07

Ex 446 (477)

Sequence---CT_Spot_neg.sld [Open]

Sample Name: JC8neg

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT23	08	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			8	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Negative Blood extract

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT24	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

JOB
2/15/07

Ex 446
(475)

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT25	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: JC9neg

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT26	09	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			9	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(479)

JDP
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT27	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT28	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(480)

JbB
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: JC10neg

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT29	10	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			10	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT30	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(481)

JHP
2/15/17

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT31	11	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: Pos. Control EDTA blood ext.

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT32	12	C:\Xcalibur\Data\EDTA\Brewer\CT_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(482)

JDP
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	CT33	11	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 1uL EDTA+ A

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	13	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			13	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(483)

JDP
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	11	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 1uL EDTA+ B

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	14	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			14	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(484)

JMB
2/15/17

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	11	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 1uL EDTA+ C

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	15	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			15	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(485)

JPP
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	11	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 5uL EDTA+ A

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	16	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			16	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(486)

JDP
2/16/17

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	11	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 5uL EDTA+ B

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	17	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			17	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(487)

JDP
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	11	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 5uL EDTA+ C

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	18	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			18	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(488)

JnP
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	11	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 10uL EDTA+ A

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	19	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			19	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(489)

JMP
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	11	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 10uL EDTA+ B

Comment:

Study:
Client:
Laboratory:
Company:
Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	20	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			20	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(490)

JDB
2/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

C ment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	11	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Sample Name: 10uL EDTA+ C

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	12	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			12	5.0		0.000

Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

****

Ex 446
(491)

JDP
1/15/07

Sequence---CT_Spot_neg.sld [Open]

Sample Name: Negative Blood extract

Comment:

Study:

Client:

Laboratory:

Company:

Phone:

Sample Type	File Name	Sample ID	Path
Unknown	Spot01	11	C:\Xcalibur\Data\EDTA\Brewer\Spot Size_Negative

Inst Method	Proc Method	Cal File	Position	InjVol	Level	Sample Wt
C:\Xcalibur\methods\EDTA_Neg_Swabs			11	5.0		0.000

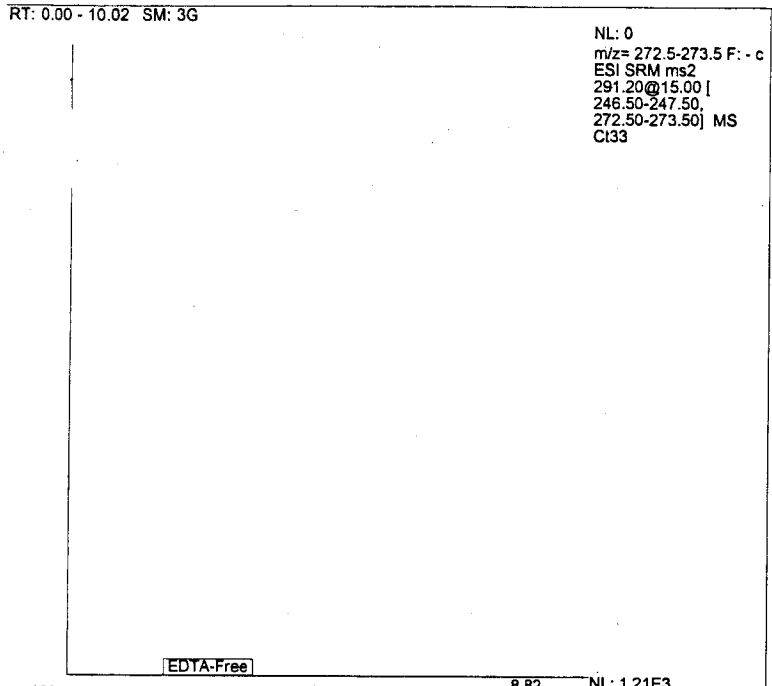
Sample Vol	ISTD Amt	Dil Factor
0.000	0.000	1.000

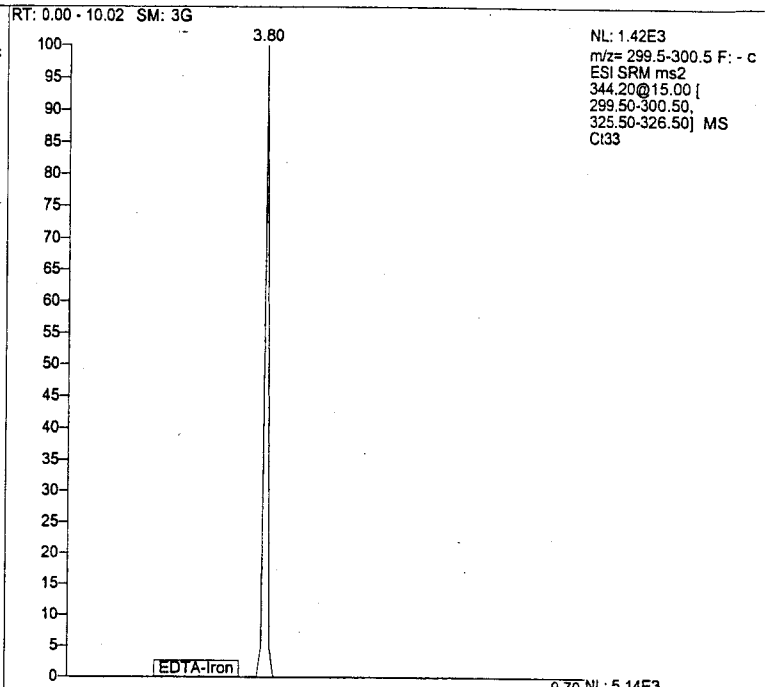
****

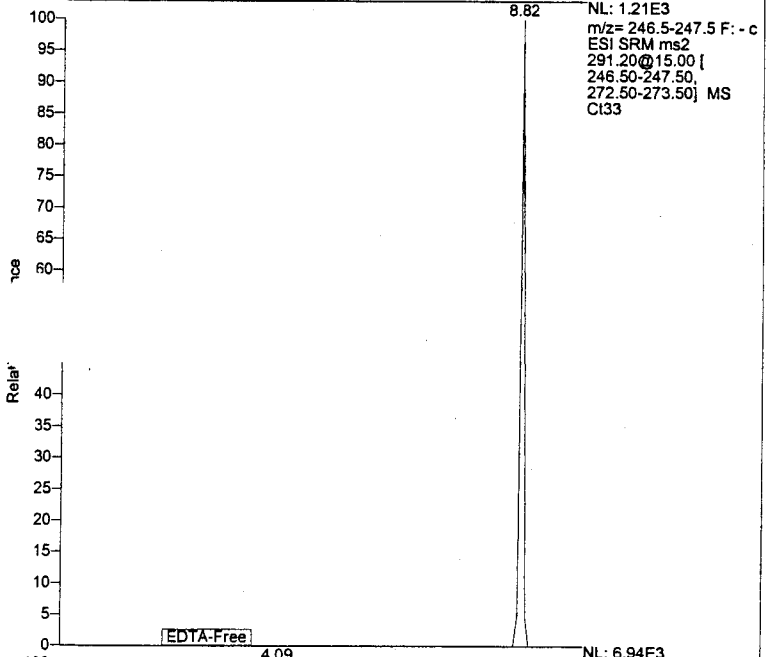
Ex 446
(492)

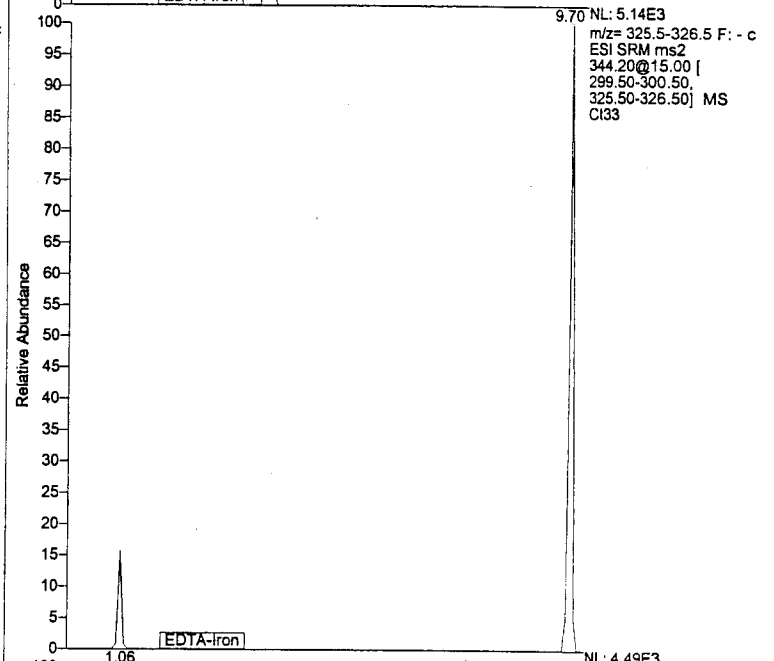
TPB
2/15/07

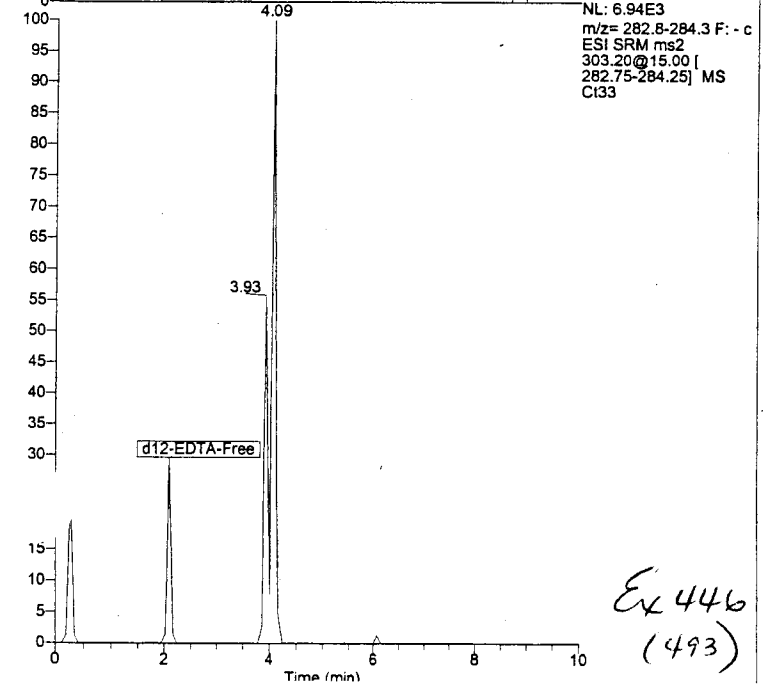
C:\Xcalibur\...\Spot Size_Negative\Ct33 02/16/07 03:47:38 Negative Blood extract 3pβ

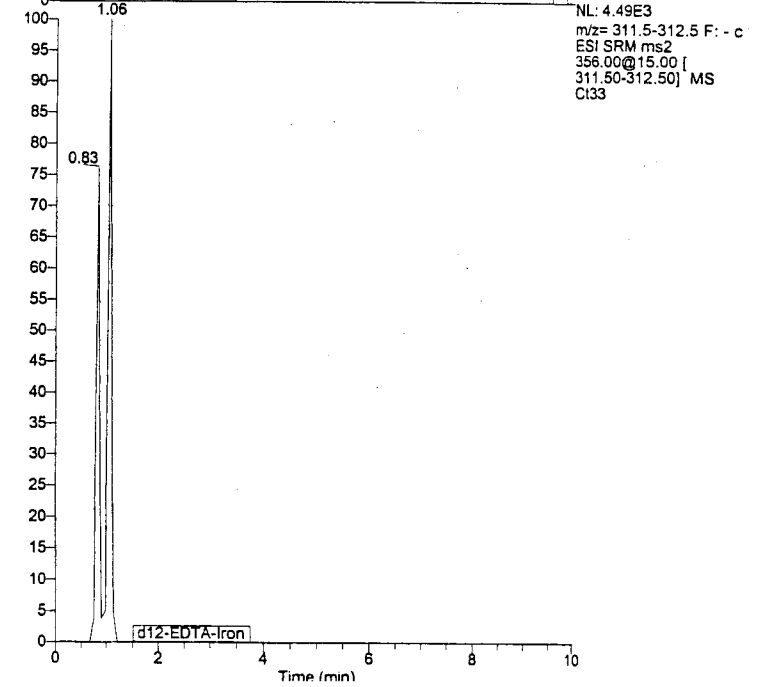






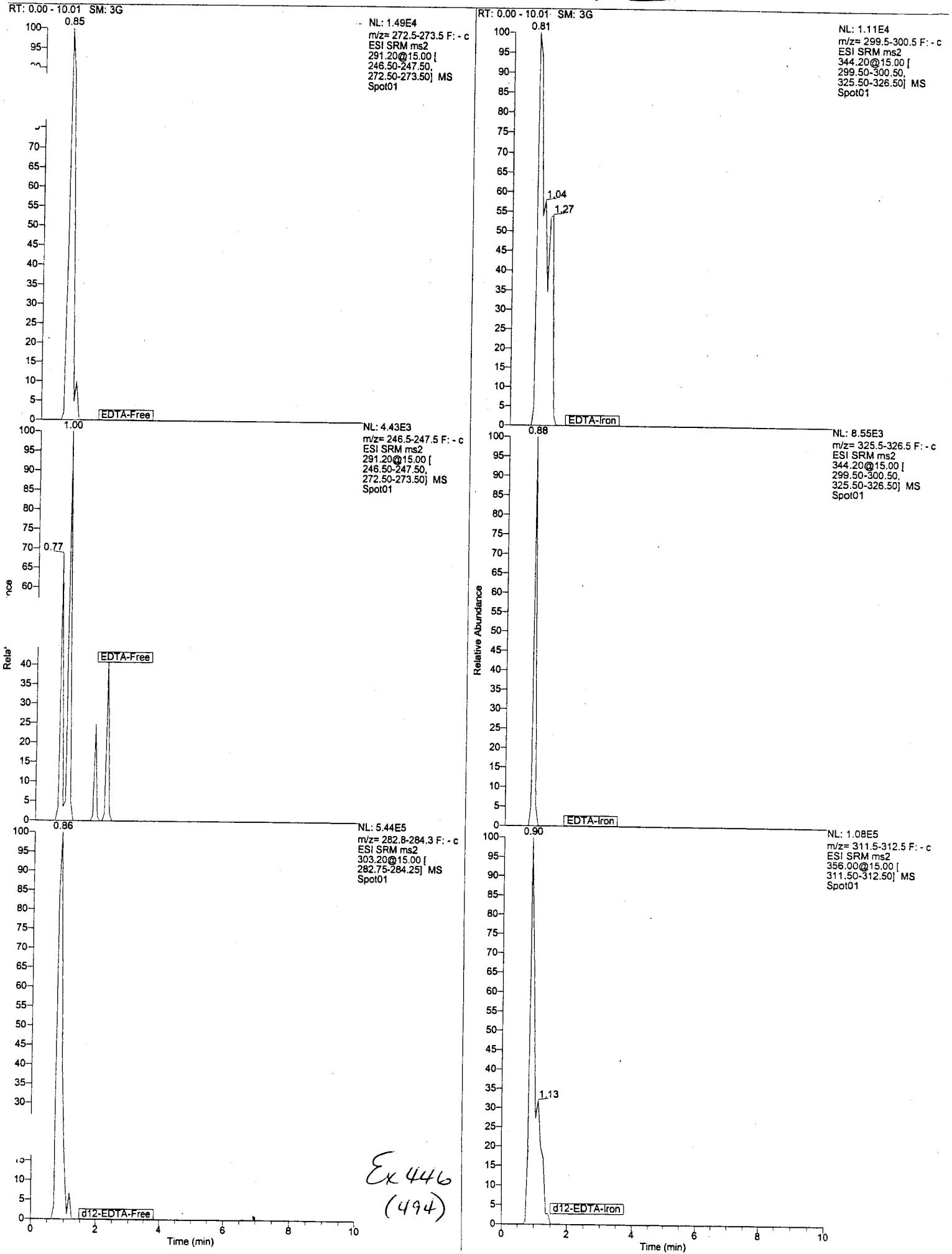






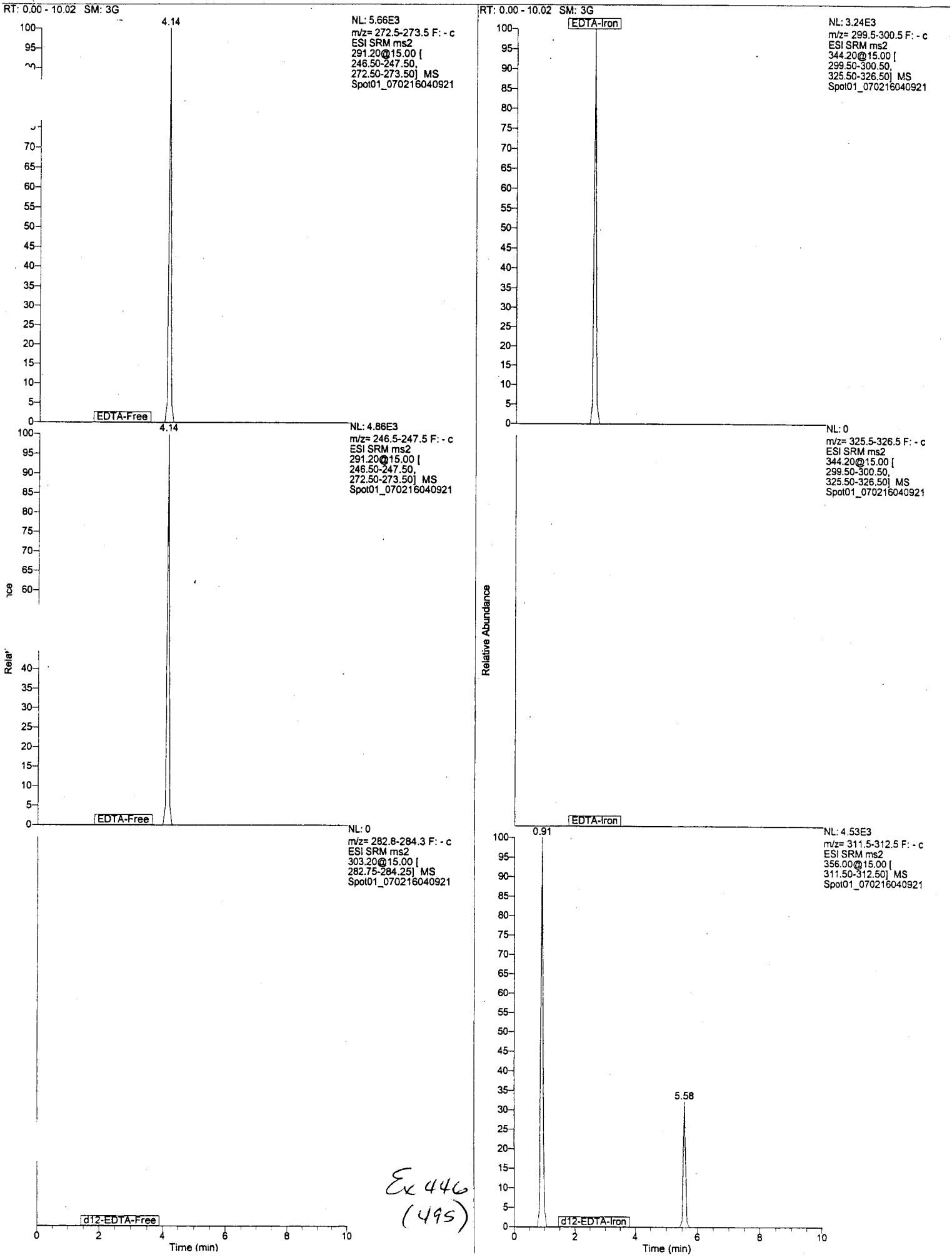
Ex 446
(493)

C:\Xcalibur\...\Spot01 02/16/07 03:58:32 1uL EDTA+ A JDB



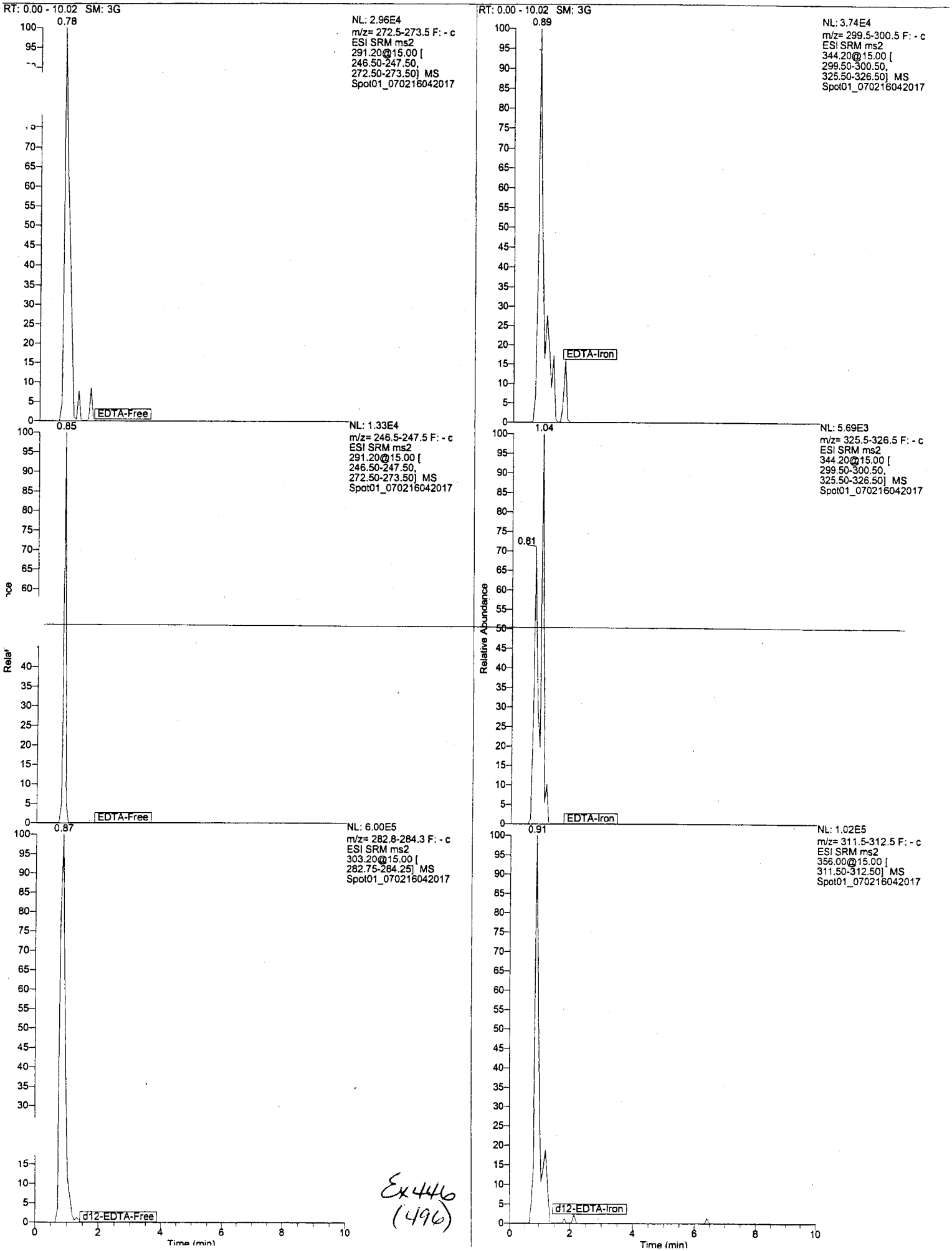
Ex 446
(494)

C:\Xcalibur\...\Spot01_070216040921 02/16/07 04:09:21 Negative Blood extract DvB

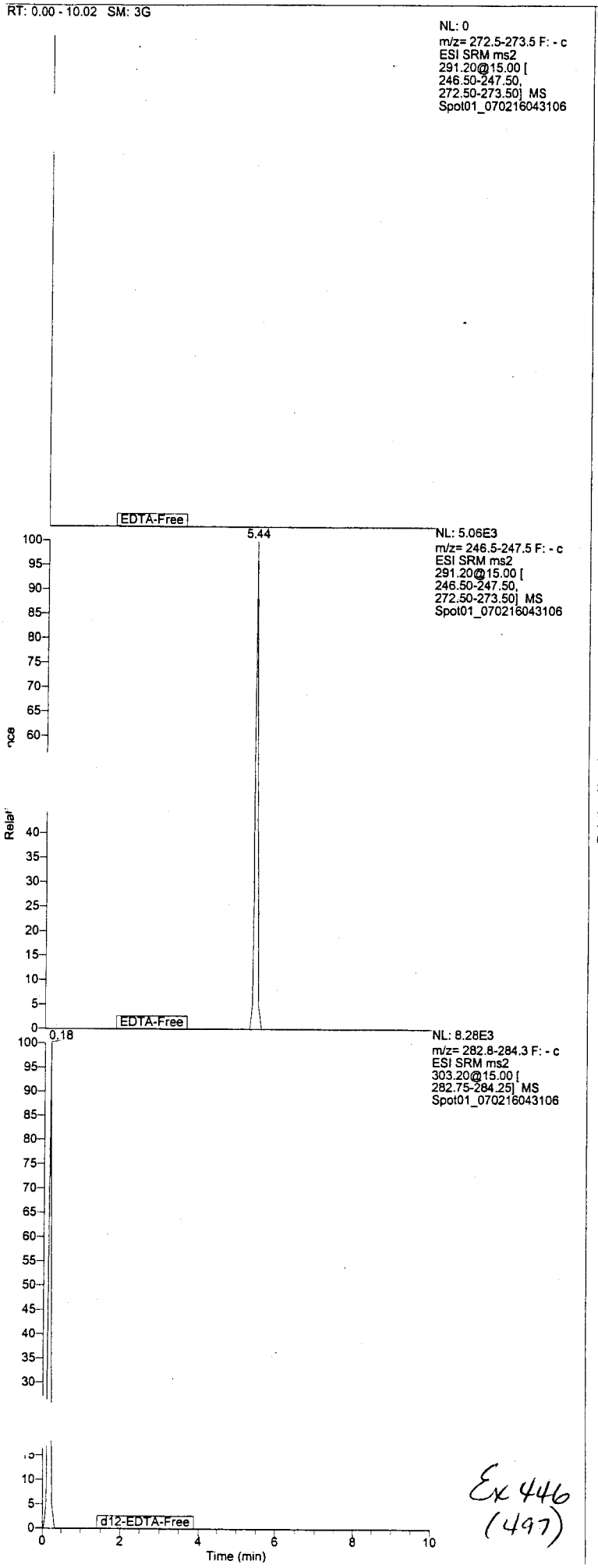


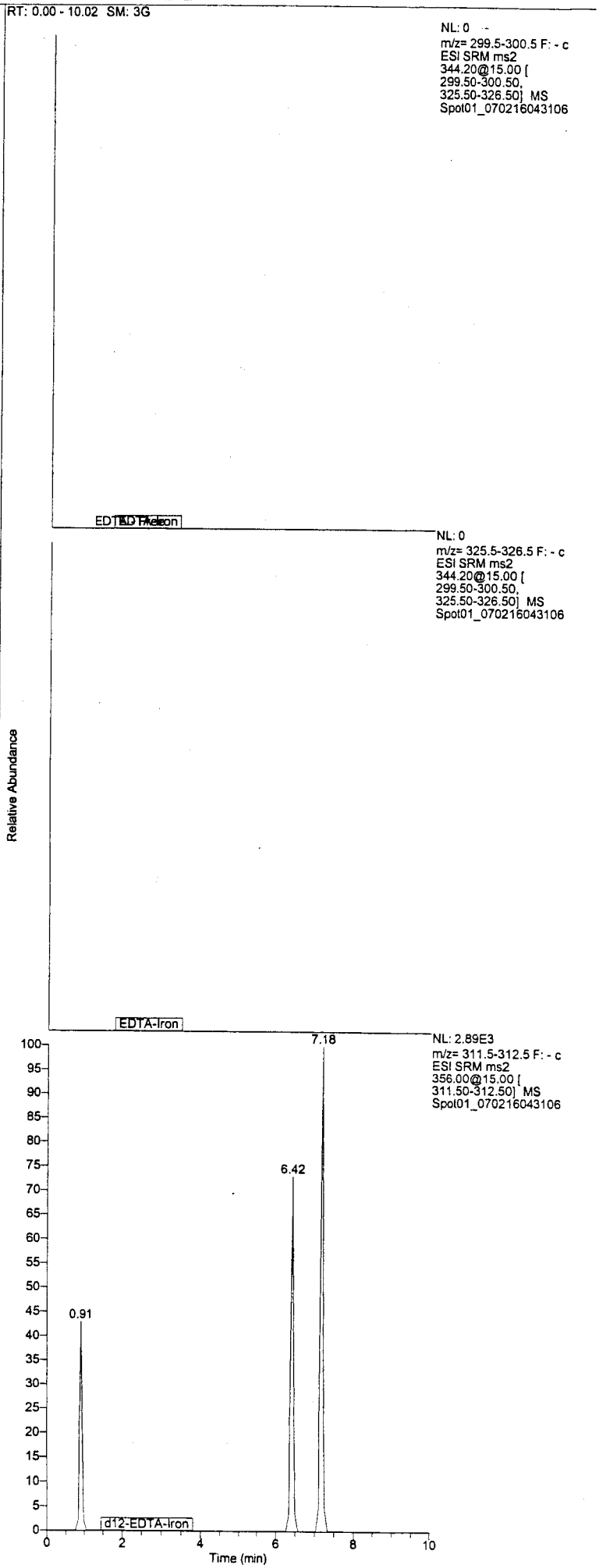
Ex 446
(495)

C:\Xcalibur\...\Spot01_070216042017 02/16/07 04:20:17 1uL EDTA+ B JnB



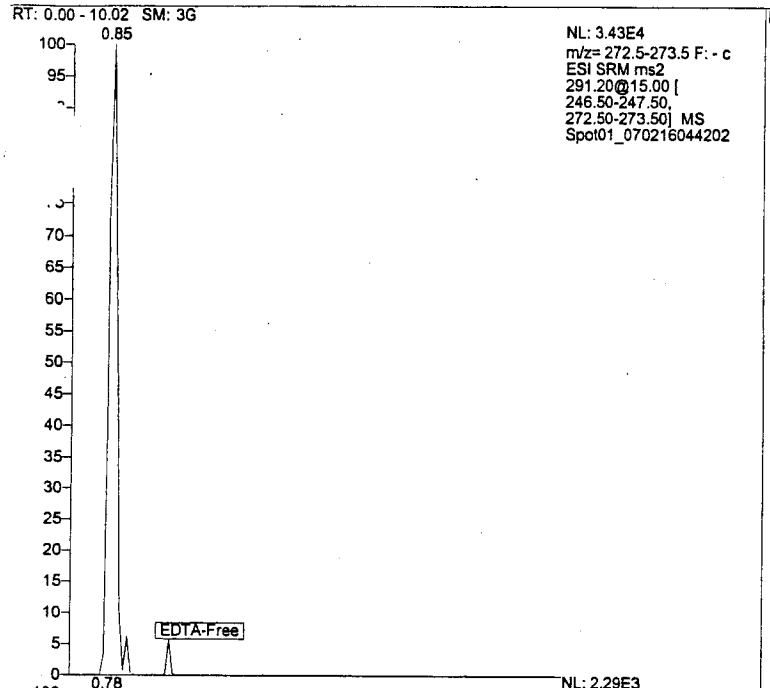
Ex 446
(496)

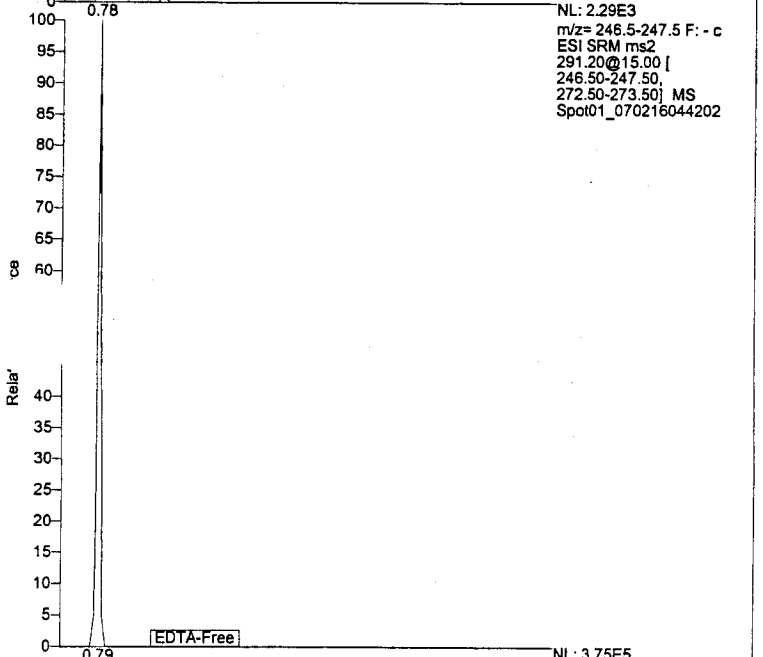

RT: 0.00 - 10.02 SM: 3G
NL: 0
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Spot01_070216043106
EDTA-Free
NL: 5.06E3
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Spot01_070216043106
5.44
EDTA-Free
NL: 8.28E3
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Spot01_070216043106
0.18
d12-EDTA-Free
Time (min)

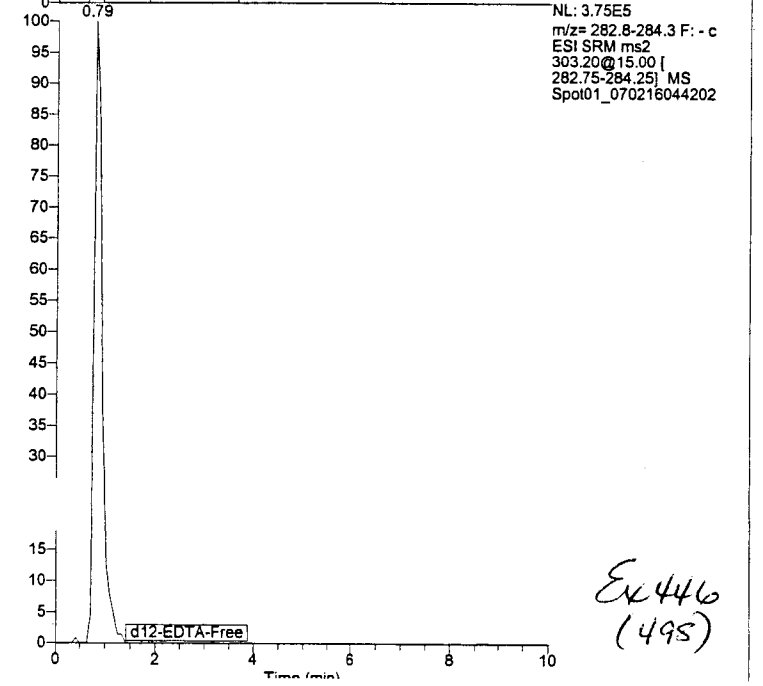

RT: 0.00 - 10.02 SM: 3G
NL: 0
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Spot01_070216043106
EDTA-Iron
NL: 0
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Spot01_070216043106
Relative Abundance
EDTA-Iron
NL: 2.89E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Spot01_070216043106
7.18
6.42
0.91
d12-EDTA-Iron
Time (min)

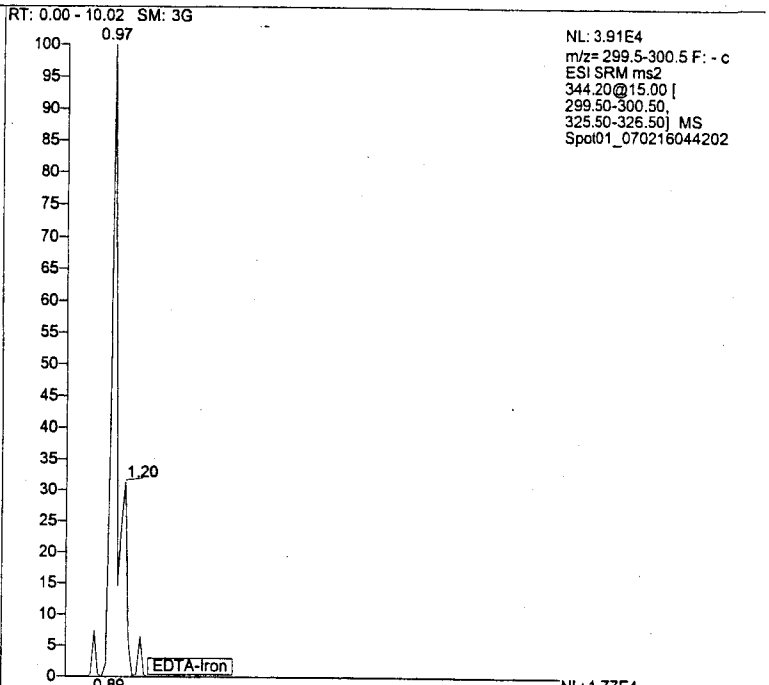
Ex 446
(497)

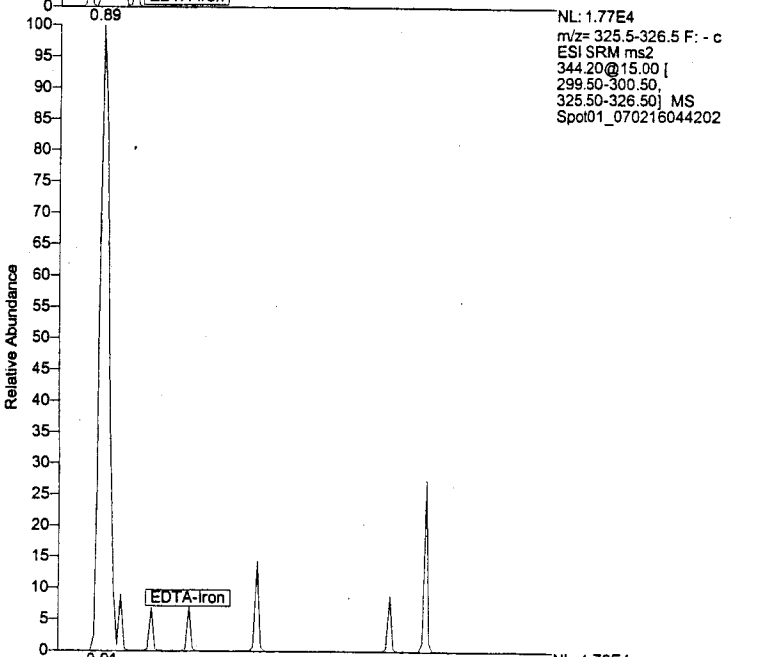
C:\Xcalibur\...\Spot01_070216044202 02/16/07 04:42:02 1uL EDTA+ C JMB

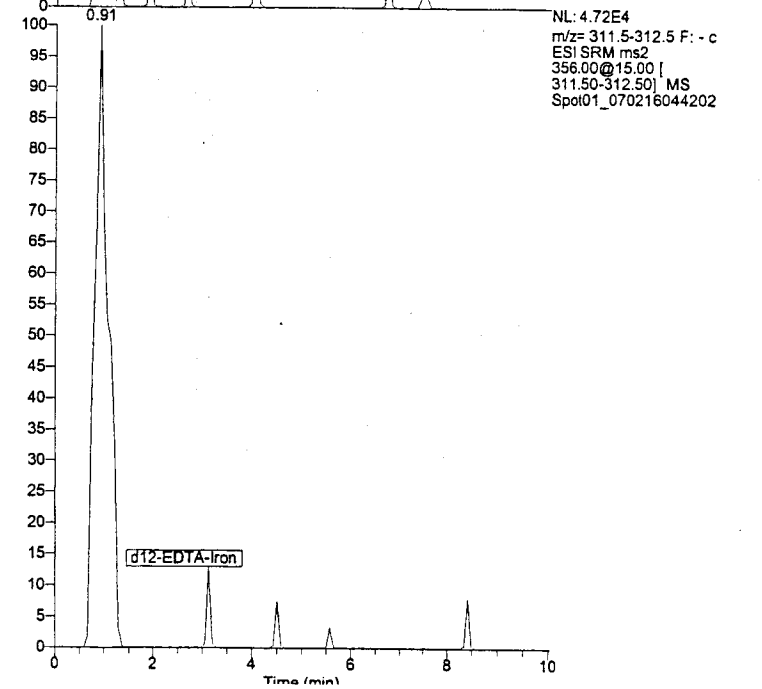






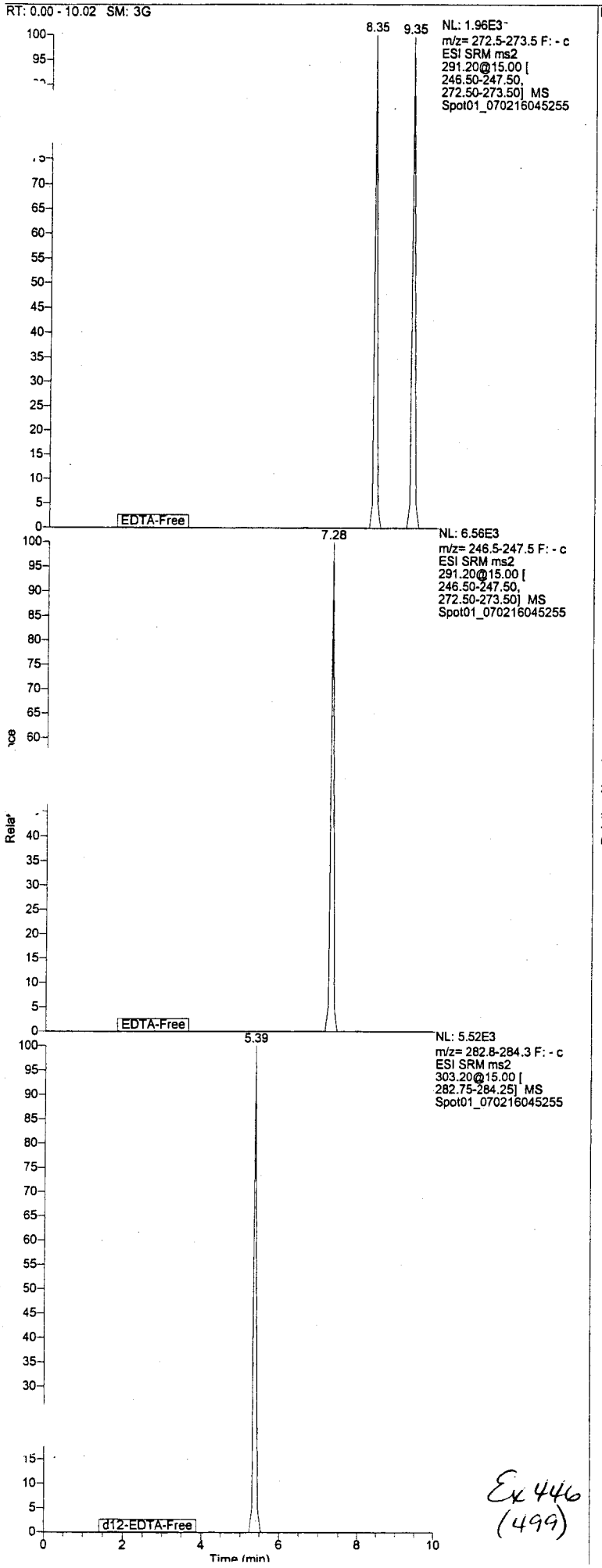


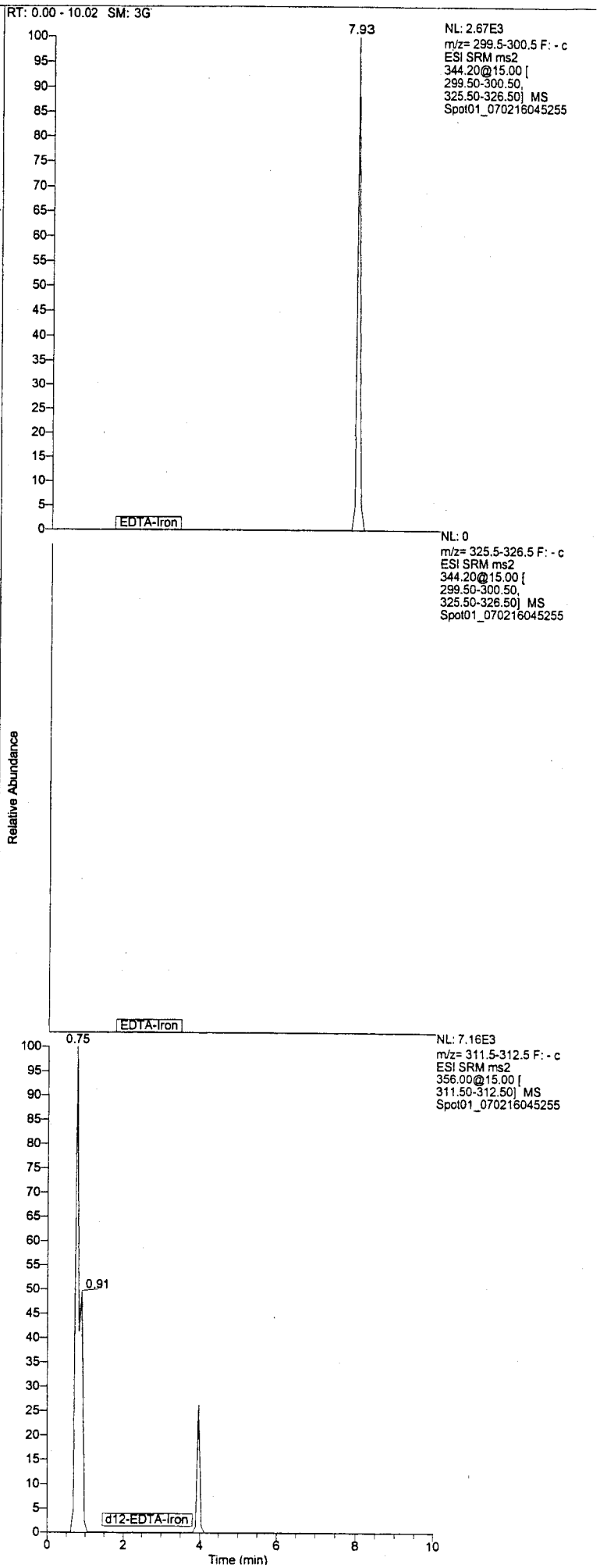




EX446
(495)

C:\Xcalibur\...\Spot01_070216045255 02/16/07 04:52:55 Negative Blood extract JDB

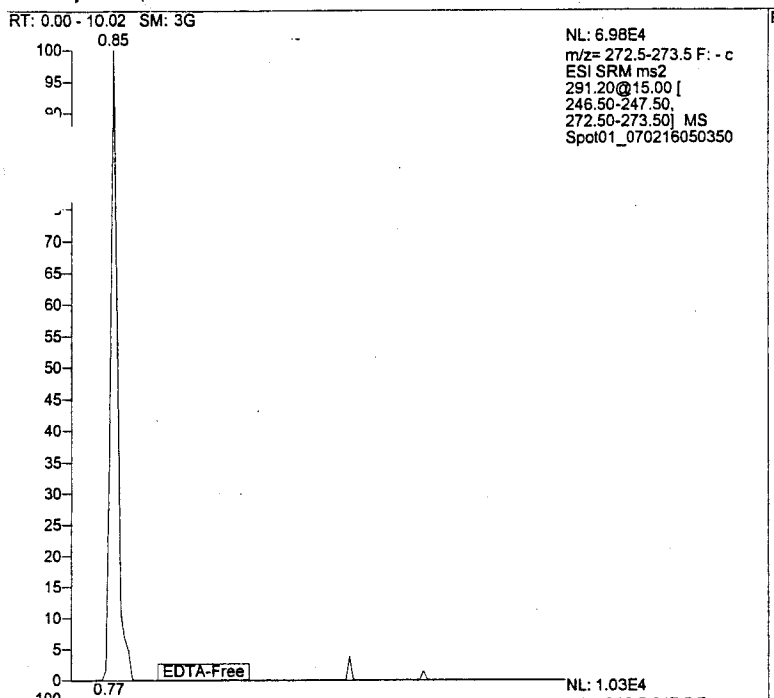


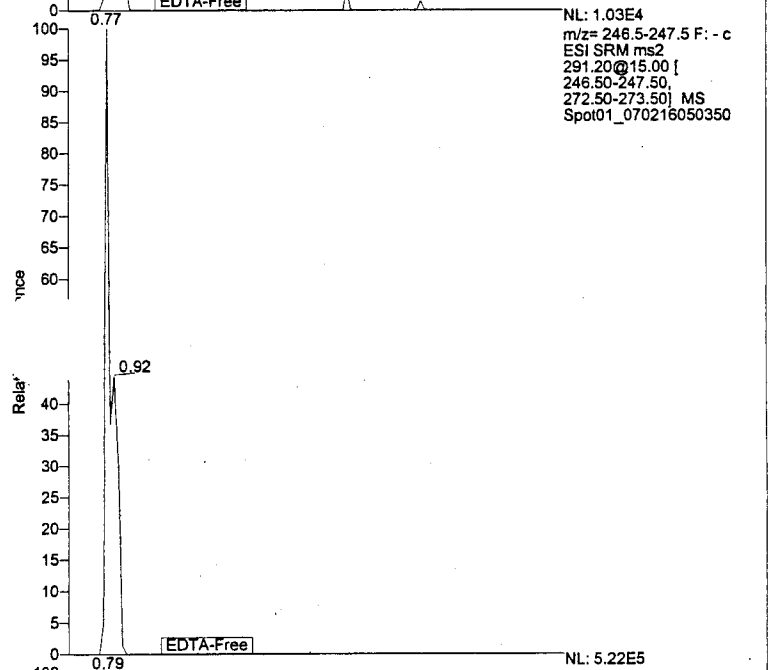


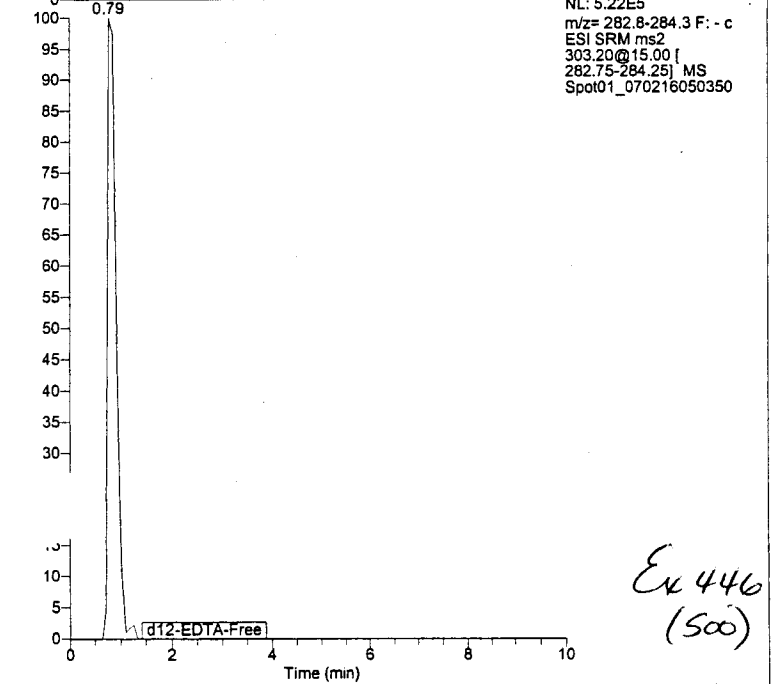
Relative Abundance

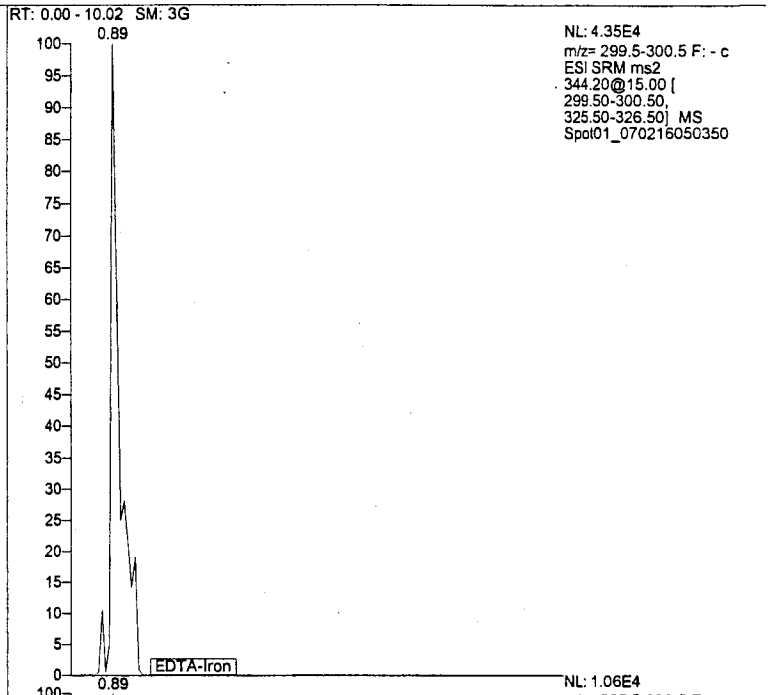
Ex 446
(499)

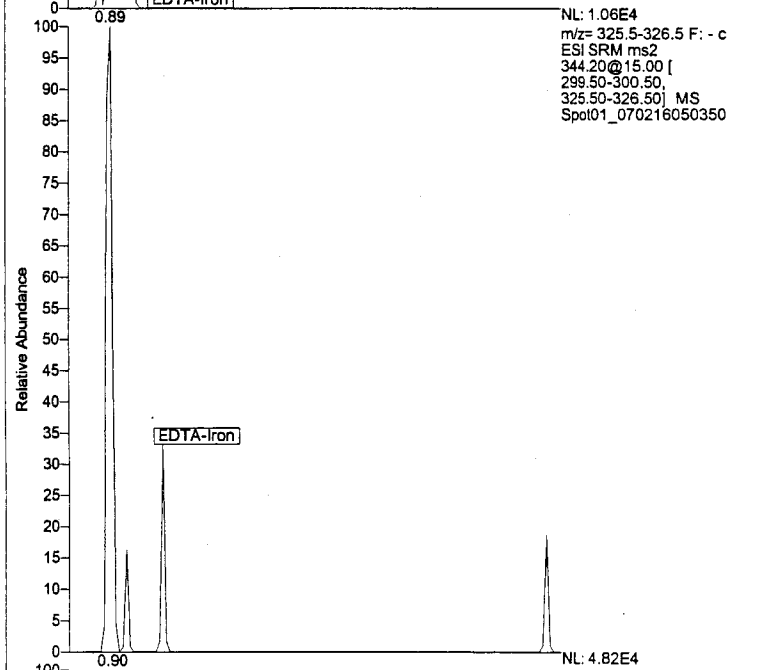
C:\Xcalibur\...\Spot01_070216050350 02/16/07 05:03:50 5uL EDTA+ A JDB

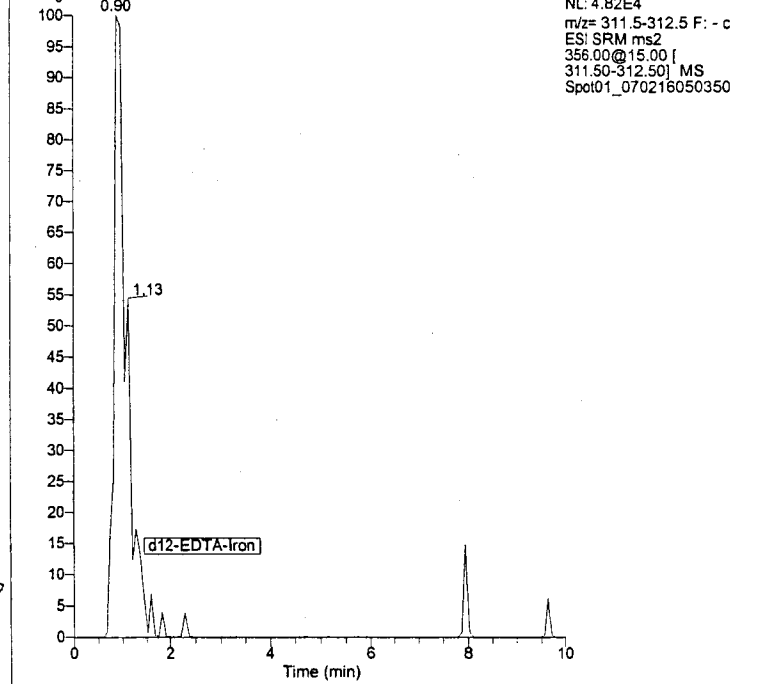




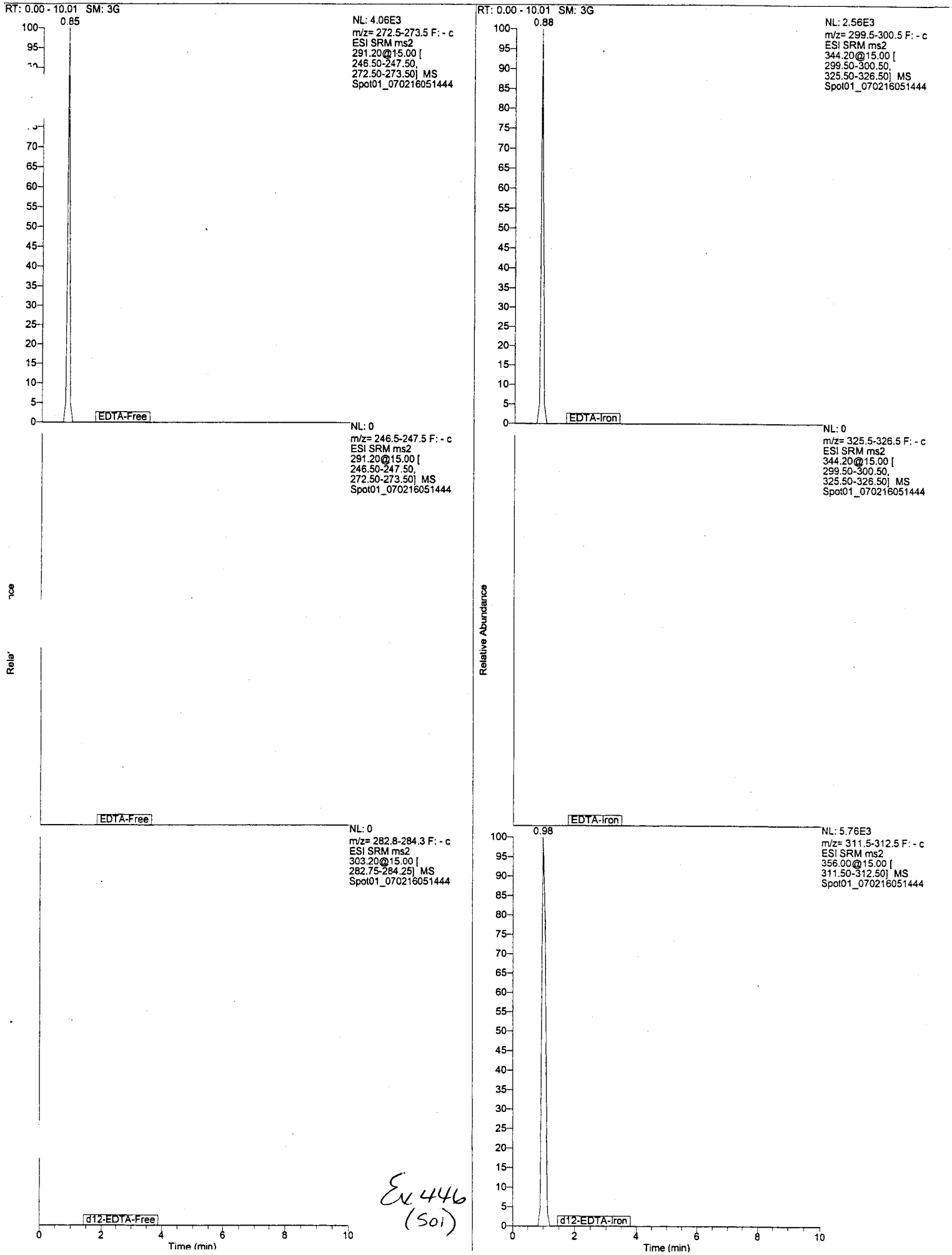






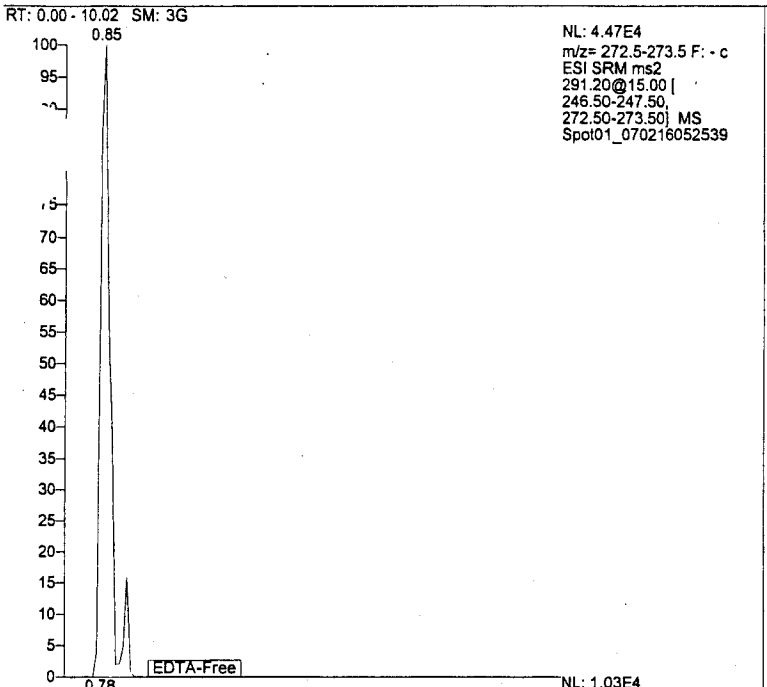


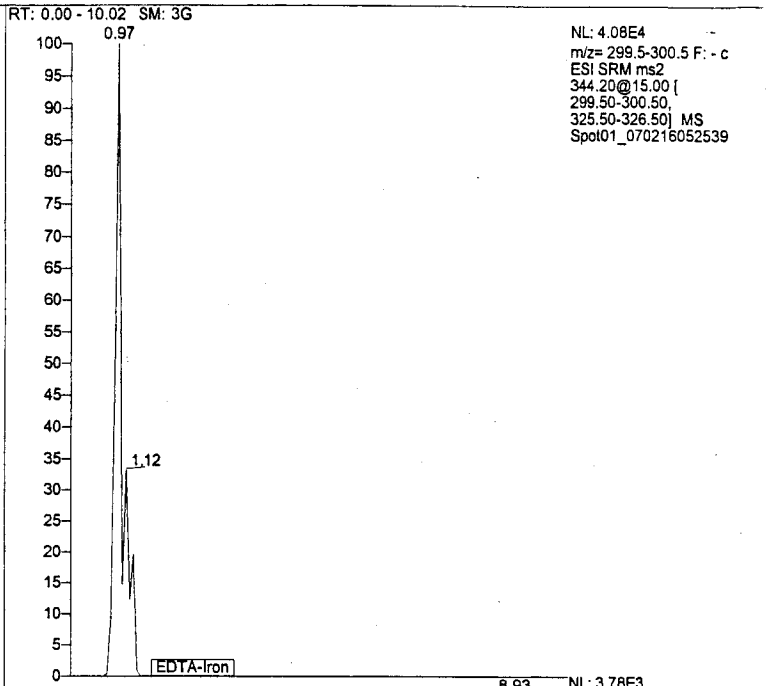
Ex 446
(500)

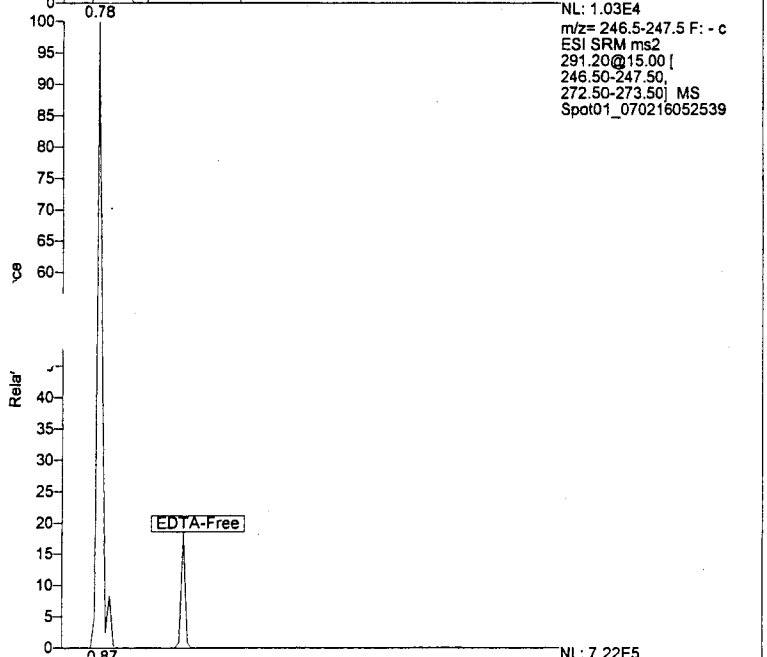

RT: 0.00 - 10.01 SM: 3G
0.85
NL: 4.06E3
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Spot01_070216051444
EDTA-Free
NL: 0
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Spot01_070216051444
EDTA-Free
NL: 0
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Spot01_070216051444
d12-EDTA-Free
Time (min)
RT: 0.00 - 10.01 SM: 3G
0.88
NL: 2.56E3
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Spot01_070216051444
EDTA-Iron
NL: 0
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Spot01_070216051444
EDTA-Iron
0.98
NL: 5.76E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Spot01_070216051444
d12-EDTA-Iron
Time (min)
Relative Abundance

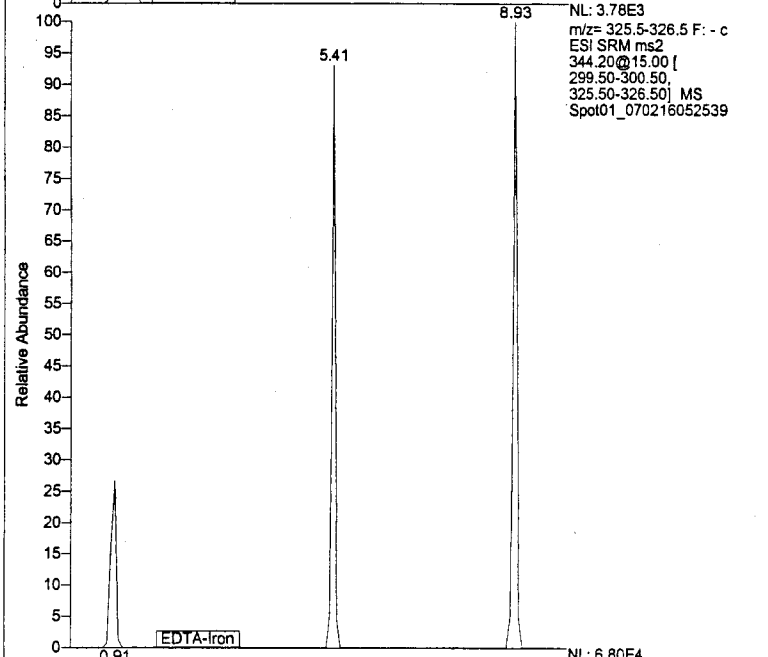
EN 446 (Soi)

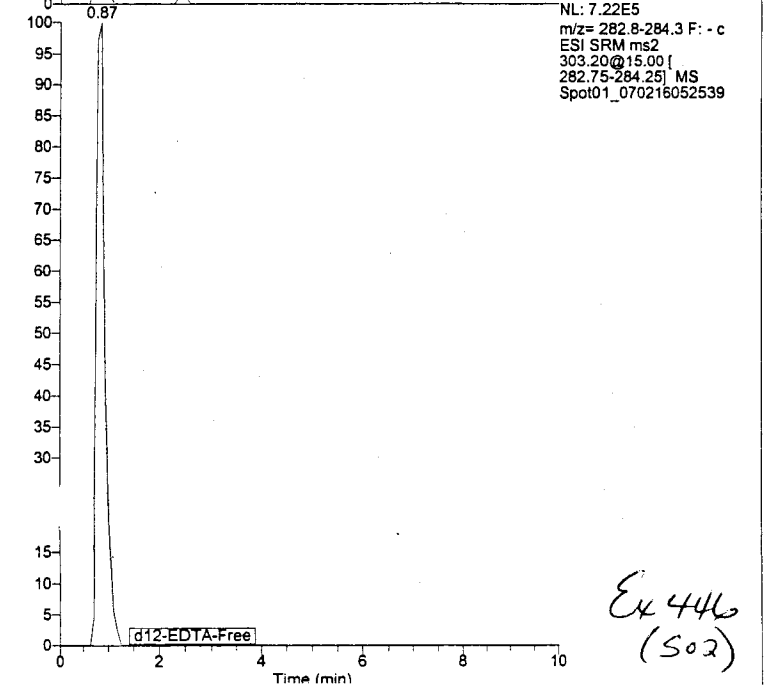
C:\Xcalibur\...\Spot01_070216052539 02/16/07 05:25:39 5uL EDTA+ B JDB

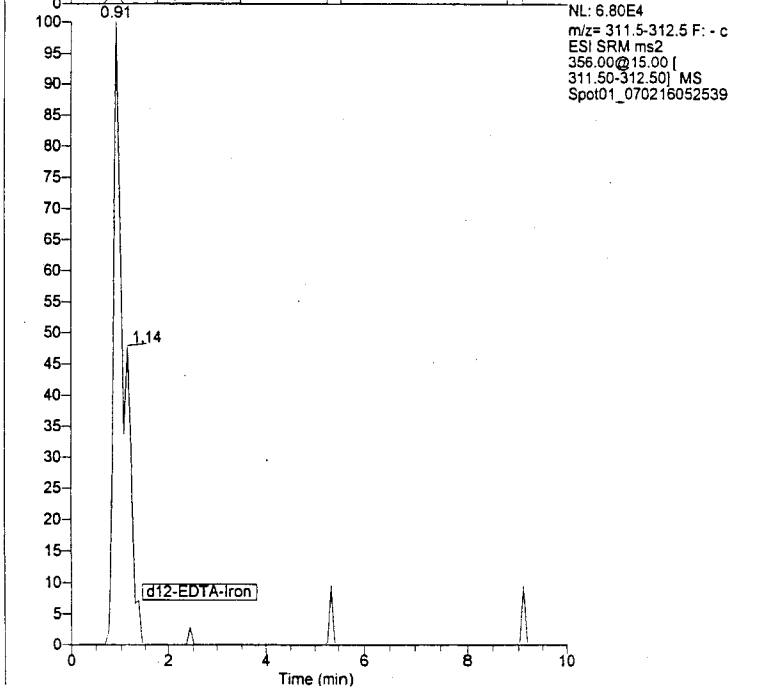






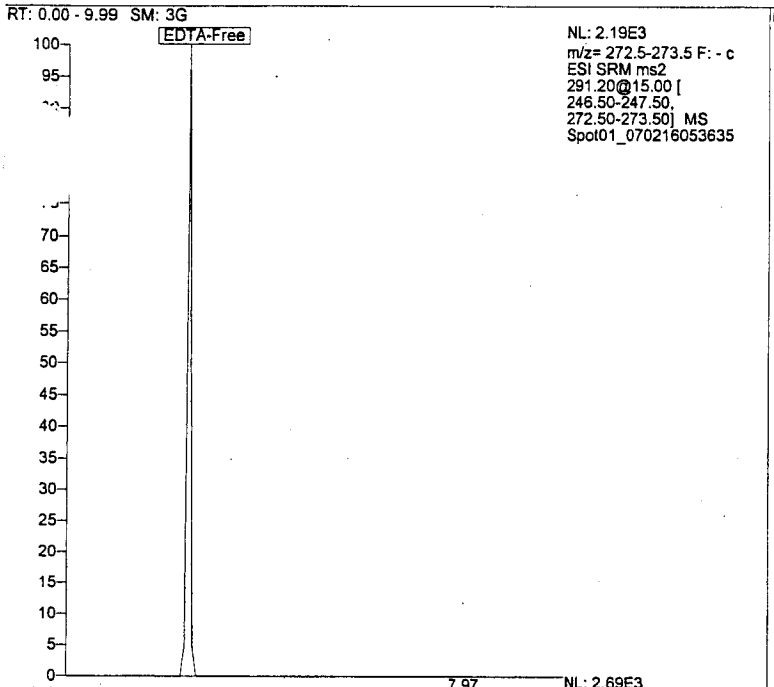


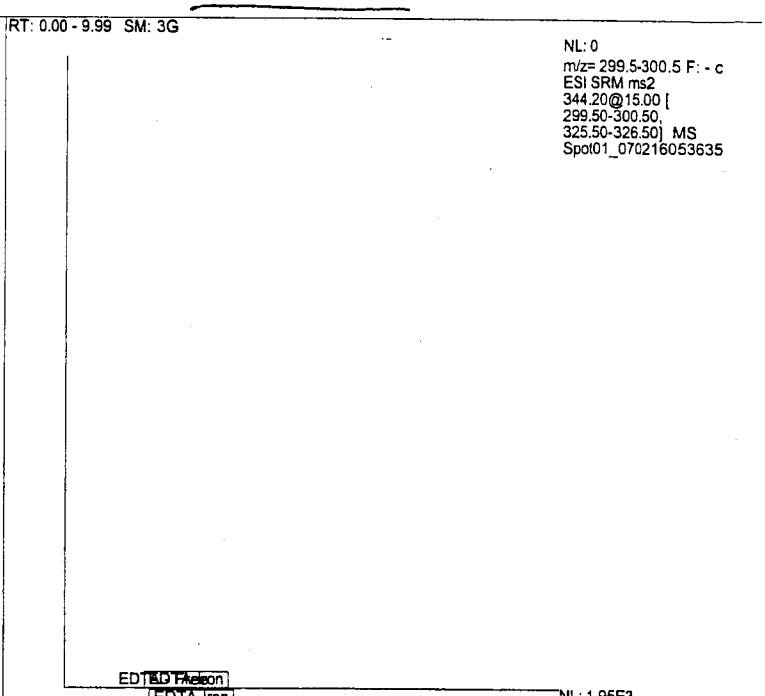


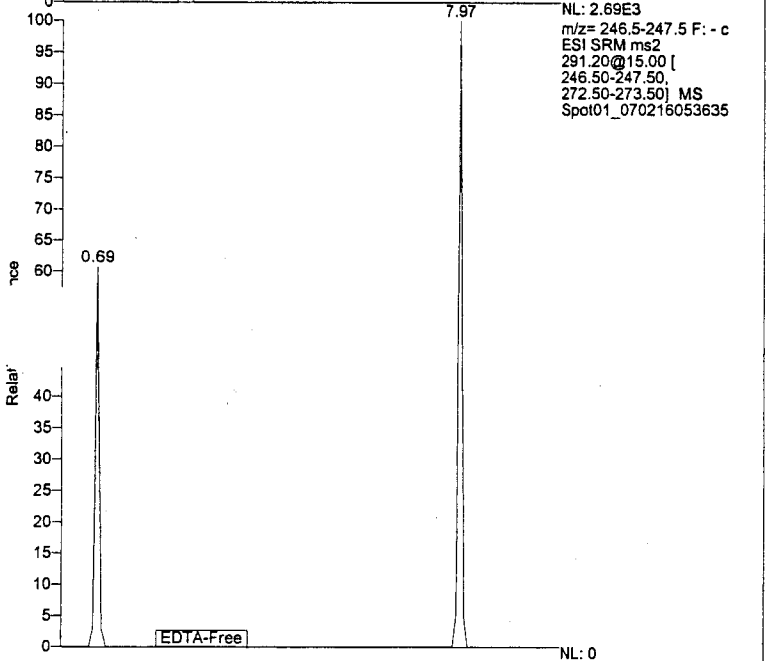


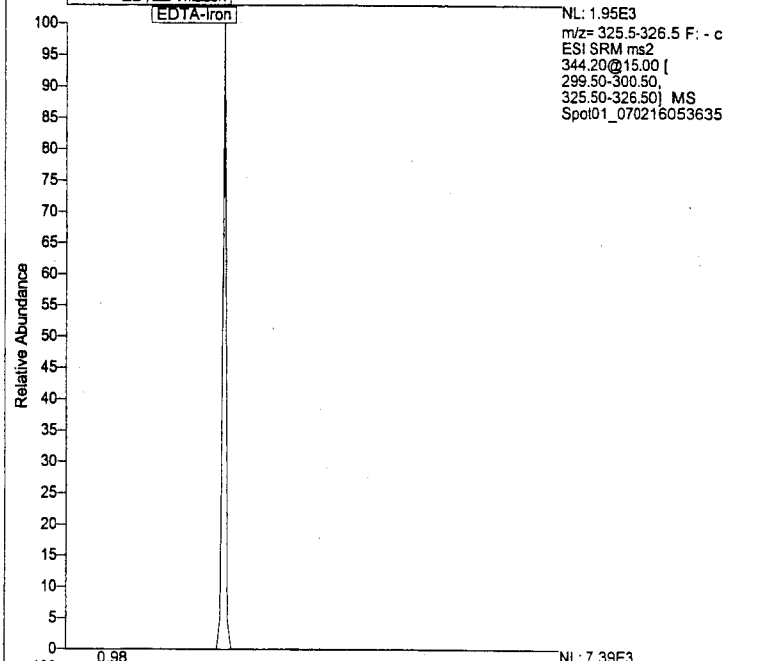
Ex 446 (502)

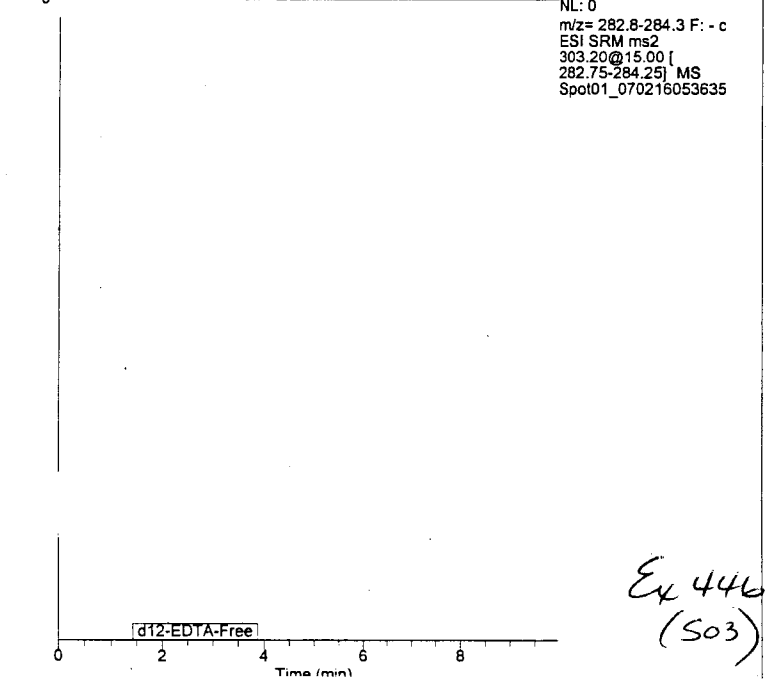
C:\Xcalibur\...\Spot01_070216053635 02/16/07 05:36:35 Negative Blood extract JDB

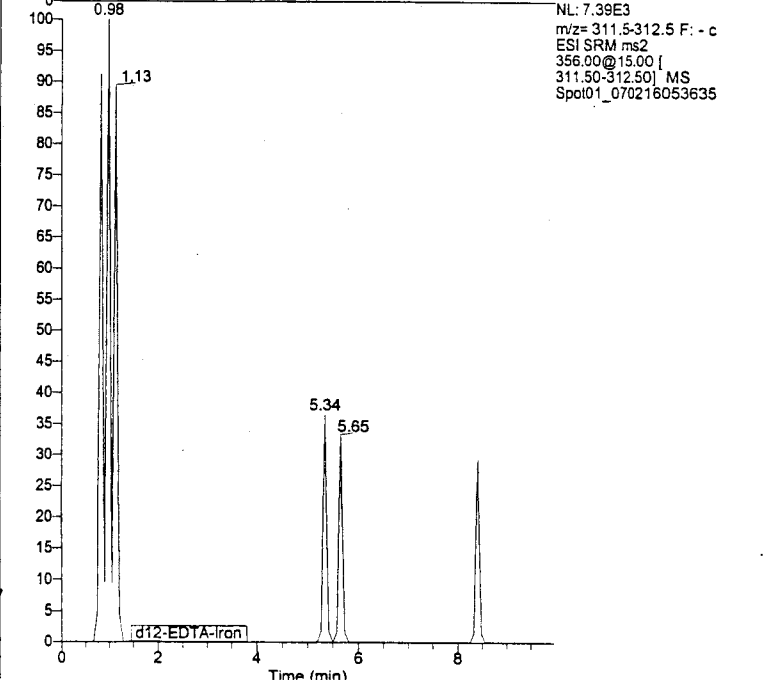






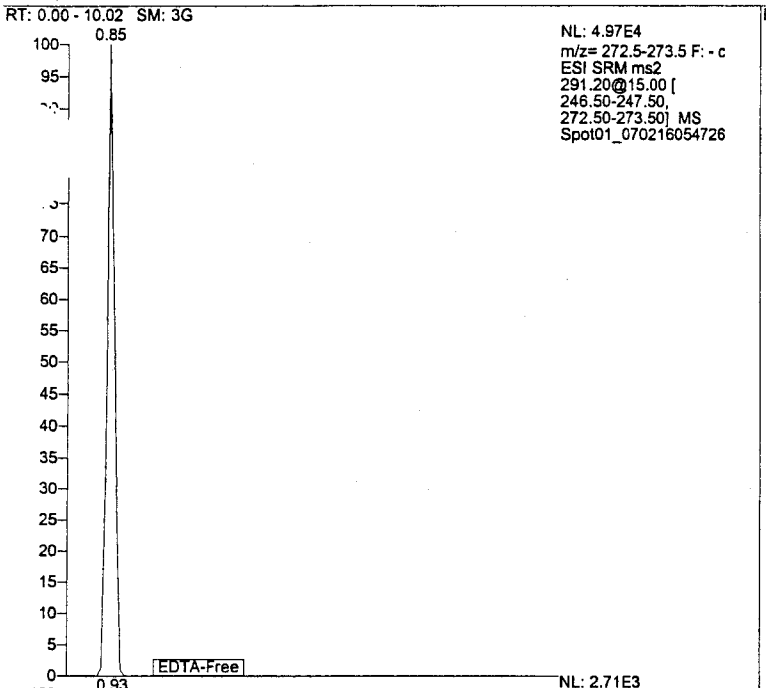


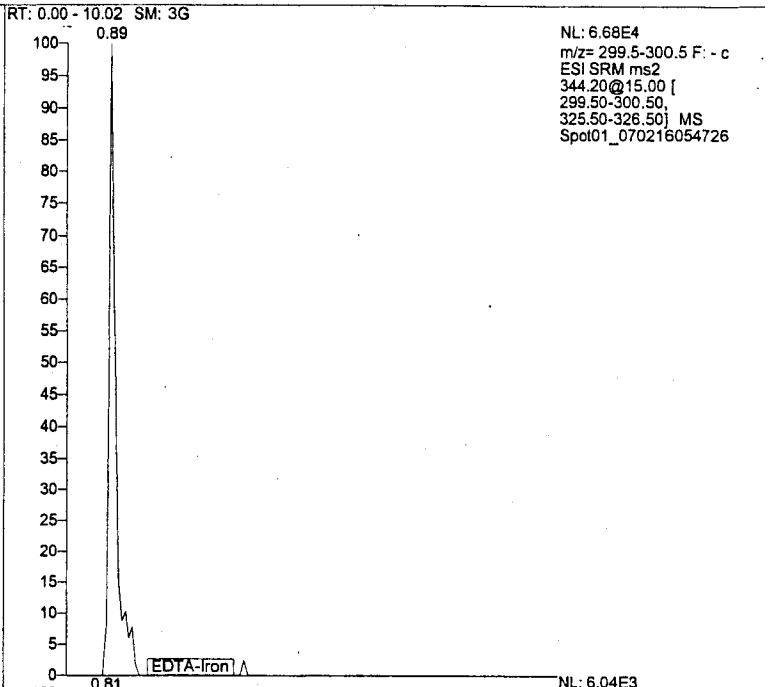


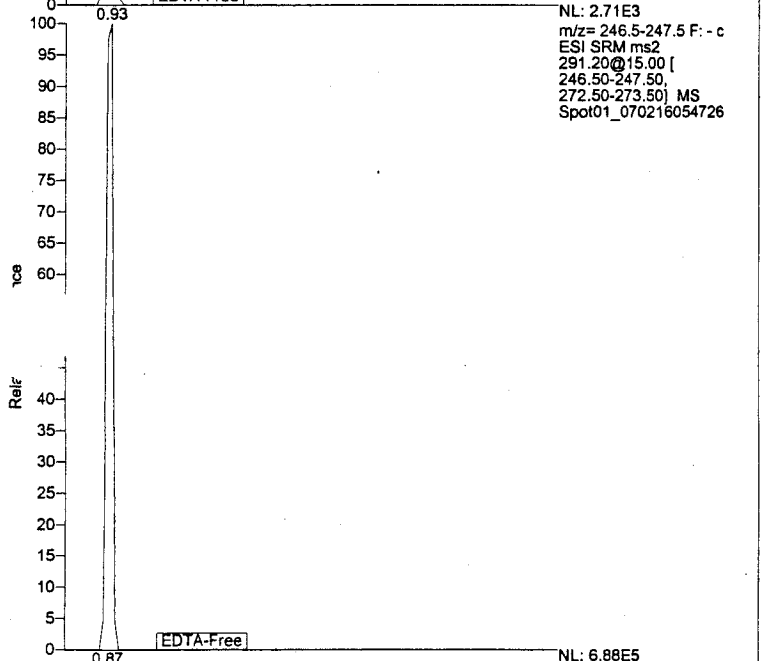


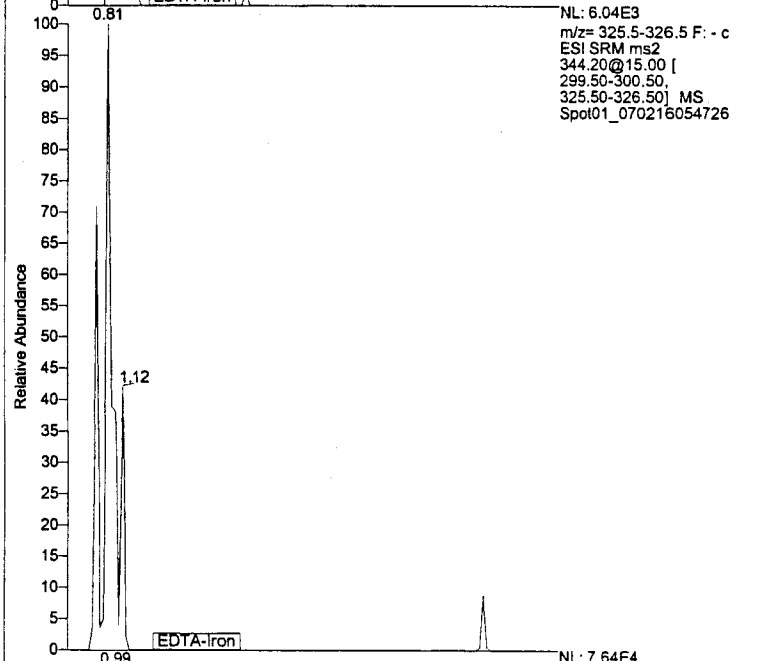
Ex 446
(503)

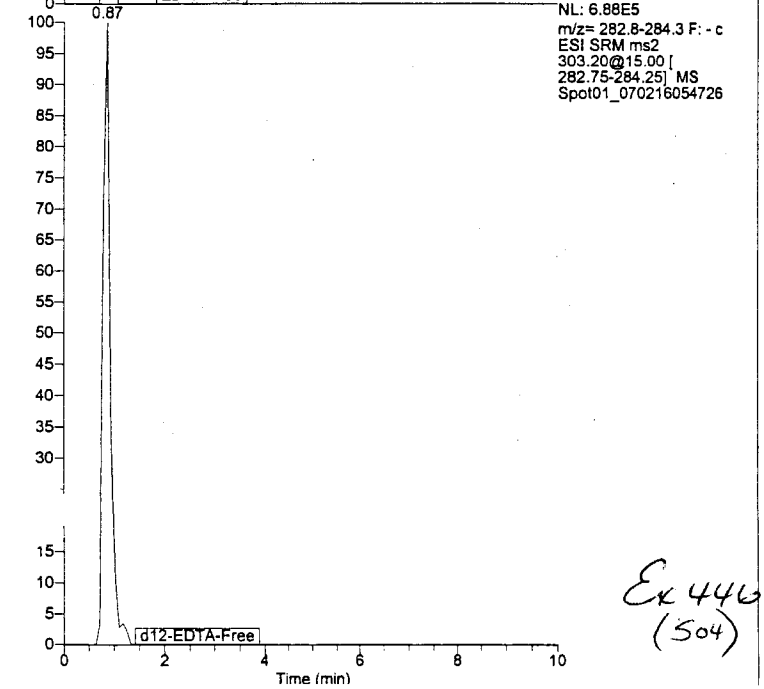
C:\Xcalibur\...\Spot01_070216054726 02/16/07 05:47:26 5uL EDTA+ C JDB

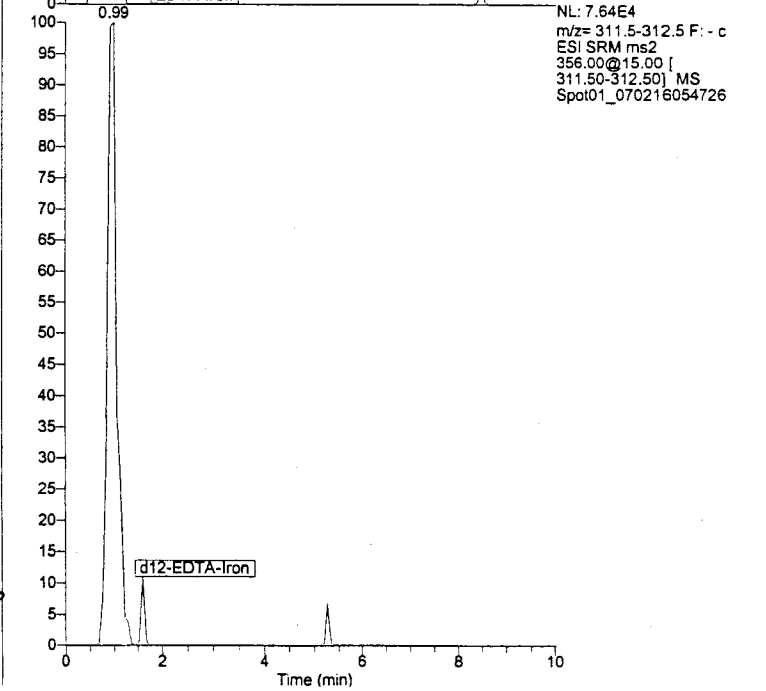






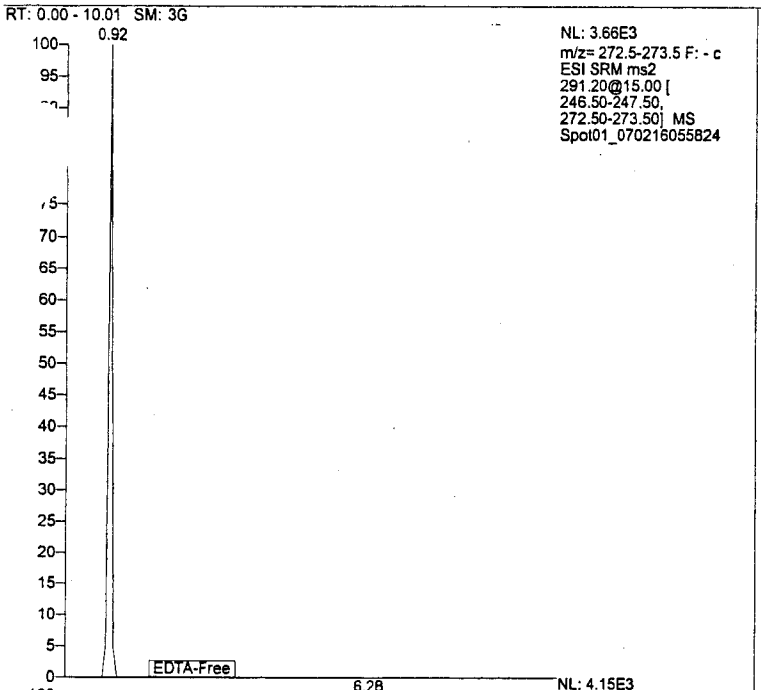


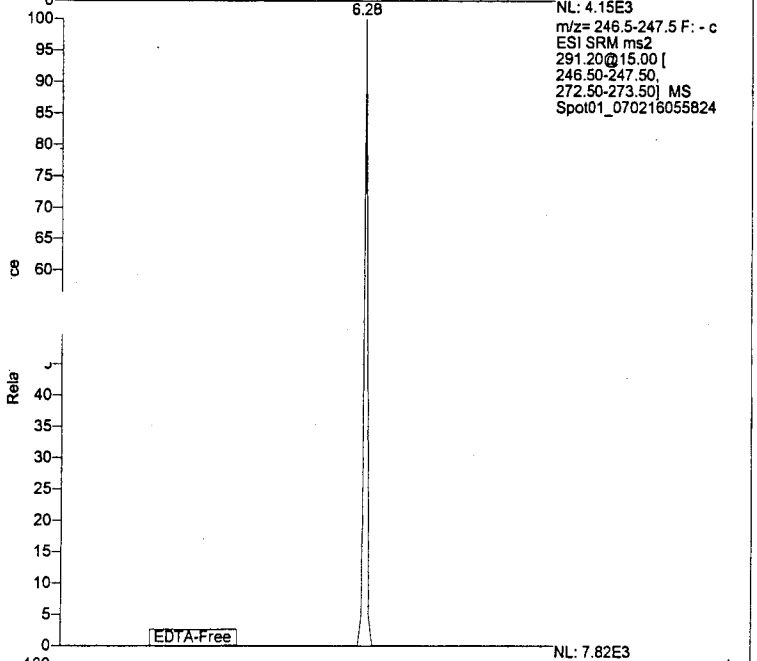


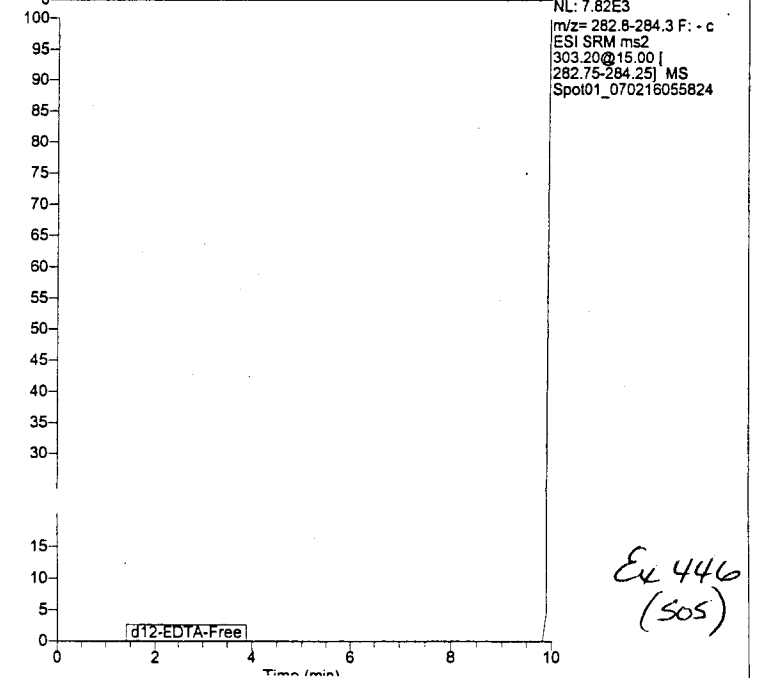


Ex 446
(504)

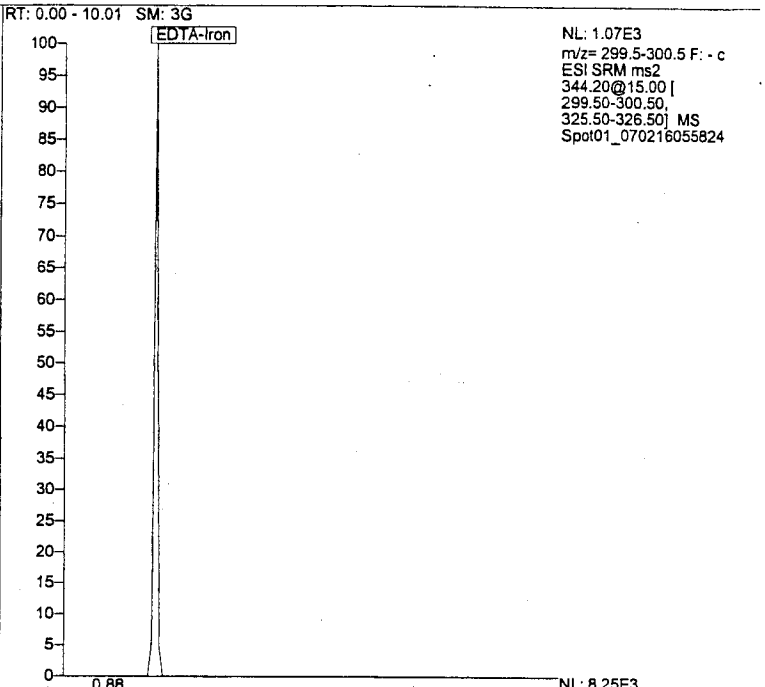
C:\Xcalibur\...\Spot01_070216055824 02/16/07 05:58:24 Negative Blood extract DJB

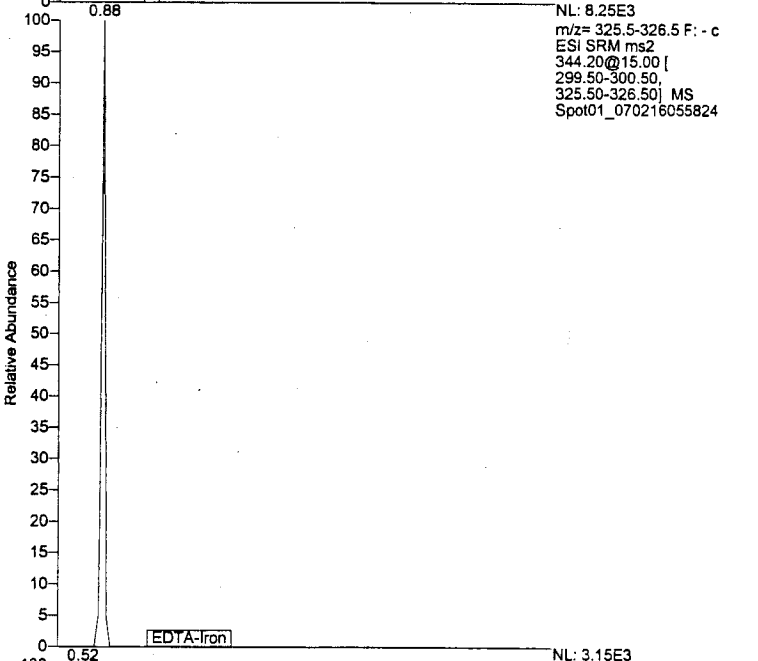


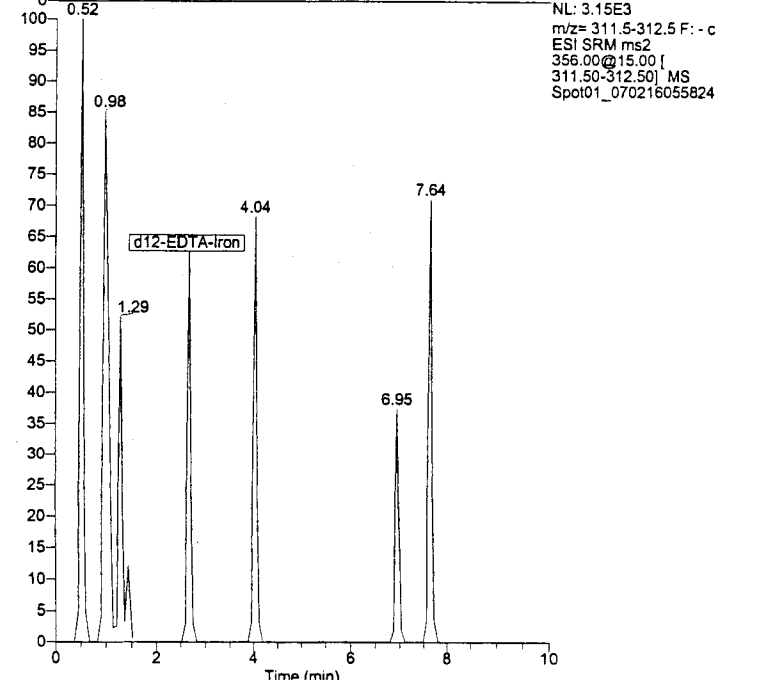




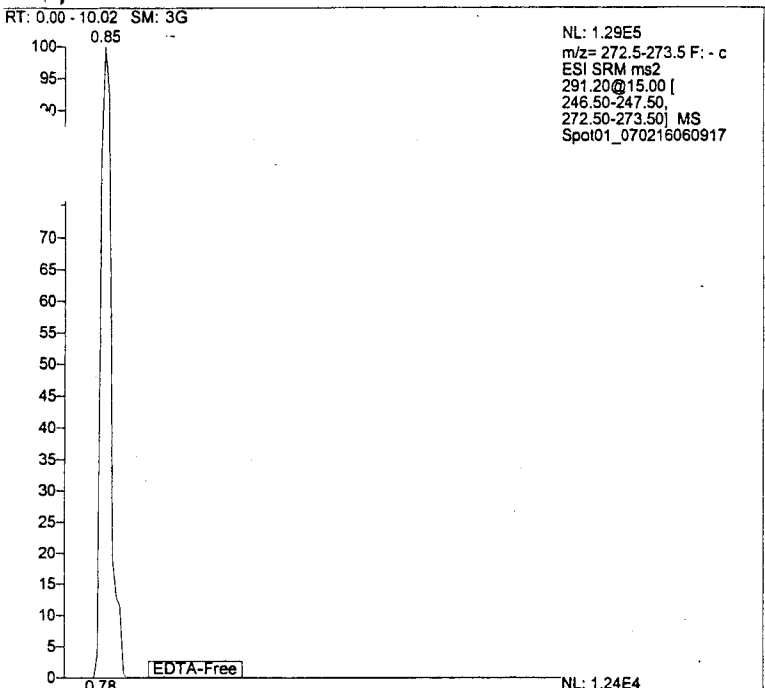
EL 446 (505)

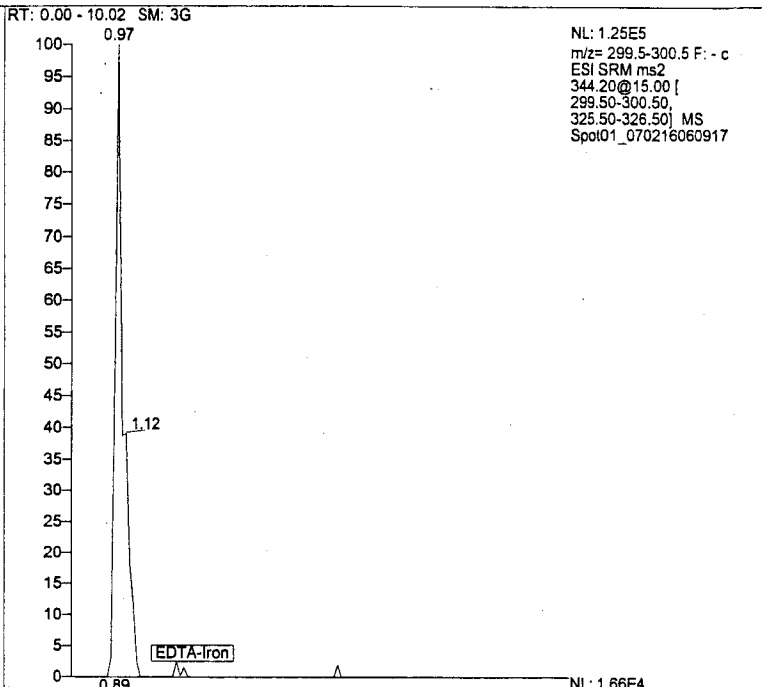


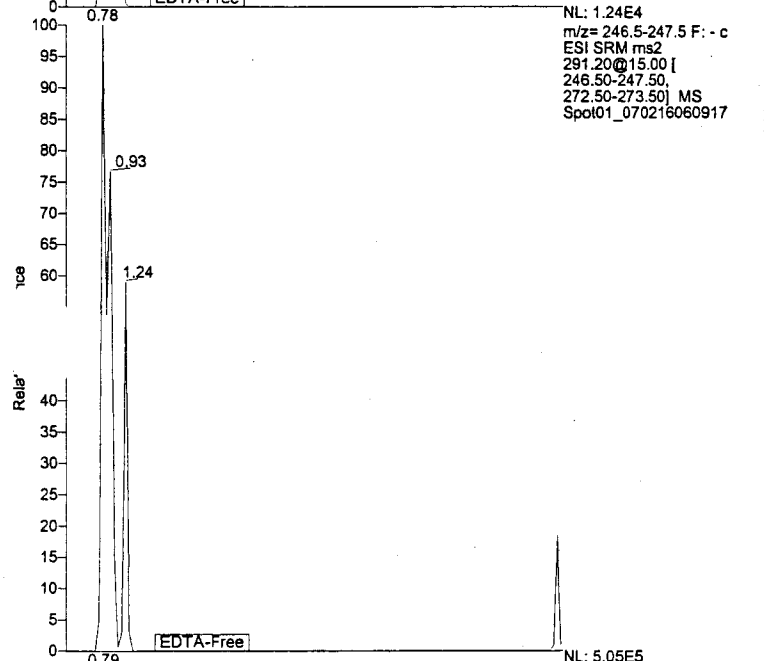


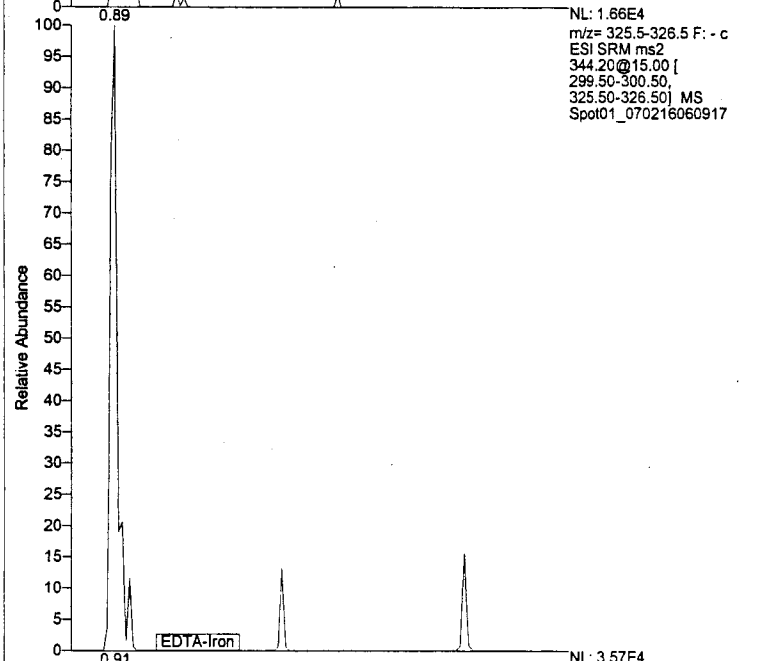


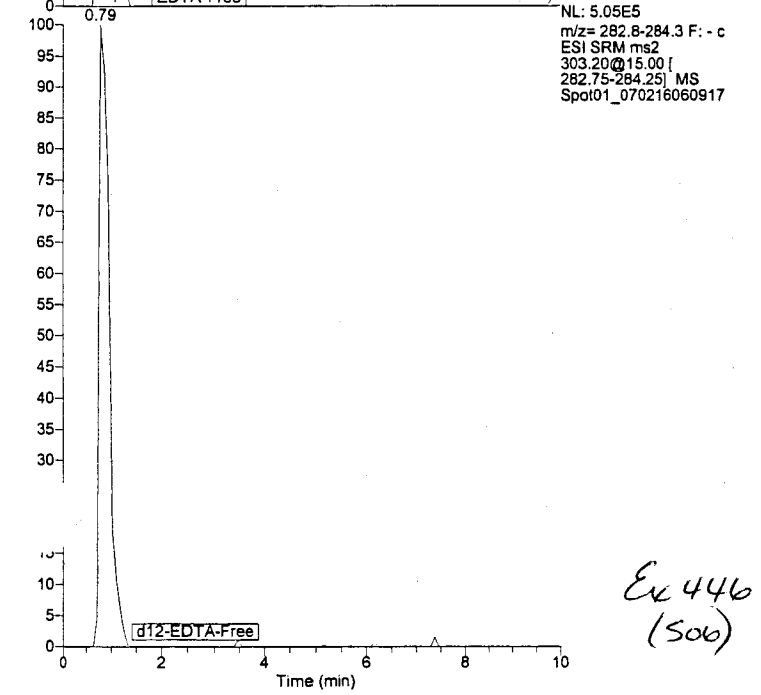
C:\Xcalibur\...\Spot01_070216060917 02/16/07 06:09:17 10uL EDTA+ A DB

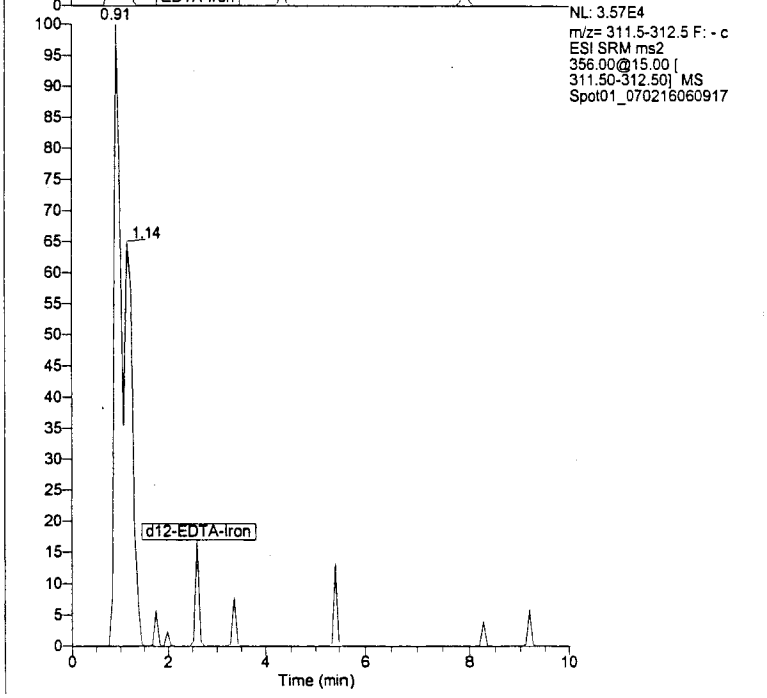






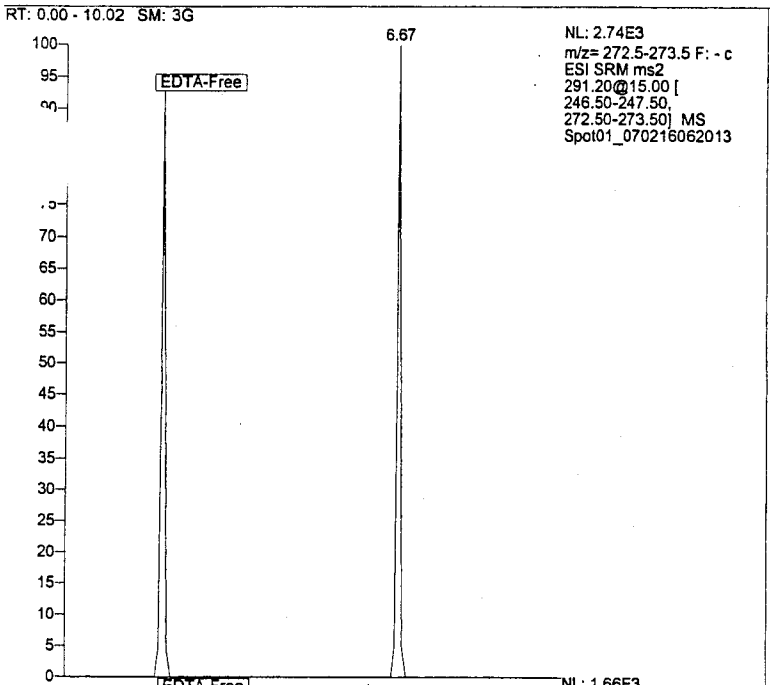


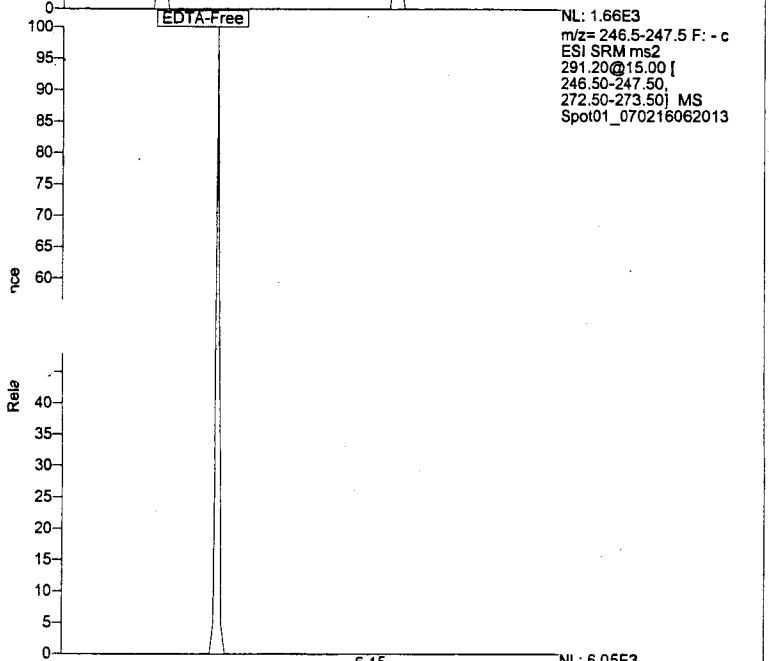


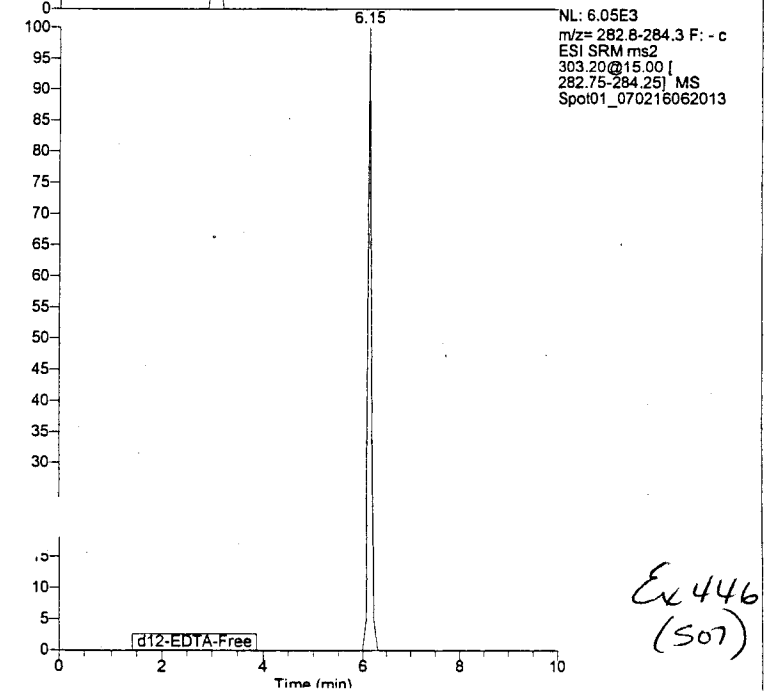


Ex 446
(506)

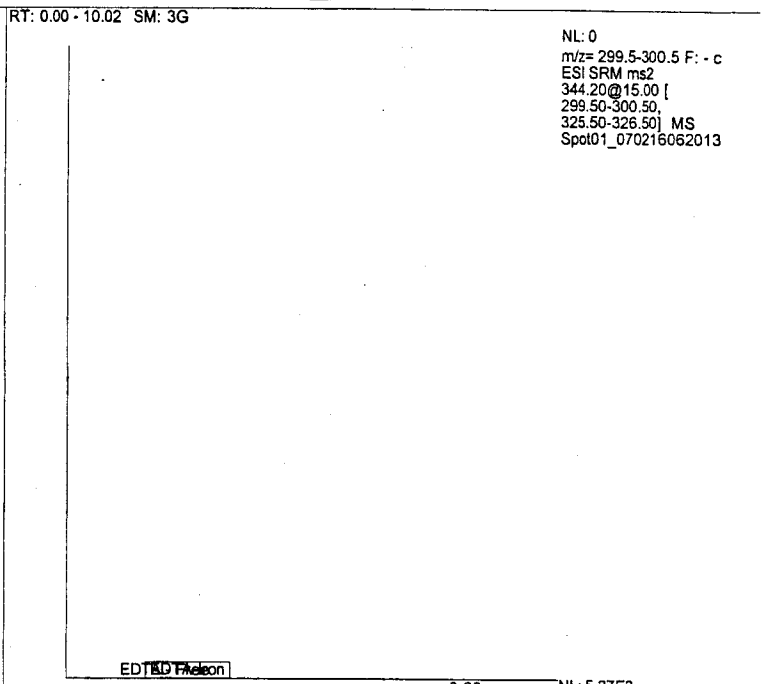
C:\Xcalibur\...\Spot01_070216062013 02/16/07 06:20:13 Negative Blood extract JMB

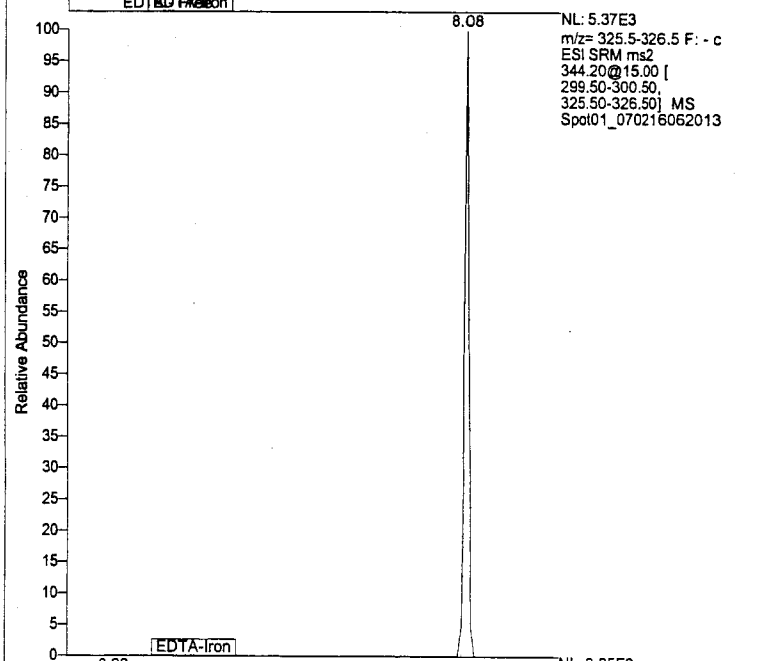


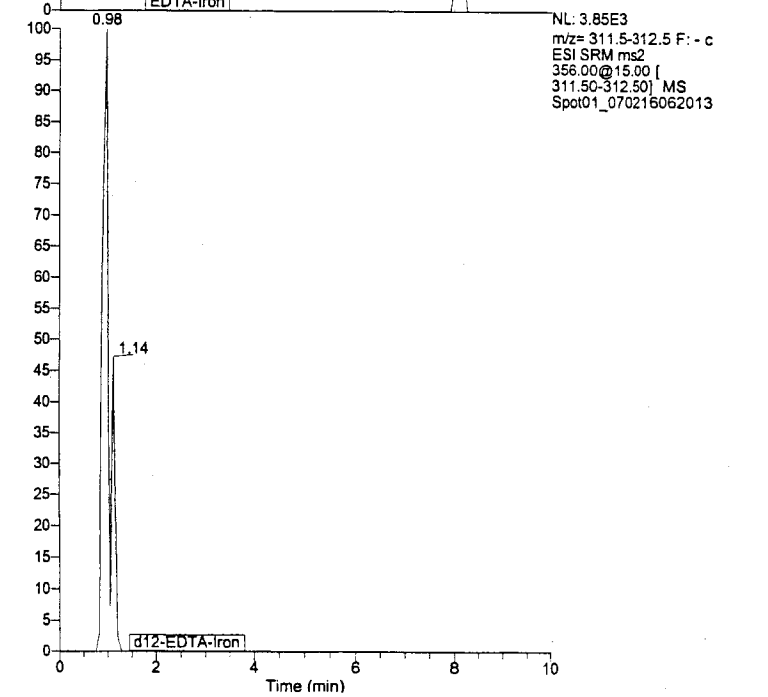




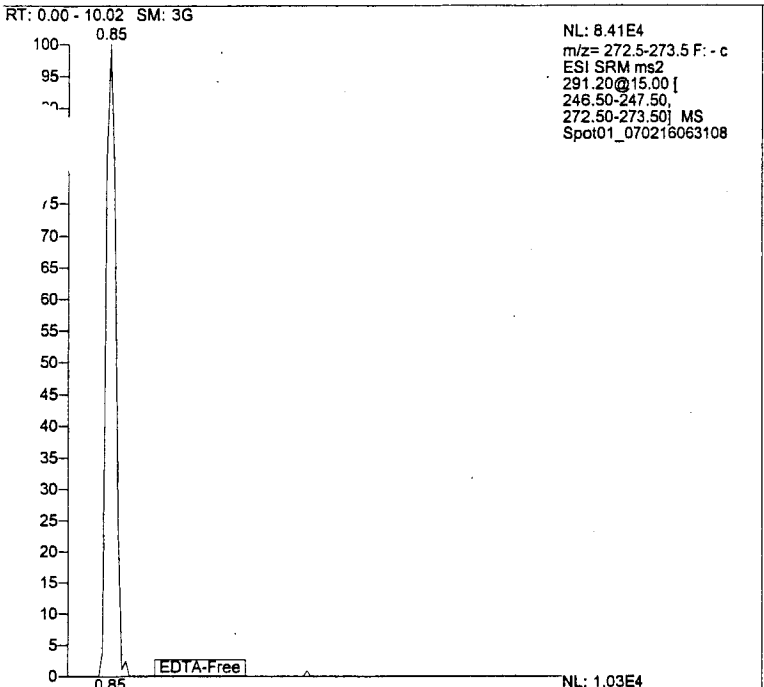
Ex 446 (507)

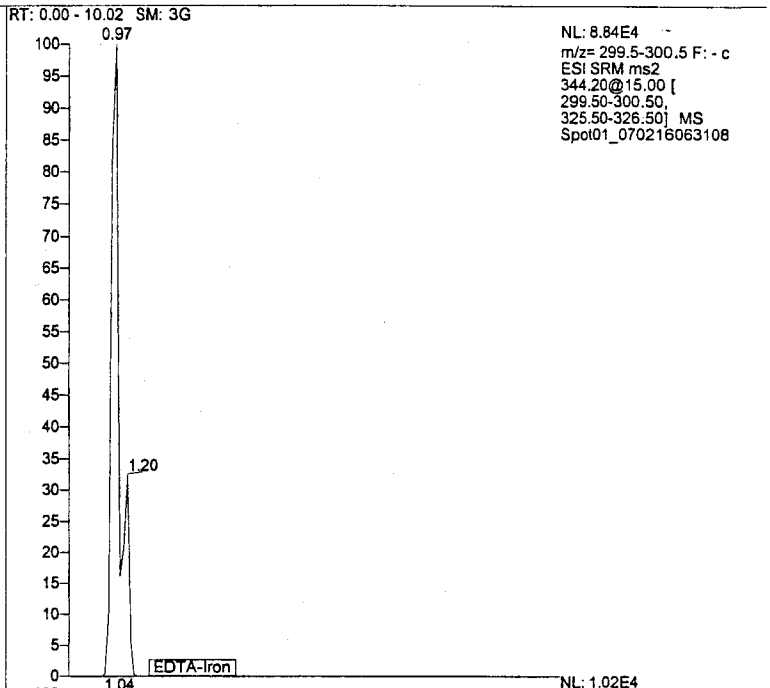


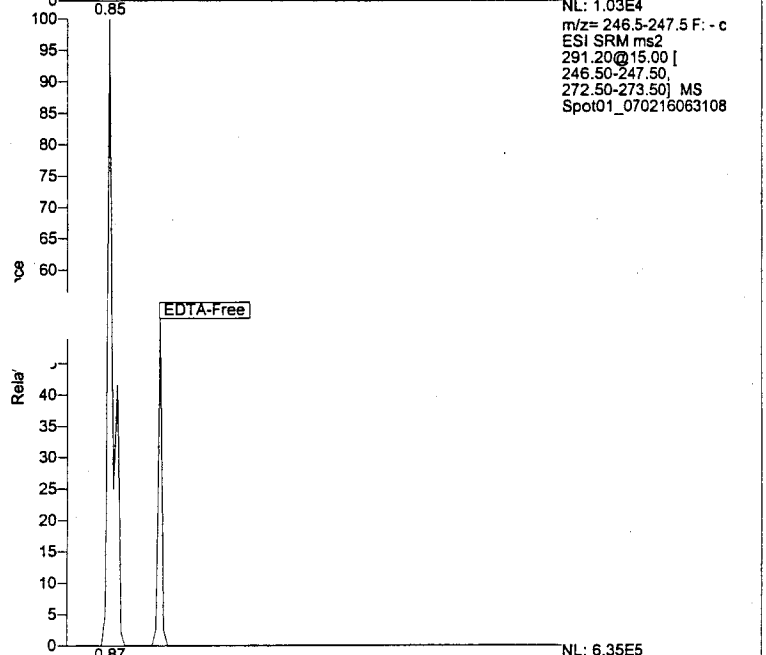


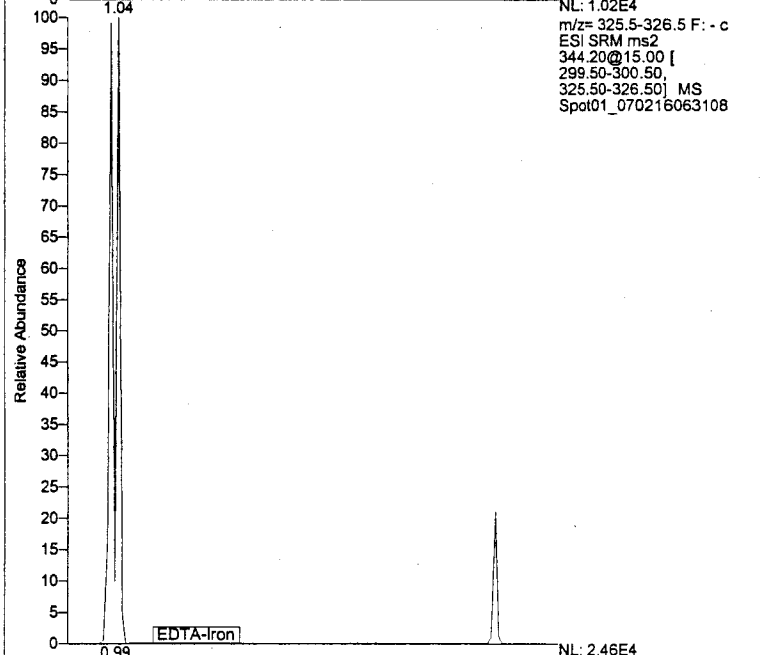


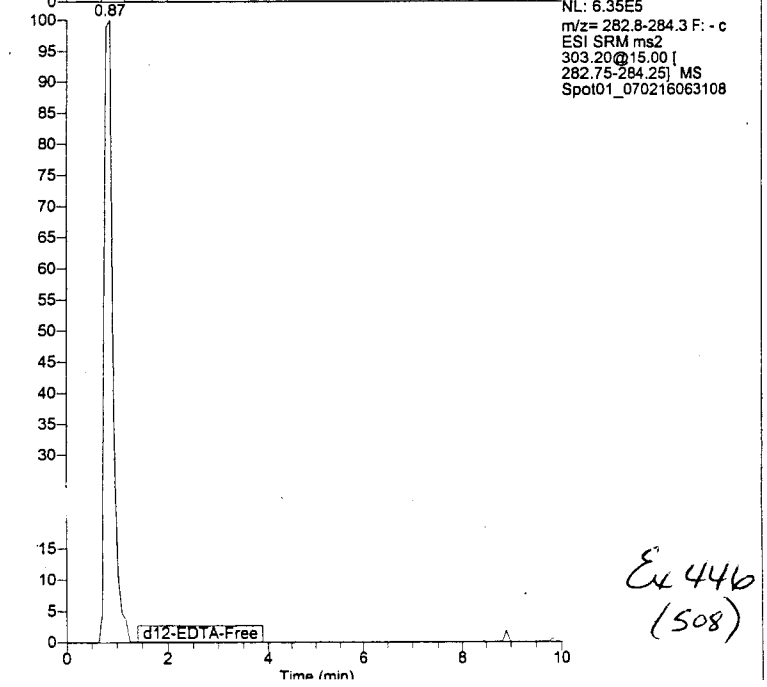
C:\Xcalibur\...\Spot01_070216063108 02/16/07 06:31:08 10uL EDTA+ B TMB

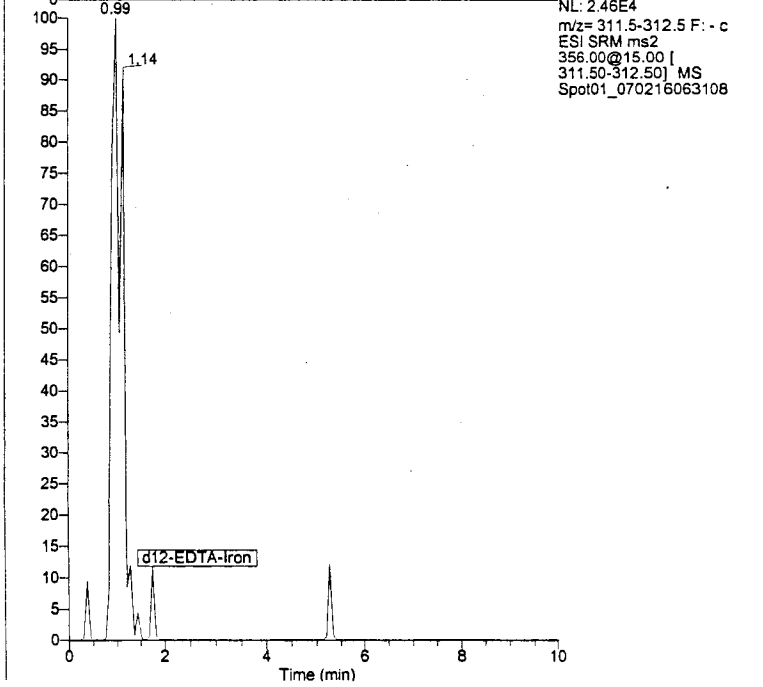




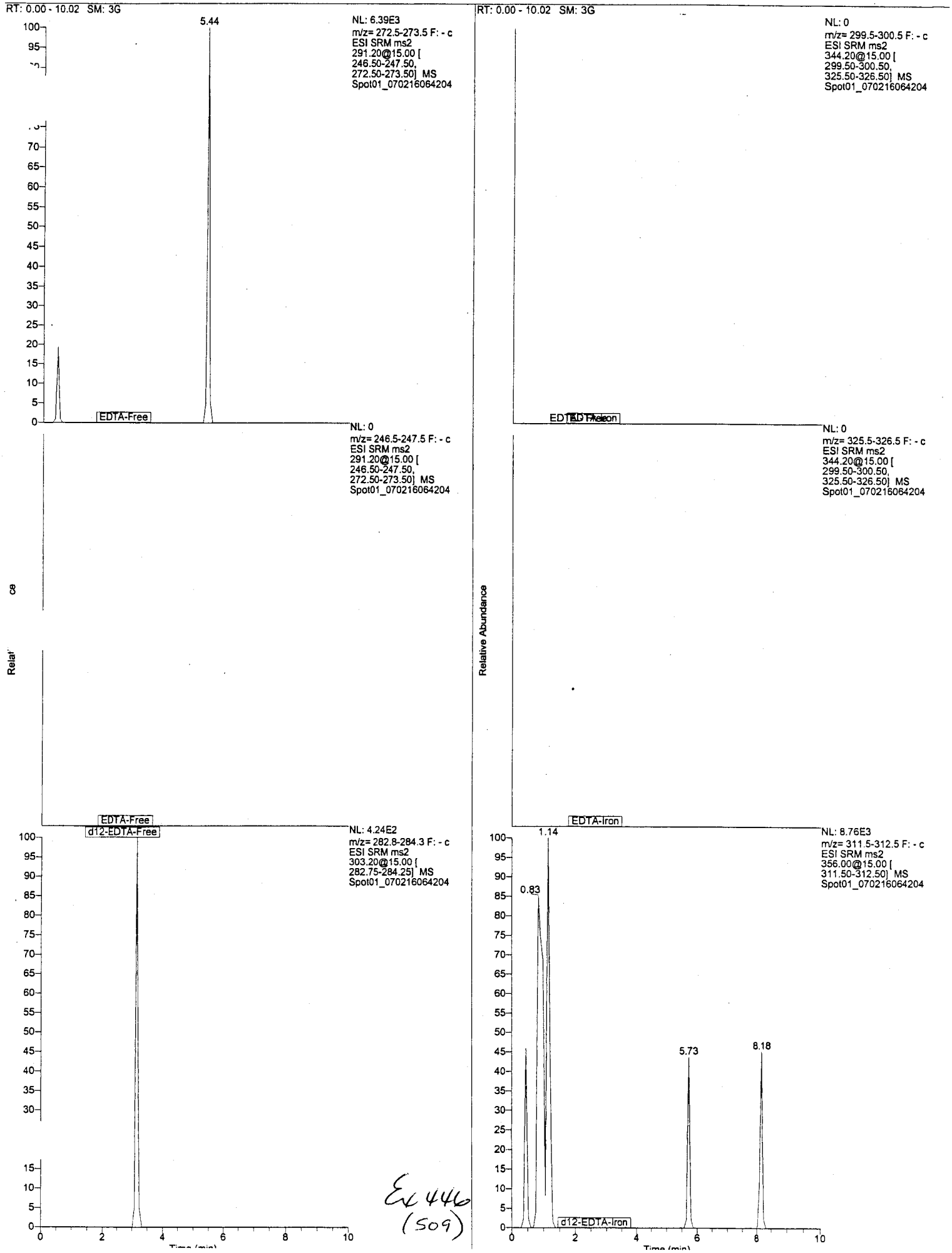






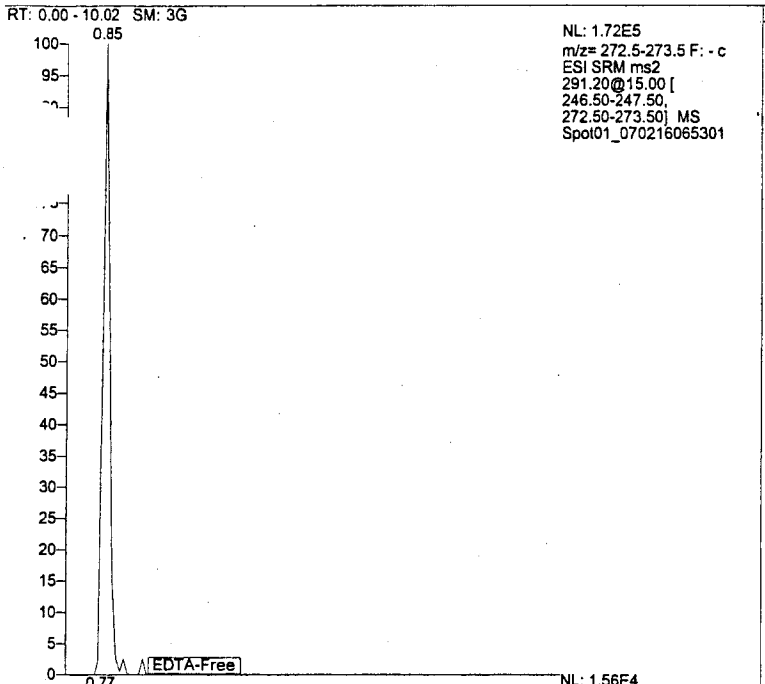


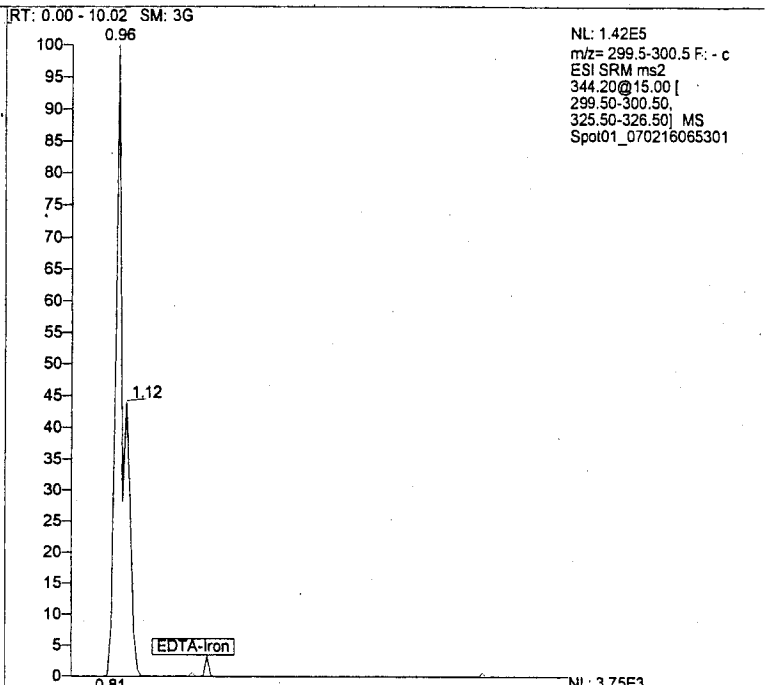
Ex 446
(508)

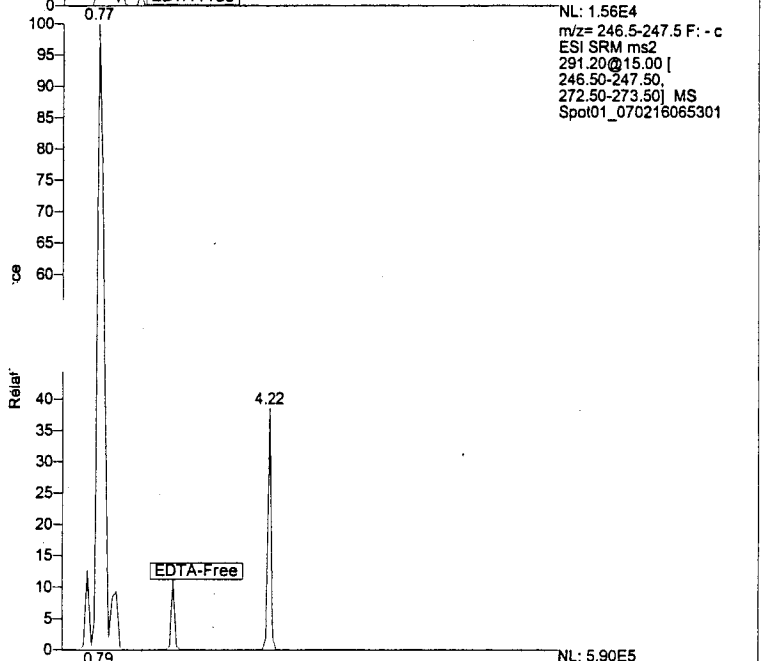

RT: 0.00 - 10.02 SM: 3G
5.44
NL: 6.39E3
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Spot01_070216064204
EDTA-Free
NL: 0
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Spot01_070216064204
EDTA-Free
d12-EDTA-Free
NL: 4.24E2
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Spot01_070216064204
Relative Abundance
Time (min)
RT: 0.00 - 10.02 SM: 3G
NL: 0
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Spot01_070216064204
NL: 0
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Spot01_070216064204
EDTA-Iron
1.14
0.83
5.73
8.18
NL: 8.76E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Spot01_070216064204
d12-EDTA-Iron
Relative Abundance
Time (min)

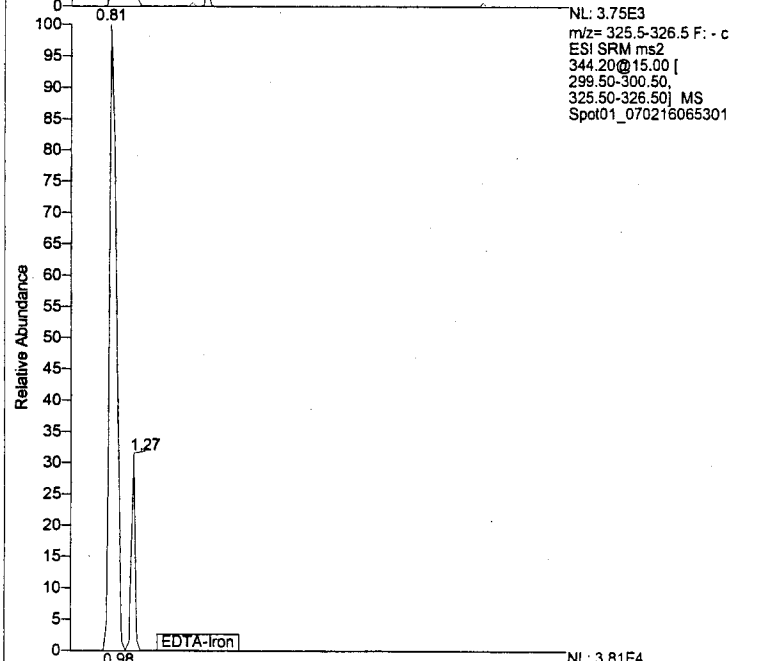
EL 446
(509)

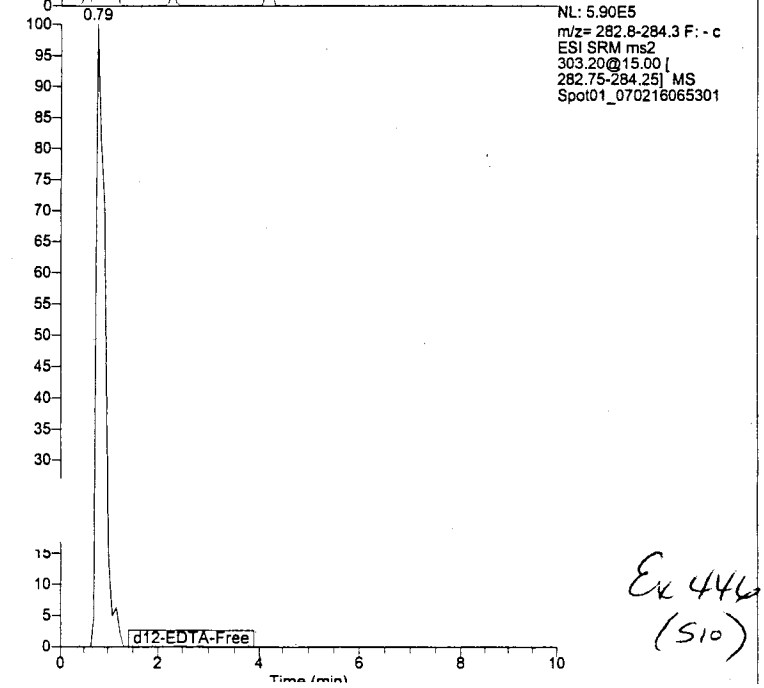
C:\Xcalibur\...\Spot01_070216065301 02/16/07 06:53:01 10uL EDTA+ C TDB

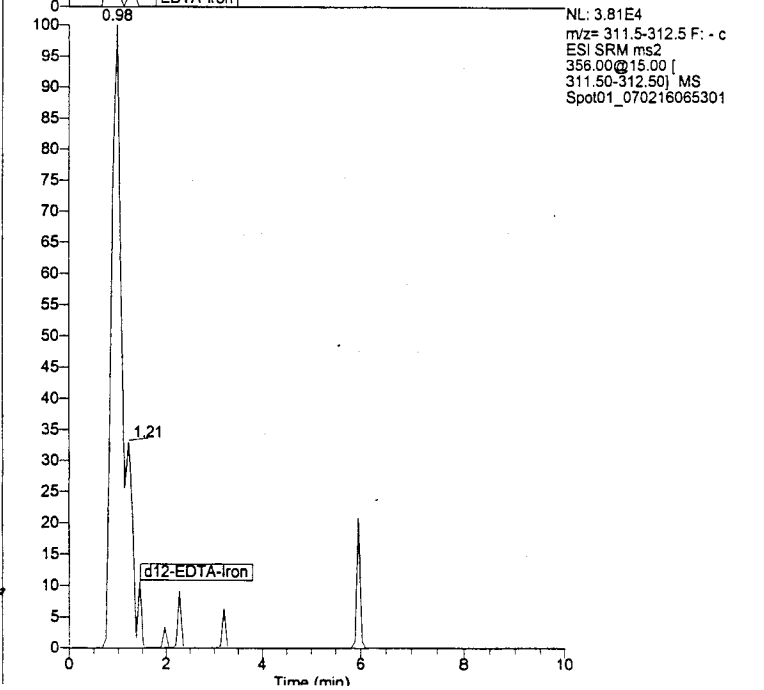




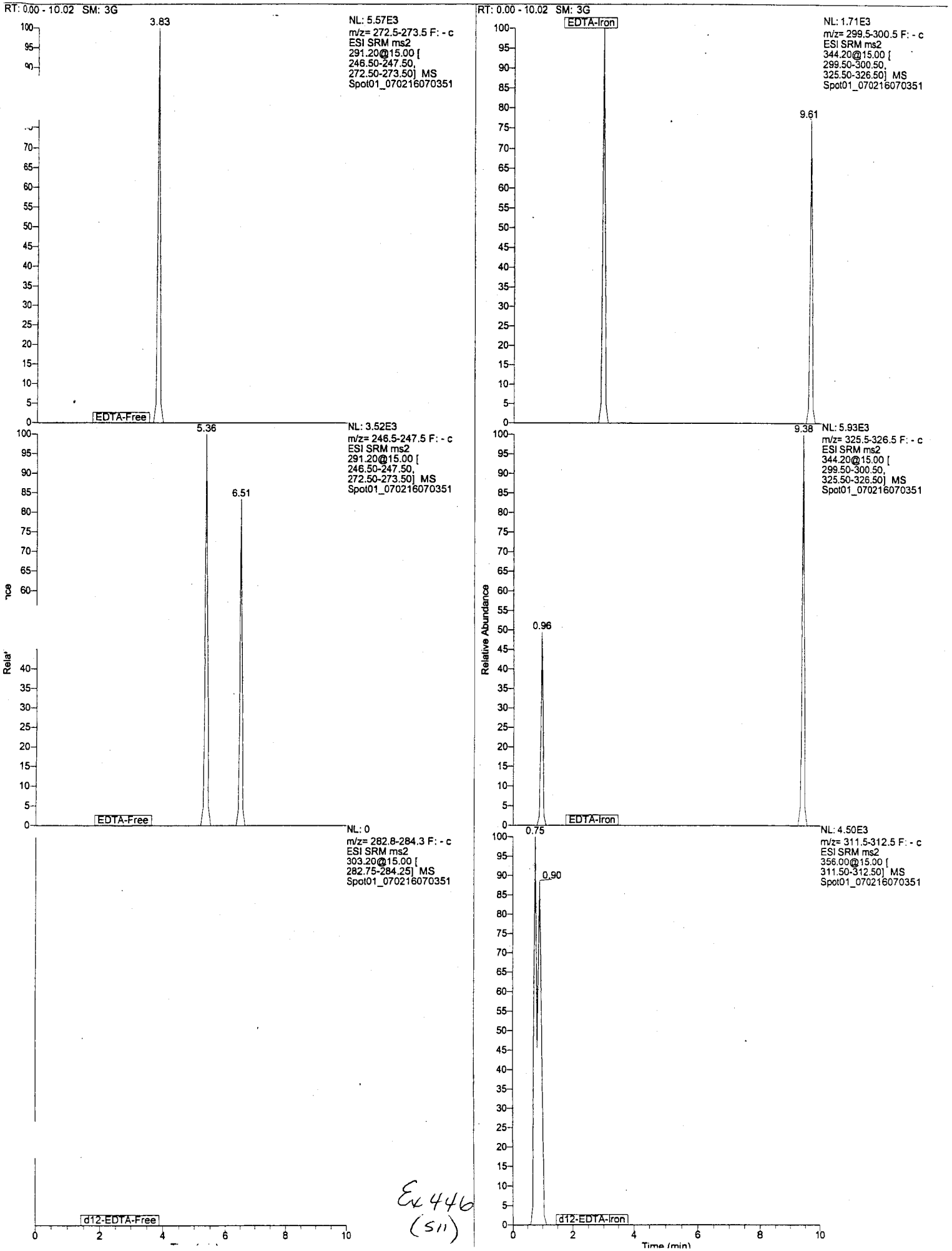








Ex 446
(S10)

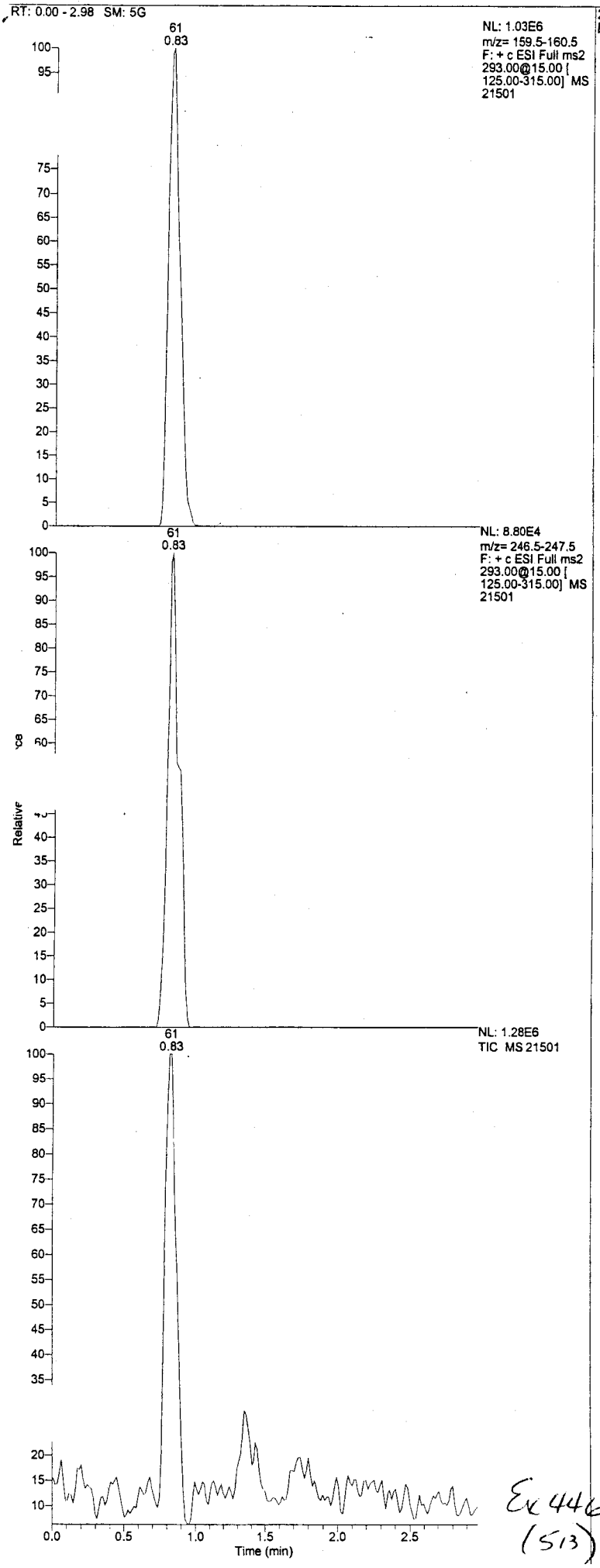

RT: 0.00 - 10.02 SM: 3G
3.83
NL: 5.57E3
m/z= 272.5-273.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Spot01_070216070351
EDTA-Free
5.36
6.51
NL: 3.52E3
m/z= 246.5-247.5 F: - c
ESI SRM ms2
291.20@15.00 [
246.50-247.50,
272.50-273.50] MS
Spot01_070216070351
EDTA-Free
NL: 0
m/z= 282.8-284.3 F: - c
ESI SRM ms2
303.20@15.00 [
282.75-284.25] MS
Spot01_070216070351
d12-EDTA-Free
RT: 0.00 - 10.02 SM: 3G
EDTA-Iron
9.61
NL: 1.71E3
m/z= 299.5-300.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Spot01_070216070351
9.38
NL: 5.93E3
m/z= 325.5-326.5 F: - c
ESI SRM ms2
344.20@15.00 [
299.50-300.50,
325.50-326.50] MS
Spot01_070216070351
0.96
EDTA-Iron
0.75
0.90
NL: 4.50E3
m/z= 311.5-312.5 F: - c
ESI SRM ms2
356.00@15.00 [
311.50-312.50] MS
Spot01_070216070351
d12-EDTA-Iron
Relative Abundance
Time (min)

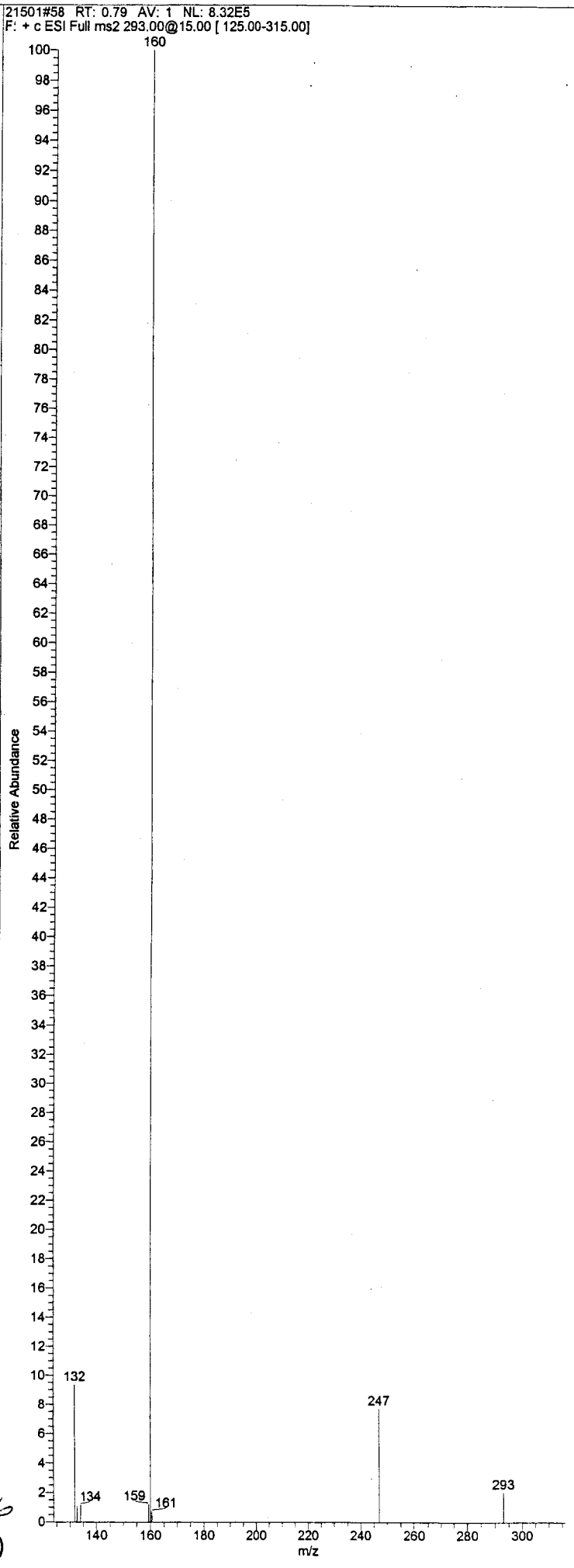
Ex 446 (511)

TM / DIAGNOSTICS

Ex 446
(512)

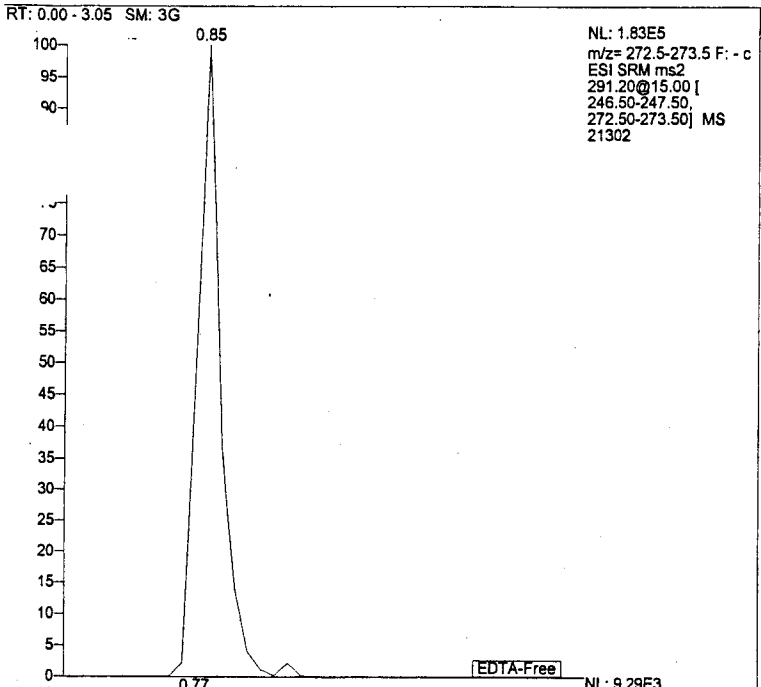
C:\Xcalibur\...\EDTA\Brewer\021507\21501 02/15/07 11:13:10 100 ppm EDTA tm

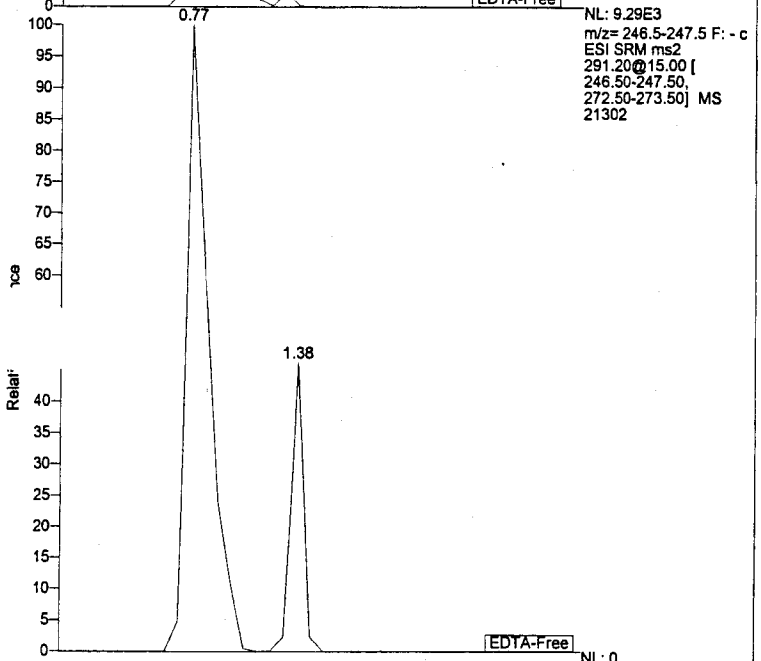


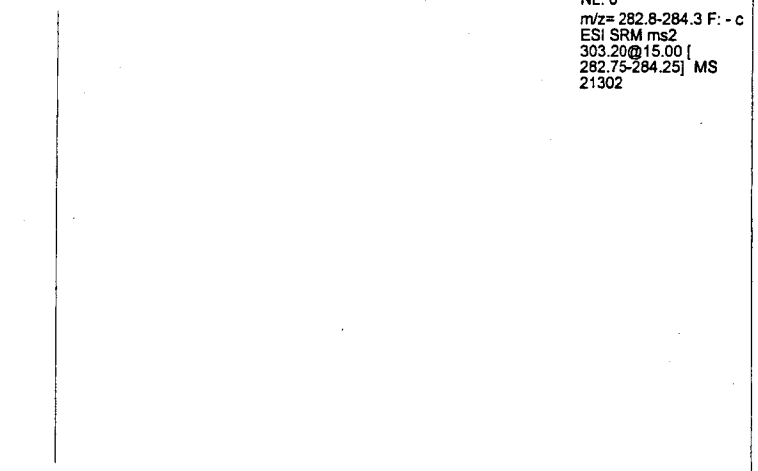


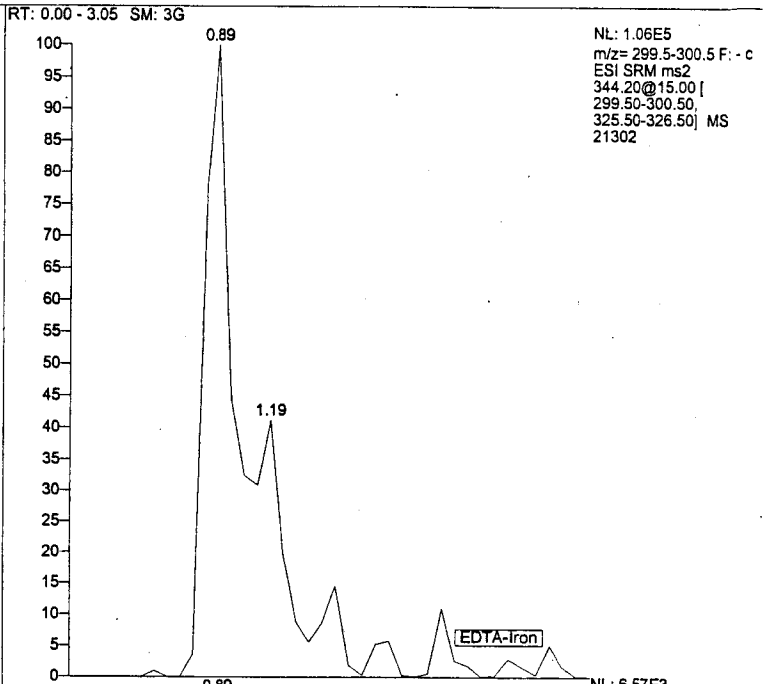
Ex 446
(513)

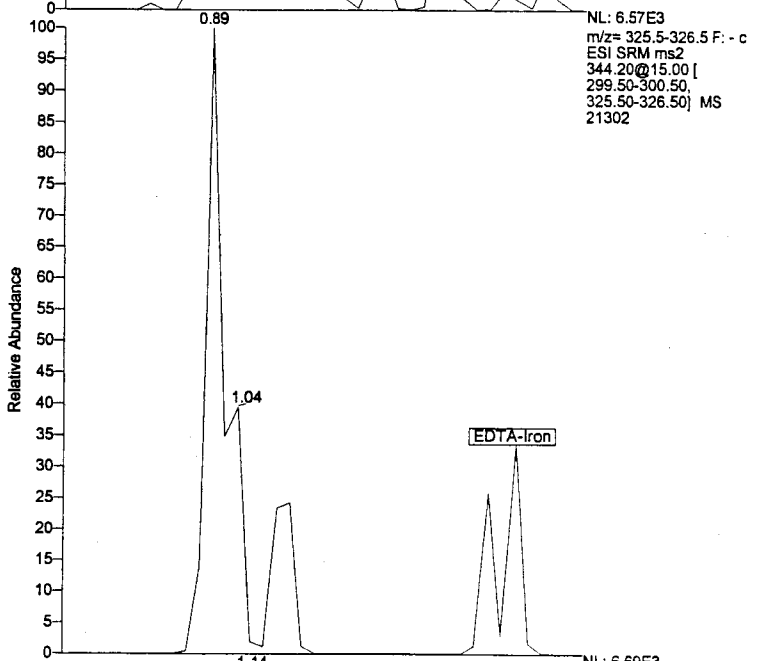
C:\Xcalibur\...\EDTA\Brewer\021307\21302 02/13/07 08:50:58 100 ppm EDTA JoB

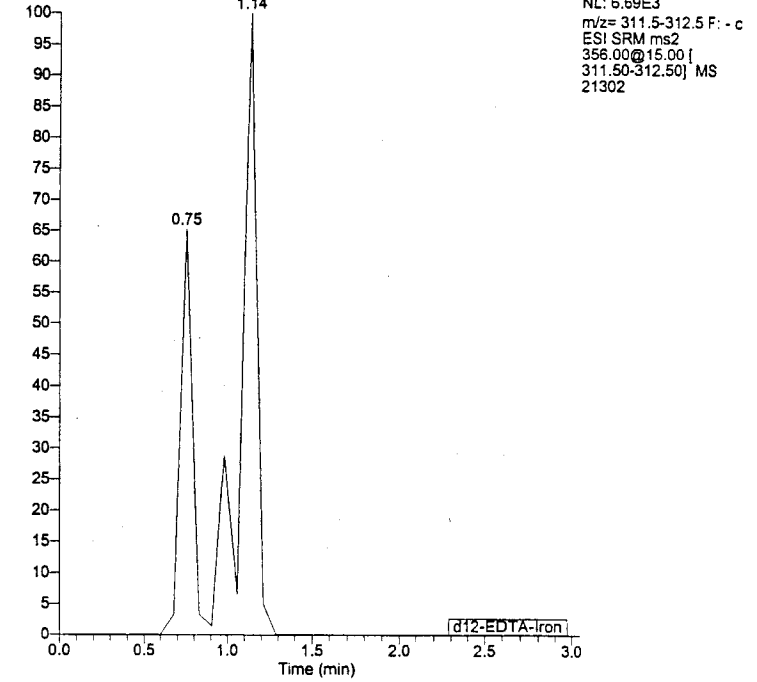








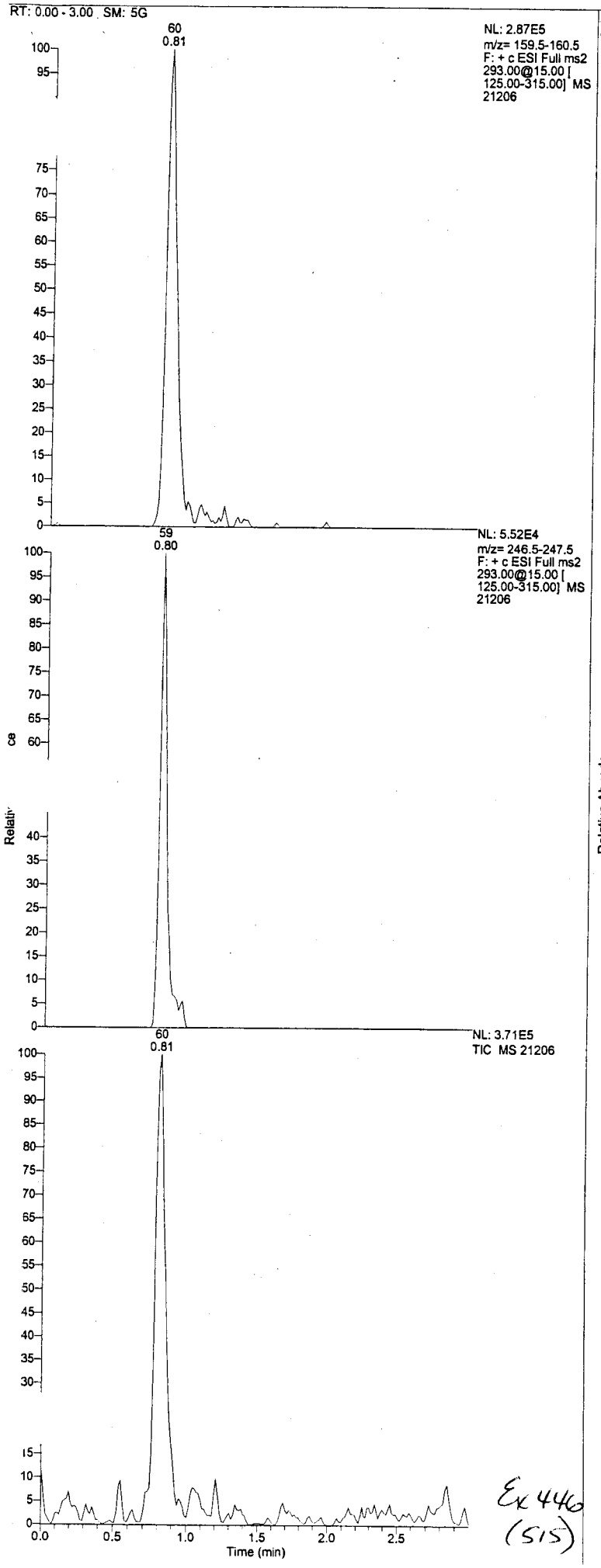




Ex 446
(514)

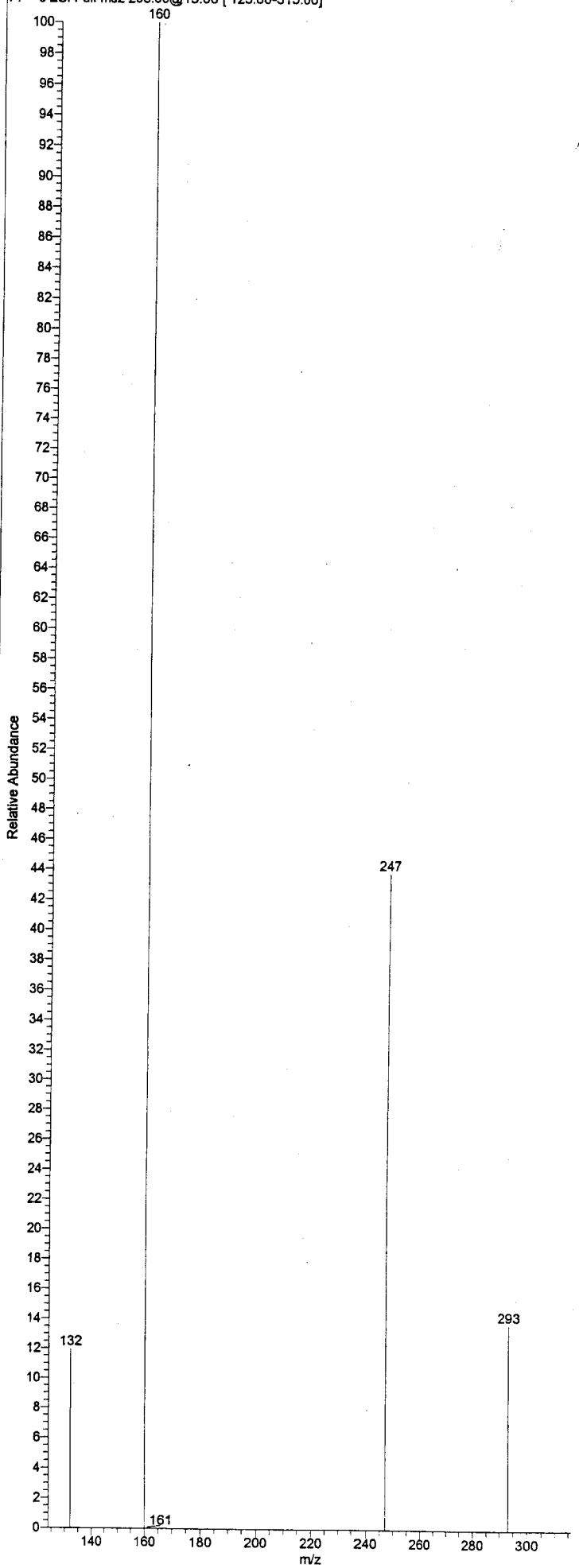
C:\Xcalibur\...\EDTA\Brewer\021207\21206 02/12/07 02:33:58 100 ppm EDTA TM JDB

RT: 0.00 - 3.00 SM: 5G

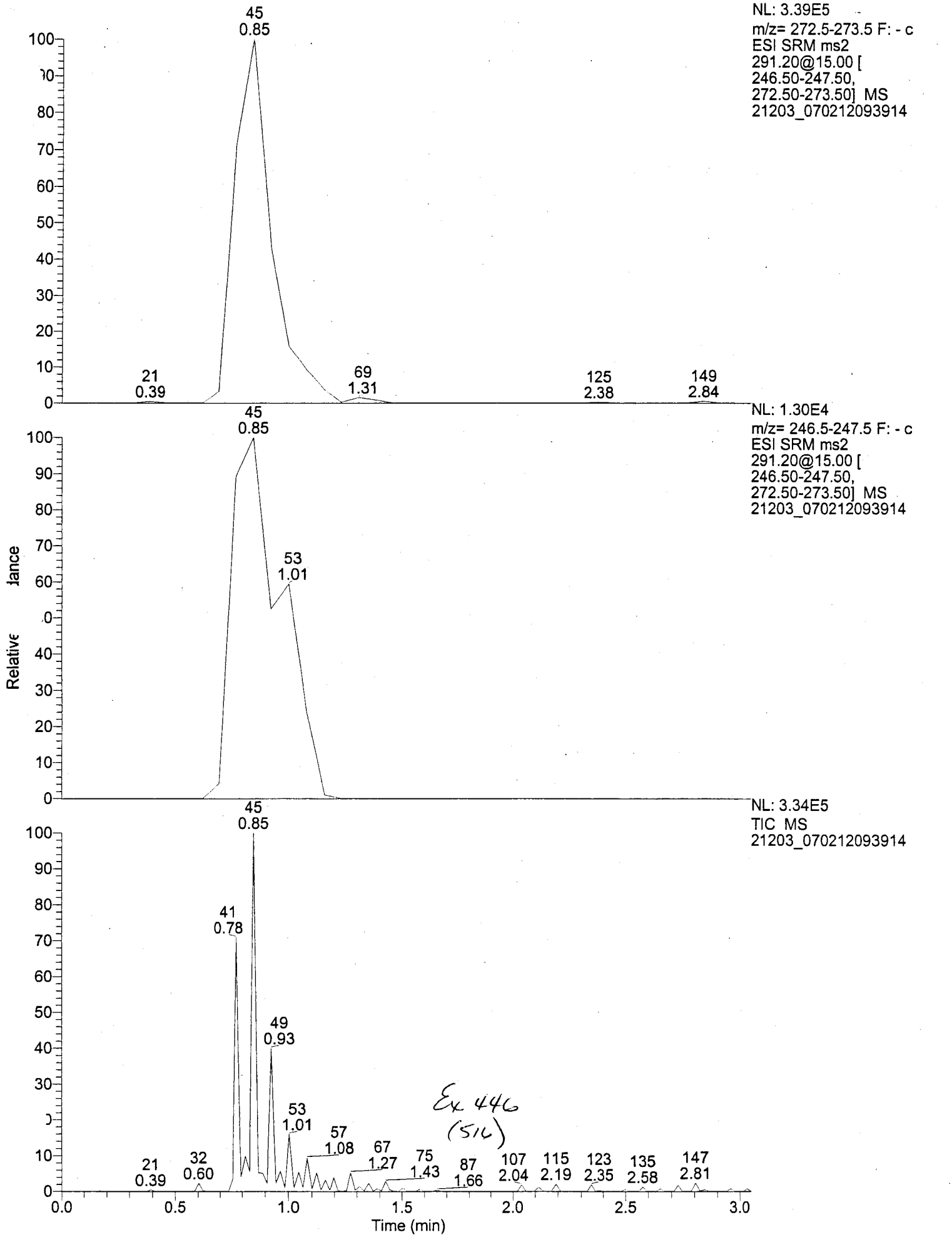


Ex 446
(515)

21206#57 RT: 0.77 AV: 1 NL: 5.06E4
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

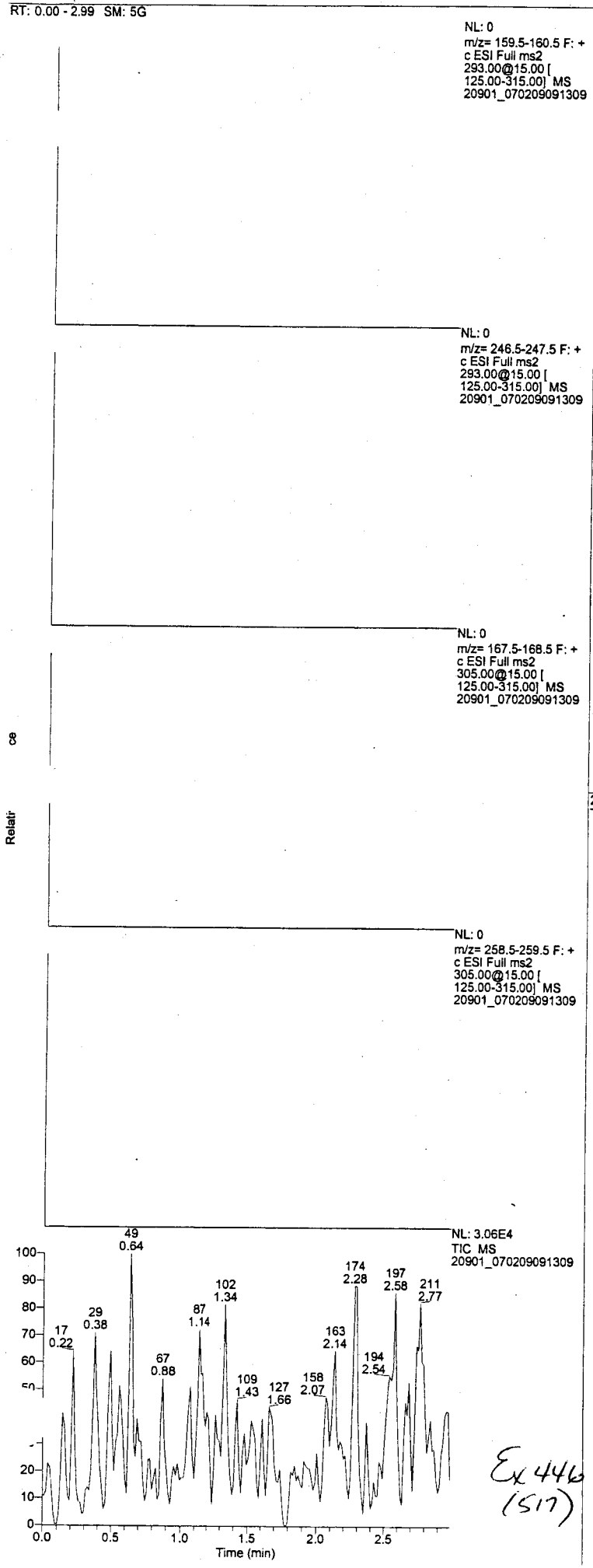


RT: 0.00 - 3.05 SM: 3G

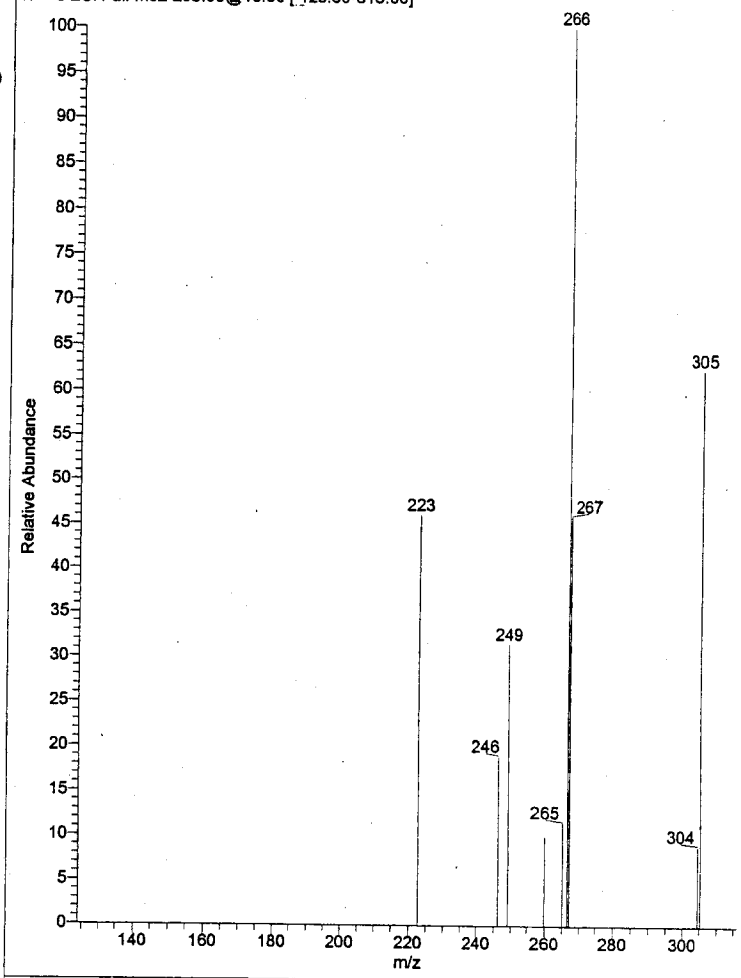


C:\Xcalibur\...\20901_070209091309 02/09/07 09:13:09 DI H2O JDB

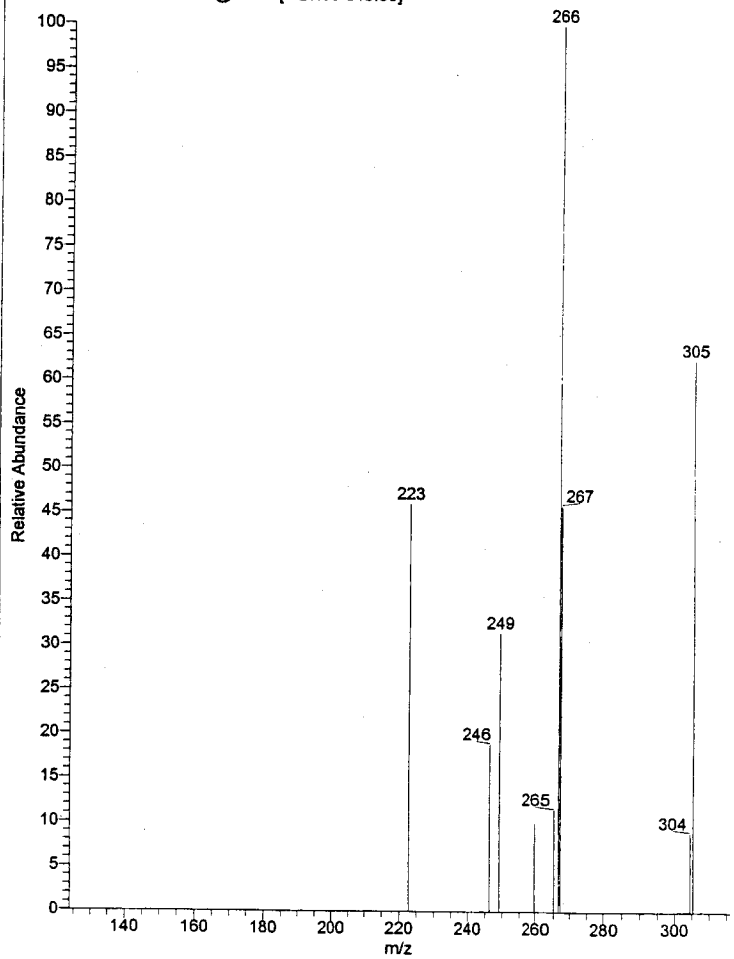
RT: 0.00 - 2.99 SM: 5G



20901_070209091309#63-73 RT: 0.82-0.96 AV: 11 NL: 2.15E3
T: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

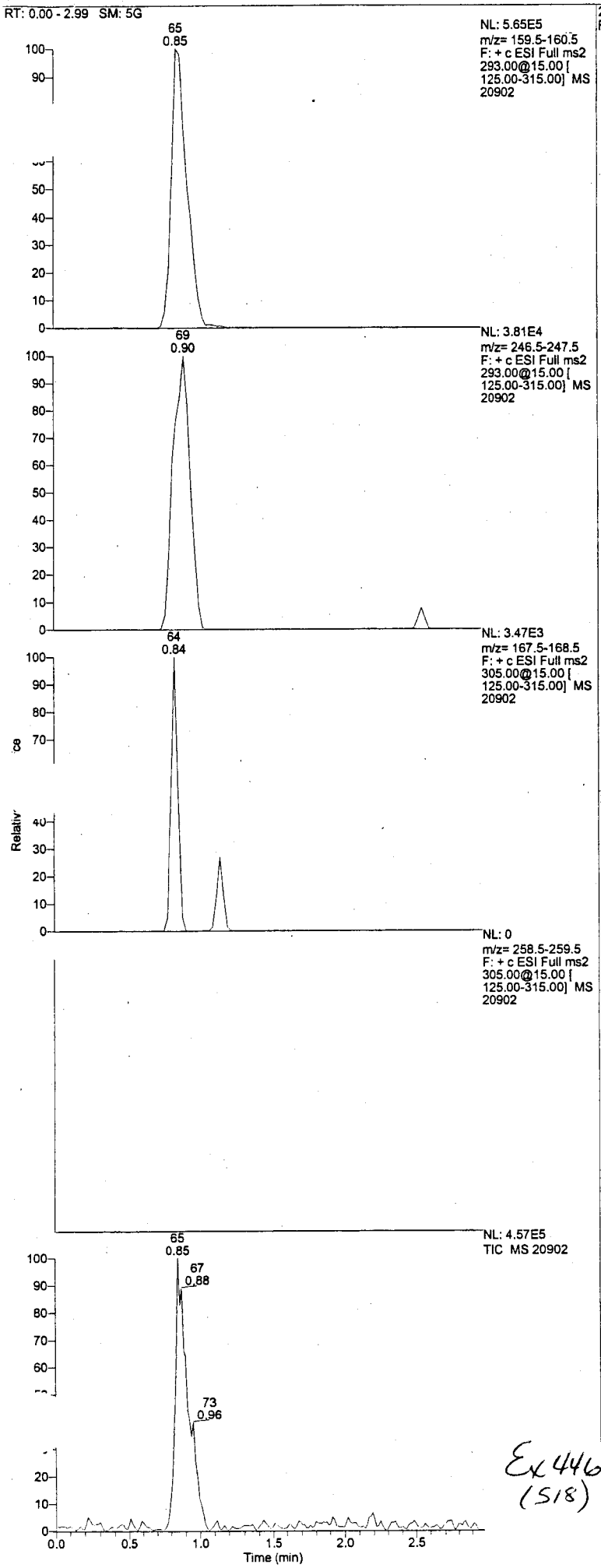


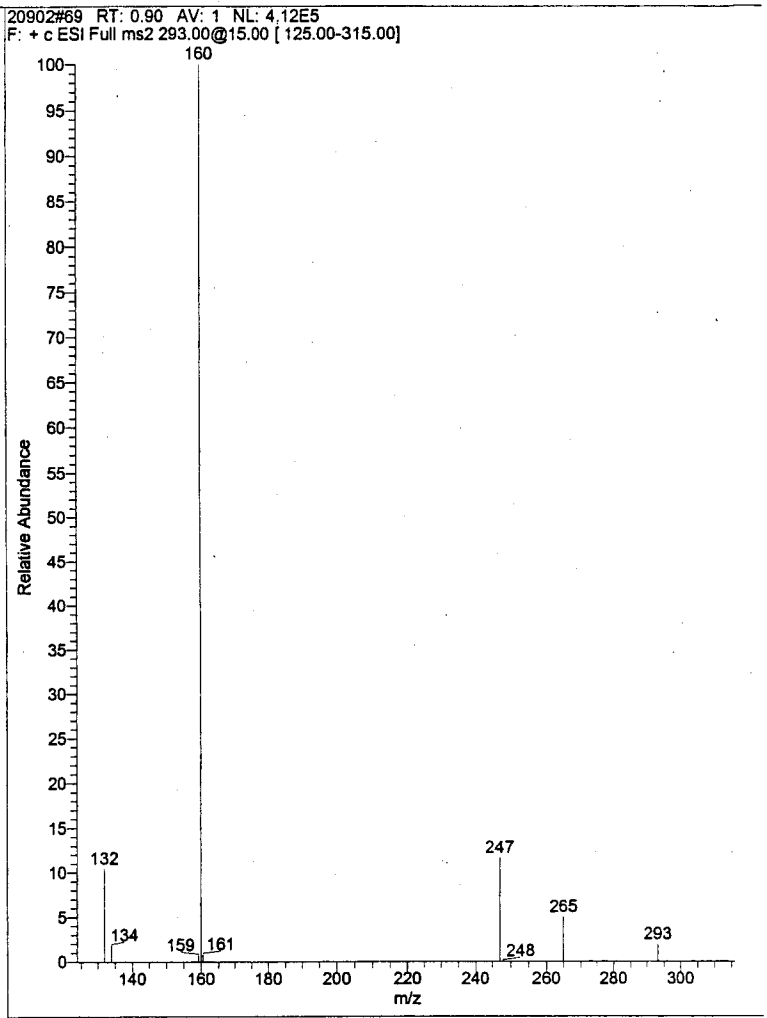
20901_070209091309#63-73 RT: 0.82-0.96 AV: 11 NL: 2.15E3
T: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

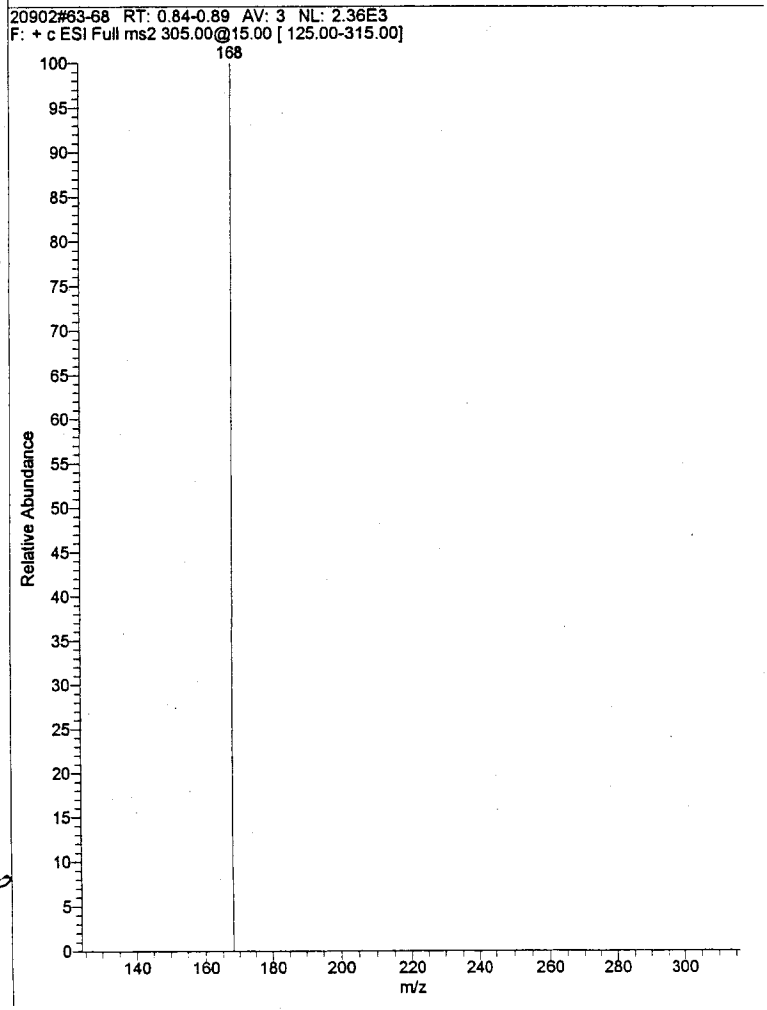


Ex 446
(517)

C:\Xcalibur\...\EDTA\Brewer\020907\20902 02/09/07 09:17:22 100 ppm EDTA (testmix) JDB



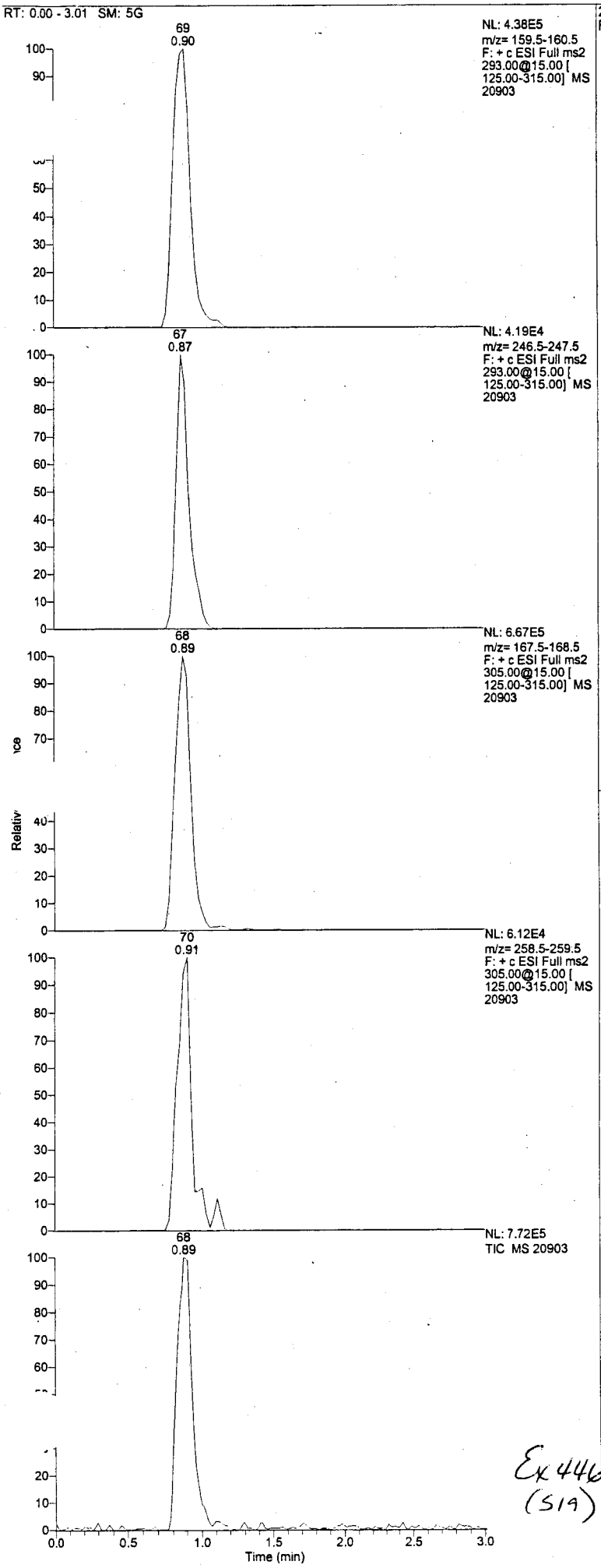




Ex 446
(S18)

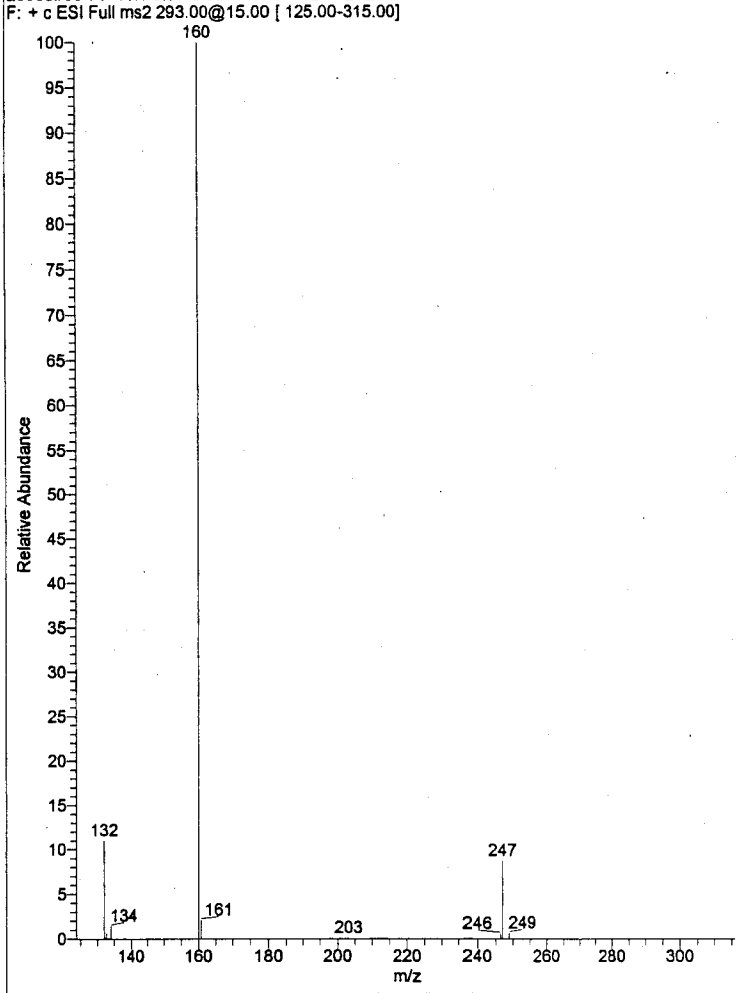
C:\Xcalibur\...\EDTA\Brewer\020907\20903 02/09/07 09:25:27 333 ppm each EDTA, d12 EDTA (2.5 uL) JDB

RT: 0.00 - 3.01 SM: 5G

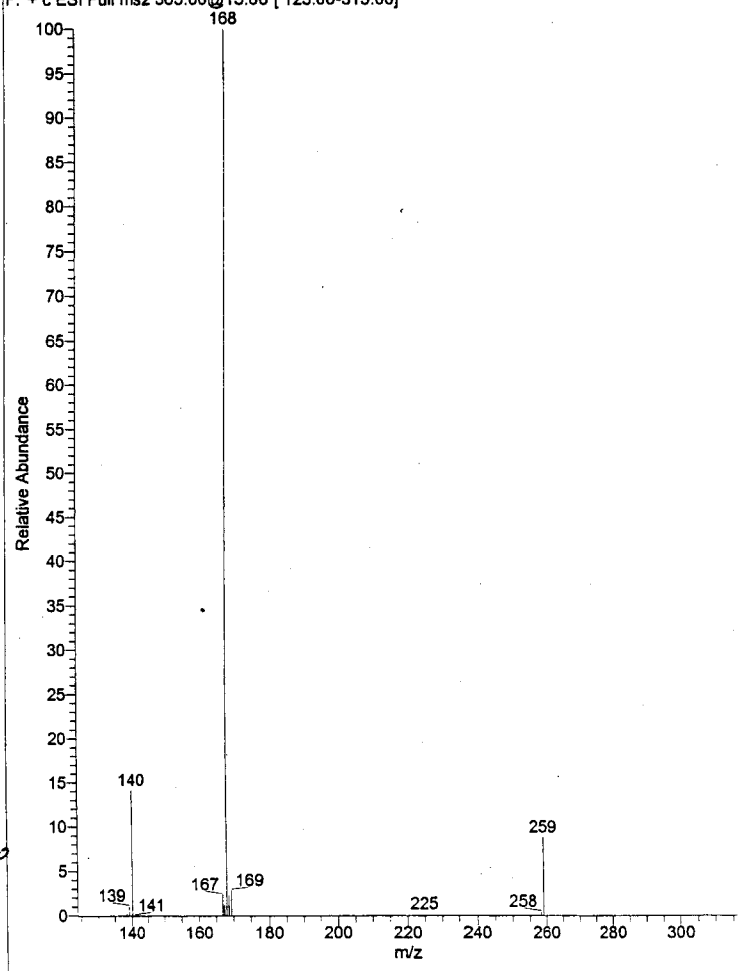


Ex 446
(519)

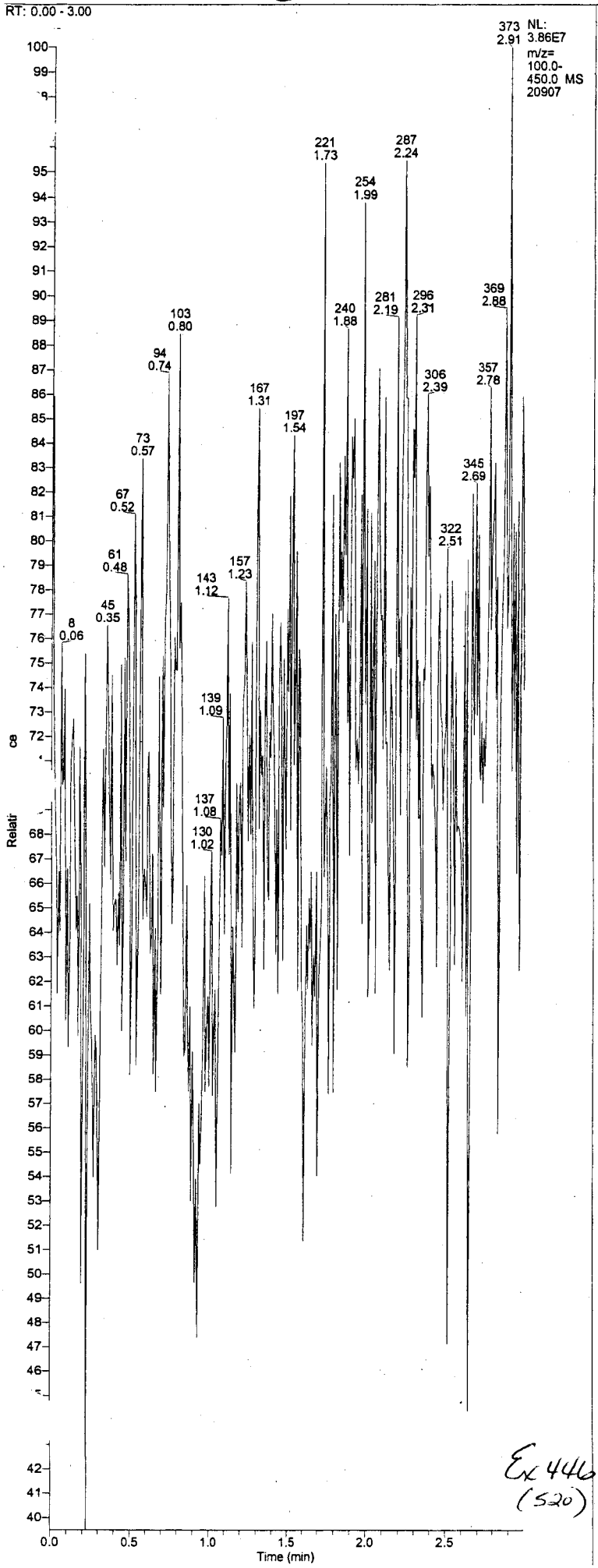
20903#66-71 RT: 0.87-0.93 AV: 3 NL: 4.25E5
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]



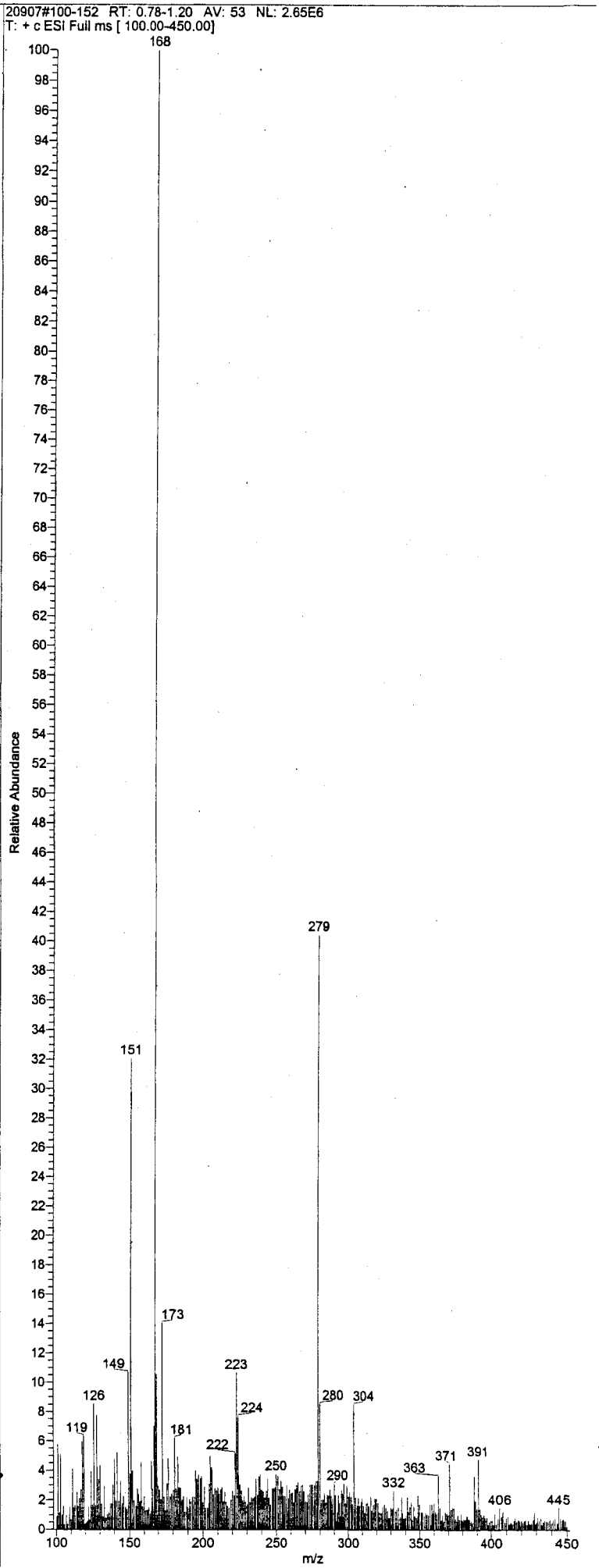
20903#66-71 RT: 0.86-0.91 AV: 3 NL: 6.70E5
F: + c ESI Full ms2 305.00@15.00 [125.00-315.00]



C:\Xcalibur\...\EDTA\Brewer\020907\20907 02/09/07 09:48:42 10mM Ni(II) aq JOB

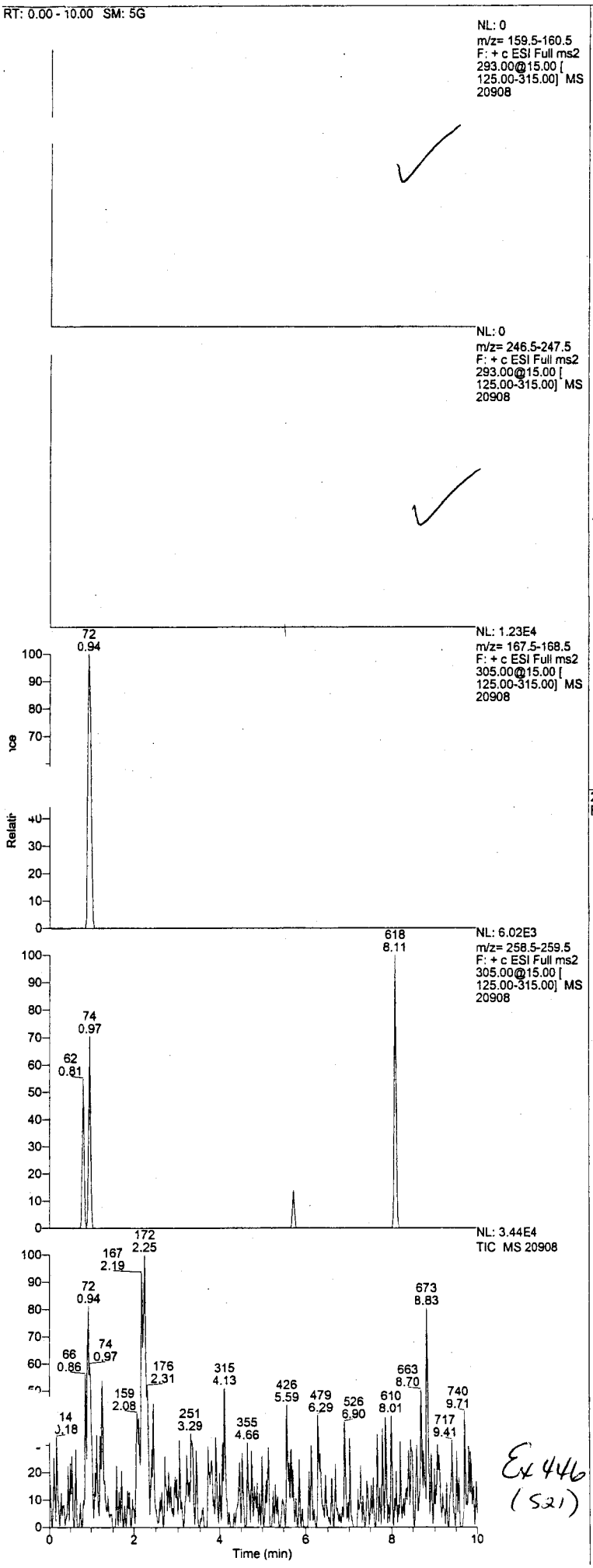


Ex 446 (520)



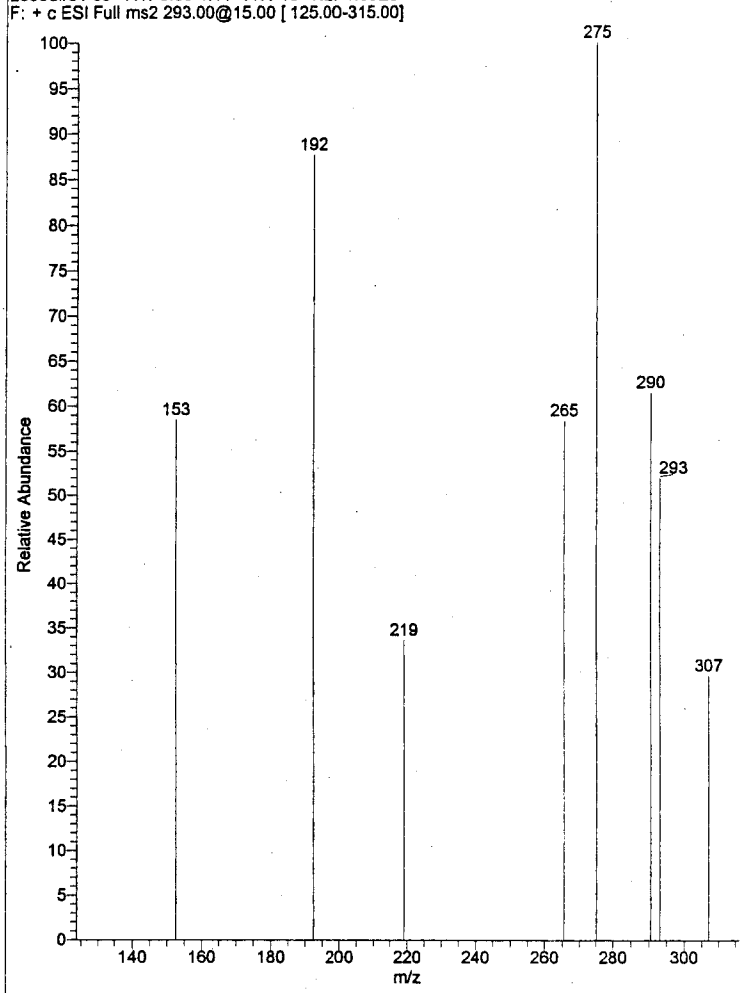
C:\Xcalibur\...\EDTA\Brewer\020907\20908 02/09/07 09:59:35 EDTA neg swab (2/2/07) JoB

RT: 0.00 - 10.00 SM: 5G

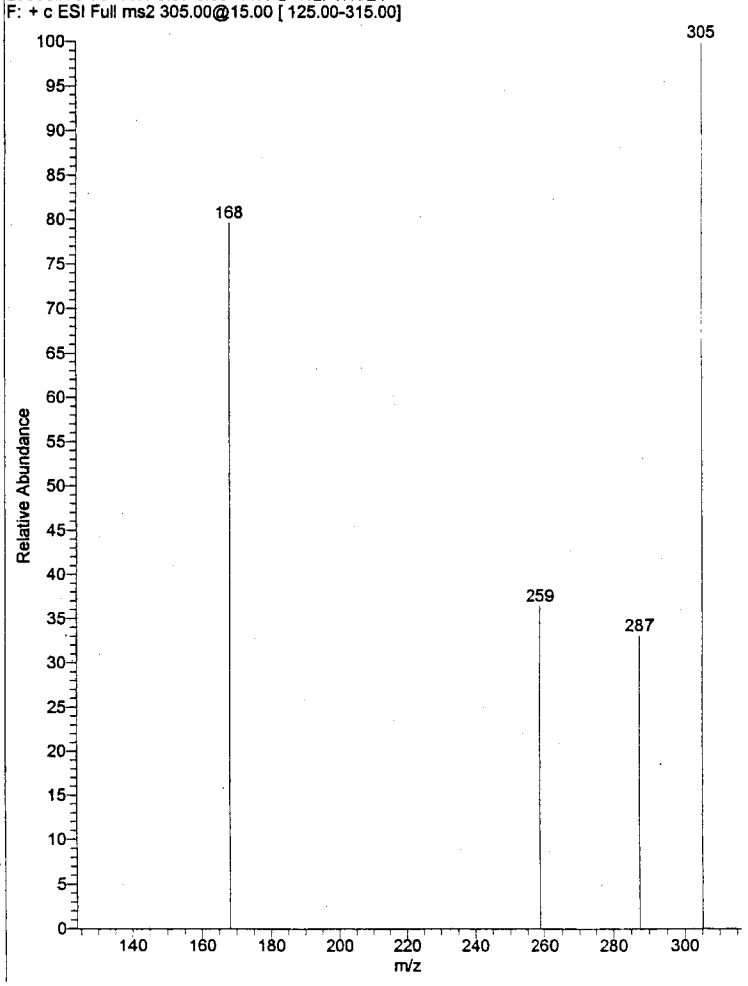


Ex 446 (521)

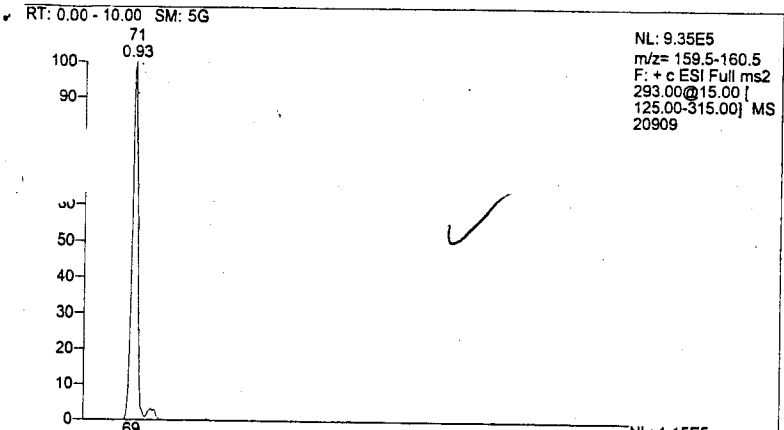
20908#61-85 RT: 0.80-1.11 AV: 13 NL: 1.09E3
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

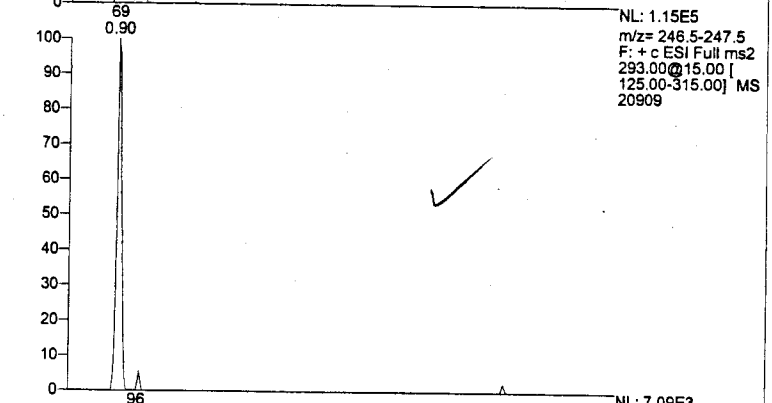


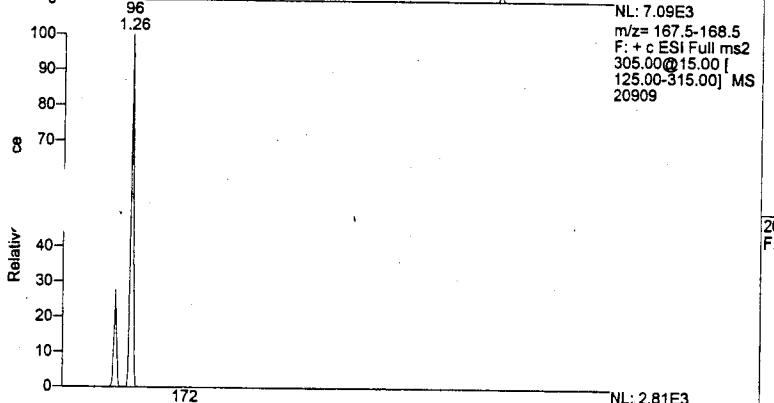
20908#73-76 RT: 0.97-0.99 AV: 2 NL: 1.19E4
F: + c ESI Full ms2 305.00@15.00 [125.00-315.00]

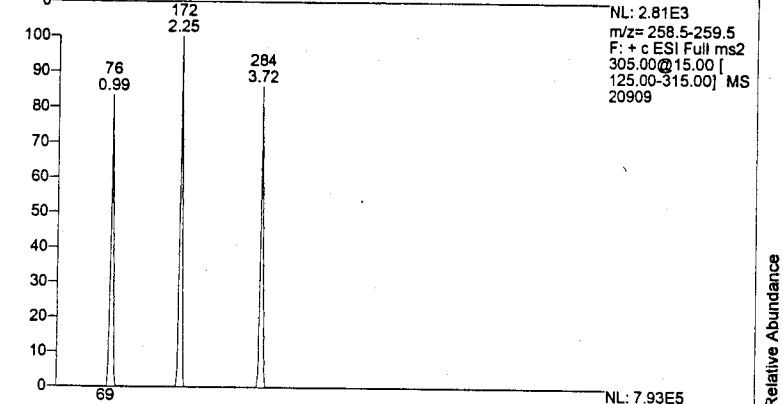


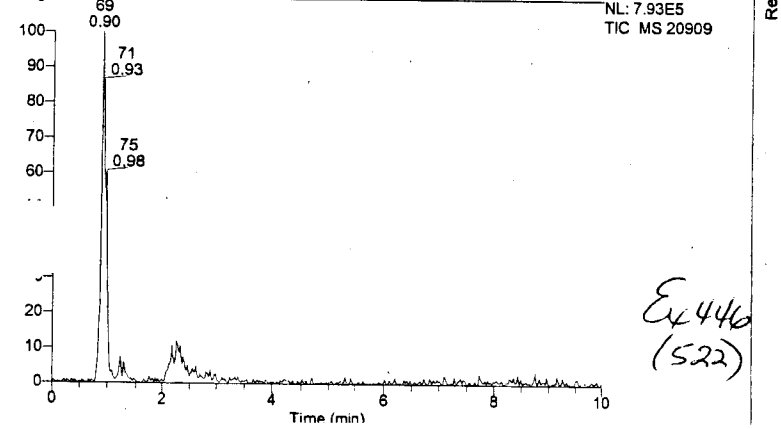
C:\Xcalibur\...\EDTA\Brewer\020907\20909 02/09/07 10:10:53 EDTA pos swab (2/2/07) JOB



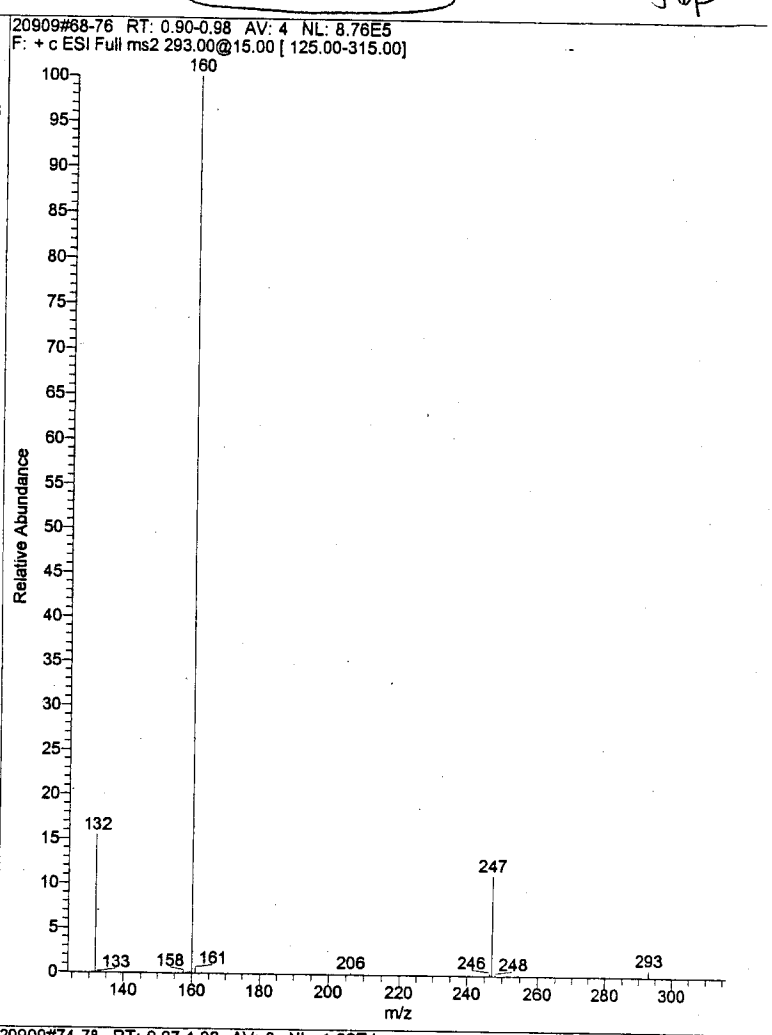


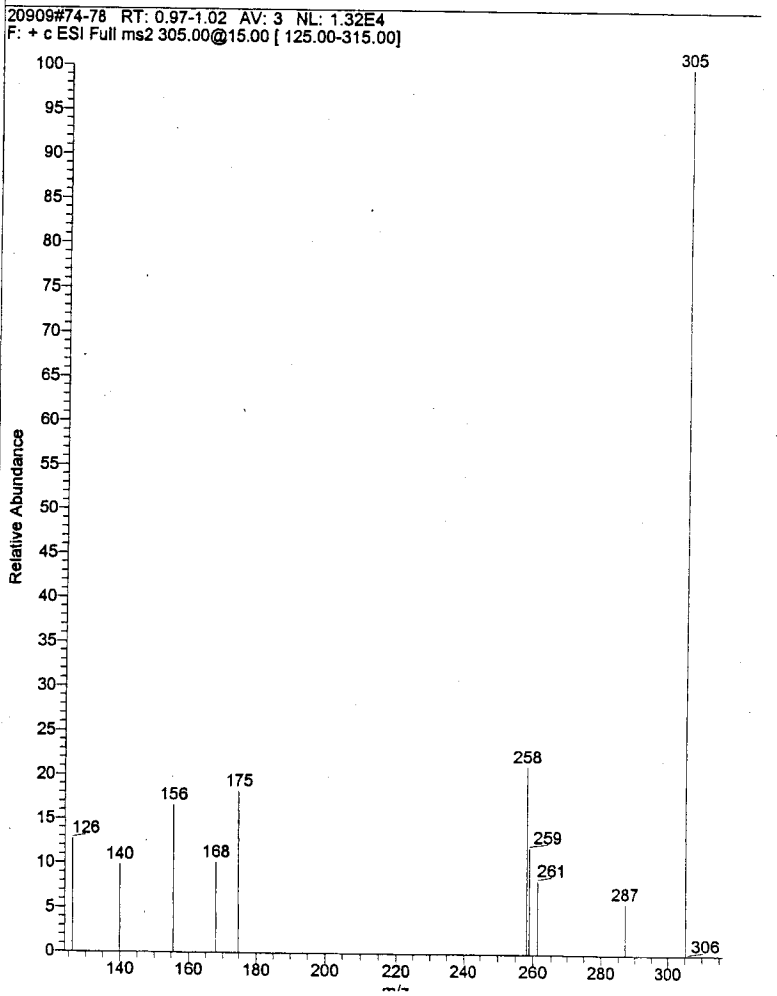






E4446 (522)





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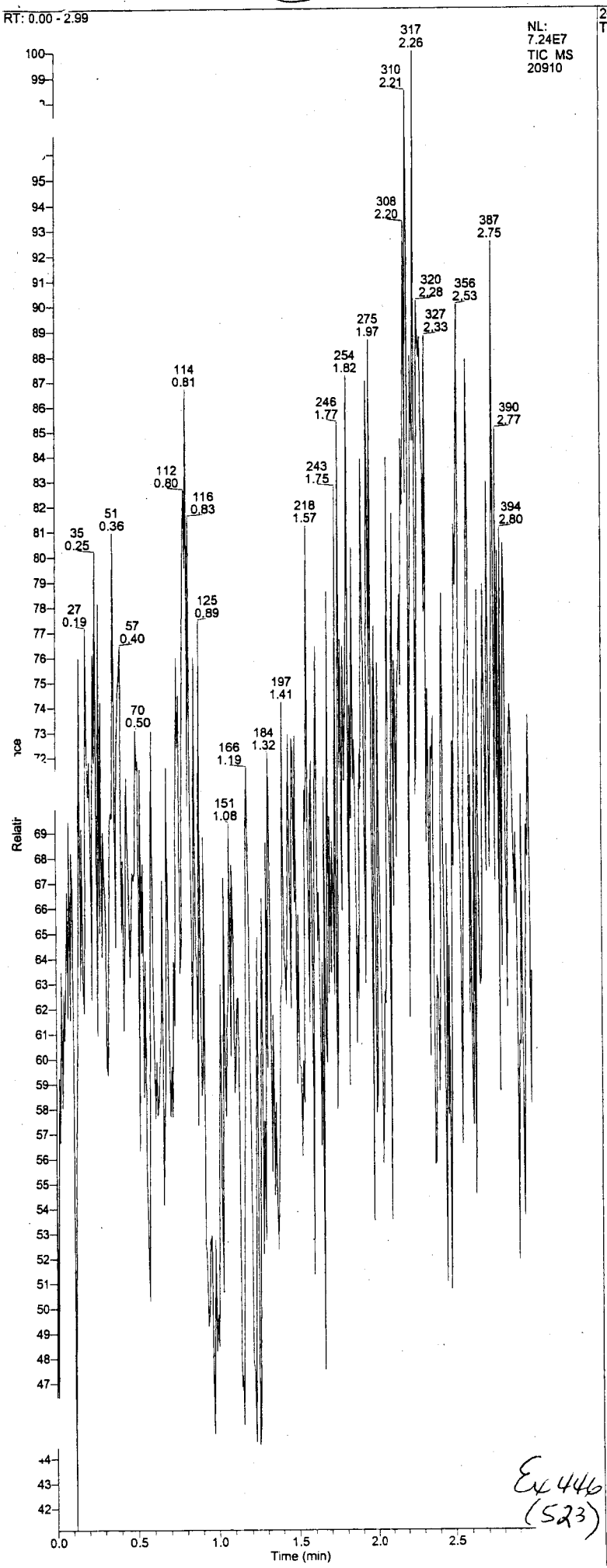

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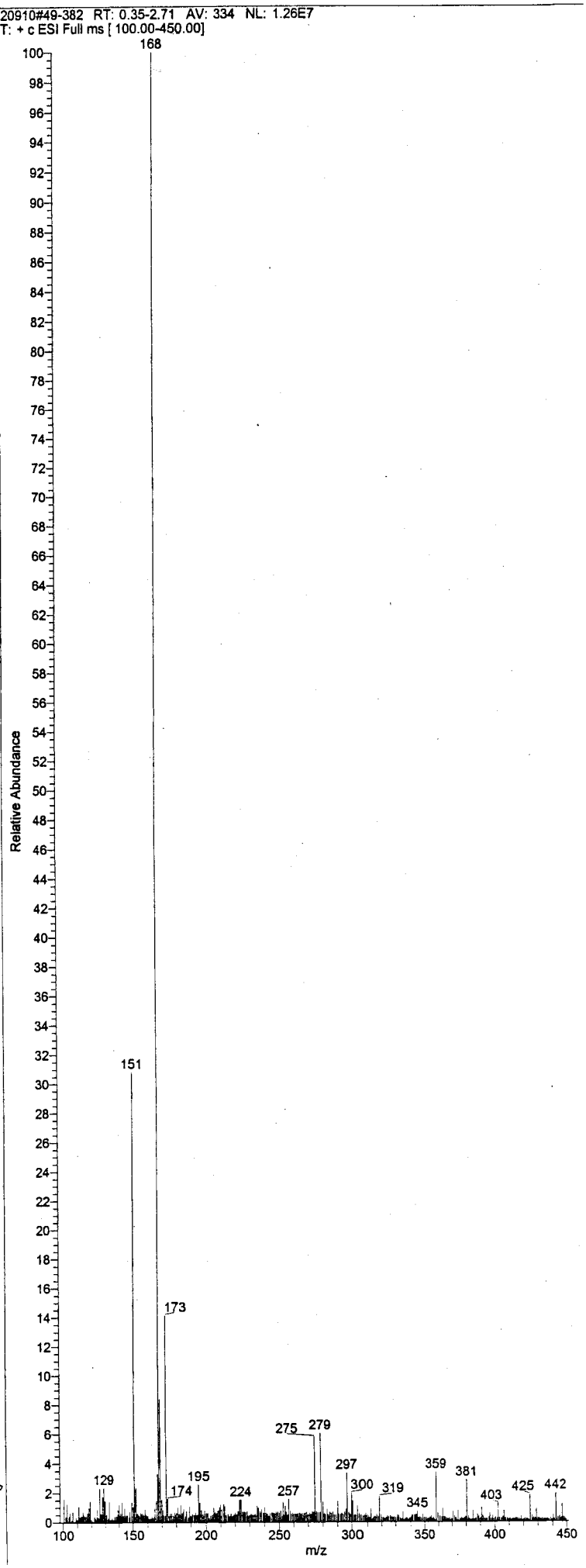

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Ex 446
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The Analysis of EDTA in Dried Bloodstains by Electrospray LC–MS–MS and Ion Chromatography*

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Abstract

Analytical methods were developed to determine the presence of ethylenediaminetetraacetic acid (EDTA) in dried bloodstains to provide probative information when allegations of evidence tampering have been made in criminal cases. A simple screening method using ion chromatography to analyze stains was found to be quantitative to the 5 ppm level. The presence of EDTA was then confirmed using negative and positive ion mode liquid chromatography–tandem mass spectrometry (LC–MS–MS) methods. A blind trial of these methods on 42 samples correctly determined the bloodstains that did and did not contain the preservative EDTA. One interesting observation in these results was the adsorption and postanalysis release of EDTA in the chromatographic system. In order to avoid cross contamination of samples resulting from this phenomena, it was found to be necessary to use EDTA-free blood extracts as blanks in the LC–MS analysis of bloodstains.

Introduction

The collection of blood at crime scenes and for legal proceedings is a common practice used to inculpate or exculpate individuals associated with evidentiary blood at crime scenes. Allegations of "planting" blood evidence from collected reference specimens has occurred in some criminal investigations, and this issue may be resolved by the determination of exogenous components that would not ordinarily be present in authentic crime scene evidence. Ethylenediaminetetraacetic acid (EDTA, also known as edetic acid, $C_{10}H_{16}N_2O_8$, molecular weight, 292.24) (Figure 1), a chemical commonly added to collected blood specimens, can be used to implicate the origin of a dried bloodstain as coming from this type of preserved specimen tube. The purpose of the EDTA in the tube is to prevent coagulation and enzymatic degradation by chelating metals in the blood that behave as catalysts and/or cofactors.

EDTA-preserved blood tubes use the salt forms of EDTA: the disodium, dipotassium, or tripotassium salt. The concentration of EDTA in its free acid form in a drawn blood tube is 1000–2000 mg/L (ppm), depending on the volume of blood and the capacity of the tube. The free acid and salt forms are all water soluble at this concentration. EDTA is stable on storage and on boiling in aqueous solutions, but it does decarboxylate when heated to temperatures of 150°C (1). It is an excellent complexing agent and forms water-soluble chelates with nearly all heavy metals. Therefore, aqueous extractions of dried bloodstains should readily isolate EDTA in solution.

EDTA is used as a chelating agent in a variety of materials, and several chromatographic methods have been developed for its determination. A number of the methods employ reversed-phase ion-pair liquid chromatography (LC) for analysis of EDTA in foodstuffs (2,3), water (4,5), radioactive waste (6), and pharmaceuticals (7). Gas chromatographic (GC) methods have also been developed for the detection of EDTA in foodstuffs (3). Ion chromatography (IC) is an additional logical approach for the analysis of EDTA (6). All of the previously mentioned LC methods use ultraviolet detectors and lack the specificity of liquid chromatography–mass spectrometry (LC–MS). An additional level of selectivity in the analysis of EDTA can be added by the use of LC–MS–MS. A simple extraction technique coupled with positive ion and negative ion LC–MS–MS methods was developed for the analysis of EDTA in preserved dried bloodstains. Pneumatically assisted electrospray (ES) was used to ionize the chromatographic effluent before mass spectral analysis of the charged species. A secondary method using ion chromatography was developed to provide a quantitative presumptive test for the presence of EDTA as well as corroborate the results of the LC–MS–MS analysis. In a blind trial conducted on 42 bloodstains using the same extraction protocol for IC and LC–MS–MS, problems occurred in the LC–MS–MS analysis. Subsequently, a refined extraction method was developed for LC–MS–MS analysis of dried blood.

*This is publication number 97-04 of the Laboratory Division of the Federal Bureau of Investigation. Names of commercial manufacturers are provided for identification only, and inclusion does not imply endorsement by the Federal Bureau of Investigation.

Ex 446 (525)

Experimental

Chemicals

American Chemical Society reagent-grade ammonium hydroxide, disodium EDTA, cupric sulfate, and high-performance liquid chromatography (HPLC)-grade acetonitrile were purchased from Sigma Chemical (St. Louis, MO). HPLC-grade methanol was obtained from EM Science (Gibbstown, NJ). Certified sulfuric acid (2.5M) and sterile Vacutainer blood tubes without additives (red top) and with EDTA (K_3) (lavender top) were obtained from Fisher Scientific (Fairlawn, NJ). Nanopure water from a Barnstead (Dubuque, IA) water purification system was used for samples and mobile phases. The deuterated standard of EDTA-d_{12} (Figure 1) was purchased from Cambridge Isotope Laboratories (Andover, MA).

Instrumentation

Ion chromatographic analysis was carried out using a Waters 510 HPLC pump (Milford, MA) coupled to a Hamilton (Reno, NV) PRP X-100 ion chromatographic column. Sample detection was performed using a Spectroflow-773 tunable absorbance detector (Kratos Analytical Instruments, Westwood, NJ.) Instrument control and signal processing were performed using a Millenium 2010 chromatography manager (Waters, Milford, MA). Additional ion chromatographic analyses were performed using a Waters 600 analytical HPLC coupled to Waters 990 photodiode array detector.

The LC–MS–MS work was performed on a Hewlett-Packard (Palo Alto, CA) HP 1090 ternary LC with autosampler connected to a Finnigan MAT (San José, CA) TSQ 700 triple-stage quadrupole MS using a Finnigan electrospray interface. Argon was used as a collision gas for MS–MS. The instrument was set up to scan the mass range in 0.5–2 s. A flow rate of 0.3 mL/min was used on a Hamilton (Reno, NV) PRP-1 polymeric column (2.1 × 150 mm).

Procedure

Test samples were made by drawing whole blood samples into unpreserved and EDTA-containing tubes. The bloodstains were prepared on the same day by applying between 2 and 50 µL of unpreserved or EDTA-containing blood onto sterile cotton linen. Additional samples were prepared using liquid whole blood from a laboratory volunteer.

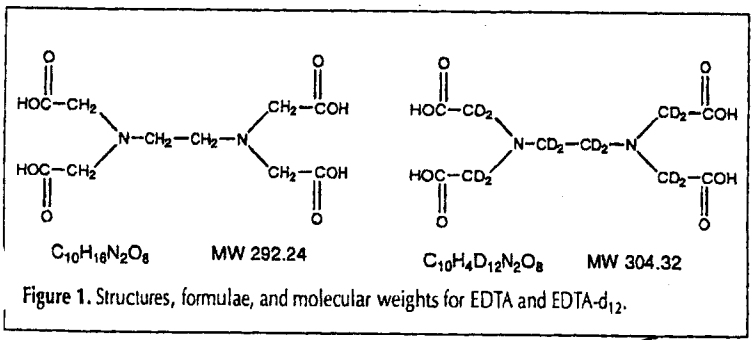

Figure 1. Structures, formulae, and molecular weights for EDTA and EDTA-d_{12}.

For the initial study, 25 to 50% of the stained area (up to $^1/_2$ cm^2) was cut out of the cotton swatch. The cutting was placed in 50 or 100 µL of 0.025M copper (II) sulfate (enough to cover sample). The samples were soaked in solution for 3 or more hours before vortex mixing and centrifuging at 3000–9000 rpm for 10 min. After passing the sample through a 0.2-µm nylon syringe filter, injections of 25 µL were made for IC analysis. The IC mobile phase was 3mM sulfuric acid/methanol (95:5). The flow rate was 2 mL/min with a detector wavelength of 254 nm. This wavelength was subsequently changed following analysis by a UV photodiode array detector that indicated the peak absorbance maximum for the copper/EDTA complex occurs at 243 nm. Following IC, the residual copper extract samples were diluted with 25 µL of water and 1-µL injections were made for positive ion LC–MS–MS. This preparation method was used for all IC analyses and for the initial run by LC–MS–MS of the 42 blind trial samples. Subsequent LC–MS–MS analysis samples were prepared by the procedure given in the next paragraph.

A different extraction procedure was developed for LC–MS–MS analysis to eliminate copper (II) sulfate, which caused arcing problems in the electrospray interface, from the extract. This procedure was used to prepare samples for both negative and positive ion LC–MS–MS but not IC analysis. A portion (up to $^1/_2$ cm^2) of the bloodstain was extracted by inserting the sample into Millipore (Bedford, MA) Ultrafree-MC centrifugal filters made of a polysulfone membrane (type PTTK) with a nominal molecular weight cutoff of 30,000 Daltons. After the addition of 25 µL of water, the sample was allowed to sit at room temperature for 45 min. The filter tubes were centrifuged for approximately 10 min, and the filtrate was collected for analysis.

Positive ion LC–MS–MS data were collected by scanning for product ions of $(M + H)^+$ at 293 u from 128–296 u at a collision offset of –20 V. The interface was set for a spray voltage of 4 kV, a sheath gas pressure of 90 psi, and an auxiliary gas flow of 5 units. The interface capillary was maintained at 200°C. A mobile phase of acetonitrile and water (5:95) with 0.06% ammonium hydroxide was used.

Negative ion LC–MS–MS data were acquired by scanning for the 300 u product ion from the iron adduct of EDTA at 344 u. A scan window of 298–302 u was employed. The collision offset for selected reaction monitoring of the 44 u mass loss was 20 V. The interface was set for a spray voltage of 4.5 kV, a sheath gas pressure of 50 psi, and an auxiliary gas flow of 5 units. The heated capillary was set for 200°C. The optimized-column mobile phase for negative ion was found to be acetonitrile and water (80:20) with 0.03% ammonium hydroxide.

The isotopic pattern calculation was performed on the ChemPuter from the Department of Chemistry at the University of Sheffield, Sheffield, England[1]. The experimental isotope pattern was calculated by adding six scans together and the same number of background scans were subtracted to obtain the result.

[1]The web address for the ChemPuter is http://www.shef.ac.uk/~chem/chemputer/isotopes.html.

Ex 446 (526)

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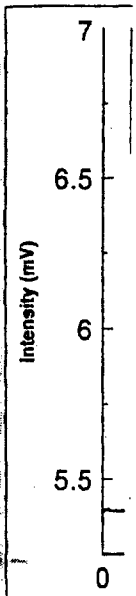

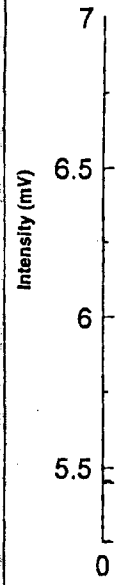

Figure 2. Io
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Results and Discussion

A review of the literature indicated that the best chromatographic approach for the determination of EDTA used HPLC or IC analysis of colored complexes formed between EDTA and copper or iron (2–6). Although procedures exist for performing this analysis by GC, these methods require time-consuming sample derivatization and are prone to matrix interferences (3). Recent applications published by Hamilton indicated acceptable separations of EDTA-copper complexes could be carried out using the ion exchange column PRP X-100 with sulfuric acid/methanol mobile phases (8). Initial IC testing with UV detection was carried out on standard samples of 50–100 ppm EDTA dissolved in 0.025M copper (II) sulfate. The results with this eluent system were encouraging; therefore, further tests were carried out on samples of liquid blood. IC samples were prepared from 200 µL of whole blood (EDTA preserved) by first diluting it to 2 mL with water and then diluting the solution 1:1 with 0.05M copper (II) sulfate. This preparation was centrifuged for 7 min, which left a clear supernatant with a brown precipitate at the bottom. A large excess of copper (II) sulfate helped to ensure conversion of all free EDTA to the copper complex. The

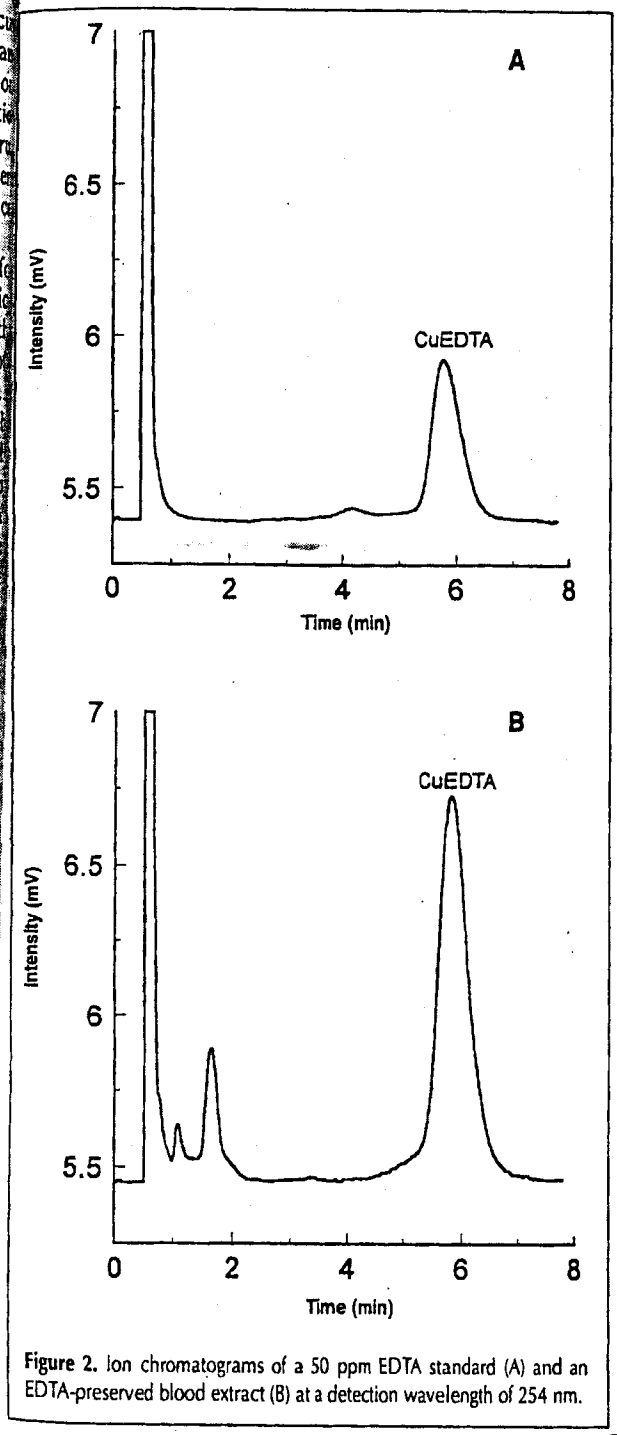

Figure 2. Ion chromatograms of a 50 ppm EDTA standard (A) and an EDTA-preserved blood extract (B) at a detection wavelength of 254 nm.

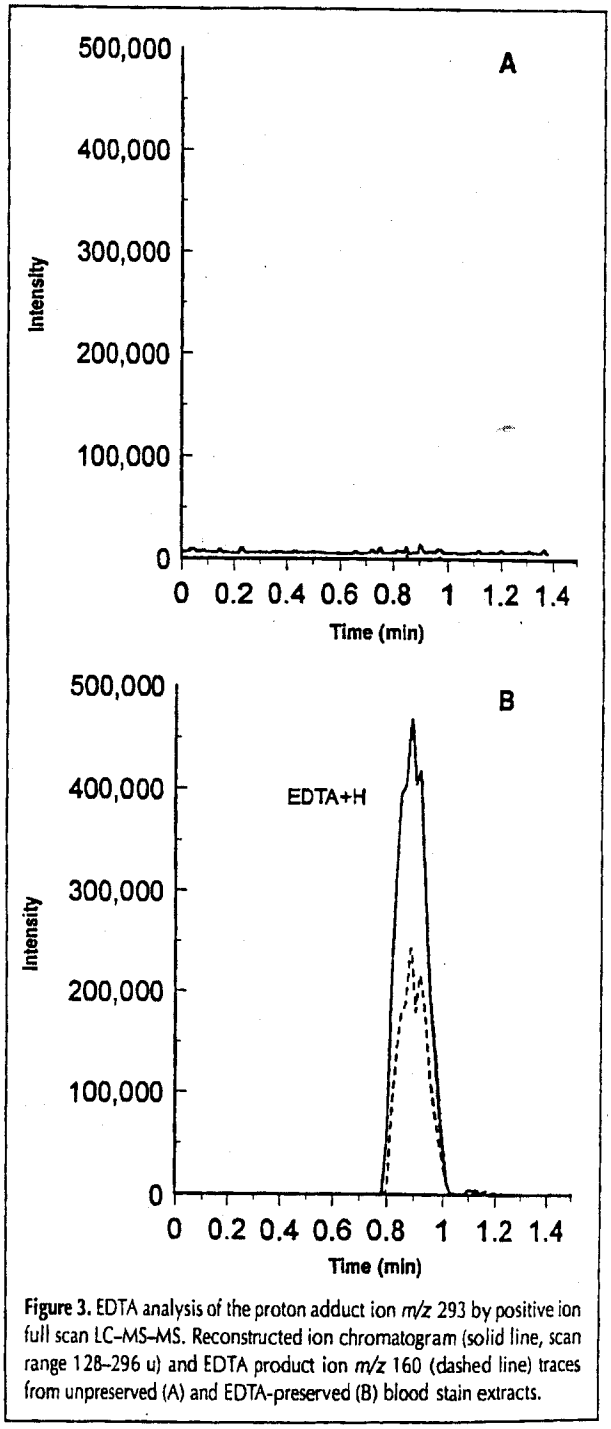

Figure 3. EDTA analysis of the proton adduct ion *m/z* 293 by positive ion full scan LC–MS–MS. Reconstructed ion chromatogram (solid line, scan range 128–296 u) and EDTA product ion *m/z* 160 (dashed line) traces from unpreserved (A) and EDTA-preserved (B) blood stain extracts.

Ex 446
(527)

result of this analysis is shown in Figure 2. Similar analyses using $FeCl_3$ and $Fe_2(SO_4)_3$ to complex with EDTA did not produce precipitates and showed large interfering peaks. As a result, all further tests were performed using Cu(II) sulfate at a concentration of 0.025M in each sample. In a set of serial dilutions in water, the analysis was shown to have a linear range from zero to greater than 500 ppm EDTA with a minimum detectable quantity of 5 ppm EDTA for the injected sample.

A Hamilton PRP-1 column was used to separate EDTA for MS detection in order to reduce interference from other compounds in the blood. Other blood components were retarded on the column and minimized peak overlap with EDTA, which has a short retention time. IC mobile and stationary phases were not used for the MS procedure because of the strong buffer ion concentrations required for ion exchange that led to electrical arcs in the electrospray interface. An additional reason

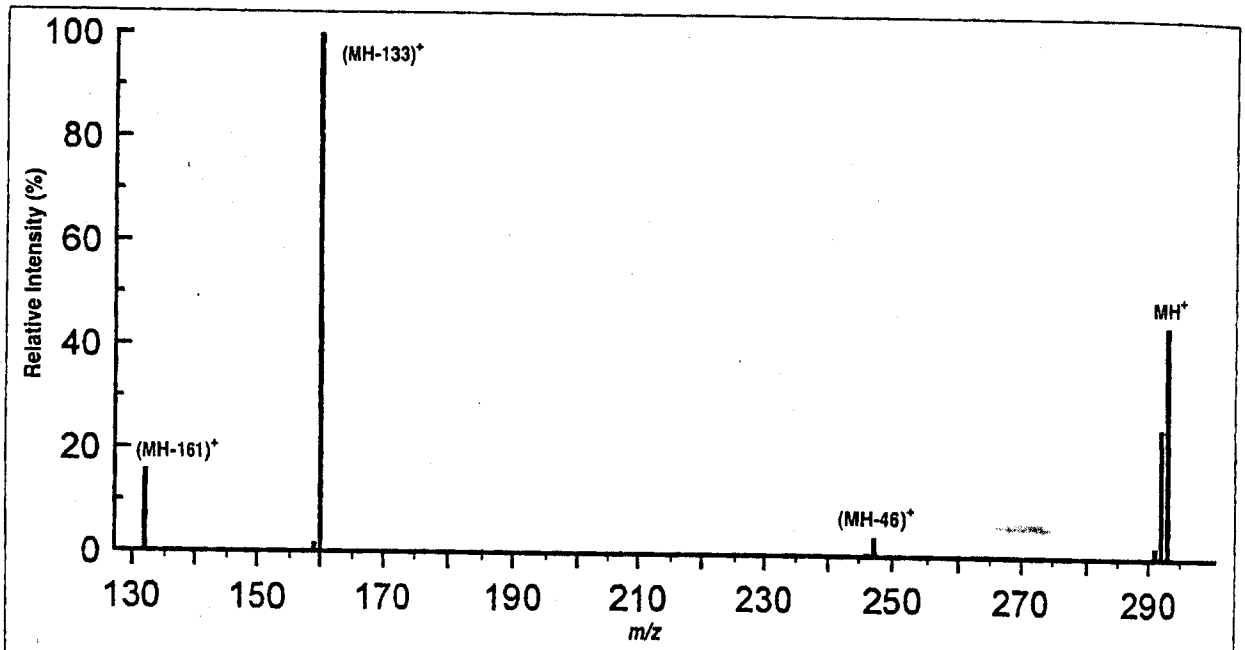

Figure 4. Positive ion LC–MS–MS product spectrum from collision-induced dissociation of EDTA ion (M+H)+ at *m/z* 293.

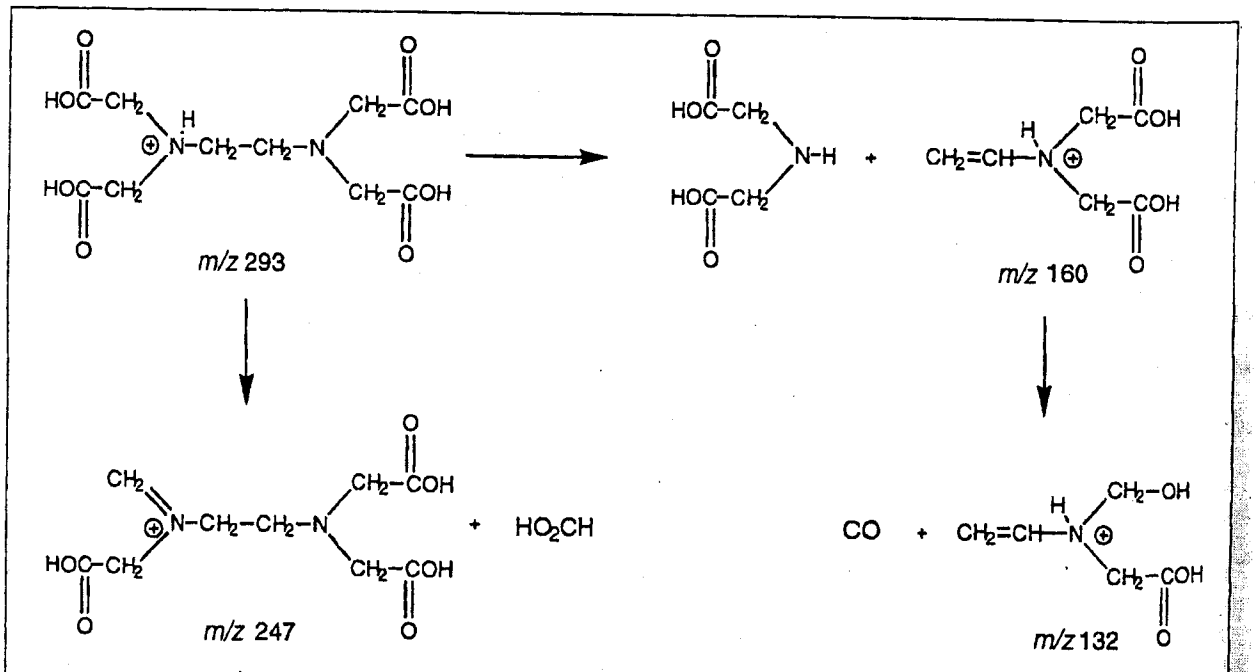

Figure 5. Fragments observed by collision-induced dissociation of EDTA proton adduct ion (*m/z* 293) in positive ion LC–MS–MS analysis of EDTA-preserved blood.

Ex 446
(528)

for keeping the ionic strength of the mobile phase minimized is higher ion concentrations may suppress analyte ionization (9).

Standard sample solutions (10–100 ppm) of disodium EDTA in water yielded abundant adduct ions by electrospray sample loop injection (flow injection analysis, FIA) or LC–MS in the positive ion mode. However, analyses of disodium EDTA with or without chromatography gave variable spectra as a result of the formation of numerous metal complexes with EDTA. The EDTA adduct ion $(M + H)^+$ at *m/z* 293 was observed even though the mobile phase was alkaline. Other charged species observed in standard sample solutions and their proposed identities were as follows: *m/z* 315, $(M + Na)^+$; *m/z* 317, $(M - 2H + Al^{III})^+$; *m/z* 337, $(M - H + 2Na)^+$; *m/z* 346, $(M - 2H + Fe^{III})^+$; *m/z* 368, $(M - 3H + Fe^{III} + Na)^+$; and *m/z* 390, $(M - 4H + Fe^{III} + 2Na)^+$. The electrospray interface is constructed of stainless steel and aluminum and accounts for the presence of Fe^{III} and Al^{III} complexes in analyses of disodium EDTA. The electrolytic nature of electrospray has been shown to form iron complexes from the stainless steel spray needle (10).

Negative ion FIA–MS (sample loop injection) or LC–MS generated several ions with samples of disodium EDTA including $(M-H)^-$ at *m/z* 291 and the doubly charged ion $(M - 2H)^{-2}$ at *m/z* 145. Adduct ions were also observed in negative ion mode with the same metals (Fe^{III} and Al^{III}) as positive ion mode. Complexed species indicated from FIA–MS or LC–MS and their proposed identities were: *m/z* 313, $(M - 2H + Na)^-$; *m/z* 315, $(M - 4H + Al^{III})^-$; *m/z* 335, $(M - 3H + 2Na)^-$; and *m/z* 344, $(M - 4H + Fe^{III})^-$. The negative ion spectra were also poorly reproducible in the relative intensity of the various ions for standard runs of disodium EDTA.

Positive ion LC–MS was also examined using a series of mixtures of acetonitrile and 0.06% ammonium hydroxide. The mobile phase (5:95) was selected as it gave the best response and consistency for EDTA analysis. This mobile phase gave $(M + H)^+$ ions at *m/z* 293 for the disodium EDTA standard (10 to 100 ppm) and was the base peak for EDTA in preserved blood samples. No prominent fragment ions were observed in the spectrum of EDTA, and thus it was necessary to analyze samples by LC–MS–MS to obtain structurally significant ions for identification (Figure 3). In the blood samples, no interferences were found in the reconstructed ion (RIC) trace for the MS–MS of ion 293. MS–MS of the 293 ion generated three product ions with a base peak at 160 u and two smaller ions at masses 132 and 247 u (Figure 4). The three-product ions (132, 160, 247) and their associated losses are consistent with the known EDTA-d_{12} spectrum which has product ions at *m/z* 140, 168, and 259, respectively (Figure 5). The neutral losses correspond to carbon monoxide, a di-carboxylic acid secondary amine, and formic acid.

A blind trial of the analysis procedures was performed independently by IC and by positive ion LC–MS–MS. Forty-two dried bloodstain extracts prepared for IC analysis were analyzed in the blind study to determine if EDTA preserved blood could be distinguished from unpreserved blood spots. Although the samples were diluted with 25 µL of water before LC–MS–MS, some electrical arcs still occurred in the electrospray interface because of the copper (II) sulfate in the samples. The volume of the original bloodstain samples ranged from 2 to 50 µL. Although all stains containing EDTA ($n = 21$) were correctly identified using the IC technique, LC–MS–MS correctly determined 20 of the 21 positive samples. Both techniques correctly identified all of the negative samples ($n = 21$). A stain sample that gave a positive result in the IC test had indications of EDTA by the LC–MS–MS procedure but was considered too weak to be called positive on a single result. This was an extract of half of the smallest EDTA blood spot (2 µL) in the 42 samples (i.e., approximately 1 µL). At the time of the testing all other positives gave significant area counts (several hundred thousand) for the 160 u product ion, and both 132 and 247 u ions were present. The false-negative result had an area count of 80,000 for ion 160, and both other product ions were present. A conservative decision was made to interpret the sample as negative until further testing could be completed.

A revised extraction method was developed for LC–MS–MS after the initial testing to eliminate the cupric sulfate-induced arcing problems in the interface and to obtain more concentrated sample extracts. A simple procedure was devised to extract the stains in centrifugal filters after a 45-min soaking in water. A molecular weight cutoff of 30,000 Da was chosen to remove particulate matter, blood cells, and large proteins from the filtrate while still maintaining an adequate flow through the filter disc. A retest by positive ion mode LC–MS–MS of several dried stains (preserved and unpreserved blood spots) produced positive results for all of the EDTA containing blood with area counts of several hundred thousand for the 160 ion and negative results for all unpreserved spots. The sample that previously had been deemed a negative (2-µL EDTA blood spot) gave an area count of 1,000,000 for ion 160 by the revised method. Two of the positive samples were extracted a second time, and it was found that, on average, 91% of the EDTA response (peak area for 160 product ion) was in the first extract.

In the negative ion mode, the ferric ion complex with EDTA at *m/z* 344, $(M-4H+Fe^{III})^-$, consistently appeared in analyses of the disodium EDTA standard and was the base peak for EDTA in preserved blood samples. The identity of the 344 ion was verified by comparing the calculated isotopic formula for $(C_{10}H_{12}N_2O_8Fe)$ with experimental data (Table I). No prominent structural

Table I. Calculated and Experimental Intensity (%) of the Molecular Ion Cluster $(C_{10}H_{12}N_2O_8Fe)$ for the Iron Complex with EDTA $(M - 4H+Fe^{III})^-$ Observed by Negative Ion LC–MS–MS for an EDTA Standard

m/z	Predicted intensity	Experimental intensity
342	6.3	6.4
343	0.8	2.1
344	100	100
345	14.7	14.7
346	2.9	2.1
347	0.3	0.6
348	0	0.2

EX446
(529)

peaks were observed in the LC–MS spectrum of this complex. MS–MS of the 344 ion gave a strong signal for the product ion at *m/z* 300 $(M - CO_2)^-$. A second LC–MS–MS method, in negative ion mode, was developed to screen for the presence of EDTA in bloodstains by observing the mass-to-charge ratio transition from 344 to 300 in the selected reaction monitoring mode (SRM). This method of MS–MS operation is selective and sensitive because the instrument scans over a limited mass range while measuring a specific transition resulting from collision-induced dissociation. The MS–MS ion trace of *m/z* 300 and the RIC show no extraneous signals other than the major peaks for the EDTA derived species from blood extracts (Figure 6). Some known ($n = 4$) and unknown ($n = 2$) blood samples were successfully tested as a trial of the negative ion method.

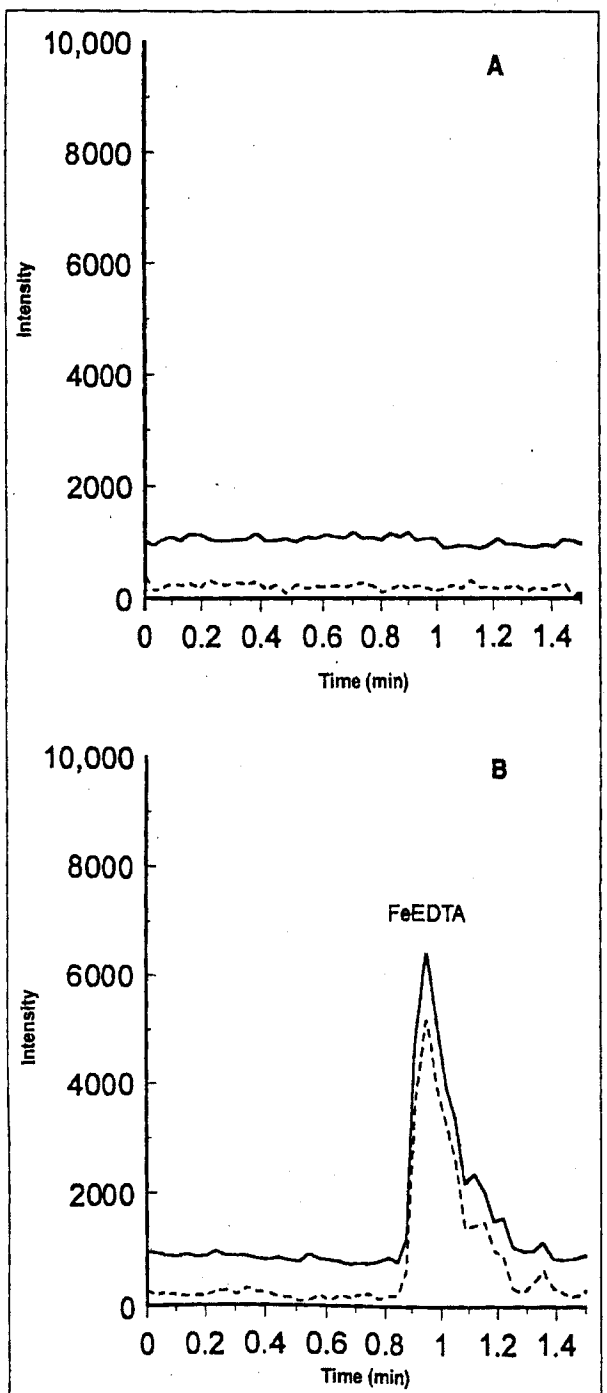

Figure 6. EDTA analysis of the iron adduct ion at *m/z* 344 by negative ion SRM LC–MS–MS. Reconstructed ion chromatogram (solid line, scan range 297–302 u) and EDTA product ion *m/z* 300 (dashed line) traces from unpreserved (A) and EDTA-preserved (B) blood stain extracts.

A comparison of negative ion and positive ion LC–MS–MS of a blood stain revealed an 80-fold difference in intensity of EDTA signal for the 160 ion (positive) over the 300 ion (negative). Because of the use of MS–MS in both techniques, responses from other components in the blood were not observed, and, therefore, both analyses were deemed selective. The positive ion mode is a better means of confirmation because there are three structurally significant product ions compared with only one for the negative ion mode.

The IC, negative ion and positive ion LC–MS–MS methods were used on investigative case samples ($n = 9$) to determine if evidentiary crime scene blood might have been tampered evidence. A phenolphthalein-presumptive test for blood (11) was positive on all tested stains ($n = 4$). In addition, all extracts of the stains appeared red in color as supportive evidence for the presence of blood. All three techniques were negative for EDTA in the bloodstains. However, in the positive ion SRM (*m/z* transition 293 to 160) LC–MS–MS analysis of one of the stains from a sock, a small peak appeared with the correct retention time for EDTA. IC and negative ion results did not indicate EDTA in the sample and positive ion full scan LC–MS–MS could not confirm all three EDTA product ions from the parent ion at 293 u. In known EDTA samples, the three techniques had consistently agreed, and intensities were strong enough for the known samples to confirm the three product ions by MS–MS of the parent ion at 293 u. A potential criticism of the evaluation of the results is the inability to determine the exact quantity of blood in a sample. In these studies, it was found that sample sizes as small as 1 µL of blood generated more than adequate signal for EDTA. A sample of this size would leave a dried blood spot of 0.1 cm^2 on a swatch of cotton linen.

A study was conducted to determine if the small unconfirmed signal observed in the case sample could result from a carry-over in the system. An EDTA-d_{12} standard was used for this work to prevent interference from endogenous levels of EDTA. After several injections of the EDTA-d_{12} standard (500 ppm), blank samples of different substances were run. No signal by LC–MS–MS was observed after a single injection of blanks such as water, mobile phase, or salt solutions. However, injection of EDTA free blood extracts, following a blank injection, gave a response for the EDTA-d_{12} compound that decreased with each repetition (Figure 7). The low signal in the case mentioned above was likely a result of an interference resulting from the previously injected standard. It is theorized that, when a blank blood matrix is injected onto the system, residual EDTA adsorbed onto sites in the column and tubing is released by competing metal ions in the blood extract. Therefore, it is recommended that the only suitable blank after injections of EDTA is EDTA-free blood extract. Multiple injections of EDTA-free blood extract should be made until no observable

EDTA peak is detected before any case samples are analyzed by this method.

EDTA is an additive in a variety of foods, such as pickles, canned mushrooms, and salad dressings, and potentially could occur in human blood at low levels. However, in a study of radio-labeled EDTA, ingested samples were found to be eliminated mainly by excretion with minimal gastrointestinal tract absorption (12). Thus, it is unlikely that measurable quantities would be found in the blood by the employed techniques. Attempts to measure EDTA in unpreserved blood by the use of samples up to 1 mL in volume were negative. A search of the literature did not find any measurements of EDTA in blood from dietary consumption levels.

Another concern in the analysis of bloodstains is the stability of EDTA in the dried spots after extended periods of storage. Samples of EDTA preserved bloodstains (n = 2) were analyzed after 2 years of storage at room temperature. LC–MS–MS analysis of the additive-free samples were negative and the preserved samples were positive for EDTA.

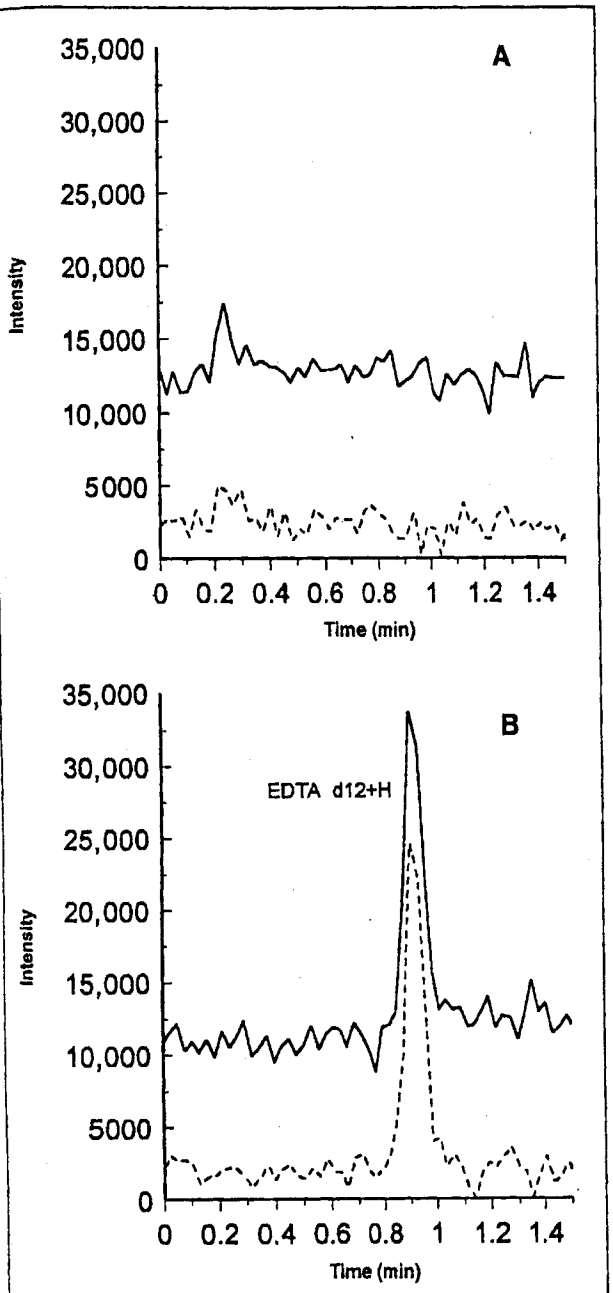

Figure 7. Sample carry over experiment to demonstrate the matrix effect of blood extracts. The top chart (A) is an injection of mobile phase after five injections of 500 ppm EDTA-d_{12} standard. The chart on the bottom (B) shows the results of injecting an extract from a non-EDTA-preserved blood tube after the blank analysis in (A). EDTA-d_{12} analysis of the proton adduct ion m/z 305 by positive ion full scan LC–MS–MS. Reconstructed ion chromatogram (solid line) and EDTA-d_{12} product ion m/z 168 (dashed line) traces.

Conclusion

Methods described herein demonstrate the ability to determine if tampering of bloodstains may have occurred using EDTA-preserved blood. The coupling of a quantitative technique with the specificity of the LC–MS–MS procedures is a powerful analytical protocol. Specific procedures were developed to identify and control the effect of matrix interference on the samples. Matrix interference can be minimized by injecting EDTA-free blood extracts before the analysis of any samples. The accuracy of the determination was supported through the use of simulated investigative case samples. Experiments on aged, dried blood indicate it is possible to determine EDTA in stains after at least 2 years of storage.

Acknowledgments

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Ex 446
(531)

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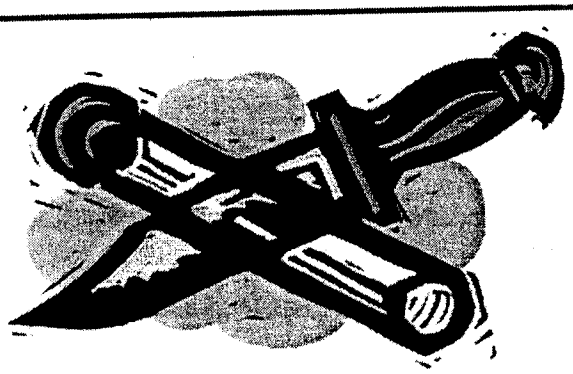
Ex 446
(532)

Analytical Chemistry

August 1, 1997

Analytical Chemistry 1997, 69, 477A-480A.



Determining EDTA in Blood

A murder trial sheds light on the need for a better analytical method

Robin L. Sheppard
Jack Henion
Cornell University

It is not often that a story on the national evening news uses words such as "liquid chromatography" and "mass spectrometry". Analytical chemists who heard such reports during the murder trial *The State of California v. Orenthal James Simpson* immediately perked up their ears, amazed that the analytical details of FBI laboratory testing were actually making headlines.

The subject of the testing was EDTA (ethylenediaminetetraacetic acid) issue was whether police had "planted" or tampered with blood evidence in an attempt to shore up the case against Simpson. The possible outcomes of the testing were simple: Either EDTA was present or it wasn't. Qualitative testing began (*1*), but as is usually the case, nothing is that simple. There was evidence of some EDTA, at levels much lower than in EDTA-preserved blood. The questions "How much EDTA is there?" and "Are the detected levels consistent with 'normal' levels or those that would result from tainted blood collected in EDTA anticoagulant blood tubes?" arose immediately.

The lead prosecutor, Marcia Clark, tried her best to present this scientific evidence. But how do you convince a jury of citizens that knows little about analytical chemistry that the EDTA came not from a lavender-stoppered tube but from a bleeding O.J. Simpson? Although it may not have been the only weak point in the prosecution's case, it certainly was a factor in the trial's outcome. Because of this criminal case, determining EDTA in human blood has become a topic of renewed interest.

What was wrong with the laboratory testing? First, it was not clear whether the method had ever been

used before. Most likely the method was developed quickly under a great deal of time pressure. In retrospect, FBI chemists now believe that the EDTA detected may have been injection carryover in the LC/MS/MS (*2*) instrumentation because a water blank instead of a matrix blank had been run before the sample. Second, the EDTA concentration was not rigorously quantitated. Certainly, the volume of the blood stain could have been estimated. EDTA is present at about 4.5 mM (~1300 ppm) in EDTA-preserved blood, which would be a very concentrated sample and easily detected by electrospray LC/MS/MS. It appeared that the amount of EDTA detected in the forensic blood samples was orders of magnitude below 4.5 mM. Regardless of what happened in the Simpson trial, it became apparent that a definitive and valid method for determining EDTA in human blood was needed.

EDTA BASICS

EDTA is a metal-complexing agent that has been popular since its commercialization in the early 1950s (*3*). The free-acid structure with a molecular weight of 292.1 is shown in Figure 1a, and a three-dimensional representation of EDTA complexed with nickel(II) with a molecular weight of 347.0 is shown in Figure 1b. EDTA has four acidic protons that are sequentially ionized at solution pH values of 2.0, 2.67, 6.16, and 10.26, respectively (*4*). The disodium salt is commonly used as an anticoagulant (*5*), and the familiar lavender-stoppered blood collection tubes contain enough EDTA to give a final concentration of ~ 4.5 mM. Upon mixing with blood, the EDTA immediately chelates the available calcium. Because calcium is necessary for the formation of fibrin, coagulation cannot take place (*5*).

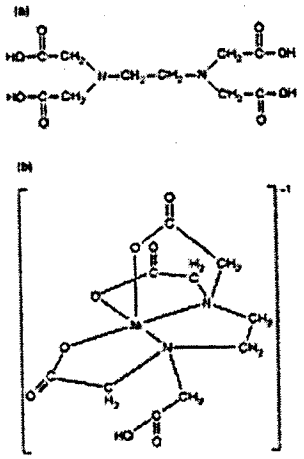


Figure 1. (a) EDTA's free-acid structure and (b) three-dimensional structure of Ni-EDTA.

EDTA is also used extensively as a food preservative, a water-softening agent, and to deliver trace minerals in animal feeds (*6*). Despite its ubiquitous presence, metabolism studies have shown that little, if any, EDTA should be present in human blood. In 1954, a metabolism study using $^{14}C_4$-labeled calcium-EDTA given intravenously showed that EDTA was detectable in the plasma but not in the blood cells (*7*). On average, 95% of an oral dose was recovered in the urine and feces within three days of administration with no EDTA detected in the plasma, and the remaining 5% was detected in the urine within 18 h. More recent metabolism studies using the NaFe(III)-EDTA complex report that it dissociates during digestion and confirm that only about 5% of the EDTA is absorbed and excreted in urine (*8*).

DETERMINING EDTA IN BLOOD AND PLASMA

Although there are numerous published methods for determining EDTA in various matrices such as

mayonnaise (*9*), wastewater (*10*), and ophthalmic solutions (*11*), our lab and one other group have recently attempted to develop improved methods that could be used for the forensic measurement of EDTA in biological matrices (*12*, *13*).

There are numerous ways to prepare samples containing free or chelated EDTA. The first and most obvious is no sample preparation at all. If the pH of the sample is not adjusted and if metal contamination can be eliminated, the natural distribution of EDTA and its metal chelates can be determined. This is not the most sensitive approach because the EDTA signal would be distributed among several different metal species or peaks. In addition, quantitation would be difficult because any change in the sample matrix could change the complex equilibria. Therefore, all the methods used to measure EDTA itself focus on preparing a singular EDTA-containing peak or compound.

For GC, the analyte must be reasonably volatile and thermally stable. Because EDTA is not volatile, it is usually derivatized by esterifying the four carboxyl groups with methanol, butanol, or isopropyl alcohol (*13-15*). This liquid-liquid extraction is a labor-intensive, multistep procedure that is difficult to automate. For LC or capillary electrophoresis (CE), the most logical way to convert all available EDTA to a single compound is to add an excess of one metal that strongly complexes with EDTA. Although iron(III) and copper(II) are commonly used (*11*, *16*), we found that nickel(II) complexes with EDTA just as effectively, and it has several advantages: Heating is not required, the complex is stable down to pH\t3, and the complex gives a higher signal by ion spray MS than either the iron or copper complexes.

DEVELOPING A CE/MS/MS METHOD

Several reports in which CE was used to determine metals using EDTA as a complexing reagent have been published in the literature (*17*, *18*). Using these reports as a starting point, we predicted that an "MS-friendly" CE separation could be developed that would minimize the chemical additives introduced into the mass spectrometer.

The intricacy of interfacing the capillary to a mass spectrometer is the reason the technique is considered nonroutine. Although many qualitative CE/MS applications have been reported, very few quantitative methods have been described in the literature (*19*, *20*). Therefore, we used this opportunity to achieve two goals: to offer an improved technique for measuring EDTA in biological matrices and to demonstrate that CE/MS can be used in a routine manner for quantitative bioanalytical applications.

Although MS may not be the most sensitive CE detector available, it offers a high level of selectivity as well as wider applicability than, for example, laser-induced fluorescence (*21*). For example, the full-scan CE/MS separation shown in Figure 2a displays the EDTA distributed as five different metal complexes. Figure 2b shows the same EDTA standard in which the predominant ion *m/z* 347, caused by Ni-EDTA, has been extracted from the total ion current data. This type of selectivity can be exploited by using the selected ion monitoring mode in which only the ion(s) of interest would be detected.

Ex 446
(535)

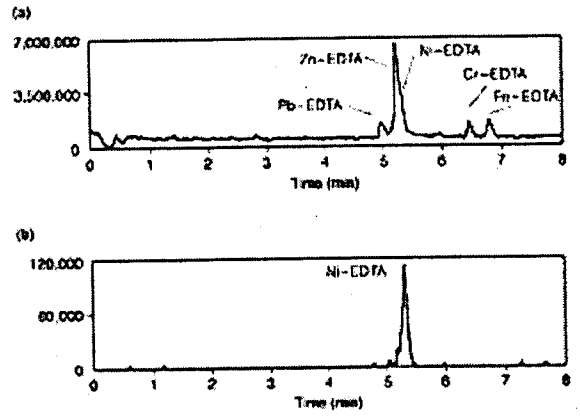


Figure 2. (a) Full-scan CE/MS electropherogram of five EDTA-metal chelates and (b) extracted ion electropherogram of Ni-EDTA.

Additional selectivity can be achieved using MS/MS by selecting a precursor ion that is characteristic of the analyte, fragmenting it into one or more ions, and selectively detecting one specific fragment from the selected precursor ion. For example, a full-scan single mass spectrum of the Ni-EDTA complex in Figure 3a shows the deprotonated molecular anion at *m/z* 347, which corresponds to the $[Ni^{2+}.H^{+}.EDTA^{4-}]^{-}$ ion. This precursor ion may be fragmented using collision-induced dissociation within the second-quadrupole region of a triple-quadrupole mass spectrometer. The full-scan product ion mass spectrum from the fragmentation of *m/z* 347 in Figure 3b shows product ions *m/z* 329 and 257, which are characteristic of the Ni-EDTA complex.

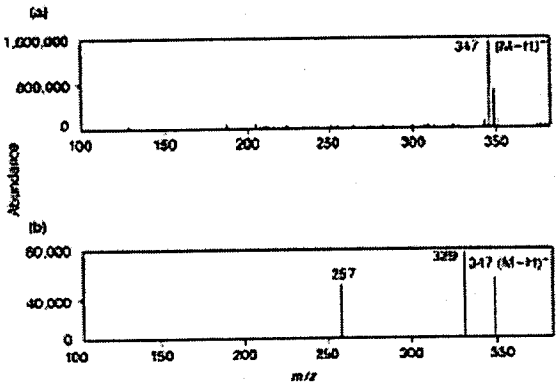


Figure 3. (a) Full-scan mass spectrum and (b) full-scan product ion scan of Ni-EDTA.

This additional selectivity can be put to use in the selected reaction monitoring (SRM) mode by monitoring only the specified transition *m/z* 347 fragmenting to 329. Further selectivity is achieved via the initial CE separation, which gives a characteristic migration time for Ni-EDTA. It is highly unlikely that a chemical compound other than Ni-EDTA would result in this characteristic transition at the specified migration time under the described experimental conditions.

To illustrate this point, blank and Ni-EDTA-spiked plasma samples were simply diluted with water, filtered, and analyzed by CE with UV detection and CE with SRM detection. The blank plasma in Figure 4a and the 1-μM Ni-EDTA-spiked plasma in Figure 4b are indistinguishable by CE/UV because of the excessive chemical background detected at 200 nm. In contrast, when analyzed by SRM-CE/MS, the same blank plasma sample is free of all matrix peaks (Figure 4c); whereas the Ni-EDTA-spiked

Ex 446
(536)

plasma displays only the targeted Ni-EDTA peak (Figure 4d). This example clearly shows the advantage of using tandem MS for this type of targeted analysis.

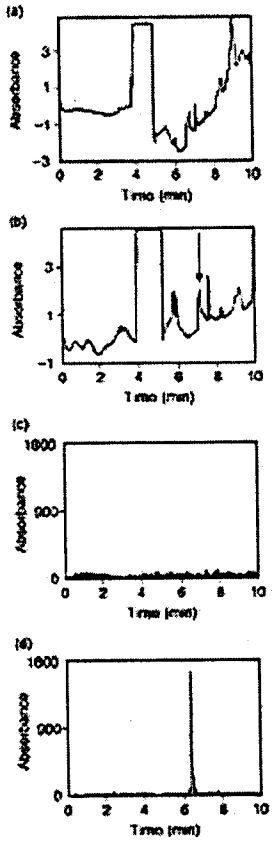


Figure 4. UV detection versus SRM detection.

CE/UV analysis of (a) blank plasma and (b) Ni-EDTA-spiked plasma. SRM-CE/MS analysis from *m/z* 347 to 329 of (c) blank plasma and (d) Ni-EDTA-spiked plasma.

To further minimize the possibility of interference, we developed an automated anion-exchange solid-phase extraction procedure. The complete SRM-CE/MS procedure uses 100 μL of plasma, to which is added 50 ng of ($^{13}C_4$)EDTA internal standard, brought to pH 9-10 with ammonium hydroxide, and complexed using nickel nitrate. The sample is diluted 1:45 with 0.05% formic acid (pH 3) and then extracted using strong anion-exchange solid-phase extraction media. The sample is eluted, evaporated, and reconstituted in 30 μL of water. The extract is injected for 0.1 min at 950 mbar inlet pressure onto a 50 μm X 60 cm amine-coated capillary. The separation is performed using a CE running buffer of 30 mM ammonium formate at pH 3 (adjusted with formic acid) and -30 kV with 50-mbar inlet pressure throughout the run. A homemade self-aligning liquid junction CE/MS interface is used with a makeup liquid of 5 mM ammonium formate in 95% methanol at 10μL/min (*22*, *23*). A triple-quadrupole mass spectrometer is used in the negative-ion mode with SRM of the transitions *m/z* 347-329 for Ni-EDTA and *m/z* 351-333 for the internal standard Ni-($^{13}C_4$)EDTA. The complete method and validation are described in this issue in reference 24.

Using this sample preparation procedure and the SRM-CE/MS method, we achieved a detection limit of 7.3 ng/mL EDTA in human plasma and a lower level of quantitation (LLQ) of 15 ng/mL (~6 fmol injected). If this method was used to determine whether a forensic blood stain had been "planted", this

Ex 446
(537)

LLQ corresponds to "planting" 1-3 nL of EDTA-preserved blood. Because it would be physically difficult to manipulate such a small volume, any such forensic sample would probably contain at least 1 μL. This hypothetical scenario illustrates the excellent sensitivity and potential forensic usefulness of the method. Similarly, the GC/MS/MS method developed by Ballard and colleagues demonstrated a comparable detection limit of 10 ng/sample, which corresponds to ~7 or 8 nL of EDTA-preserved blood (*13*).

CONCLUSIONS

Many techniques have been used over the years to determine EDTA in various matrices, and most can be adapted to biological samples. However, SRM-CE/MS provides the highest specificity and the best detection level of any method currently published.

We have been able to demonstrate that typical human plasma samples do contain detectable EDTA, but at levels that are lower than the LLQ reported in this work. The LLQ of our method, at 15 ng/mL, is a factor of 10^5 lower than the typical concentration found in EDTA-preserved blood (4.5 mM or 1.3 X 10^6 ng/mL). But more importantly, we have demonstrated that CE/MS methods can be used for routine bioanalytical analysis with acceptable precision, accuracy, and adequate detection levels for quantitation of trace-level concentrations (*24*).

CE/MS techniques will undoubtedly become an important forensic technique because of the low volumes of sample required for analysis, as well as the ability to use the mass spectrometer to achieve selectivity higher than with any other on-line detector.

The question of blood-evidence tampering in a criminal trial has led not only to improved analytical techniques for the determination of EDTA, but also to the demonstration that a relatively new technique is ready to be used as credible scientific evidence in the courtroom.

REFERENCES

ChemCenter ACS Pubs Div. Home Page

Ex 446
(538)

Performance Monitoring Protocol (QA/QC) for the Finnigan LCQ LC/MS (ESI)

1 Scope

This document addresses the performance monitoring (QA/QC) of the Finnigan LCQ LC/MS system consisting of a Finnigan LCQ MS and a Waters LC. It is an analytical instrument used to analyze a wide variety of evidence and must be maintained in such a way as to verify its reproducibility from analysis to analysis and its reliability in court.

2 Principle

The LCQ system is comprised of a Waters Liquid Chromatograph (LC) and a Finnigan ion trap LCQ Mass Spectrometer (MS). The instrument is configured with an API source that is capable of both electrospray (ESI) and atmospheric pressure chemical (APCI) ionization. The instrument is primarily used in ESI mode. However, this protocol can also be used for APCI provided the method of ionization is clearly labeled in the resulting data and documentation. Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Policy."

3 Equipment/Materials/Reagents

a. Instrumentation - Finnigan LCQ MS, API Source, Waters Alliance 2690/2695 LC, and Data System with XCalibur software (or equivalent)

b. API Gas - Nitrogen, 99.99% (high purity or equivalent)

c. Ion Trap Gas - Helium, 99.99% (high purity or equivalent)

d. Methanol, HPLC grade

e. Deionized Water, 18 MΩ Milli-Q or equivalent

f. Acetonitrile, HPLC grade

g. Acetic Acid, reagent grade

Ex 446
(539)

h. Ultramark 1621 (Finnigan or equivalent)

i. Caffeine (Sigma or equivalent)

j. MRFA (L-methionyl-arginyl-phenylalanyl-alanine acetate) (Finnigan or equivalent)

k. Ammonium Hydroxide (NH_4OH), reagent grade

l. Codeine (Sigma or equivalent)

m. Brucine (Sigma or equivalent)

n. Reserpine (Sigma or equivalent)

o. Volumetric glassware

p. Infusion Syringe - 10 to 500 µL LC syringe (Hamilton or equivalent)

4 Standards and Controls

4.1 Testmix

The Testmix is used to assess daily operating performance, mass assignment, and continued integrity of the system. To prepare, weigh 5.0 mg caffeine, 1.0 mg codeine, 1.0 mg brucine, and 1.0 mg reserpine into a 100-mL volumetric flask. Bring to the mark with methanol and mix well. Store the solution in the refrigerator. It has a shelf-life of three years. This preparation may be appropriately scaled up.

4.2 Calibration Solution

The calibration solution is used for coarse tuning and calibrating the mass spectrometer over the entire mass range. This procedure only needs to be performed when the instrument has been moved, down for a long period of time, undergone a major repair, or warranted based on system performance.

The calibration solution is a solution of caffeine, MRFA, and Ultramark 1621 in acetonitrile:methanol:water containing 1% acetic acid. To prepare this solution, follow the procedure in the LCQ 'Getting Started' manual. Store the solution in the refrigerator. It has a shelf-life of three years. This preparation may be appropriately scaled up.

5 Calibration

The calibration procedure should be performed as needed, when the instrument has been moved, down for a long period of time, undergone a major repair, or warranted based on system performance.

a. Load a 250 µL syringe with the calibration solution.

b. Using capillary tubing, connect the infusion syringe to the ESI probe assembly, and place in the syringe pump.

c. Set the syringe pump to the correct syringe type and set the pump rate to10 µL/minute.

d. Load the tune file "ESI_TUNE" (or equivalent).

e. Check that instrument is in POSITIVE ION mode and collecting CENTROID data.

f. Set the detector using the parameters listed in the 'Instrumental Conditions' section of this protocol.

g. Turn on the syringe pump and verify that the solution is flowing out the ESI needle.

h. Engage the ESI probe and turn on the MS.

i. In Tune Plus, open the Calibrate dialog box, choose the 'Automatic' tab and check the individual tests or 'Select All' and then 'Start.'

j. When the calibration is complete, it will display whether or not the calibration was successful.

- If the procedure fails, repeat the calibration.
- When the procedure passes, print the report and evaluate the calibration solution spectrum using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the spectrum of the calibration solution.

k. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Policy." If any requirements fail, the IOSS

Ex 446
(541)

Manager will determine the corrective action to be taken.

6 Sampling

Not applicable.

7 Procedures

7.1 Daily Checks

The following steps are to be performed daily. Enter the appropriate information in the QA/QC log for tracking purposes.

a. Record the remaining disk space on the hard drive. Use Windows Explorer program (WindowsNT) to verify that the hard disk has at least 100 MB of free disk space. Do not use if less than 100 MB remain.

b. Record the line pressure of the building nitrogen supply (API gas). The regulator should read between 60 and 100 p.s.i. If it cannot maintain this pressure, contact IOSS. If the nitrogen is supplied by a gas cylinder, record the tank pressure. Change the tank if less than 100 p.s.i. remaining.

c. Record the line pressure of the building helium supply (ion trap gas). The regulator should read between 30 and 60 p.s.i. If it cannot maintain this pressure, contact IOSS. If the helium is supplied by a gas cylinder, record the tank pressure. Change the tank if less than 100 p.s.i. remaining.

d. Check the Ion Gauge to ensure that no significant leaks are present in the system. Do not use if the pressure is higher than 1×10^{-4} torr.

e. Prepare the instrument for analysis of Testmix. Verify that the instrument has the correct source probe installed (ESI), the correct tune file loaded (esi_tune or equivalent), positive ion mode selected, and centroid data being collected. If a column is installed, remove it from that system and replace it with the infusion capillary tube.

f. Perform an analysis of the Testmix prior to the analysis of evidence using parameters listed in the 'Instrumental Conditions' section of this protocol. Start the HPLC pump. Engage the ESI probe and turn on the MS. Start an acquisition using a filename such

as 'testmix' (or equivalent). Make three 5 µL injections of the Testmix solution approximately 10 seconds apart by using the manual loop injector, and then stop the data collection. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the TIC and spectra for all components in the Testmix.

g. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Policy." If any requirements fail, contact IOSS.

7.2 As Needed Checks

a. Re-cut or replace the sample capillary as needed.

b. Clean or replace the heated capillary as needed.

8 Instrumental Conditions

Refer to the "General Instrument Maintenance Policy" for procedures on minor deviations.

8.1 Testmix

Liquid Chromatograph	
Mobile Phase:	95:5 methanol:water + 0.03% ammonium hydroxide
Flow Rate:	0.2 mL/min
Column:	None
Inj Volume:	5 µL
Number of Inj:	3

Mass Spectrometer	
Ionization:	ESI
Tune File:	esi_tune
Scan Mode:	Full Scan
Scan Range:	100-650 m/z

Ex 446
(543)

8.2 Calibration

Mass Spectrometer

Ionization:	ESI
Tune File:	esi_tune
Scan Mode:	Full Scan
Scan Range:	100-2000 m/z

9 Decision Criteria

9.1 Testmix

Verify the results of the Testmix. The following ions should be observed in the three Testmix injections:

- Caffeine 195 m/z
- Codeine 300 m/z
- Brucine 395 m/z
- Reserpine 609 m/z

9.2 Calibration

Verify the results of the calibration. The calibration will indicate if the procedure was successful. The individual ions for the calibration solution are:

• Caffeine	195 m/z	greater than 5%
• MRFA	524 m/z	greater than 50%
• Ultramark	1022 m/z	greater than 5%
	1122 m/z	greater than 20%
	1222 m/z	greater than 50%
	1322 m/z	greater than 50%
	1422 m/z	greater than 80%
	1522 m/z	greater than 50%
	1622 m/z	greater than 50%
	1722 m/z	greater than 20%
	1822 m/z	greater than 10%
	1922 m/z	present

10 Calculations

Not applicable.

11 Uncertainty of Measurement

Not applicable.

12 Limitations

Only properly trained personnel shall perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

13 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis. Many instrument components are held at temperatures of 250°C and higher. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

14 References

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Policy" (Inst 001) *Instrument Operation and Support Subunit SOP Manual.*

"Liquid Chromatograph General Maintenance Protocol" (Inst 003) *Instrument Operation and Support Subunit SOP Manual.*

"Mass Spectrometer General Maintenance Protocol" (Inst 004) *Instrument Operation and Support Subunit SOP Manual.*

FBI Laboratory Safety Manual.

Rev. #	Issue Date	History
0	06/21/06	New document which replaces original titled "Performance Monitoring Protocol (QA/QC) for the Finnigan LCQ LC/MS (ESI)."

Approval

Chemistry Unit Chief: _____ Date: 6/12/06
Marc A. LeBeau

QA Approval

Quality Manager: _____ Date: 6/15/2006
Robert B. Stacey

Issuance

IOSS Manager: _____ Date: 6/21/06
Jeffrey N. Leibowitz

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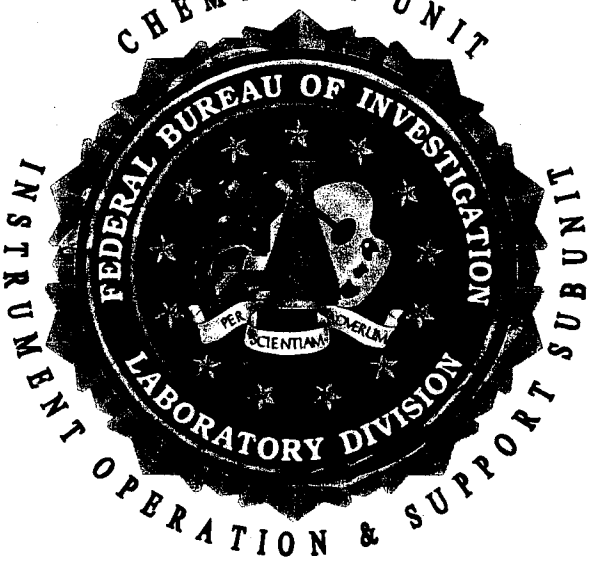
E4 446
(546)

INSTRUMENT MAINTENANCE AND CALIBRATION

Ex 446
(547)

Instrument Logbook



LCQ-1
(LC000117)

Date	Init	Comments
12/21/06 07	CLM	TM; oleandomycin m.p.
1/7/06 JDB	CLM	TM, 060103000Z, Nitroglycerin
1/31/07	JML/JDB	ESI TM; EDTA STDs, Water blanks
2/1/07	JML/JDB	ESI TM;
2/4/07	JDB	TM ESI; VALIDATION (EDTA)
2/5/07	JDB	TM ESI; VALIDATION (EDTA)
2/6/07	JDB	TM ESI; VALIDATION (EDTA)
2/7/07	JAC	TM ESI
2/7/07	JDB	VALIDATION (EDTA)
2/9/07	JDB	TM ESI; VALIDATION (EDTA)
2/12/07	JDB	TM ESI; VALIDATION (EDTA)
2/13/07	JDB	EDTA TM; VALIDATION (EDTA)
2/15/07	JDB	EDTA TM; VALIDATION (EDTA)
2/16/07	JDB	EDTA TM; 070201013

Ex 446
(549)

(555)

6/26/06 New SOP- Inst 202-0

1/26/07 Inter octopole lens reading 333V - problem traced to Main System PCB. Replaced PCB with one from LCQ-2. System OK.

Instrument Name: LCQ-1
Serial Number: LC000117

Month: January
Year: 2007

QUALITY ASSURANCE/QUALITY CONTROL LOG

Refer to the "Performance Monitoring Protocol (QA/QC) for the Finnigan LCQ LC/MS (ESI)" for procedures, standards, and decision criteria.

SOP 202

	Daily Checks							Annual/As Needed				
Day	Initials	Disk Space	Helium	Nitrogen	Ion Gauge	Testmix	Pass/Fail	Initials	Heated Cap	Skimmer	Tune/Calib	Pass/Fail
1												
2												
3												
4	JW	394MB	40	94	$1.73 x 10^{-5}$	5Mix	Pass					
5												
6												
7												
8												
9												
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30												
31	JNL	380 MB	40	90	$2.71 x 10^{-5}$	ESI TM	Pass					

Ex 446
(551)

Instrument Name: **LCQ-1**
Serial Number: **LC000117**

QUALITY ASSURANCE/QUALITY CONTROL LOG

Month: **Februar**
Year: **2007**

Refer to the "Performance Monitoring Protocol (QA/QC) for the Finnigan LCQ LC/MS (ESI)" for procedures, standards, and decision criteria. SOP 202

	Daily Checks							Annual/As Needed				
Day	Initials	Disk Space	Helium	Nitrogen	Ion Gauge	Testmix	Pass/Fail	Initials	Heated Cap	Skimmer	Tune/Calib	Pass/Fail
1	JM	337 MB	40	88	2.2x10^{-5}	ESI TM	PASS					
2	JDB	371 MB	40	95	1.53x10^{-5}	ESI TM	PASS					
3												
4												
5	JDB	366 MB	40	96	1.37x10^{-5}	ESI TM	PASS					
6	JDB	365 MB	40	96	1.55x10^{-5}	ESI TM	PASS					
7	JAC	358 MB	40	96	2.26x10^{-5}	ESI TM	PASS					
8												
9	JDB	347 MB	48	96	1.39x10^{-5}	ESI TM	PASS					
10												
11												
12	JDB	337 MB	40	86	2.41x10^{-5}	ESI TM	PASS					
13	JDB	323 MB	40	90	2.09x10^{-5}	EDTA TM⊖	PASS					
14												
15	JDB	319 MB	40	96	1.50x10^{-5}	EDTA TM⊕	PASS					
16	JDB	295 MB	40	96	1.57x10^{-5}	EDTA TM⊕	PASS					
17												
18												
19												
20												
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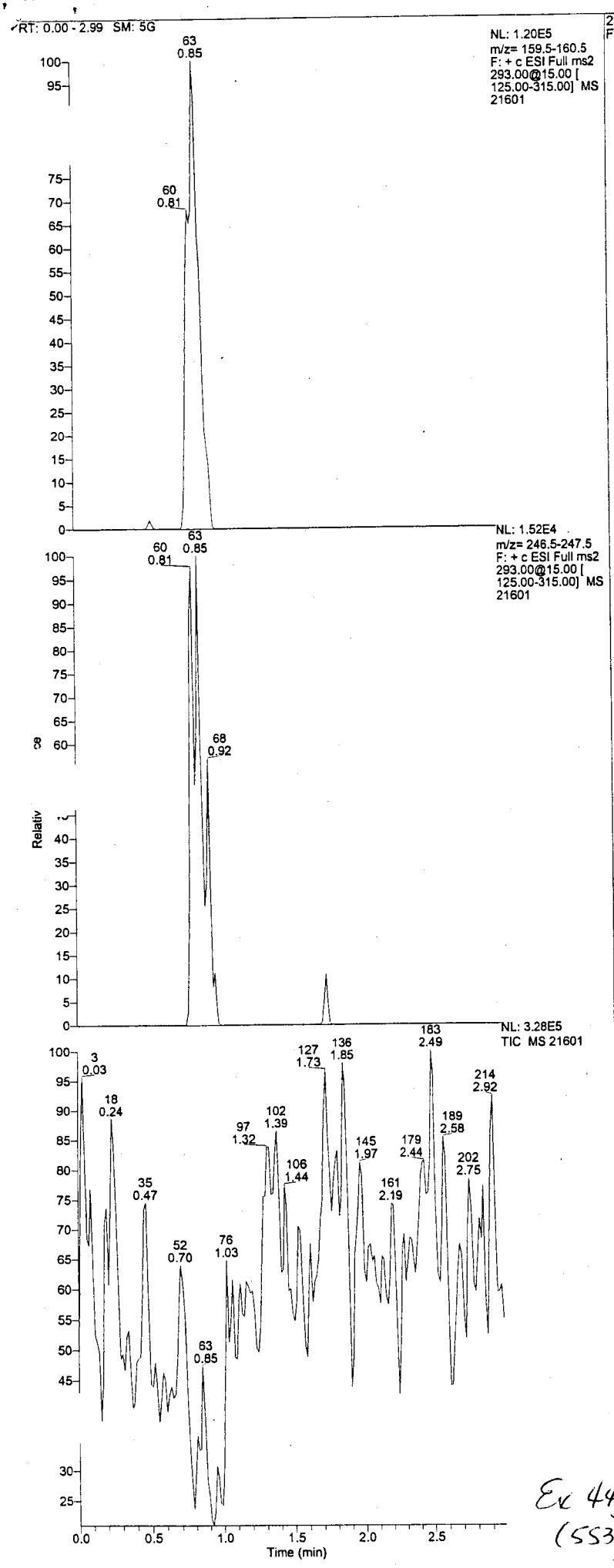
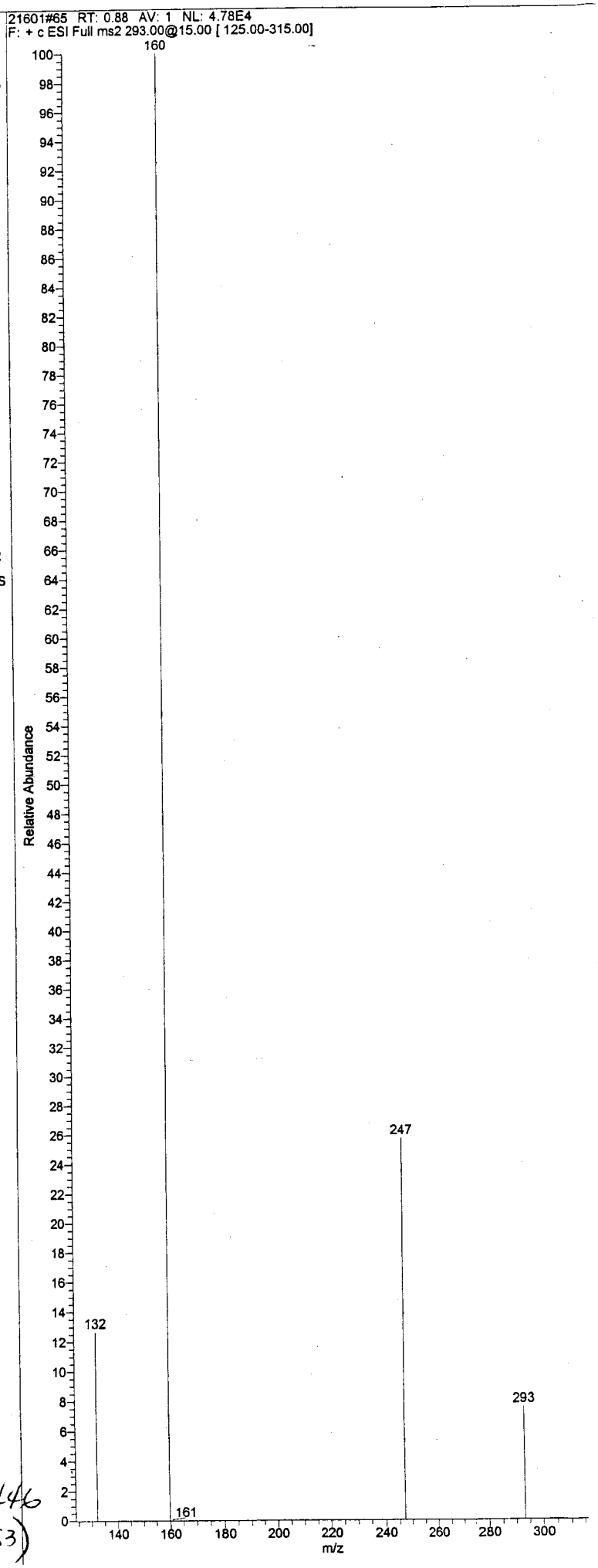
31												

(ESS) 9th 23

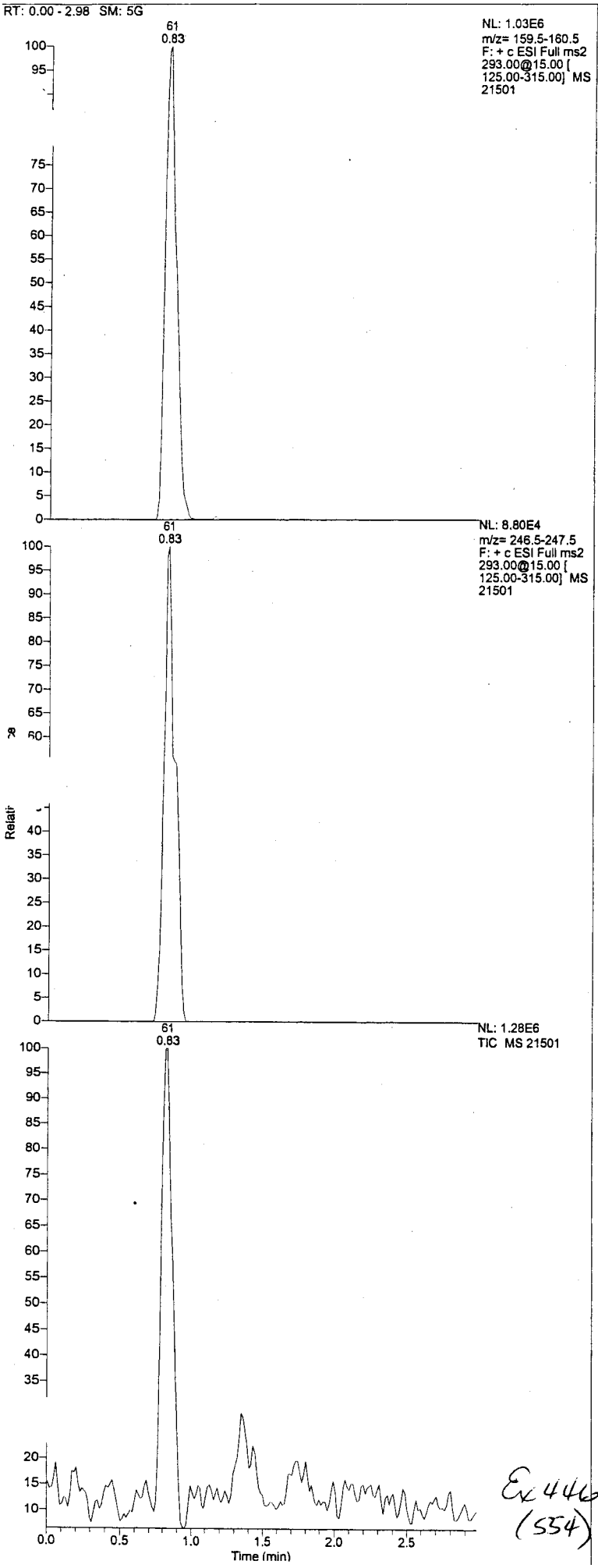

RT: 0.00 - 2.99 SM: 5G
NL: 1.20E5
m/z= 159.5-160.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21601
63
0.85
60
0.81
NL: 1.52E4
m/z= 246.5-247.5
F: + c ESI Full ms2
293.00@15.00 [
125.00-315.00] MS
21601
68
0.92
NL: 3.28E5
TIC MS 21601
3
0.03
18
0.24
35
0.47
52
0.70
76
1.03
97
1.32
102
1.39
106
1.44
127
1.73
136
1.85
145
1.97
161
2.19
179
2.44
183
2.49
189
2.58
202
2.75
214
2.92
Relativ
Time (min)


21601#65 RT: 0.88 AV: 1 NL: 4.78E4
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]
160
247
132
161
293
Relative Abundance
m/z

Ex 446
(553)

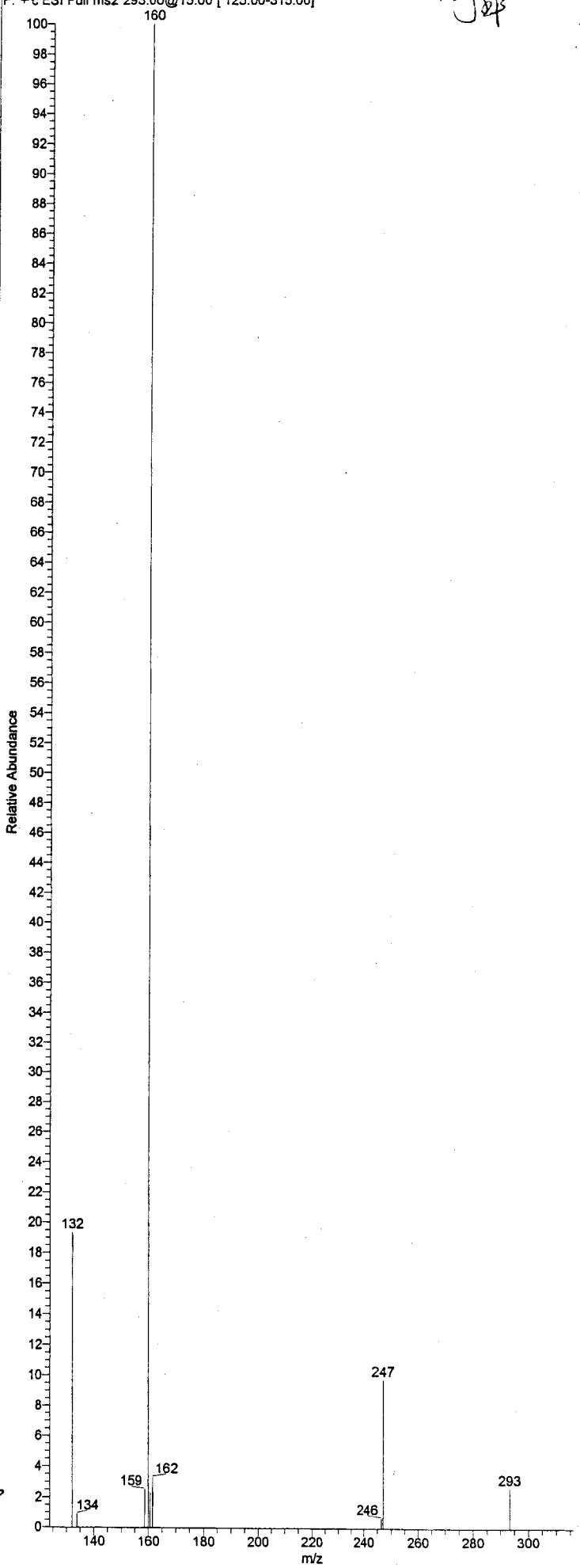
C:\Xcalibur\...\EDTA\Brewer\021507\21501 02/15/07 11:13:10 100 ppm EDTA tm

RT: 0.00 - 2.98 SM: 5G



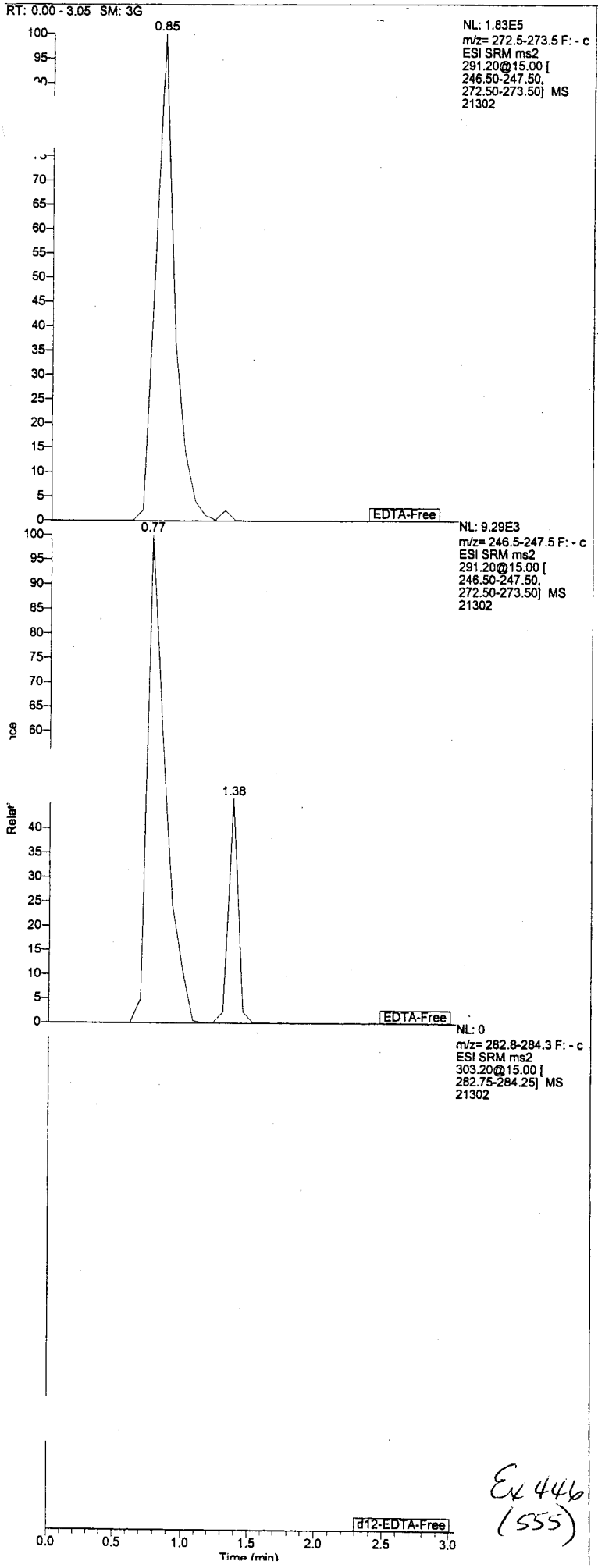
21501#64 RT: 0.87 AV: 1 NL: 6.10E5
F: + c ESI Full ms2 293.00@15.00 [125.00-315.00]

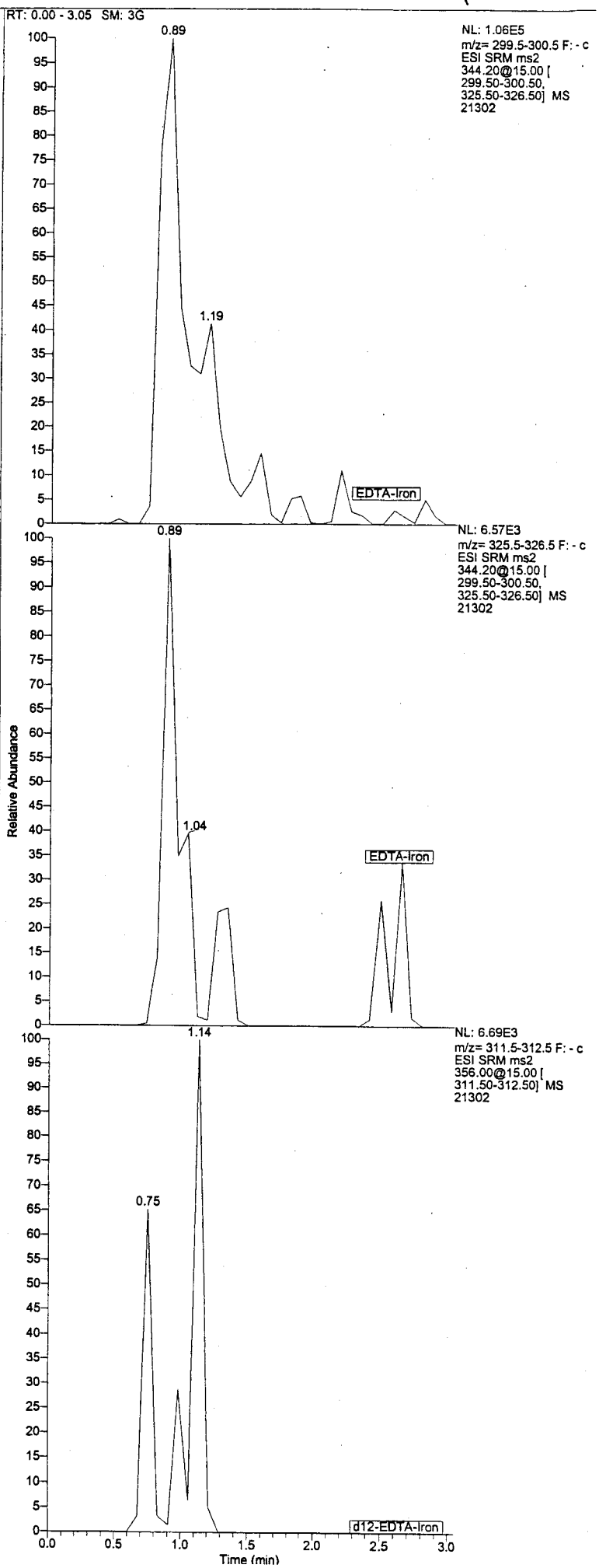
JDB



Ex 446
(554)

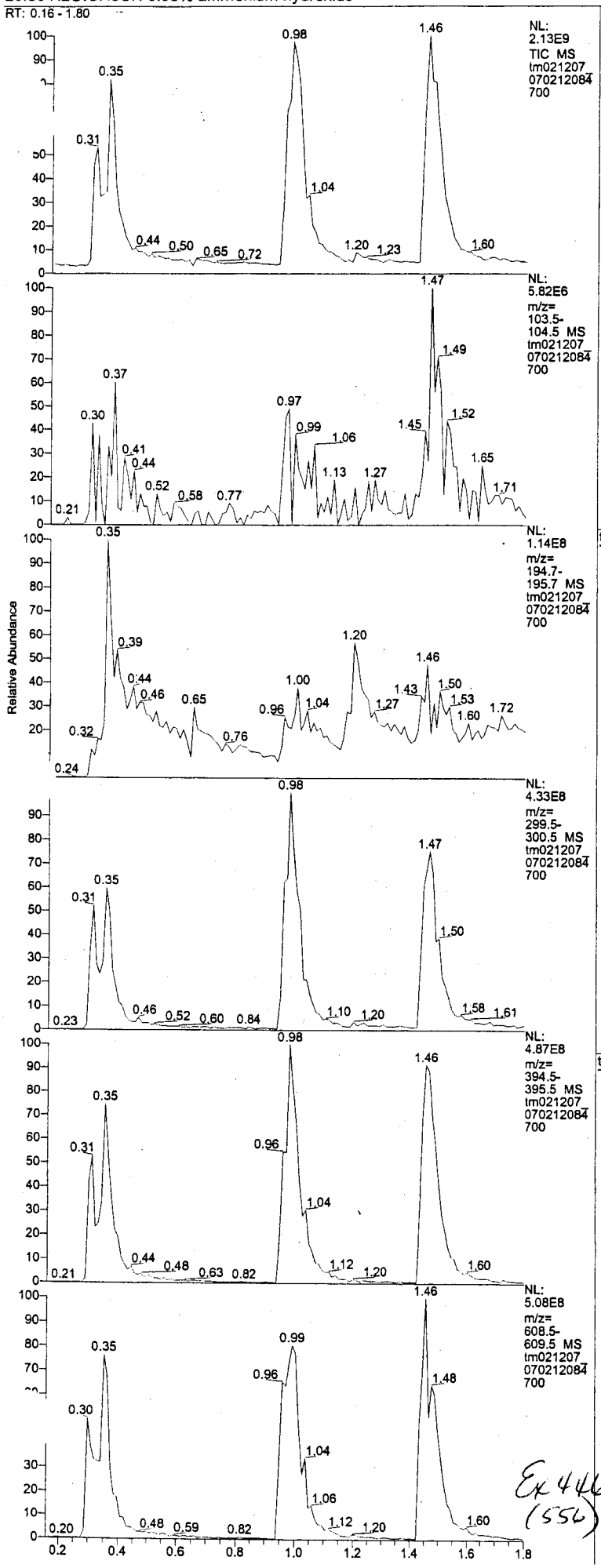
C:\Xcalibur\...\EDTA\Brewer\021307\21302 02/13/07 08:50:58 100 ppm EDTA JDB

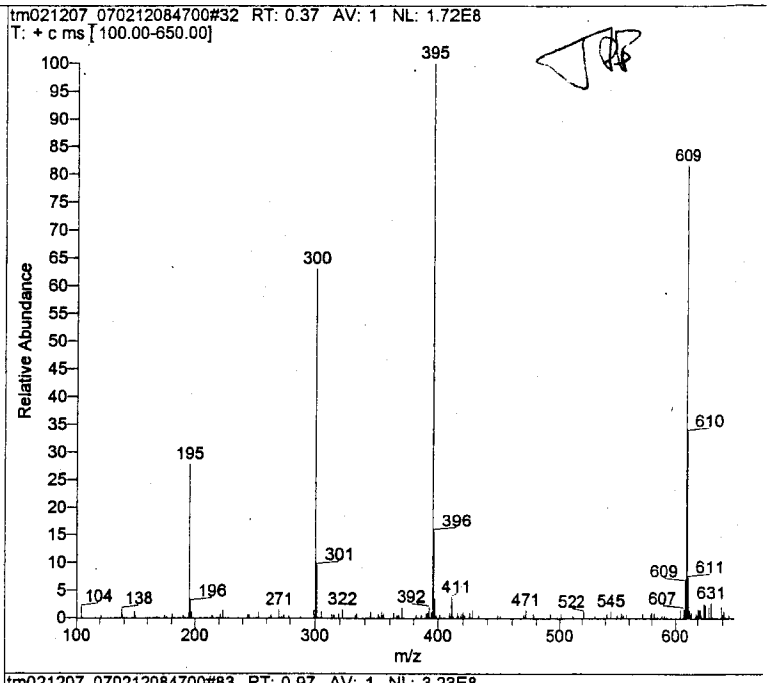


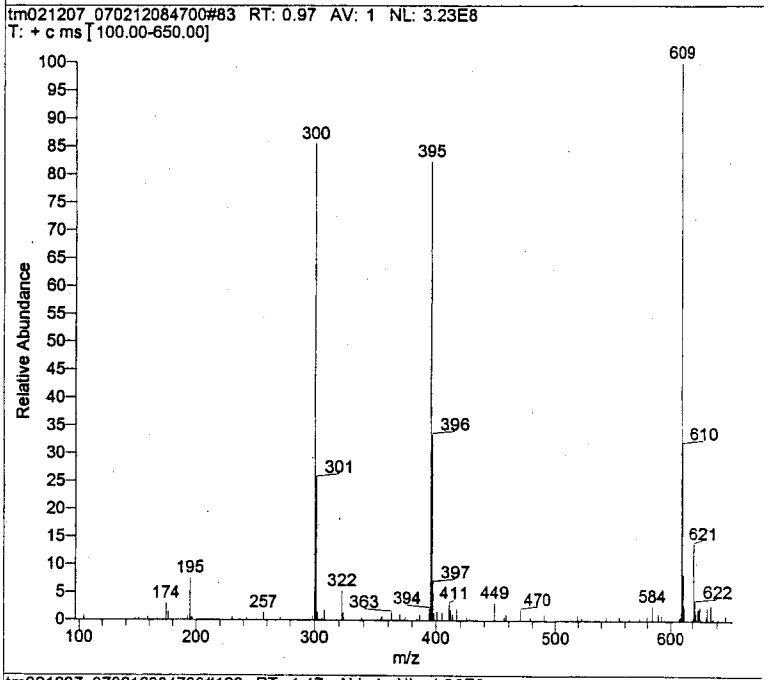


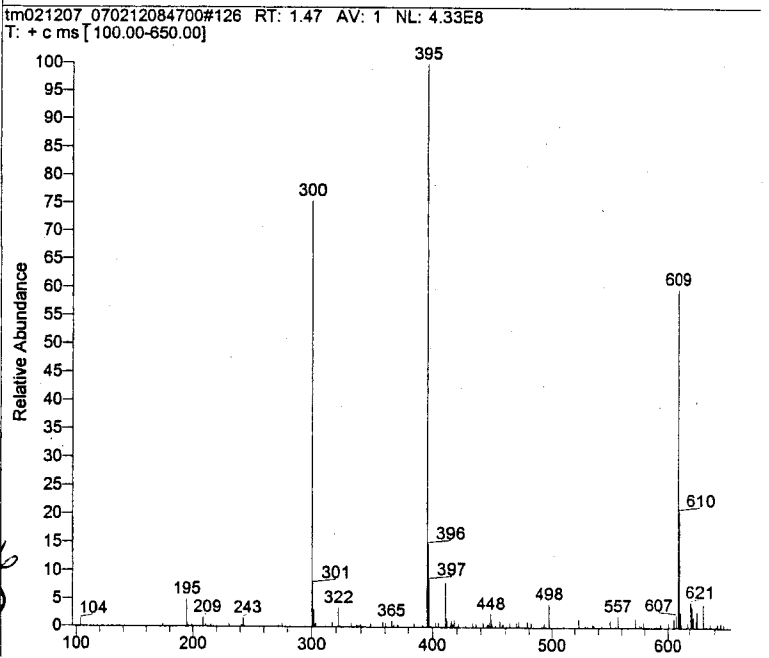
Ex 446
(555)

C:\Xcalibur\...\tm021207_070212084700 02/12/07 08:47:01 tm lc/ms lot 0703
20:80 H2O:CH3CN 0.03% ammonium hydroxide



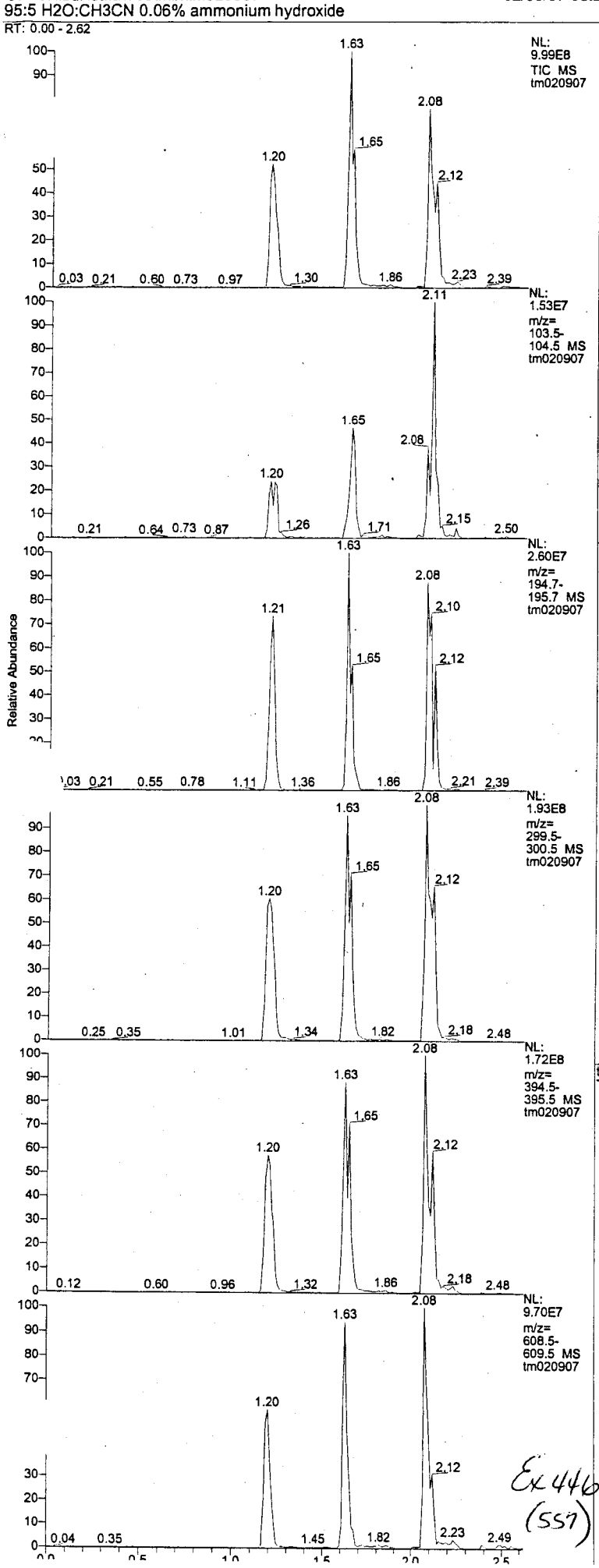


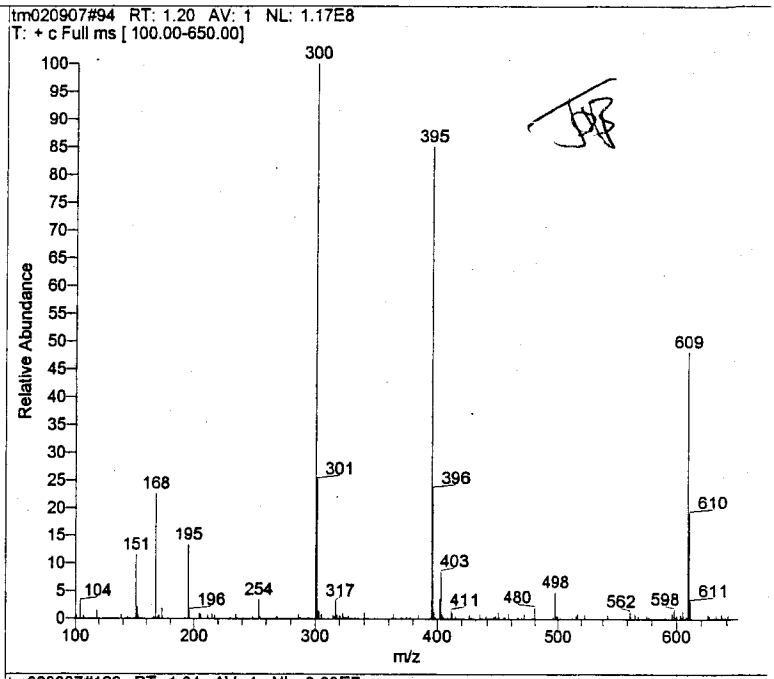


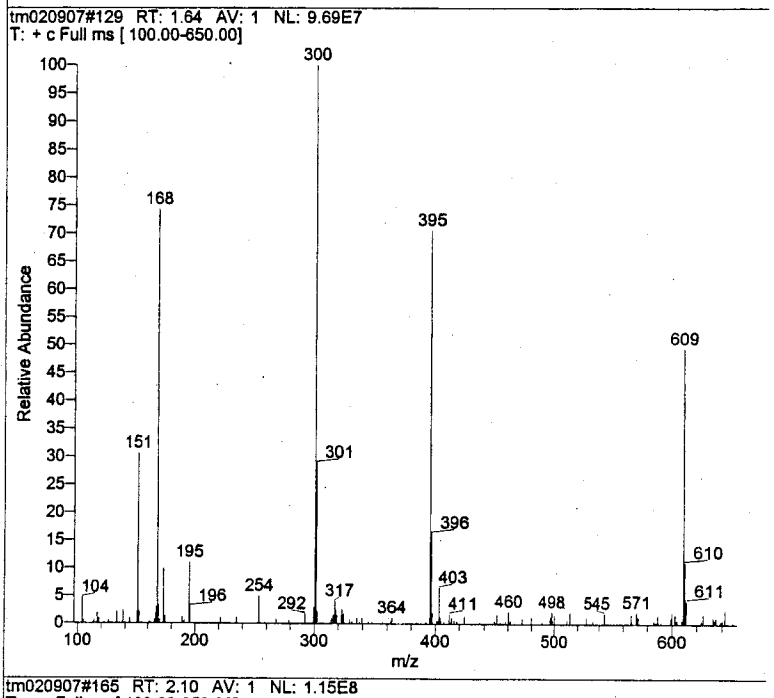


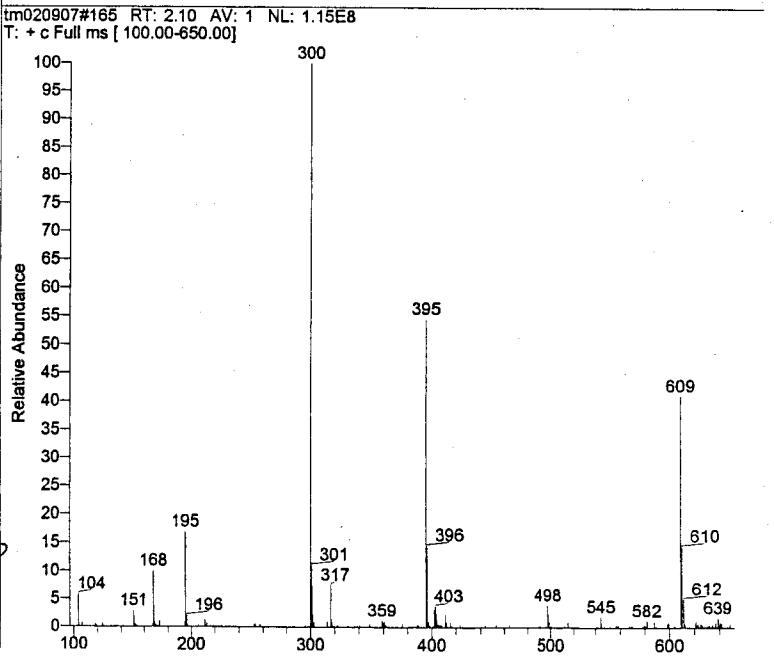
Ex 446
(556)

C:\Xcalibur\data\Testmix\tm020907 02/09/07 08:25:52 Lot 0703 ESI testmix
95:5 H2O:CH3CN 0.06% ammonium hydroxide



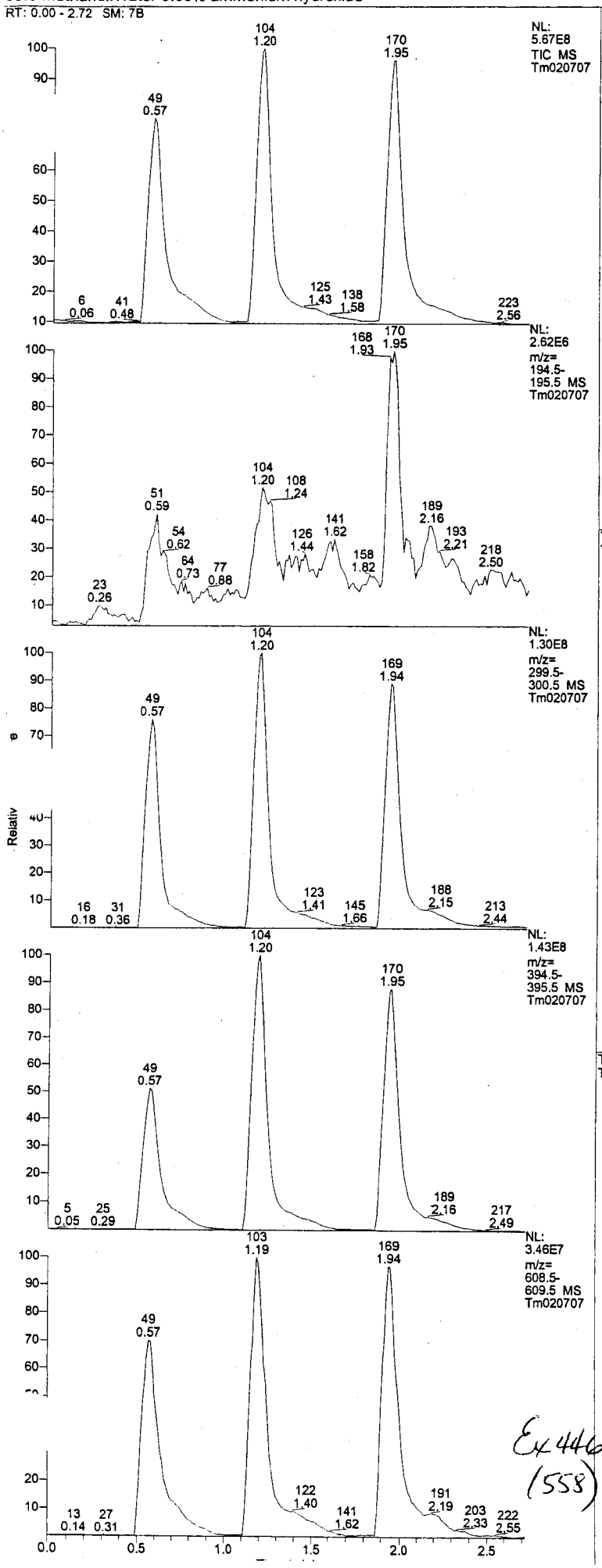


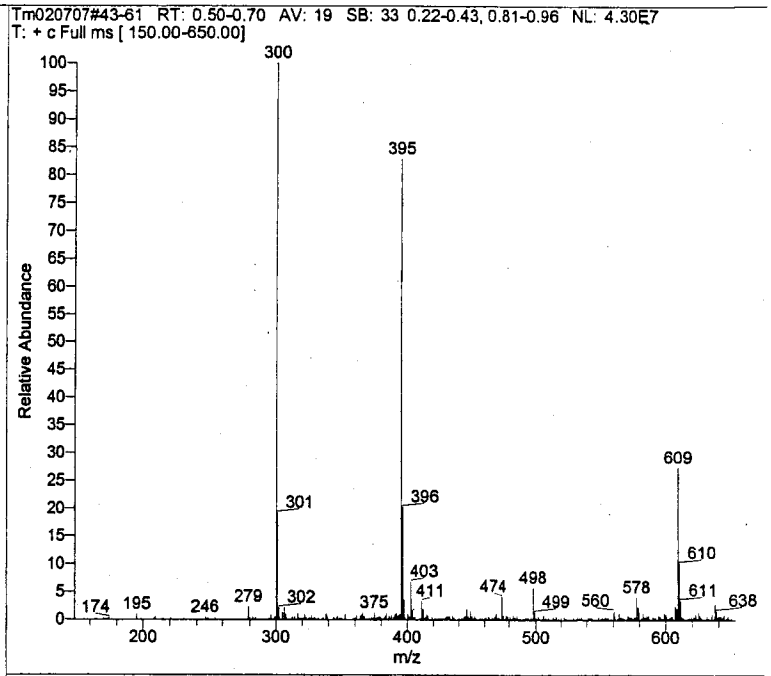


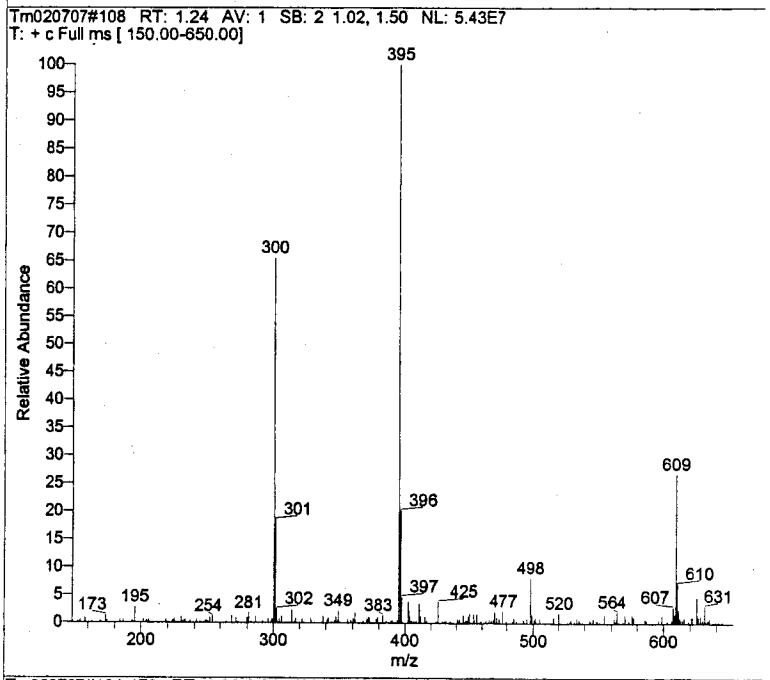


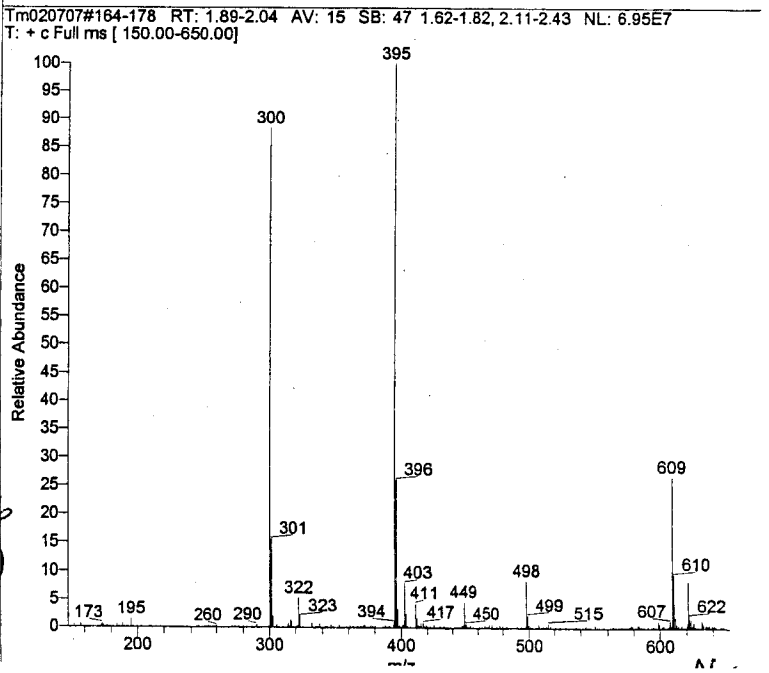
Ex 446
(557)

C:\Xcalibur\data\Testmix\Tm020707 02/07/07 10:50:28 Testmix
95:5 Methanol:Water 0.03% ammonium hydroxide

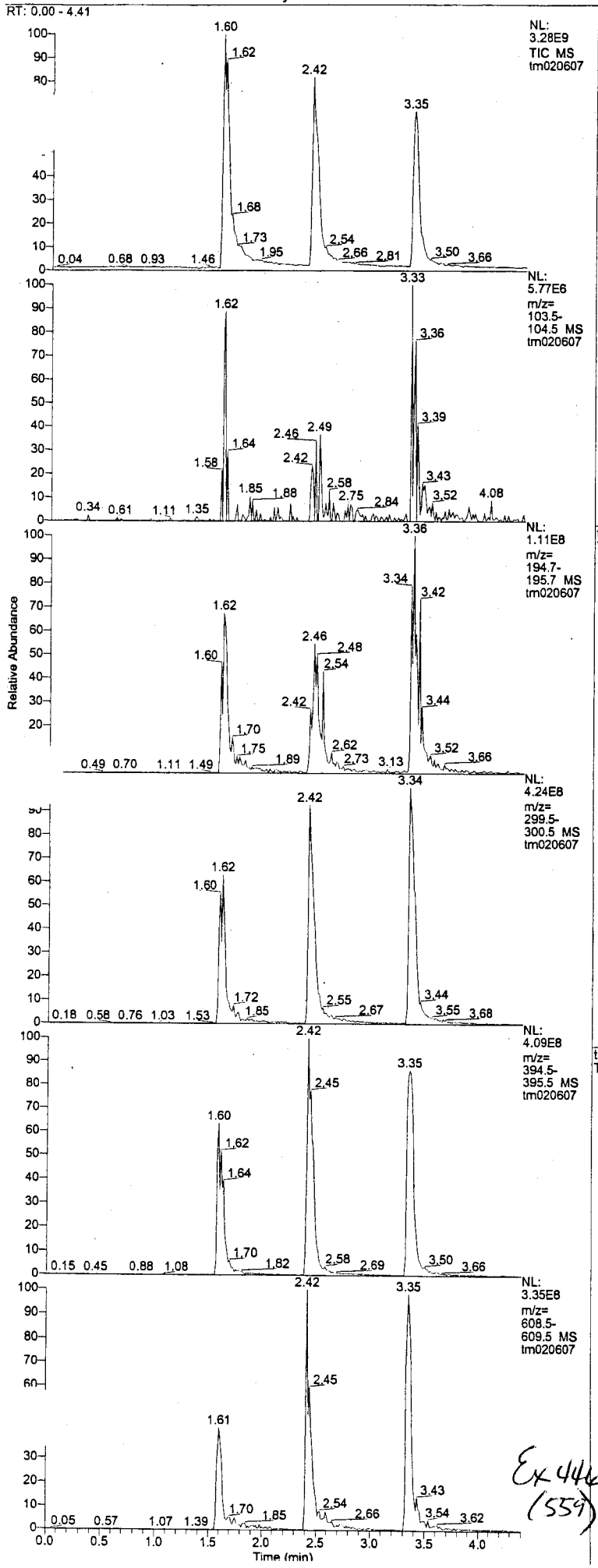


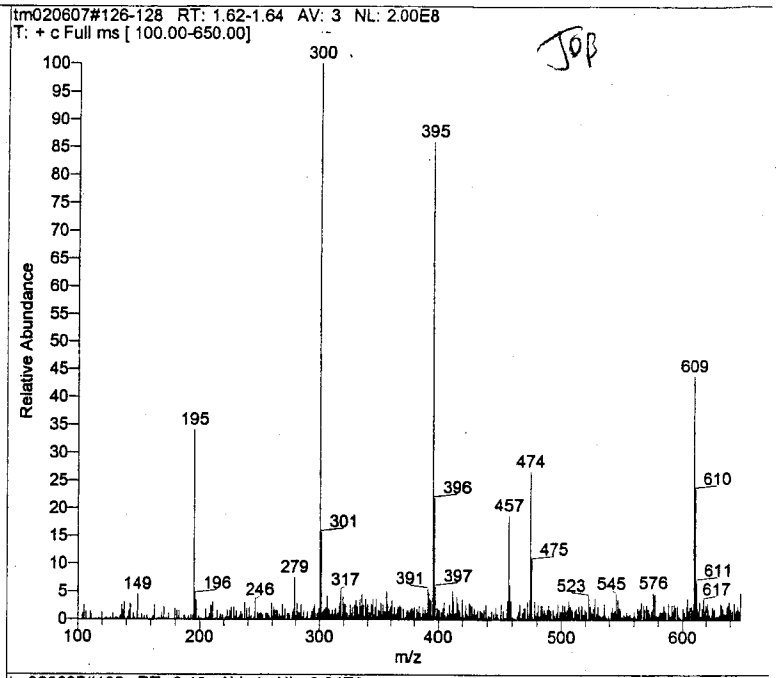


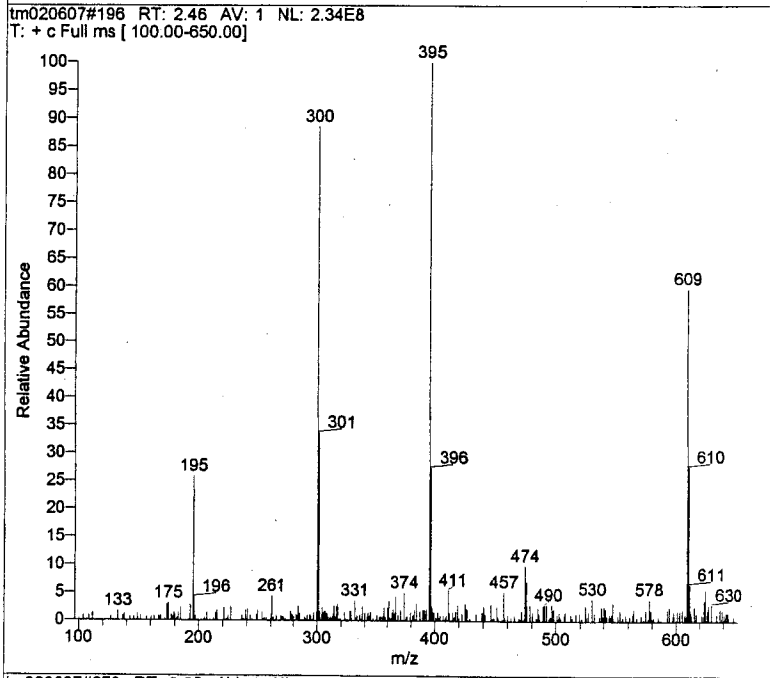


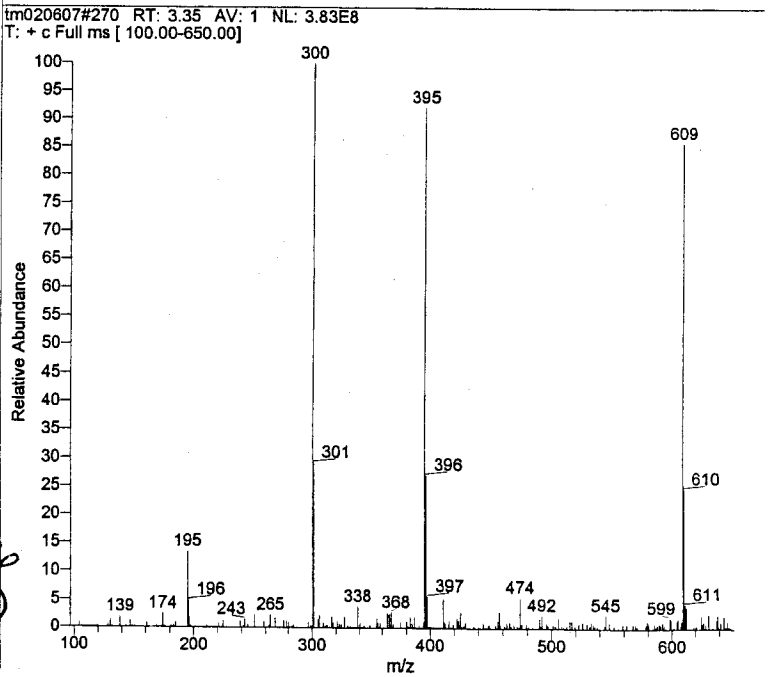


C:\Xcalibur\data\Testmix\tm020607 02/06/07 08:53:12 ESI TM Lot 0703
95:5 Water/ACN + 0.06% ammonium hydroxide





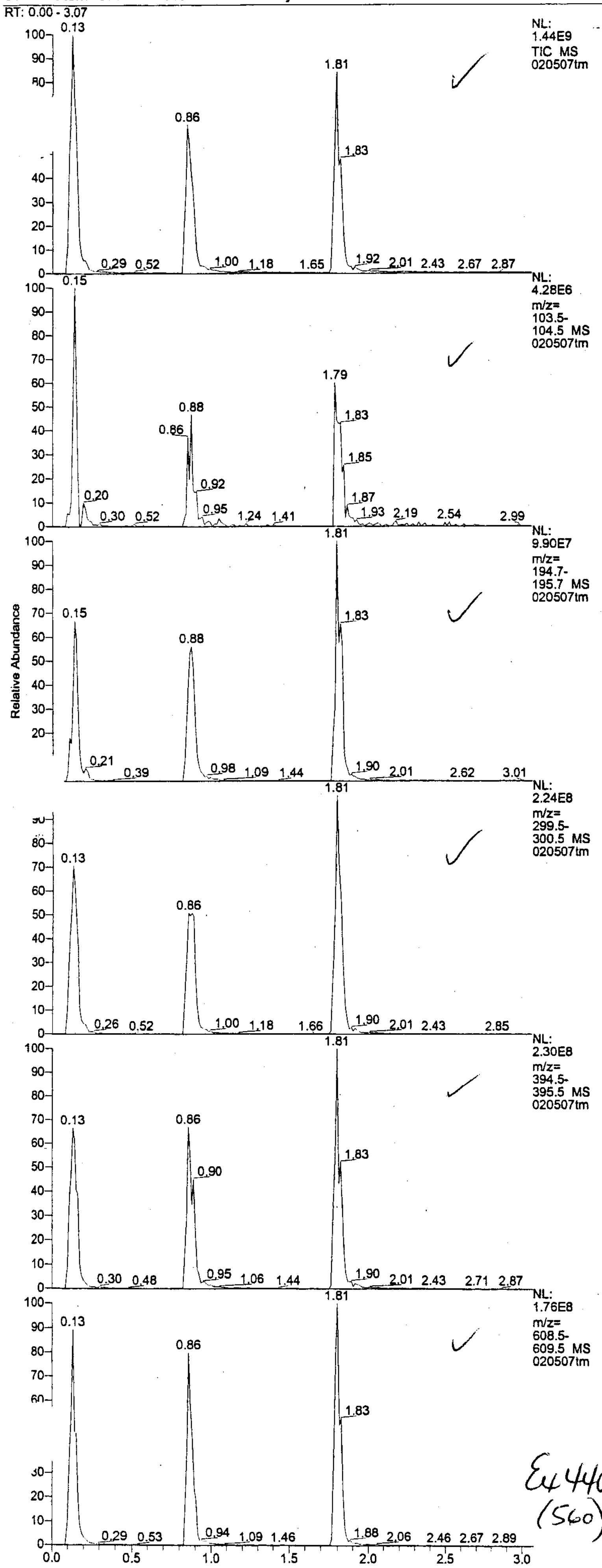


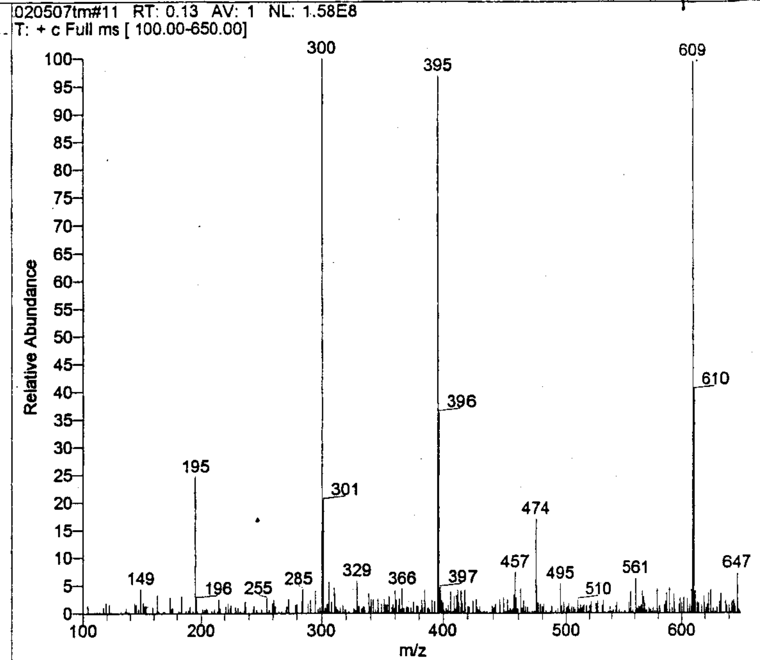


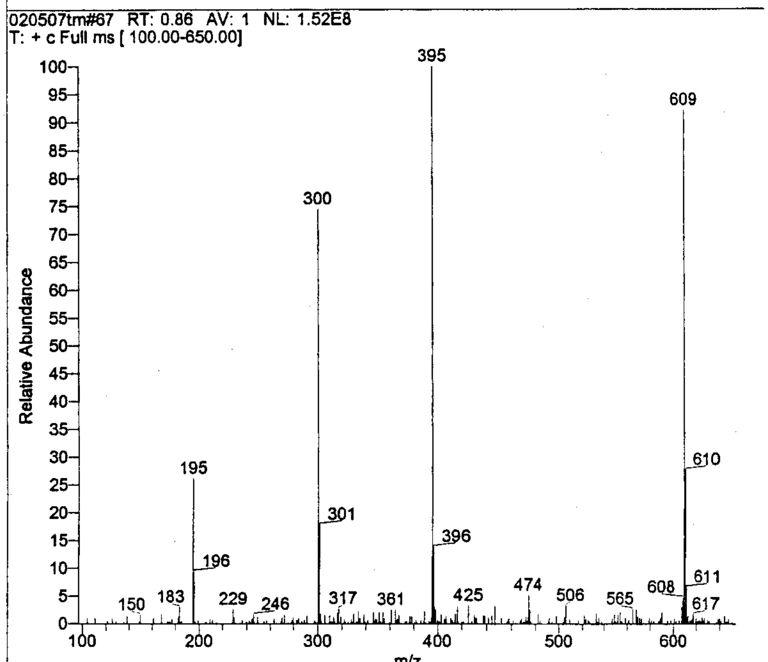
Ex 446 (559)

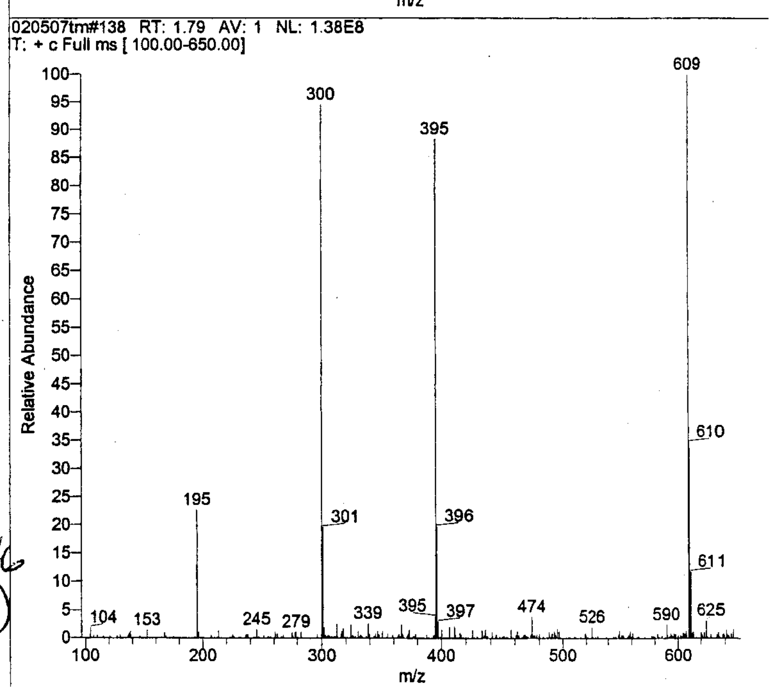
C:\Xcalibur\data\Testmix\020507tm 02/05/07 10:13:16 ESI tm lot 0703 JDR

95:5 Water/ACN + 0.06% ammonium hydroxide

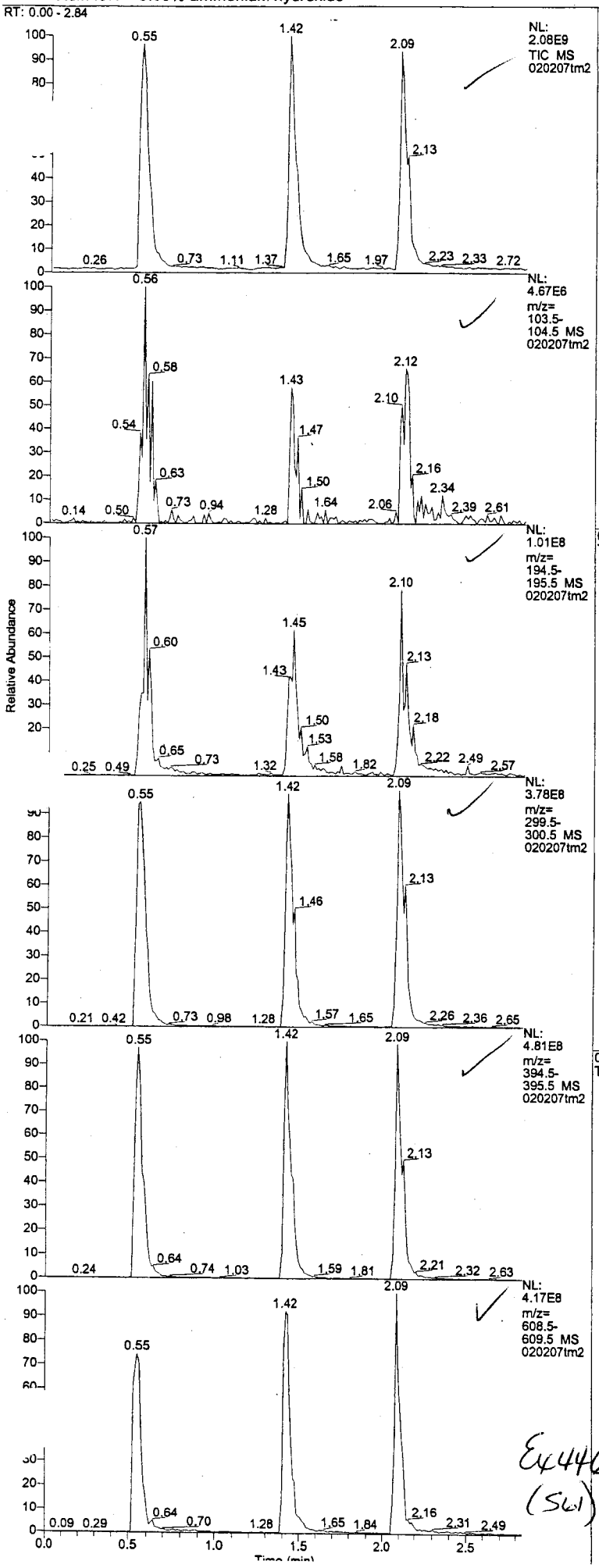


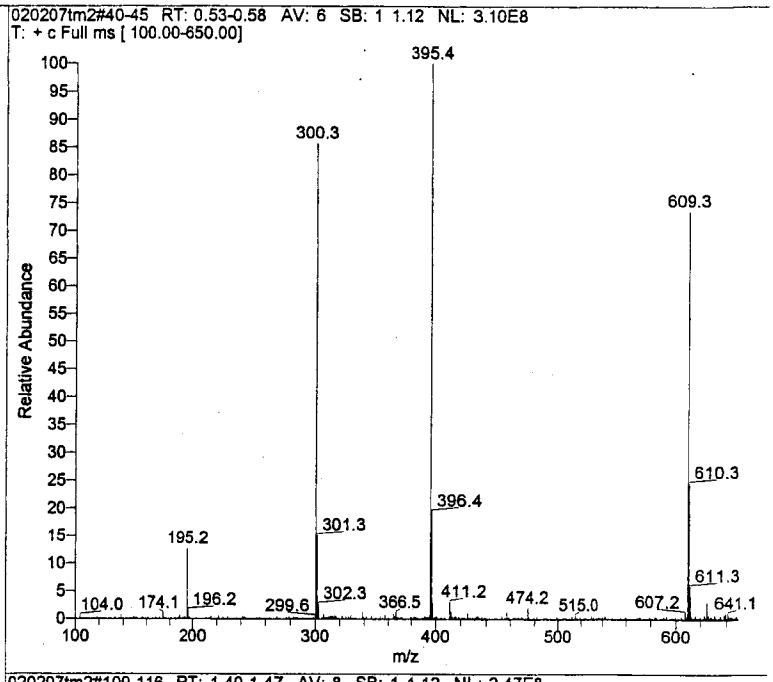


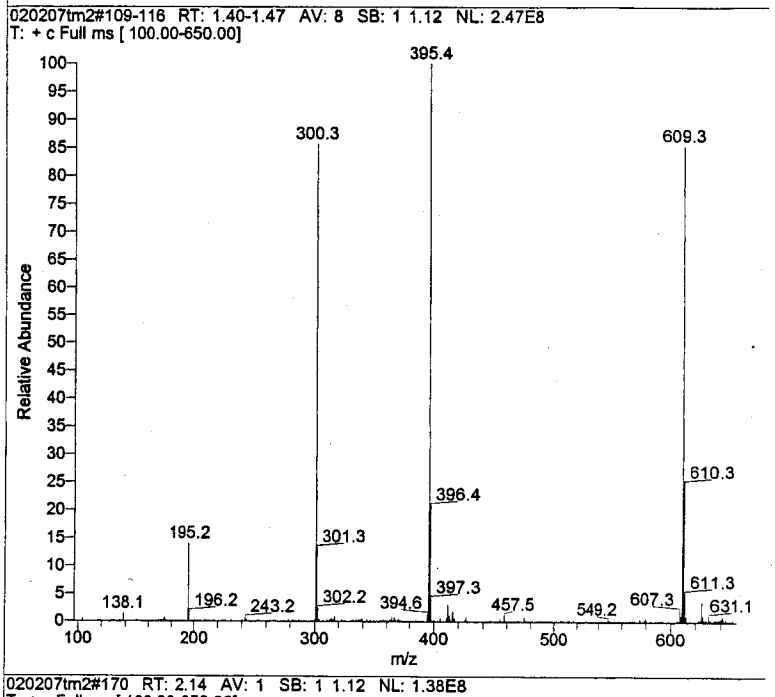


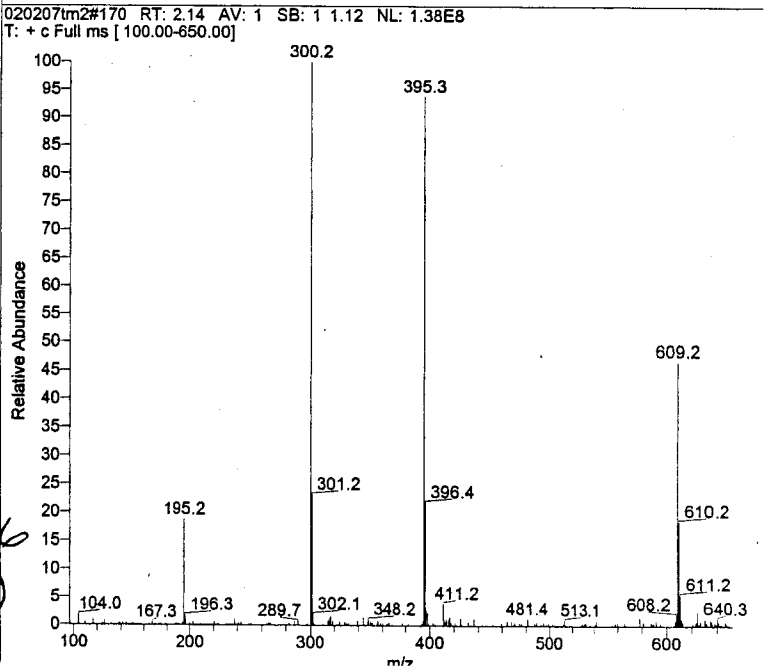


C:\Xcalibur\data\Testmix\020207tm2 02/02/07 01:18:31 ESI tm lot 0703
95:5 Water/ACN + 0.06% ammonium hydroxide



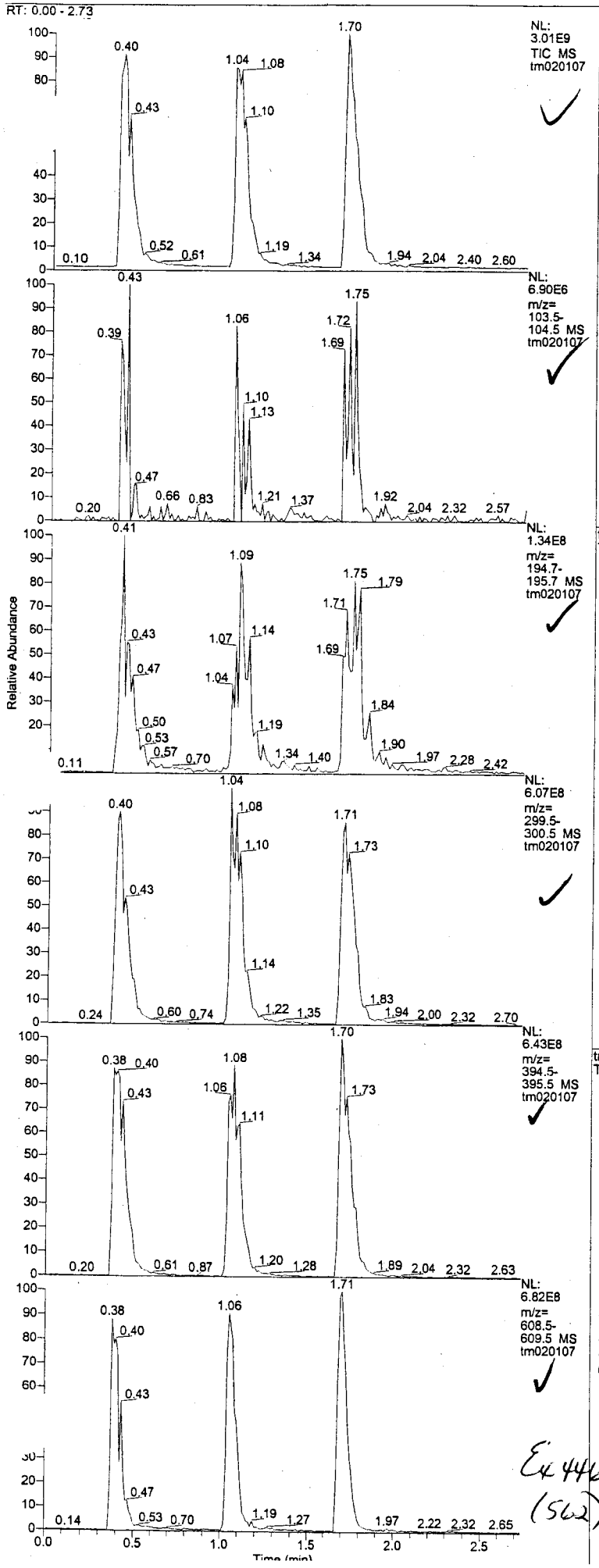


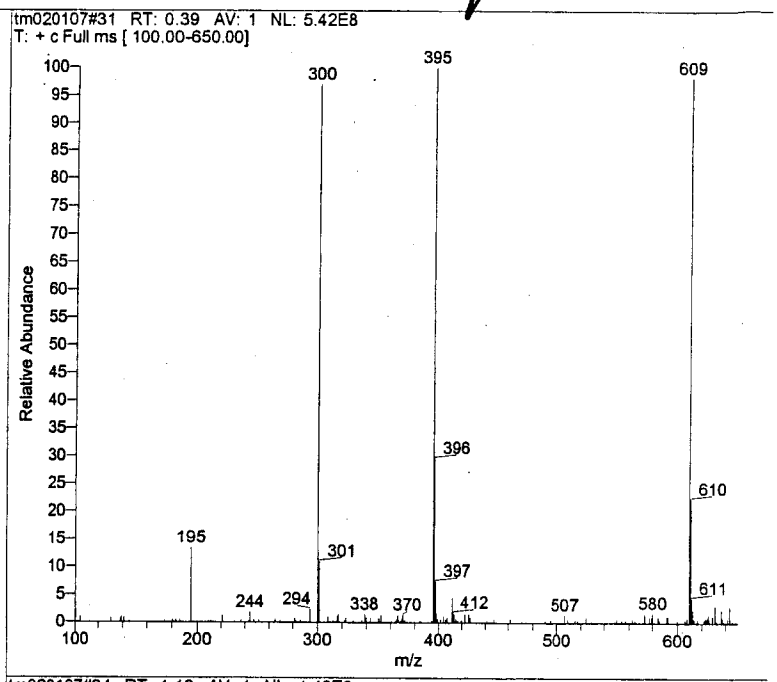


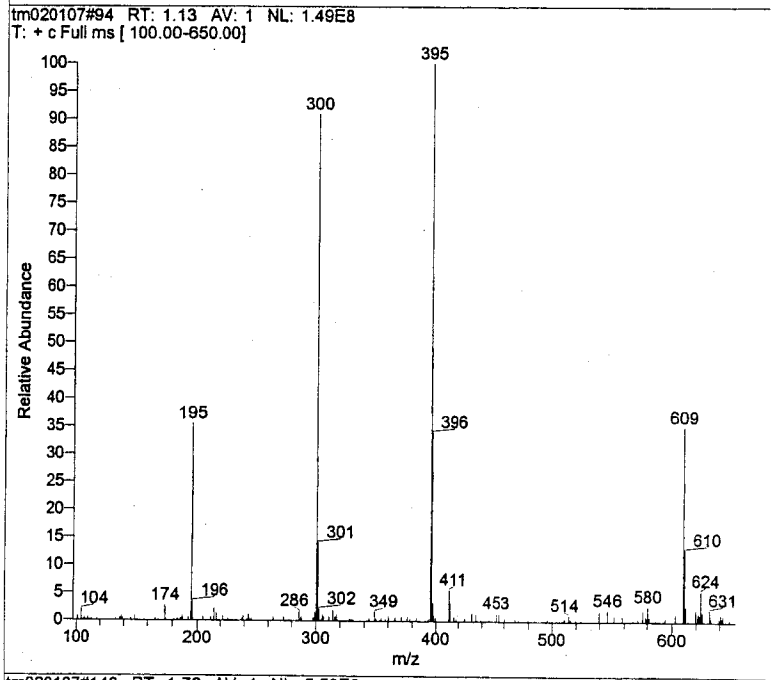


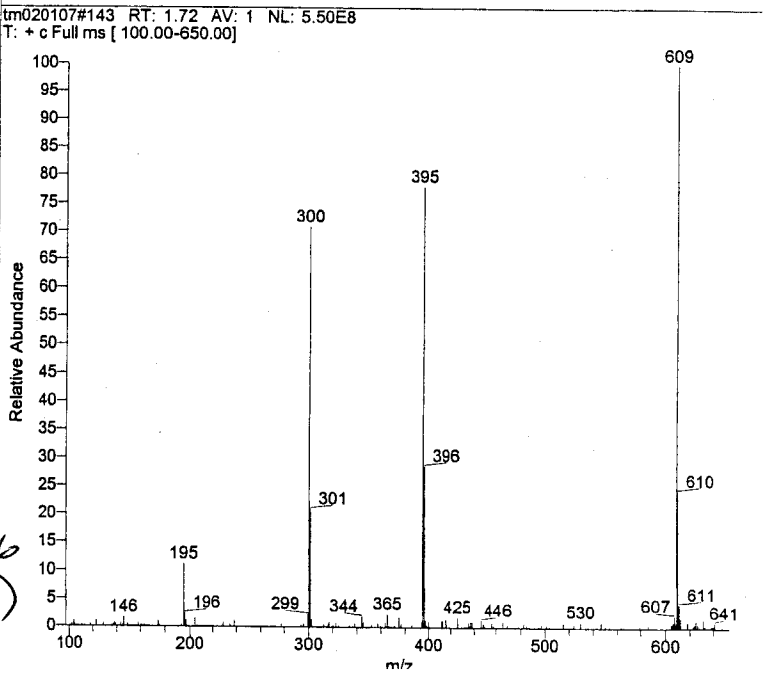
E4446
(561)

C:\Xcalibur\data\Testmix\tm020107 02/01/07 09:40:18 ESI Testmix - Lot #0703

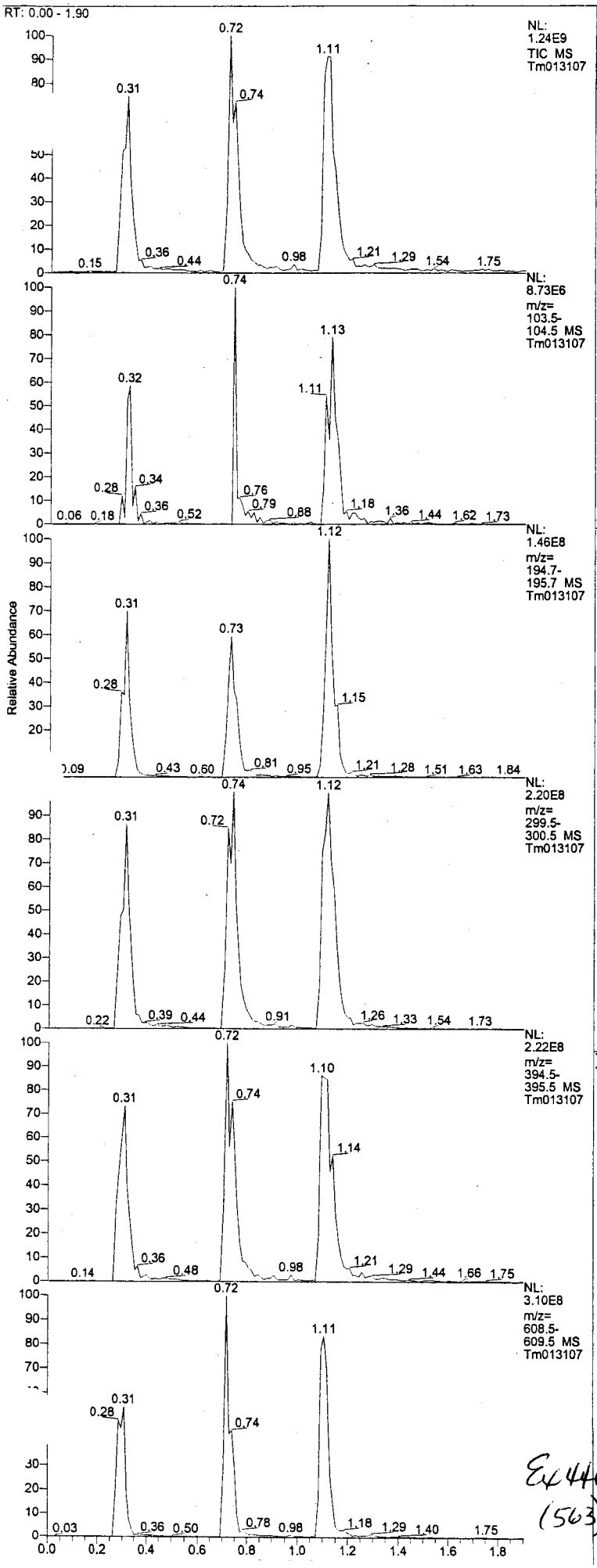


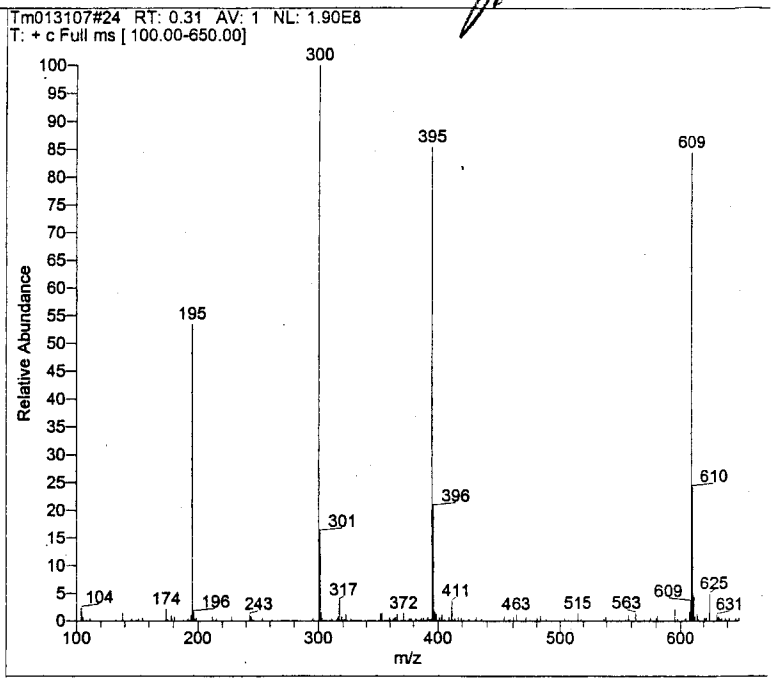


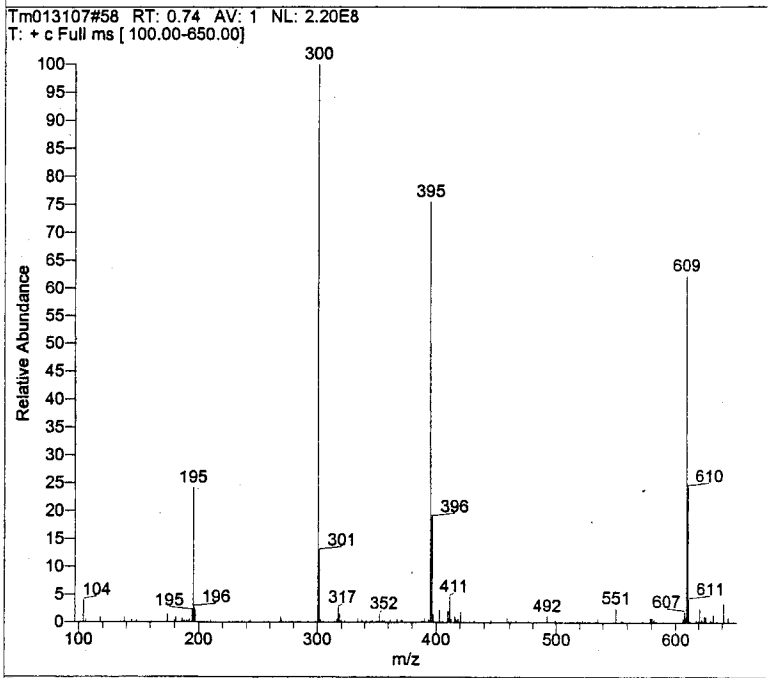


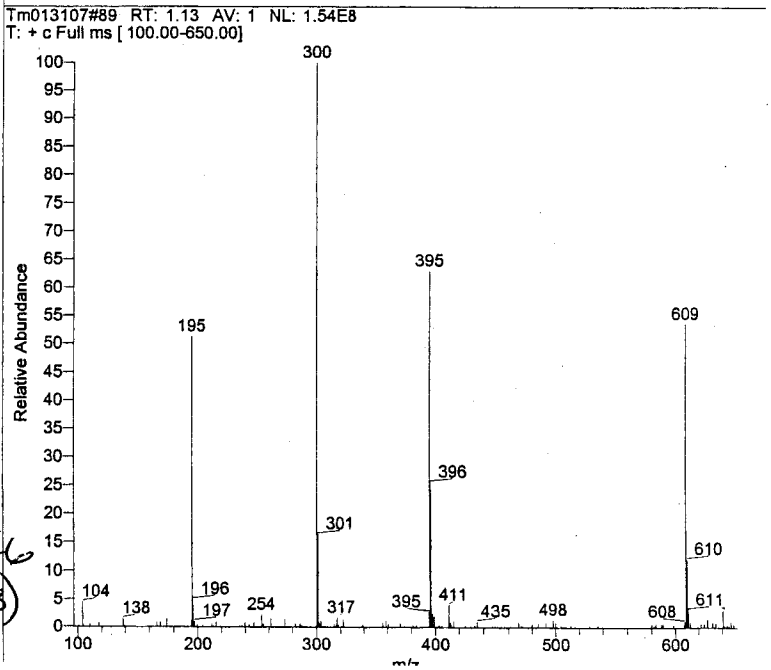


Ex 446 (562)


RT: 0.00 - 1.90
NL: 1.24E9 TIC MS Tm013107
NL: 8.73E6 m/z= 103.5-104.5 MS Tm013107
NL: 1.46E8 m/z= 194.7-195.7 MS Tm013107
NL: 2.20E8 m/z= 299.5-300.5 MS Tm013107
NL: 2.22E8 m/z= 394.5-395.5 MS Tm013107
NL: 3.10E8 m/z= 608.5-609.5 MS Tm013107
Relative Abundance


Tm013107#24 RT: 0.31 AV: 1 NL: 1.90E8
T: + c Full ms [100.00-650.00]
Relative Abundance
m/z


Tm013107#58 RT: 0.74 AV: 1 NL: 2.20E8
T: + c Full ms [100.00-650.00]
Relative Abundance
m/z

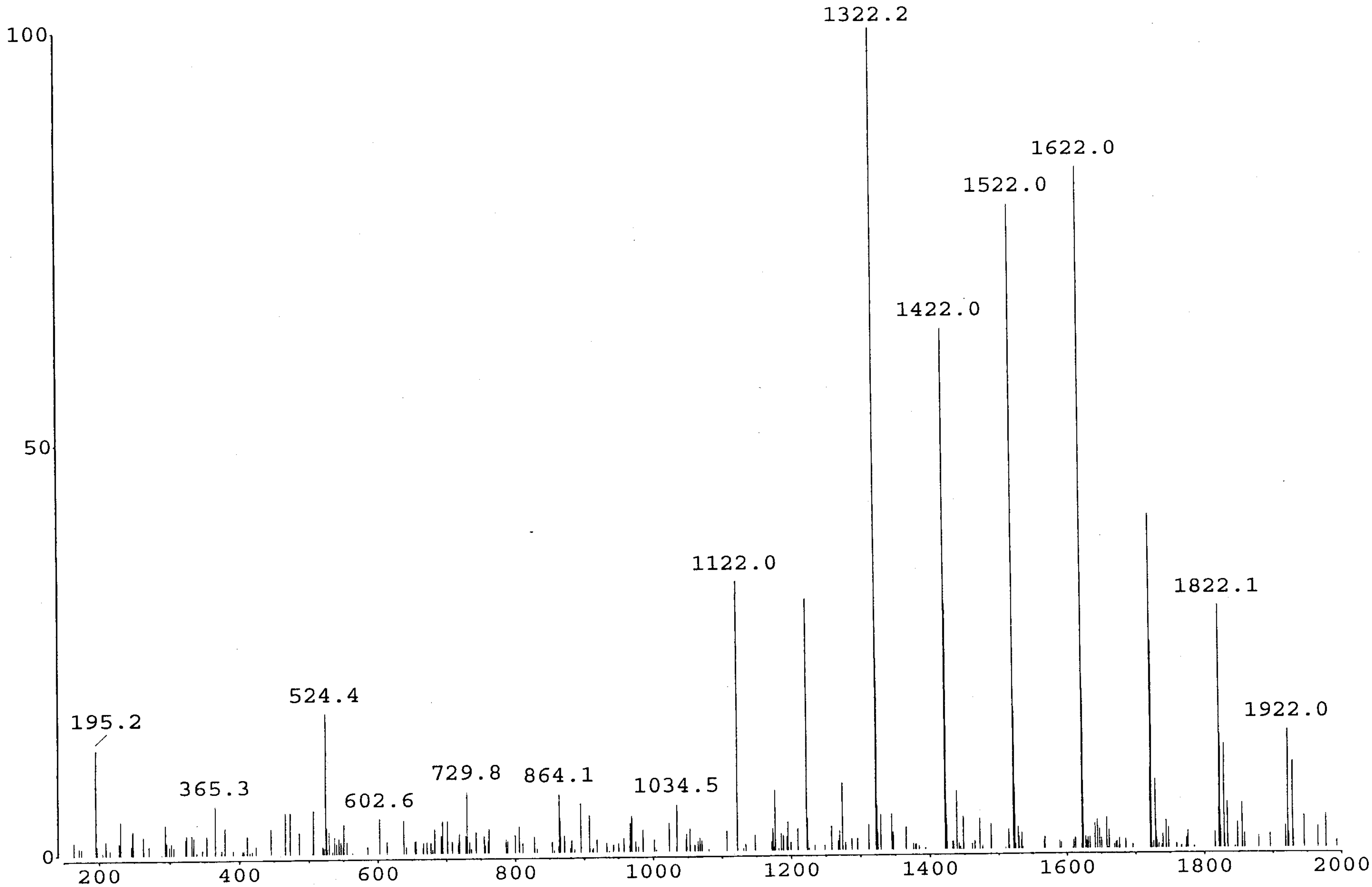

Tm013107#89 RT: 1.13 AV: 1 NL: 1.54E8
T: + c Full ms [100.00-650.00]
Relative Abundance
m/z

Ex 446
(563)

LCQ-1 Serial # LC000117 QA/QC Tune/Calibration - Passed
12/15/05

LCQ Instrument Control 15 Dec 2005 11:27 AM

S#: 2919 IT: 0.19 ST: 1.38 NL: 4.36e+008



Ex 446 (564)

LCQ-1 Serial# LC000117 Tune/Calibration 12/15/05 JNL

```
10:13:37:  Calibrating instrument...
10:13:38:  Tuning octapole resonance frequency
10:13:39:  Octapole resonance frequency is: 2447.000000
10:13:39:  Octapole frequency now set to 2447.000000
10:13:42:  Octopole Frequency Calibration Check PASSED
10:13:42:  Octopole Frequency Calibration SUCCESSFUL
10:13:42:
10:13:47:  Setting multiplier gain...
10:13:47:  Finding gain at multiplier voltage = -700.000000 V
10:14:34:  Finding gain at multiplier voltage = -800.000000 V
10:15:27:  Finding gain at multiplier voltage = -900.000000 V
10:17:42:  Finding gain at multiplier voltage = -1000.000000 V
10:18:18:  Multiplier Gain is: 92537.500000 at -1000.000000 V
10:18:18:  Finding gain at multiplier voltage = -1100.000000 V
10:19:16:  Multiplier Gain is: 152912.500000 at -1100.000000 V
10:19:16:  Finding gain at multiplier voltage = -1150.000000 V
10:19:59:  Multiplier Gain is: 200312.500000 at -1150.000000 V
10:19:59:  Finding gain at multiplier voltage = -1200.000000 V
10:21:07:  Multiplier Gain is: 268950.000000 at -1200.000000 V
10:21:07:  Finding gain at multiplier voltage = -1250.000000 V
10:22:09:  Multiplier Gain is: 382875.000000 at -1250.000000 V
10:22:09:  Finding gain at multiplier voltage = -1230.000000 V
10:23:11:  Multiplier Gain is: 332925.000000 at -1230.000000 V
10:23:11:  Multiplier Gain Calibration SUCCESSFUL
10:23:11:
10:23:11:  SUMMARY of CALIBRATION:
10:23:11:  Octopole Frequency Calibration SUCCESSFUL
10:23:11:  Multiplier Gain Calibration SUCCESSFUL
10:23:11:  All requested calibration(s) SUCCESSFULLY completed!
10:23:11:   Saving All Calibrations...
10:23:13:  Calibration is FINISHED.

10:26:37:  Calibrating instrument...
10:26:39:  Optimizing normal scan resolution...
10:26:39:  Searching for calibration masses...
10:26:49:  Peak found at m/z 195.071350
10:27:07:  Peak found at m/z 524.181641
10:27:15:  Peak found at m/z 1221.974121
10:27:24:  Peak found at m/z 1521.888672
10:27:33:  Peak found at m/z 1821.803223
10:27:54:  Optimizing resolution...
10:31:10:  Smoothing data...
10:31:22:  Optimum res ej amp for mass 195.071350 at 2.082704v
10:31:22:  Optimum res ej amp for mass 524.181641 at 4.762085v
10:31:22:  Optimum res ej amp for mass 1221.974121 at 12.497646v
```


EX 446
(565)

```
10:31:22:  Optimum res ej amp for mass 1521.888672 at 11.665340v
10:31:22:  Optimum res ej amp for mass 1821.803223 at 8.265011v
10:31:22:  old resej slope: 0.005582 old resej intercept: 0.984897
10:31:22:  new resej slope: 0.005407 new resej intercept: 1.507543
10:31:23:  Resolution optimization is finished!
10:31:27:  Calibrating masses for normal scan...
10:31:27:  Averaging spectra...
10:33:00:  Checking normal scan resolution and mass calibration...
10:33:00:  Averaging spectra...
10:34:04:  m/z 195.088200 found at m/z 195.071350
10:34:04:    (diff: -0.016850) with FWHM: 0.419571
10:34:04:  m/z 524.265000 found at m/z 524.248169
10:34:04:    (diff: -0.016831) with FWHM: 0.470647
10:34:04:  m/z 1221.991000 found at m/z 1222.040771
10:34:04:    (diff: 0.049771) with FWHM: 0.536244
10:34:04:  m/z 1521.972000 found at m/z 1522.021729
10:34:04:    (diff: 0.049729) with FWHM: 0.564163
10:34:04:  m/z 1821.953000 found at m/z 1821.936279
10:34:04:    (diff: -0.016721) with FWHM: 0.547290
10:34:04:  Normal Scan Resolution & Mass Calib. SUCCESSFUL
10:34:04:
10:34:05:  Calibrating masses for AGC prescan...
10:34:05:  Searching for calibration masses...
10:34:13:  Peak found at m/z 195.408203
10:34:20:  Peak found at m/z 524.584961
10:34:27:  Peak found at m/z 1223.031006
10:34:33:  Peak found at m/z 1522.291992
10:34:40:  Peak found at m/z 1821.552979
10:34:40:  Averaging spectra...
10:35:18:  Checking AGC scan mass calibration...
10:35:18:  Averaging spectra...
10:36:04:  m/z 195.088200 found at m/z 194.688202
10:36:04:    (diff: -0.399998) with FWHM: 2.906278
10:36:04:  m/z 524.265000 found at m/z 523.864990
10:36:04:    (diff: -0.400010) with FWHM: 4.625562
10:36:04:  m/z 1221.991000 found at m/z 1223.031006
10:36:04:    (diff: 1.040006) with FWHM: 4.221591
10:36:04:  m/z 1521.972000 found at m/z 1522.291992
10:36:04:    (diff: 0.319992) with FWHM: 4.695783
10:36:04:  m/z 1821.953000 found at m/z 1821.552979
10:36:04:    (diff: -0.400021) with FWHM: 5.253312
10:36:04:  AGC Scan Mass Calibration SUCCESSFUL
10:36:04:
10:36:04:  SUMMARY of CALIBRATION:
10:36:04:  Normal Scan Resolution Calibration SUCCESSFUL
10:36:04:  Normal Scan Mass Calibration SUCCESSFUL
10:36:04:  AGC Scan Mass Calibration SUCCESSFUL
10:36:04:  All requested calibration(s) SUCCESSFULLY completed!
```


Ex 446
(566)

```
10:36:04:   Saving All Calibrations...
10:36:05:  Calibration is FINISHED.

10:50:55:  Calibrating instrument...
10:50:56:  Searching for calibration masses...
10:51:05:  Peak found at m/z 195.113037
10:51:13:  Peak found at m/z 1521.996826
10:51:13:  Calibrating waveforms...
10:51:14:  Scanning mass 195.088200 at q of 0.830000
10:51:47:  Done scanning
10:51:47:  Scanning mass 1521.972000 at q of 0.830000
10:52:41:  Done scanning
10:52:41:  Calculating calibration for q of 0.830000
10:52:48:  Scanning mass 195.088200 at q of 0.250000
10:53:19:  Done scanning
10:53:20:  Scanning mass 1521.972000 at q of 0.250000
10:54:24:  Done scanning
10:54:24:  Calculating calibration for q of 0.250000
10:54:30:  Waveform/frequency calibration complete
10:54:30:  Checking waveform isolation frequency calibration...
10:54:42:  Isolating m/z 525.246216...
10:54:50:  95.574436 percent of m/z 525.246216 retained
10:54:50:  99.846417 percent of m/z 524.246216 ejected
10:54:50:  99.954871 percent of m/z 526.246216 ejected
10:54:50:  Isolation efficiency is OK
10:54:50:  Checking MS/MS activation frequency calibration...
10:55:51:  Expected frequency: 68.150000 kHz...
10:55:51:   Observed frequency: 68.099782 kHz
10:55:51:  Frequency calibration for ion activation is OK
10:55:51:  Waveform/Frequency Calibration SUCCESSFUL
10:55:51:
10:55:52:  Calibrating Injection RF...
10:55:52:  Searching for calibration masses...
10:56:01:  Peak found at m/z 195.113037
10:56:09:  Peak found at m/z 524.289917
10:56:17:  Peak found at m/z 1222.082275
10:56:25:  Peak found at m/z 1521.996826
10:56:33:  Peak found at m/z 1821.977783
10:56:33:  Optimizing Injection RF for maximum ion intensities...
11:01:28:  Smoothing data...
11:01:28:  Normalizing data...
11:01:37:  Max intensity of m/z 195.113037 at inj. RF of 38.000000
11:01:37:  Max intensity of m/z 524.289917 at inj. RF of 60.000000
11:01:37:  Max intensity of m/z 1222.082275 at inj. RF of 106.000000
11:01:37:  Max intensity of m/z 1521.996826 at inj. RF of 124.000000
11:01:37:  Max intensity of m/z 1821.977783 at inj. RF of 148.000000
11:01:37:  old ionrfslope: 0.062334 old ionrfint: 30.211300
11:01:37:  new ionrfslope: 0.066392 new ionrfint: 25.005436
```


Ex 446
(567)

```
11:01:43:  Injection RF calibration is finished.
11:01:44:  Checking Injection RF Calibration at m/z 195.088200
11:02:06:  Calculated Inj RF: 37.957654;  Observed Inj RF: 37.957654
11:02:07:  Checking Injection RF Calibration at m/z 1821.953000
11:02:27:  Calculated Inj RF: 145.967817;  Observed Inj RF: 143.967817
11:02:27:  Injection RF Calibration SUCCESSFUL
11:02:27:
11:02:27:  SUMMARY of CALIBRATION:
11:02:27:  Waveform/Frequency Calibration SUCCESSFUL
11:02:27:  Injection RF Calibration SUCCESSFUL
11:02:27:  All requested calibration(s) SUCCESSFULLY completed!
11:02:27:   Saving All Calibrations...
11:02:29:  Calibration is FINISHED.

11:08:08:  Calibrating instrument...
11:08:09:  Optimizing zoom scan resolution...
11:08:09:  Searching for calibration masses...
11:08:58:  Peak found at m/z 195.093170
11:09:17:  Peak found at m/z 524.219971
11:09:36:  Peak found at m/z 1221.976074
11:09:55:  Peak found at m/z 1521.886963
11:10:40:  Peak not found between m/z 1801.953000 and 1841.953000!
11:10:40:  Signal at m/z 1821.953000 too weak!
11:11:13:  Optimizing resolution...
11:13:19:  Smoothing data...
11:13:32:  Optimum res ej amp for mass 195.093170 at 1.252738v
11:13:32:  Optimum res ej amp for mass 524.219971 at 1.710324v
11:13:32:  Optimum res ej amp for mass 1221.976074 at 3.923381v
11:13:32:  Optimum res ej amp for mass 1521.886963 at 4.828431v
11:13:32:  old resej slope: 0.001686 old resej intercept: 1.222810
11:13:32:  new resej slope: 0.002697 new resej intercept: 0.576090
11:13:33:  Resolution optimization is finished!
11:13:37:  Calibrating masses for zoom scan...
11:13:37:  Averaging spectra...
11:14:20:  Checking zoom scan resolution and mass calibration...
11:14:20:  Averaging spectra...
11:17:01:  m/z 195.088200 found at m/z 195.133179
11:17:01:    (diff: 0.044979) with FWHM: 0.117575
11:17:01:  m/z 524.265000 found at m/z 524.250000
11:17:01:    (diff: -0.015000) with FWHM: 0.131892
11:17:01:  m/z 1221.991000 found at m/z 1222.005859
11:17:01:    (diff: 0.014859) with FWHM: 0.137947
11:17:01:  m/z 1521.972000 found at m/z 1521.991943
11:17:01:    (diff: 0.019943) with FWHM: 0.151088
11:17:01:  m/z 1821.953000 found at m/z 1821.933105
11:17:01:    (diff: -0.019895) with FWHM: 0.150135
11:17:01:  Zoom Scan Resolution & Mass Calibration SUCCESSFUL
11:17:01:
```


Ex 446
(568)

```
11:17:01:  SUMMARY of CALIBRATION:
11:17:01:  Zoom Scan Resolution Calibration SUCCESSFUL
11:17:01:  .Zoom Scan Mass Calibration SUCCESSFUL
11:17:01:  All requested calibration(s) SUCCESSFULLY completed!
11:17:01:   Saving All Calibrations...
11:17:02:  Calibration is FINISHED.
```


Ex 446
(569)

CVs

Ex 446
(570)

CURRICULUM VITAE

Last Modified: 12/18/2006

Marc A. LeBeau, Ph.D.

FBI Laboratory
Chemistry Unit, Room 4220
Federal Bureau of Investigation
2501 Investigation Parkway
Quantico, VA 22135
(703) 632-7408 FAX (703) 632-7411

PROFESSIONAL EXPERIENCE

Sept 2000 - Present

Unit Chief / Supervisory Chemist
FBI Laboratory
Federal Bureau of Investigation
Washington, D.C.

Manage the daily operation of the Chemistry Unit. Responsibility includes acceptance and assignment of cases, review of technical reports, training of personnel, coordination of Unit's response to major crimes, and performance of forensic examinations on evidence submitted to the Chemistry Unit.

Oct 1994 – Sept 2000

Forensic Chemist / Toxicologist
FBI Laboratory
Federal Bureau of Investigation
Washington, D.C.

Performed forensic examinations on evidence submitted to the Chemistry Unit. Casework involves toxicological analyses, drug and drug residue examinations, bank dye assays, ink comparisons and investigations into product tampering claims.

June 1990 – Oct 1994

Forensic Toxicology Supervisor
Saint Louis County Medical Examiner's Office
Saint Louis, Missouri

Managed the daily operation of the forensic toxicology laboratory; reviewed and signed official laboratory reports; performed medicolegal analyses on postmortem and antemortem biologicals; testified in court on validity of laboratory results; researched/developed new analytical methods.

Sept 1988 – Apr 1990

Chemistry Instructor
University of New Haven
West Haven, Connecticut

Taught general chemistry lecture and laboratory courses.

Ex 446
(571)

July 1989 – Sept 1989 **Intern**
National Medical Services
Willow Grove, Pennsylvania

Assisted supervisor of forensic casework; learned principles of environmental and forensic toxicology; assisted scientists with case analyses.

Summers 1985 & 1986 **Laboratory Technician**
Monsanto Chemical Company
Saint Louis, Missouri

Performed extractions and instrumental analyses.

EDUCATION

Aug 1984 – May 1988 **Central Missouri State University**
Warrensburg, Missouri
Bachelor of Arts Degree
Majors: Chemistry & Criminal Justice

Sept 1988 – June 1990 **University of New Haven**
West Haven, Connecticut
Master of Science Degree
Major: Forensic Science

Aug 1990 – July 1994 **Saint Louis University**
Saint Louis, Missouri
Graduate Course Work
Major: Pathology
Concentration: Toxicology

Sept 1997 – Dec 2005 **University of Maryland - Baltimore**
Baltimore, Maryland
Doctorate of Philosophy
Toxicology

PROFESSIONAL TRAINING

Jun 1992:	*An International Symposium on Forensic Toxicology.* Sponsored by The FBI Lab.
Jun 1993:	*Professional Practice in Toxicology: A Review.* Sponsored by the American Association for Clinical Chemistry.
Dec 1994:	*Chromatographic Methods in Forensic Science.* Sponsored by The FBI Laboratory.
Feb 1995:	*XSQ 7000 Operations Course.* Finnigan MAT Institute.
Mar 1995:	*Forensic Examiner Training Course.* Federal Bureau of Investigation.
Oct 1995:	*Fundamentals of Forensic Toxicology: A Basic Course.* Society of Forensic Toxicologists - 25th Anniversary Meeting.
Oct 1995:	*Advanced Forensic Toxicology: Pharmacologic and Interpretative Topics.* Society of Forensic Toxicologists 25th Anniversary Meeting.
Oct 1995:	*Advanced Forensic Toxicology: Analytical Topics.* Society of Forensic Toxicologists - 25th Anniversary Meeting.
Jun 1996:	*GCQ Operations Course.* Finnigan MAT Institute.
Aug 1996:	*Ethics Training.* FBI Headquarters.
Dec 1996:	*Management Training for Laboratory Supervisors.* FBI Laboratory.
Feb 1997:	*Pharmacokinetics and Interpretation of Old and New Drugs.* American Academy of Forensic Sciences - 49th Annual Meeting.
Feb 1997:	*Forensic Toxicology: A Living Science.* American Academy of Forensic Sciences - 49th Annual Meeting.
Feb 1997:	*Looking Backward and Forward: Three Decades of Toxicological Adventure.* American Academy of Forensic Sciences - 49th Annual Meeting.
Jul 1997:	*Bank Security Device Training.* ICI Americas, Inc. demonstration at FBI Academy.
Jul 1997:	*Capillary Electrophoresis.* Hewlett-Packard Company.
Feb 1998:	*Standardized Methods in the Forensic Science Laboratory.* American Academy of Forensic Sciences - 50th Annual Meeting.
Feb 1998:	*Methamphetamine: Synthesis, Pharmacology, Analysis and Toxicology.* American Academy of Forensic Sciences - 50th Annual Meeting.
Feb 1998:	*Tryptamines: Natural and Synthetic.* American Academy of Forensic Sciences - 50th Annual Meeting.
May 1998:	*The Science of Solid Phase Extraction.* United Chemical Technologies, Inc.
Oct 1998:	*Rohypnol.* 1998 SOFT/TIAFT Meeting. Albuquerque, New Mexico.
Oct. 1998:	*Chemical Ionization Mass Spectrometry.* 1998 SOFT/TIAFT Meeting. Albuquerque, New Mexico.
Feb 1999:	*Marijuana: A Forensic Symposium.* 1999 American Academy of Forensic Sciences - 51st Annual Meeting.
Apr 1999:	*Chemical Warfare Agent Awareness.* Edgewood Arsenal. Aberdeen Proving Grounds. Edgewood, Maryland

Aug 1999:	*Xcalibur Training Course.* Finnigan MAT Institute.
Feb 2000:	*The Use of Micro-Plate Immunoassay Systems in Analytical Toxicology.* 2000 American Academy of Forensic Sciences - 52nd Annual Meeting.
Feb 2000:	*Forensic Applications of Capillary Electrophoresis.* 2000 American Academy of Forensic Sciences - 52nd Annual Meeting.
Apr 2000:	*Ethics Training.* FBI Headquarters.
Oct 2000:	*The Forensic Toxicological Aspects of Chemical Terrorism.* 2000 SOFT Meeting. Milwaukee, Wisconsin.
Oct 2000:	*Forensic Applications of LC/MS at the Beginning of the 21st Century.* 2000 SOFT Meeting. Milwaukee, WI.
Oct 2000:	*Benzodiazepines: Pharmacology and Analytical Challenges.* 2000 SOFT Meeting. Milwaukee, Wisconsin.
Feb 2001:	*Raves, Nightclubs, and the New Youth Drug Culture.* American Academy of Forensic Sciences - 53rd Annual Meeting, Seattle, Washington.
Mar 2002:	International Conference on Domestic Violence, Sexual Assault, and Stalking. San Diego, California.
Apr 2002:	Club Drugs: Dancing with Death Conference. Sun Valley, Idaho.
May 2002:	Situational Leadership. Ninth House Network On-line Training.
Sept 2002:	Leading Scientific Organizations. 30th Annual FBI Symposium on Crime Lab Development, St. Louis, Missouri.
Oct 2002:	Drug Deterrence Program's Supervisory Drug-Free Workplace Training, Washington, DC.
Jan 2003:	Chemical Agents of Opportunity for Terrorism: Preparedness and Response. Atlanta, GA.
Sept 2003:	Achieving Excellence through Partnerships. 31st Annual Symposium on Crime Laboratory Development, Minneapolis, MN.
Oct 2003:	Preparation and Planning for Laboratory Accreditation by the American Board of Forensic Toxicology (ABFT), 2003 SOFT Meeting, Portland, Oregon.
Feb 2005:	Executive Development Institute. FBI Academy, Quantico, Virginia.
May 2005:	ASCLD-LAB *International* Assessor Training Course. Lansing, Michigan.
Aug 2005:	Crime Laboratory Development Symposium. Ross School of Business - University of Michigan. Ann Arbor, Michigan.
Oct 2005:	Blood Alcohol Concentration Extrapolation. 2005 SOFT Meeting, Nashville, Tennessee.
Oct 2005:	Postmortem Toxicology Interpretation. 2005 SOFT Meeting, Nashville, Tennessee.

PROFESSIONAL AFFILIATIONS / COMMITTEES

1989-Prsnt:	American Academy of the Forensic Sciences; Fellow
1988-1990:	American Chemical Society
1993:	American Association for Clinical Chemistry
1995-Prsnt:	The International Association of Forensic Toxicologists; Member
1995-Prsnt:	Society of Forensic Toxicologists; Full Member
1997-1998:	National Institute of Justice *Drug Testing Technology* Panel
1997-1999:	American Academy of Forensic Sciences Representative to the *Joint Committee on Education and Training in Toxicology.*
1997-2000:	*Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG)* - FBI Laboratory Representative
1998:	Department of Justice *Drug Facilitated Rape* Panel
1999-Prsnt:	Member of the Editorial Board of *Forensic Science Communications*
1999-2002:	Consultant to the College of American Pathologists-Forensic Identity Committee
1999-Prsnt:	American Society of Crime Laboratory Directors - Laboratory Accreditation Board (ASCLD-LAB) Certified Inspector
1999-Prsnt:	Chairman of SOFT *Drug-Facilitated Sexual Assault Ad-Hoc Committee*
1999-2001:	Member of SOFT *Continuing Education Ad-Hoc Committee*
1999-2004:	Chair of the 2004 FBI Laboratory Symposium on Forensic Toxicology and Joint Meeting of the Society of Forensic Toxicologists and The International Association of Forensic Toxicologists
2000:	Member of Office for Victims of Crime *Drug-Facilitated Sexual Assault* Panel
2002-2005:	Consultant to Quality Forensics – Toxicology Committee
2002-Prsnt:	Chairman of the *Scientific Working Group on the Forensic Analysis of Chemical Terrorism (SWGFACT)*
2002-2004:	Consultant to the Violence Against Women's Office – Committee to set *National Standards for the Sexual Assault Forensic Examination*
2002-Prsnt:	Member of The International Association of Forensic Toxicologists (TIAFT) Young Scientists Committee
2003-Prsnt:	Member of The International Association of Forensic Toxicologists (TIAFT) Systematic Toxicological Analysis Committee
2005:	Co-editor of the Special Issue of *Forensic Science International* dedicated to the 2004 Joint Meeting of SOFT and TIAFT. Vol. 153, No. 1, October 2005.
2005:	Co-editor of the Special Issue of *Journal of Chromatography B* dedicated to the 2004 Joint Meeting of SOFT and TIAFT.
2005:	Co-editor of the Special Issue of *Journal of Analytical Toxicology* dedicated to the 2004 Joint Meeting of SOFT and TIAFT. Vol. 29, No. 5, July/August 2005.

Ex 446
(515)

2006: Guest Editor of the SOFT Special Issue of *Journal of Analytical Toxicology*

2006-Prsnt: Member of *Journal of Analytical Toxicology* Editorial Board

CERTIFICATIONS & AWARDS

1995: Department of Justice, Federal Bureau of Investigation, *Performance Award.*

1996: Department of Justice, Federal Bureau of Investigation, *Performance Award.*

1997: Federal Bureau of Investigation. *Incentive Award.*

1997: Department of Justice, Federal Bureau of Investigation, *Performance Award.*

1998: Federal Bureau of Investigation. *Quality Step Award.*

1998: Department of Justice, Federal Bureau of Investigation, *Performance Award*

1999: Department of Justice, Federal Bureau of Investigation, *Performance Award*

1999: Federal Bureau of Investigation. *Quality Step Award.*

1999: Certificate of Achievement - TWA Investigation.

1999: Certificate of Achievement - JASMUR Investigation

2000: Department of Justice, Federal Bureau of Investigation, *Performance Award.*

2004: Federal Bureau of Investigation. *Director's Award for Outstanding Scientific Advancement*

PRESENTATIONS & TECHNICAL INSTRUCTION

Jun 1996: *Forensic Toxicology.* DEA International Training Conference - Alexandria, Virginia.

Jul 1996: *Forensic Chemistry and Toxicology.* Aspen Systems, Inc. - Rockville, Maryland.

Sept 1996: *Forensic Toxicology.* DEA International Training Conference - Alexandria, Virginia.

Mar 1997: *Submission of Chemistry Evidence.* Evidence Program In-Service - Quantico, Virginia.

May 1997: *Submission of Chemistry Evidence.* Evidence Program In-Service - Quantico, Virginia.

Jun 1997: *Forensic Toxicology.* DEA International Training Conference - Alexandria, Virginia.

Jul 1997: *The Use of Chromatography in Forensic Toxicology.* ***Chromatographic Methods in Forensic Science.*** Sponsored by The FBI Laboratory.

Jul 1997: *Murder by Poison.* 49th Annual Meeting of the American Association of Clinical Chemists - Atlanta, Georgia.

Mar 1998: *The Use of Chromatography and Mass Spectrometry in Forensic Toxicological Examinations.* ***Chromatographic Methods in Forensic Science.*** Sponsored by The FBI Laboratory.

Mar 1998: *Submission of Chemistry Evidence.* Evidence Program In-Service - Quantico, Virginia.

Jul 1998: *The Use of Chromatography and Mass Spectrometry in Forensic Toxicological Examinations.* ***Chromatographic Methods in Forensic Science.*** Sponsored by The FBI Laboratory.

Aug 1998: *Forensic Toxicology.* DEA International Training Conference - Alexandria, Virginia.

Sept 1998: *The Toxicological Investigation of Drug-Facilitated Sexual Assaults.* New York State Forensic Toxicology Seminar - Albany, New York.

Oct 1998: *Direct Analysis of Urine for Succinylcholine.* 1998 SOFT/TIAFT Meeting. Albuquerque, New Mexico.

Nov 1998: *Drug-Facilitated Sexual Assault.* New York Prosecutors Training Institute, Inc., Queens County, New York.

Feb 1999: *The Analysis of Biofluids for Flunitrazepam and Metabolites by Electrospray LC/MS.* American Academy of Forensic Sciences - 51st Annual Meeting, Orlando, Florida.

Apr 1999: *The Toxicological Investigation of Drug-Facilitated Sexual Assaults.* Southern Association of Toxicologists - Houston, Texas.

Jun 1999: *The Toxicological Investigation of Drug-Facilitated Sexual Assaults.* Henry Lee Institute. Meriden, Connecticut.

Jun 1999: *The Toxicological Investigation of Drug-Facilitated Sexual Assaults.* Virginia Campus Law Enforcement Association - Virginia Beach, Virginia.

Aug 1999: *Submission of Chemistry Evidence.* Evidence Supervisors Program In-Service - Quantico, Virginia.

Sept 1999: *The Investigation of Drug-Facilitated Sexual Assaults.* Salt Lake City Police and Crime Lab Personnel. Salt Lake City, Utah.

Oct 1999: *Analytical Issues Surrounding Drug-Facilitated Assaults.* 1999 SOFT Meeting. Rio Mar, Puerto Rico.

Oct 1999: *Rapid Analysis of Biofluids for GHB and GBL using Headspace GC-FID and GC-MS.* 1999 SOFT Meeting. Rio Mar, Puerto Rico.

Ex 446
(577)

Feb 2000: *Differentiation of Bufotenine and Psilocin by GC/MS(CI).* American Academy of Forensic Sciences - 52nd Annual Meeting, Reno, Nevada.

Feb 2000: *Brodifacoum Identification by Electrospray LC/MS and LC/MS/MS/MS.* American Academy of Forensic Sciences - 52nd Annual Meeting, Reno, Nevada.

Apr 2000: *The Investigation of Drug-Facilitated Sexual Assaults.* Los Angeles Police and Crime Lab Personnel. Los Angeles, California.

Apr 2000: *The Investigation of Drug-Facilitated Sexual Assaults.* Santa Monica Rape Treatment Center. Santa Monica, California.

May 2000: *Drug-Facilitated Sexual Assaults.* National Organization of Women - National Judicial Education Program. Chicago, Illinois.

May 2000: *Investigations of Drug-Facilitated Sexual Assaults.* New York State Police Sex Offense Seminar. Albany, New York.

Jun 2000: *Drug-Facilitated Sexual Assaults.* Rochester-Area Multidisciplinary Team. Rochester, New York.

Jun 2000: *Drug-Facilitated Sexual Assaults.* National Organization of Women - National Judicial Education Program. Burlington, Vermont.

Aug 2000: *The Effect of Storage Temperature on Endogenous GHB Levels in Urine.* TIAFT Meeting, Helsinki, Finland.

Oct 2000: *The Use of LC/MS and LC/MS^n to Solve Analytical Challenges in Forensic Toxicology*. 2000 SOFT Meeting. Milwaukee, Wisconsin.

Oct 2000: *A Comparison of Instrumental Approaches to Benzodiazepines Analysis: A Case Study.* 2000 SOFT Meeting. Milwaukee, Wisconsin.

Oct 2000: *Intra- and Interindividual Variations in Urinary Levels of Endogenous GHB.* 2000 SOFT Meeting. Milwaukee, Wisconsin.

Dec 2000: *Drug-Facilitated Sexual Assault.* Ohio University Drug Facilitated Sexual Assault Conference. Athens, Ohio.

Jan 2001: *Drug-Facilitated Sexual Assaults.* Third National Symposium on Victims of Federal Crime. Washington, DC.

Feb 2001: *Homicidal Poisonings.* American Academy of Forensic Sciences - 53rd Annual Meeting, Seattle, Washington.

Feb 2001: *Managing and Testifying in Drug-Facilitated Sexual Assault Cases.* American Academy of Forensic Sciences - 53rd Annual Meeting, Seattle, Washington.

Feb 2001: *Drug-Facilitated Sexual Assault: An FBI Perspective.* American Academy of Forensic Sciences - 53rd Annual Meeting, Seattle, Washington.

Feb 2001: *Solving Investigative Challenges in Drug Facilitated Rape.* American Academy of Forensic Sciences - 53rd Annual Meeting, Seattle, Washington.

Feb 2001: *Advances in Drug Detection in Drug Facilitated Rape.* American Academy of Forensic Sciences - 53rd Annual Meeting, Seattle, Washington.

Mar 2001: *Investigations of Drug-Facilitated Sexual Assaults.* California Sexual Assault Investigators Association Meeting - Shell Beach, California.

Apr 2001: *Investigations of Drug-Facilitated Sexual Assaults.* Southern Indiana Sexual Assault Training Project - New Albany, Indiana.

May 2001: *Homicidal Poisonings.* California Association of Toxicologists Quaterly Meeting, San Diego, California.

May 2001: *Toxicological Investigations of Drug-Facilitated Sexual Assaults.* First National Sexual Assault Response Team Conference - San Antonio, Texas.

Aug 2001: *Intra- and Interindividual Variations in Urinary Levels of Endogenous GHB.* 2001 TIAFT Meeting. Prague, Czech Republic.

Sept 2001: *Toxicological Investigations and Evidence Collection in Drug-Facilitated Sexual Assault Cases.* 7th International Congress of Therapeutic Drug Monitoring and Clinical Toxicology - Washington, DC.

Oct 2001: *Does Creatinine-Normalization Decrease the Variations in Urinary Levels of Endogenous GHB?* 2001 SOFT Meeting. New Orleans, Louisiana.

Feb 2002: *The Interpretation of GHB in Biological Specimens* as part of *GHB: Old Substance, New Problem* workshop. 2002 AAFS Meeting, Atlanta, Georgia.

Mar 2002: *Drug-Facilitated Sexual Assault – Toxicology: What You Need to Know.* 2nd International Conference on Domestic Violence, Sexual Assault and Stalking. San Diego, California.

Mar 2002: *Overcoming the Challenges of Drug-Facilitated Sexual Assault Investigations.* 2002 Pittsburgh Conference. New Orleans, Louisiana.

Mar 2002: *Drug-Facilitated Sexual Assault Investigations.* 2002 Missouri Office of Prosecution Services Spring Statewide Training, Osage Beach, Missouri.

April 2002: *Club Drugs: Dancing with Death.* Sponsored by United States Attorney's Office, Sun Valley, Idaho.

Aug 2002: *Toxicological Investigations of Drug-Facilitated Sexual Assault.* 2002 Drug Enforcement Administration Victim Witness Conference, Las Vegas, Nevada.

Sept 2002: *Fentanyl in Hair by LC/MS and LC/MS/MS.* 2002 TIAFT Meeting. Paris, France.

Oct 2002: *Managing and Testifying in Drug-Facilitated Sexual Assault Cases.* 2002 SOFT Meeting, Dearborn, Michigan.

Dec 2002: *Toxicological Investigations of Drug-Facilitated Sexual Assault.* New York City Alliance Against Sexual Assault, New York, New York.

May 2003: *Evidence Collection in DFSA Cases: Clinical and Forensic Issues.* Gamma Hydroxybutyrate: Spotlight on GHB Conference. Orlando, Florida.

May 2003: *Drug-Facilitated Sexual Assault: Why We Fail...How We Can Succeed.* Second National Sexual Assault Response Team Conference – New Orleans, Louisiana.

May 2003: *Toxicological Investigations of Drug-Facilitated Sexual Assault* Colorado Coalition Against Sexual Assault – Breckenridge, Colorado.

Aug 2003: *Toxicological Investigations of Drug-Facilitated Sexual Assault.* Kane County Coalition Against Sexual Assault, Illinois.

Sept 2003: *Drug-Facilitated Sexual Assault: The U.S. Perspective.* 2003 International Congress on Therapeutic Drug Monitoring and Clinical Toxicology, Basel Switzerland.

Nov 2003: *The Challenges of Drug-Facilitated Sexual Assault.* New Zealand and Australia Forensic Science Society, Sydney, Australia.

Nov 2003: *Drug-Facilitated Sexual Assault.* 2003 TIAFT Meeting, Melbourne, Australia.

Mar 2004: *DFSA Investigations: What's New With DFSA.* California Sexual Assault Investigators Association Meeting - Shell Beach, California.

Apr 2004: *Forensic Chemistry.* Saudi Arabia National Police – Riyadh, Saudi Arabia.

Apr 2004: *Drug-Facilitated Sexual Assault.* The Other Half of the Solution: Men Taking a Stand to End Sexual Violence. New Jersey Coalition Against Sexual Assault – Long Branch, New Jersey.

May 2004: *Application of Analytical Chemistry at the FBI Laboratory.* Analytica Conference 2004, Munich, Germany.

June 2004: *Evidence Collection for Drug-Facilitated Sexual Assault.* Attorney General's Sexual Assault Task Force, SART Training, Eugene, Oregon.

Jun 2004: *Challenges of Drug-Facilitated Sexual Assault.* Attorney General's Sexual Assault Task Force, SART Training, Eugene, Oregon.

Jun 2004: *Drug-Facilitated Sexual Assault.* Maryland Coalition Against Sexual Assault. Columbia, Maryland.

Aug 2004: *Forensic Chemistry.* 2004 FBI Laboratory Symposium on Forensic Toxicology. Washington, DC.

Aug 2004: *Managing Cases of DFSA.* 2004 FBI Laboratory Symposium on Forensic Toxicology. Washington, DC.

Sept 2004: *The Toxicological Evidence in Drug-Facilitated Sexual Assault Cases.* Spotlight on GHB Conference. Las Vegas, Nevada.

Oct 2004: *Drug-Facilitated Sexual Assault Training for Forensic Scientists.* NEAFS Conference. Mystic, Connecticut.

Feb 2005: *Bank Security Dye Packs and the FBI Laboratory.* 2005 AAFS Meeting. New Orleans, Louisiana.

Mar 2005: *Drug-Facilitated Sexual Assault:* 2005 Federal Conference for Victims of Crime, Atlanta, Georgia.

Mar 2005: *The Challenges of Drug-Facilitated Sexual Assault.* Louisiana SANE/SART Program, Baton Rouge, Louisiana.

Jun 2005: *Recommendations for Combating Drug-Facilitated Sexual Assault,* Third National SART Training Conference, San Francisco, California.

Jul 2005: *The Toxicological Evidence in Drug-Facilitated Sexual Assault Cases.* Columbia, South Carolina.

Aug 2005: *A Comprehensive Study on the Variations of Urinary Concentrations of Endogenous GHB.* The International Association of Forensic Toxicologists Conference, Seoul, South Korea.

Sept 2005: *Drug-Facilitated Sexual Assault.* Fairfax Coalition Against Sexual Assault. Fairfax, Virginia.

Sept 2005: *Drug-Facilitated Sexual Assault.* The Peace Corps, Washington, DC.

Oct 2005: *Drug-Facilitated Sexual Assault.* The EVAW International Conference on Sexual Assault, Domestic Violence, and Stalking. Baltimore, Maryland.

Nov 2005: *Recommendations for Combating Drug-Facilitated Sexual Assault,* Los Angeles Chief of Police Conference, Los Angeles, California

Feb 2006: *Health Care Fraud.* Health Care Fraud Coordinators' Conference. Columbia, South Carolina.

Mar 2006: *Drug-Facilitated Sexual Assault*. Georgia Network to End Sexual Assault, Atlanta, Georgia.

Apr 2006: *Recommendations for Combating Drug-Facilitated Sexual Assault*, Armed Forces Institute of Pathology Conference Against Sexual Violence, Rockville, Maryland.

Apr 2006: *Drug-Facilitated Sexual Assault*. EVAW Conference, Kansas City, Missouri.

Jun 2006: *Challenges of Drug-Facilitated Crimes*. Deutsche Pharmazeutische Gesellschaft, Saar, Germany.

Jul 2006: *Drug-Facilitated Sexual Assault*. EVAW Conference, Washington, DC.

Aug 2006: *Mivacurium and Metabolites by LC/FLU and LC/MS/MS*. The International Association of Forensic Toxicologists Annual Meeting, Ljublijana, Slovenia.

Aug 2006: *Further Evidence of In Vitro Production of GHB in Urine*. The International Association of Forensic Toxicologists Annual Meeting, Ljublijana, Slovenia.

Oct 2006: *Uncertainty of Quantitative Measurements*. Society of Forensic Toxicologists Annual Meeting, Austin, Texas.

Oct 2006: *Overcoming the Toxicological Challenges of Drug-Facilitated Sexual Assault*. Society of Forensic Toxicologists Annual Meeting, Austin, Texas.

PUBLICATIONS

LeBeau, M., Montgomery, M., and Miller, M. *Direct Analysis of Urine for Succinylcholine.* In: Proceedings of the 1998 SOFT/TIAFT Meeting. Albuquerque, New Mexico.

LeBeau, M., et al. *Recommendations for the Toxicological Investigations of Drug-Facilitated Sexual Assaults.* Journal of Forensic Sciences, Volume 44, Number 1, January 1999.

LeBeau, M. *Toxicological Investigations of Drug-Facilitated Sexual Assaults.* Forensic Science Communications, Volume 1, Number 1, 1999.

Schwartz, R., Milteer, R., LeBeau, M. *Drug-Facilitated Sexual Assault (Date Rape).* Southern Medical Journal, Volume 93, Number 6, 2000.

LeBeau, M., Montgomery, M., Wagner, J., and Miller, M. *Analysis of Biofluids for Flunitrazepam and Metabolites by Electrospray Liquid Chromatography/Mass Spectrometry.* Journal of Forensic Sciences, Volume 45, Number 5, 2000.

LeBeau, M., Montgomery, M., Miller, M., and Burmeister, S. *Analysis of Biofluids for Gamma-Hydroxybutyrate (GHB) and Gamma-Butyrolactone (GBL) by Headspace GC/FID and GC/MS.* Journal of Analytical Toxicology, Volume 24, 2000.

LeBeau, M., Montgomery, M., Jufer, R., and Miller, M. *Elevated GHB in Citrate Buffered Blood.* Journal of Analytical Toxicology, Volume 24, 2000.

LeBeau, M., Miller, M., and Levine, B. *Effect of Storage Temperature on Endogenous GHB Levels in Urine.* Forensic Science International, Volume 119(2), 2001.

Darby, S., Miller, M., Allen, R., and LeBeau, M. *A Mass Spectrometric Method for Quantitation of Insulin in Blood Samples.* Journal of Analytical Toxicology, Volume 25, 2001.

Drug-Facilitated Sexual Assault: A Forensic Handbook. Marc A. LeBeau and Ashraf Mozayani, Eds. (2001), Academic Press: London.

LeBeau, M.A. *Forensic Toxicology at the FBI Laboratory.* NEACT Journal, Volume 20, Number 1, 2001.

SWGDRUG Recommendations for Education and Training, Quality Assurance, and Methods of Analysis, U.S. Department of Justice, Drug Enforcement Administration, 2001.

LeBeau, M.A., Christenson, R.H., Levine, B., Darwin, W.D., and Huestis, M.A. *Intra- and Interindividual Variations in Urinary Concentrations of Endogenous Gamma-Hydroxybutyrate (GHB),* Journal of Analytical Toxicology, Volume 26, 2002.

LeBeau, M.A. and Quenzer, C.F. *Succinylmonocholine Identified in Negative Control Tissues,* Volume 27, 2003.

LeBeau, Marc A., *Quality Assurance Guidelines for Laboratories Performing Forensic Analysis of Chemical Terrorism,* Forensic Science Communications, Volume 6, Number 2, 2004.

Montgomery, Madeline A., LeBeau, Marc A., Miller, Mark L., and Jufer, Rebecca A. *The Identification of Mivacurium and Metabolites in Biological Samples.* Journal of Analytical Toxicology, Volume 29, 2005.

Montgomery, M.A., LeBeau, M.A., Jenkins, A.J. *Qualitative Identification of Doxacurium and its Breakdown Products in Postmortem Fluids by Liquid Chromatography-Tandem Mass Spectrometry.* Journal of Analytical Toxicology, Volume 30, 2006.

LeBeau, M.A. Montgomery, M.A., Morris-Kukoski, C., Schaff, J.E., Deakin, A., Levine, B., *A Comprehensive Study on the Variations of Urinary Concentrations of Endogenous GHB,* Journal of Analytical Toxicology, Volume 30, 2006.

Jagerdeo, E., Leibowitz, J.N., Schumacher, L., Henningsen, D.A., and LeBeau, M. *Analysis of Trace Amount of Bank Dye and Lachrymators from Exploding Bank Devices by Solid-Phase Microextraction and Gas Chromatography-Mass Spectrometry,* Jounral of Chromatographic Science, Volume 44, 2006.

Ex 446
(582)

Bradley, M.J., Keagy, R.L., Lowe, P.C., Rickenbach, M.P., Wright, D.M., and LeBeau, M.A. *A Validation Study for Duct Tape End Matches*, Journal of Forensic Sciences, Volume 51, Number 3, May 2006.

LeBeau, M. Gamma-Hydroxybutyric Acid (GHB) in *Principles of Forensic Toxicology.* Barry Levine, Ed., 2nd edition, (2006), AACC Press, Washington, DC.

LeBeau, M. Montgomery, M., Morris-Kukoski, C., Schaff, J., and Deakin, A. *Further Evidence of In Vitro Production of GHB in Urine Samples*, Forensic Science International, Accepted for Publication.

LeBeau, M. Drug-Facilitated Crimes in *Encyclopedia of Forensic Science,* Olaf Drummer, Section Editor, In press.

LeBeau, M. Drug-Facilitated Sexual Assault in *Sexual Abuse and Assault: Adult Victims*, Linda Ledray, Ann Burgess, and Angelo Giardino, Eds., In press.

Ex 446
(583)

Jason D. Brewer

Federal Bureau of Investigation
Chemistry Unit, Room 4220
2501 Investigation Parkway
Quantico, VA 22135
(703) 632-7827

PROFESSIONAL EXPERIENCE

Sep 2006 - present **Forensic Chemist Examiner**
FBI Laboratory
Federal Bureau of Investigation
Quantico, VA

Perform forensic examinations on evidence submitted to the Chemistry Unit in criminal investigations in the areas of controlled substances, bank security devices, pepper sprays, inks, lubricants, secret writing and general unknowns. Interpret data, prepare written reports and provide expert witness testimony regarding results of chemical analyses.

Nov 2004 - Sep 2006 **Chemist**
FBI Laboratory
Federal Bureau of Investigation
Quantico, VA

Performed forensic examinations on evidence submitted to the Chemistry Unit in criminal investigations in the areas of controlled substances, bank security devices, pepper sprays, inks and general unknowns.

Jan 2004 - Nov 2004 **Postdoctoral Fellow (Visiting Scientist)**
Oak Ridge Institute for Science and Education (ORISE)
FBI Laboratory
Federal Bureau of Investigation
Quantico, VA

Developed new methods for the identification of organic dyes in ink at the FBI Laboratory, Counterterrorism and Forensic Science Research Unit (CTFSRU). Presented work at scientific meetings and published results in peer reviewed journals. Assisted in preparation of quarterly reports and presented results to the FBI Laboratory, Chemistry Unit.

Aug 1998 - Aug 2003 **Research Assistant**
Department of Chemistry
The University of North Carolina at Chapel Hill
Chapel Hill, NC

Designed and performed high vacuum, thin-film deposition experiments; collected and interpreted spectroscopic ellipsometry, atomic force microscopy, capacitance-voltage and other applicable data; periodically reported significant results. Researched scientific literature and presented relevant articles to research group. Generated an original research dissertation concerning plasma nitridation of silicon dioxide films for semiconductor applications.

Aug 1998 - May 2000 **Teaching Assistant**
Department of Chemistry
The University of North Carolina at Chapel Hill
Chapel Hill, NC

Instructed and evaluated students in Physical Chemistry Lab (Scanning Tunneling Microscopy) and General Chemistry Lab I and II.

May 1998 - Aug 1998 **Intern**
Virginia Semiconductor, Inc.
Fredericksburg, VA

Assisted in the manufacturing and testing of single-crystal silicon wafers. Initiated and maintained a wastewater neutralization system.

May 1996 - May 1998 **Research Assistant**
Department of Chemistry
James Madison University
Harrisonburg, VA

Investigated and synthesized several (tricarbonyl) (4-L-pyridine) (5-L'-1,10-phenanthroline) rhenium (I) perchlorates for applications as molecular reporters. Presented results at scientific meetings.

May 1997 - Aug 1997 **Intern**
National Science Foundation (NSF) / San Jose State University (SJSU) / IBM
IBM Almaden Research Center (IBM-ARC)
Almaden, CA

Studied the chemical modification of fused-silica capillaries as part of a joint NSF/SJSU/IBM-ARC summer research program. Presented results at a research symposium at IBM-ARC.

May 1995 - Aug 1995 **Intern**
FBI Training Division
Federal Bureau of Investigation
Quantico, VA

Catalogued and distributed ammunition; organized and maintained target scenarios at the Firearms Training Unit (FTU) range.

EDUCATION

1998 - 2003 **The University of North Carolina at Chapel Hill**
Chapel Hill, NC
Doctor of Philosophy Degree
Major: Chemistry

1994 - 1998 **James Madison University**
Harrisonburg, VA
Bachelor of Science Degree
Major: Chemistry

Ex 446
(555)

AWARDS AND HONORS

2006	FBI Award (casework recognition)
2001 - 2002	UNC at Chapel Hill Francis P. Venable Fellowship
2001	AVS Plasma Science and Technology Division, Coburn and Winters Award Finalist
2001	AVS Dorothy M. and Earl S. Hoffman Travel Scholarship
1998 - 1999	UNC at Chapel Hill Francis P. Venable Fellowship
1998	The Degesch America, Inc. Award Recipient

PROFESSIONAL AFFILIATIONS

Mar 2006 - Present	Scientific Working Group on the Forensic Analysis of Chemical Terrorism (SWGFACT)
2001 - 2002	American Vacuum Society (AVS)
2000 - 2001	Materials Research Society (MRS)
1996 - 2000	American Chemical Society (ACS)

PROFESSIONAL TRAINING RECEIVED

Aug 2006	Raman Microscopy, McCrone Research Institute, Chicago, IL
Jun 2006	Microsoft Office 2000: Intermediate Access, FBI Virtual Academy, Quantico, VA
Apr 2006	Microsoft Office 2000: Beginning Access, FBI Virtual Academy, Quantico, VA
Mar 2005	IR Spectrometry for Trace Analysis, FBI Academy, Quantico, VA
Feb 2005	Modern Practice of GC/MS, Shimadzu, Columbia, MD
Feb 2005	Forensic Chemist Seminar, DEA, Sterling, VA
Feb 2005	HPLC Operation and Troubleshooting, Waters, FBI Laboratory, Quantico, VA
Jan 2005	CE of Ballpoint Ink, FBI Laboratory, Quantico, VA
Jan 2005	GC-TOF MS Operation, Waters, FBI Laboratory, Quantico, VA
Dec 2004	On-line Extraction for Automating Sample Preparation, FBI Laboratory, Quantico, VA
Dec 2004	Basic Q-TOF Theory and Operation, Waters, FBI Laboratory, Quantico, VA
Mar 2004	Practical Capillary Electrophoresis, ACS, Anaheim, CA

PRESENTATIONS

Oct 2004	The 15th Annual Frederick Conference on CE/Proteomics, Frederick, MD. "Forensic Analysis of Ballpoint Pen Inks Using Capillary Electrophoresis."
May 2004	FBI Laboratory, Quantico, VA. "Ink Analysis by Capillary Electrophoresis."
Aug 2003	Original Doctoral Dissertation, The University of North Carolina at Chapel Hill, Chapel Hill, NC. "Plasma Nitridation of Silicon Dioxide Studies."
Oct 2001	AVS 48th International Symposium, San Francisco, CA. "Study of SiO_xN_y Films Deposited by Radio-Frequency Plasma Assisted Electron Cyclotron Resonance."
Mar 2001	The University of North Carolina at Chapel Hill, Chapel Hill, NC. "Next Generation Gate Dielectrics for Metal-Oxide-Semiconductor Microelectronic Devices."
Nov 2000	MRS Fall Meeting, Boston, MA. "Kinetics of Nitrogen Incorporation in Thermal Silicon Dioxide by Radio-Frequency Plasma Assisted Electron Cyclotron Resonance."

Nov 2000	MRS Fall Meeting, Boston, MA. "Study of Silicon Oxynitride Films Produced by Radio-Frequency Plasma Assisted Electron Cyclotron Resonance."
Aug 1997	NSF/SJSU/IBM-ARC Research Symposium, Almaden, CA. "The Etching and Chemical Modification of Fused-Silica Capillaries."
Nov 1996	Southeast Regional Meeting of the American Chemical Society, Greenville, SC. "Applications of Rhenium (I) Micelle Complexes as Molecular Reporters."

PUBLICATIONS

J.M. Egan, J.D. Brewer, K.A. Hagan and C.L. Strelko, "Capillary Electrophoresis of Ballpoint Pen Inks," *Journal of the American Society of Questioned Document Examiners* **9**(1), 37 (2006).

J.D. Brewer, J.M. Egan and K. Hagan, "Forensic Analysis of Blue Ballpoint Pen Inks Using Capillary Electrophoresis," *Forensic Science Communications* **7**(3), July 2005.

J.M. Egan, J.D. Brewer and K. Hagan, "Forensic Analysis of Black Ballpoint Pen Inks Using Capillary Electrophoresis," *Forensic Science Communications* **7**(3), July 2005.

A. Raveh, J.D. Brewer and E.A. Irene, "Nitridation of thermal SiO_2 films by radio-frequency plasma assisted electron cyclotron resonance: Effect of plasma modes and process parameters," *Journal of Vacuum Science and Technology A* **19**(1), 9 (2001).

A. Raveh, J.D. Brewer and E.A. Irene, "Nitridation of thermal SiO_2 films by radio-frequency plasma assisted electron cyclotron resonance: Layer structure and composition," *Journal of Vacuum Science and Technology A* **19**(1), 17 (2001).

Ex 446
(587)

EILEEN MARIE WANINGER

Federal Bureau of Investigation
Chemistry Unit, Room 4220
2501 Investigation Parkway
Quantico, VA 22135
(703) 632-7422

PROFESSIONAL EXPERIENCE

Sept 1994 - present **Forensic Chemist Examiner**
FBI Laboratory
Federal Bureau of Investigation
Washington D.C./ Quantico, VA

Performs forensic examinations on evidence submitted to the Chemistry Unit in criminal investigations in the areas of controlled substances, bank security devices, pepper sprays, and general unknowns. Supervises chemists, reviews data, and prepares written reports. Provides expert testimony regarding results of chemical analyses.

Oct 1991- August 1994 **Forensic Scientist**
Illinois State Police
Suburban Chicago Forensic Science Laboratory
Toxicology Section
Maywood, IL

Duties included the forensic analysis of biological fluids for the presence of alcohol and drugs. Prepared written reports and provided expert testimony regarding results of analyses.

Jan 1990 – Oct 1991 **Forensic Scientist**
Illinois State Police
Springfield Forensic Science Laboratory
Toxicology Section
Springfield, IL

Duties included the forensic analysis of biological fluids for the presence of alcohol and drugs. Prepared written reports and provided expert testimony regarding results of analyses.

June 1986 – Dec 1988 **Researcher**
Southern Illinois University School of Medicine
Department of Pharmacology and the Department of Internal Medicine
Springfield, IL

Performed research in the areas of pharmacokinetic analyses of various drugs. Performed a computer analysis for determining ventricular volume and function of cardiac catheterization patients in order to assist physicians.

EDUCATION

1989 - 1990	**Sangamon State University** Graduate Courses in Business Administration Springfield, IL
1984-1986	**Illinois College** Bachelor of Science Degree Majors: Chemistry and Biology Jacksonville, IL
1982-1984	**Springfield College in Illinois** Associate in Arts Degree Major: Pre-Med Springfield, IL

PROFESSIONAL TRAINING

2005	*33rd Annual Crime Laboratory Development Symposium*, (Ann Arbor, MI)
2005	*HPLC Operation and Troubleshooting*, Waters Corporation, (Quantico, VA)
2004	*On-Line Extraction for Automating Sample Prep for LC and GC*, (Quantico, VA)
2004	*Basic Q-TOF Theory and Operation*, Waters Corporation/Micromass, (Quantico, VA)
2004	*Quantitative Residue Analysis Using Mass Spectrometry*, Waters Corporation (Quantico, VA)
2004	*ASCLD/LAB – International Assessor Training (*Woodbridge, VA)
2002	*FBI Support Supervisors Management Seminar*, FBI Academy (Quantico, VA)
2002	*Forensic Microscopy*, McCrone Research Institute (Chicago, IL)
2002	*Advancements in IR Microsampling Technology,* Nicolet (Lancaster, PA)
2002	*Basic Hazardous Materials (HAZMAT) Transportation Training,* FBI Headquarters (Washington, DC)
2001	*Advancements in Analytical LC/MS Methods,* Waters Corporation, (FBI Headquarters, Washington, DC)
2001	*DEA Forensic Chemist Seminar,* Special Testing and Research Laboratory (Chantilly, VA)
1999	*Mass Spectrometry Workshop.* Mid-Atlantic Association of Forensic Scientists (Harpers Ferry, WV)
1999	*American Society of Crime Laboratory Directors Laboratory Accreditation Board (ASCLD/LAB) Inspector Training*, (Chicago, IL)
1998	*Clandestine Laboratory Investigations Workshop*, Mid-Atlantic Association of Forensic Scientists, (Rockville, MD)
1998	*LCQ Basic Operation Course*, ThermoQuest Institute (Riviera Beach, FL)
1997	*Nicolet Omnic FT-IR Training Course*, Nicolet Instrument Company (Washington , DC)
1996	*Spill Control and Containment Training*, Fisher Safety-ESP (Washington, DC)
1995	*Radiation Awareness Training, FBI Headquarters (Washington, DC)*
1995	*Finnigan XSQ 7000 Mass Spectrometer Operations Course*, Finnigan MAT Institute (Cincinnati, OH)
1994	*Chromatographic Methods in Forensic Science*, FBI Academy (Quantico, VA)
1994	*FBI Forensic Examiner Training,* FBI Academy (Quantico, VA)
1993	*Roche Grand Rounds Cobas Mira Training,* Roche Diagnostics (Schaumburg, IL)

1992	*GC/MS Confirmation from Toxi-Lab Extractions*, Toxi-Lab Inc. and Finnigan (Schaumburg, IL)
1992	*Restek Capillary Chromatography Seminar*, Restek Corporation (Oakbrook, IL)
1991	*Pharmacology*, Spring Semester, Sangamon State University (Springfield, IL)
1990	*Hewlett-Packard GC/MS Operator's Training Course*, Hewlett-Packard (Springfield, IL)
1990	*Hewlett-Packard Basic Liquid Chromatography Semina*r, Hewlett-Packard (Naperville, IL)
1990	*Environmental Toxicology*, Fall Semester, Sangamon State University (Springfield, IL)
1990	*Toxi-Lab Workshop*, Toxi-Lab Inc. (Springfield, IL)

PROFESSIONAL AFFILIATIONS

1991 - present	Midwestern Association of Forensic Scientists, member
1994 - present	Mid-Atlantic Association of Forensic Scientists, member
2000 - present	Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) FBI Laboratory representative

CERTIFICATIONS

1999 – present	Laboratory Inspector American Society of Crime Laboratory Directors Laboratory Accreditation Board (ASCLD/LAB)

PRESENTATIONS

2005	*Forensic Chemistry at the FBI Laboratory*, Annual National Meeting of SIMLA, Rome, Italy.
2005	*Forensic Chemistry and Toxicology at the FBI Laboratory*, Overview of Forensic Science: State of the Art and Prospects, University of Padua, Padua, Italy.
2003	*Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) 2002 Update*, Mid-Atlantic Association of Forensic Scientists Annual Meeting, Frederick, MD.
2000	*The FBI Laboratory Chemistry Unit,* University of Illinois Careers Symposium, Champaign-Urbana, IL.
1997	*Evidence Submission Training*, Metropolitan Police Department, Washington, DC.

PUBLICATIONS

Cavett V., Waninger E., Krutak J., Eckenrode B., May 2004. Visualization and LC/MS Analysis of Colorless Pepper Sprays. J. Forensic Sci. Vol. 49, No. 3.

Ex 446
(590)

Madeline A. Montgomery

Federal Bureau of Investigation
Chemistry Unit, Room 4220
2501 Investigation Parkway
Quantico, VA 22135
(703) 632-7425

PROFESSIONAL EXPERIENCE

Jan 2001 - present **Forensic Chemist Examiner**
FBI Laboratory
Federal Bureau of Investigation
Washington, DC
Quantico, VA

Duties include examination of evidence submitted to the FBI Chemistry Unit in criminal investigations in the area of toxicology and chemical identification. Responsible for inventorying evidence, using chemical extraction principles to isolate drugs and poisons from biological samples and food matrices, analyzing prepared extracts using analytical instrumentation, interpreting results of analyses, and providing interpretations of results in the form of written reports and oral testimony.

May 1996 – Jan 2001 **Forensic Chemist**
FBI Laboratory
Federal Bureau of Investigation
Washington, DC

Duties included examination of evidence submitted to the FBI Chemistry Unit in criminal investigations. Areas of examination included toxicology, drug and drug residue cases, analysis of bank security dye residues, and identification of unknown chemicals.

EDUCATION

1998-2000 **George Washington University**
Washington, DC
Graduate Coursework in Forensic Science and Forensic Toxicology

1992-1996 **George Washington University**
Washington, DC
Bachelor of Science Degree in Chemistry

PROFESSIONAL TRAINING

1996 *SYVA ETS Plus Training.* Behring Diagnostics (San Jose, CA)
1997 *Theories of Chromatography.* FBI Academy (Quantico, VA)
1997 *Forensic Toxicology Workshop.* Forensic Toxicology Certification Board, Inc. (Gainesville, FL)
1997 *Drug Chemist Seminar.* Drug Enforcement Administration (Fairfax, VA)
1998 *Working with your Rapid Trace.* Zymark Corporation (Hopkinton, MA)
1999 *GC-MSD Chemstation Data Analysis and Reporting.* Hewlett Packard Corporation (Atlanta, GA)
1999 *ToxiLab Training Class.* Ansys Diagnostics Inc. (Baltimore, MD)

Ex 446
(54)

1999	*Chemical Agent Awareness.* Edgewood Chemical and Biological Forensic Analytical Center (Edgewood, MD)
1999	*Excalibur Training Course.* Thermoquest Institute (Chicago, IL)
1999	*HP Macro Programming.* Hewlett Packard Corporation (Atlanta, GA)
1999	*Pathology for Toxicologists.* Annual Society of Forensic Toxicologists Meeting (San Juan, PR)
1999	*Pharmacology Reviews: Selected New Drugs.* Annual Society of Forensic Toxicologists Meeting (San Juan, PR)
1999	*Investigation of Drug Facilitated Sexual Battery.* Annual Society of Forensic Toxicologists Meeting (San Juan, PR)
1999	*Inficon Hapsite Training.* Inficon Corporation (Quantico, VA)
2000	*Capillary Electrophoresis.* Annual American Academy of Forensic Sciences Meeting (Reno, NV)
2000	*Forensic Applications of LC/MS.* Annual Society of Forensic Toxicologists Meeting (Milwaukee, WI)
2000	*Benzodiazepines: Pharmacology and Analytical Challenges.* Annual Society of Forensic Toxicologists Meeting (Milwaukee, WI)
2000	*Forensic Toxicological Aspects of Chemical Terrorism.* Annual Society of Forensic Toxicologists Meeting (Milwaukee, WI)
2001	*The Agony of Ecstasy: Clinical, Pathological, and Toxicological Aspects of MDMA.* Annual Society of Forensic Toxicologists Meeting (Seattle, WA)
2001	*Evidence Response Team Training.* FBI (Quantico, VA)
2001	*LC/MSD Techniques and Software Operation.* Agilent Technologies (Wilmington, DE)
2001	*Hazardous Materials Operations.* Hazardous Materials Response Unit, FBI (Leesburg, VA)
2003	*Chemical Agents of Opportunity for Terrorism: Preparedness and Response.* American College of Medical Technology (Atlanta, GA)
2003	*Toxicology in the Emergency Room.* Annual Society of Forensic Toxicologists Meeting (Portland, OR)
2005	*SWGFACT Ricin Workshop.* Centers for Disease Control and Prevention (Atlanta, GA)
2005	*ISO/IEC 17025:2005 Training.* ASCLD LAB (Quantico, VA)
2005	*ASCLD/LAB International Assessor Training.* ASCLD LAB (Quantico, VA)
2006	*Analysis of Organophosphate Nerve Agent Metabolites in Urine by LC/MS/MS.* Centers for Disease Control and Prevention (Atlanta, GA)
2006	*Analysis of Ricinine by LC/MS/MS.* Centers for Disease Control and Prevention (Atlanta, GA)
2006	*Analysis of Nitrogen Mustard Metabolites by LC/MS/MS.* Centers for Disease Control and Prevention (Atlanta, GA)

PRESENTATIONS

2000	*Differentiation of Psilocyn and Bufotenine by GC/MS(CI).* Annual American Academy of Forensic Sciences Meeting (Reno, NV)
2000	*Analysis of Biological Specimens for Opiates by Solvent Extraction and Derivatization with d6-Acetic Anhydride: Application to a Case of Morphine Poisoning.* Annual Society of Forensic Toxicologists Meeting (Milwaukee, WI)
2000	*Analysis of Biofluids for Flunitrazepam and Metabolites by Electrospray LC-MS.* Annual Eastern Analytical Symposium (Atlantic City, NJ)
2000	*Analysis of Biological Specimens for Gamma-Hydroxybutyrate Using Headspace GC-FID and GC-MS(EI) Techniques.* Annual Eastern Analytical Symposium (Atlantic City, NJ)
2001	*Benzodiazepines and Drug-Facilitated Sexual Assault.* Annual American Academy of Forensic Sciences Meeting (Seattle, WA)
2001	*Temazepam Analysis in Mother and Child.* Annual Society of Forensic Toxicologists Meeting (New Orleans, LA)
2001	*Drug-Facilitated Sexual Assault.* International Conference on Sexual Assault and Harassment on Campus (Kissimmee, FL)
2002	*Drug-Facilitated Sexual Assault.* Howard County Family and Sexual Violence Coordinating Council (Ellicott City, MD)
2002	*Drug-Facilitated Sexual Assault.* King County District Attorney's Office (Brooklyn, NY)

2002	*Drug-Facilitated Sexual Assault.* Northeast Colleges and Universities Security Association (Saratoga Springs, NY)
2002	*Drug-Facilitated Sexual Assault.* American Prosecutor's Research Institute (Salt Lake City, UT)
2002	*Yohimbine and 11-OH-Yohimbine Analysis by LC-MS and LC-MS-MS.* The International Association of Toxicologists Annual Meeting (Paris, France)
2002	*Drug-Facilitated Sexual Assault.* American Prosecutor's Research Institute (Knoxville, TN)
2003	*Drug-Facilitated Sexual Assault.* American Prosecutor's Research Institute (Plainfield, IN)
2003	*Post-Mortem Detection of Doxacurium And Its Breakdown Products By Liquid Chromatography Tandem Mass Spectrometry.* The International Association of Toxicologists Annual Meeting (Melbourne, Australia)
2005	*Drug-Facilitated Sexual Assault – The Drugs and the Challenges.* Nevada Coalition Against Sexual Assault (Reno, Nevada)
2005	*Drug-Facilitated Sexual Assault and Toxicology.* Department of Defense Sexual Assault Team Training (Bethesda, MD)
2005	*Drug-Facilitated Sexual Assault: Challenges and Solutions.* The National Center for Victims of Crime Annual Conference (Washington, DC)
2005	*Mivacurium and Metabolites from Biological Samples.* Annual Society of Forensic Toxicologists Meeting (Nashville, TN)
2005	*Advanced Topics in Drug-Facilitated Sexual Assault.* The Eighth Annual Sex Crimes Information Sharing Conference (Egg Harbor, NJ).
2005	*Chromatography Applications for Forensic Toxicology.* FBI Academy Chromatography School (Quantico, VA).
2006	*Preventing Drug-Facilitated Sexual Assault.* New York State Coalition Against Sexual Assault Conference (Buffalo, NY).
2006	*Drug-Facilitated Sexual Assault.* Colorado Association of Sex Crimes Investigators (Aspen, CO).
2006	*Drug-Facilitated Sexual Assault: When The Toxicology Report is the Smoking Gun.* Chemical Society of Washington (Washington, DC)
2007	*Finding the Needle in the Haystack: Improving the Toxicological Investigation of Drug-Facilitated Crimes – Benzodiazepines.* Annual American Academy of Forensic Sciences Meeting (San Antonio, TX)

PROFESSIONAL AFFILIATIONS

1999-present	Society of Forensic Toxicologists - Full Member
2000-present	Society of Forensic Toxicologists Drug-Facilitated Sexual Assault Committee Member
2000-2002	American Academy of Forensic Sciences - Student Member
2002-2006	Scientific Working Group on the Forensic Analysis of Chemical Terrorism Member
2004-present	The International Association of Forensic Toxicologists - Member
2006-present	Qualified ASCLD/LAB-*International* Assessor

PUBLICATIONS

Gillette, R., Doyle, J.M., Miller, M.L., Montgomery, M.A., and Mushrush, G.W. Capillary Electrophoresis Screening of Poisonous Anions Extracted from Biological Samples. *J. Chrom B.* **831**: 190-195 (2006).

LeBeau, M.A., Montgomery, M.A., Wagner, J.R., and Miller, M.L.. Analysis of Biofluids for Flunitrazepam and Metabolites by Electrospray Liquid Chromatography/Mass Spectrometry. *J. Forensic Sci.* **45**: 1133-1141 (2000).

LeBeau, M.A., Montgomery, M.A., Miller, M.L., and Burmeister, S.G. Analysis of Biofluids for Gamma-Hydroxybutyrate (GHB) and Gamma-Butyrolactone (GBL) by Headspace GC/FID and GC/MS. *J. Anal. Toxicol.* **24**: 421-428 (2000).

LeBeau, M.A., Montgomery, M.A., Jufer, R.A., and Miller, M.L.. Elevated GHB in Citrate Buffered Blood. *J. Anal. Toxicol.* **24**: 383-384 (2000).

Ex 446

LeBeau, M.A., Montgomery, M.A., Morris-Kukoski, C., Schaff, J.E., Deakin, A., and Levine, B. A Comprehensive Study on the Variations in Urinary Concentrations of Endogenous gamma-hydroxybutyrate (GHB). *J. Anal. Toxicol.* **30**: 98-105 (2006).

Montgomery, M.A., LeBeau, M.A., and Jenkins, A.J. Qualitative Identification of Doxacurium and Its Breakdown Products in Postmortem Fluids by Liquid Chromatography-Tandem Mass Spectrometry. *J. Anal. Toxicol.* **30**: 57-60 (2006).

Montgomery, M.A., LeBeau, M.A., Miller, M.L., and Jufer, R.A. The Identification of Mivacurium and Metabolites in Biological Samples. *J. Anal. Toxicol.* **29**: 637-642 (2005).

PROFICIENCY / COMPETENCY TESTS

Ex 446
(595)

competency test

Date: 2/15/2007

To: LEBEAU, MARC A. (LD) (FBI)

From: Jay A. Clark

RE: Competency Test

Items for EDTA/Blood Competency test are being submitted to you in five (5) separate white envelopes, each marked with a unique identifier, as follows:

JC1

JC4

JC7

JC9

JC10

Each envelope contains one (1) swab with a dried blood stain. Please determine if the stain is from EDTA preserved blood or non-preserved blood.

Issued By: Jay A. Clark

Date: 2-15-07

Received By: [signature]

Date: 2-15-07

Ex 446
(596)

Memo

Date: 2/16/2007

To: Jay Clark, Cynthia Morris-Kukoski

From: Marc LeBeau

RE: Competency Test Results

The results of my competency test for the EDTA in Bloodstains SOP are as follows:

JC1: Negative for EDTA

JC4: Positive for EDTA

JC7: Negative for EDTA

JC9: Positive for EDTA

JC10: Negative for EDTA

Ex 446
(597)

Memo

To: Marc A. LeBeau
From: Cynthia L. Morris-Kukoski
Date: February 26, 2007
Subject: Competency EDTA

Dr. LeBeau successfully completed his competency test for the EDTA in Bloodstains SOP. He 100% correctly identified which blood samples contained EDTA and which ones did not.

Ex 446
(598)

competency test

Date: 2/15/2007

To: MONTGOMERY, MADELINE A. (LD) (FBI)

From: Jay A. Clark

RE: Competency Test

Items for EDTA/Blood Competency test are being submitted to you in five (5) separate white envelopes, each marked with a unique identifier, as follows:

JC2

JC3

JC5

JC6

JC8

Each envelope contains one (1) swab with a dried blood stain. Please determine if the stain is from EDTA preserved blood or non-preserved blood.

Issued By: Jay A. Clark

Date: 2-15-07

Received By: Madeline A. Montgomery

Date: 2-15-07

Ex 446
(599)

Memo

To: M. LeBeau
From: M. Montgomery MM
Date: 2/16/07
Subject: EDTA Competency Test Conclusions

I have drawn the following results after reviewing the data acquired by J. Brewer on 2/15/07:

Sample JC2: Positive for EDTA
Sample JC3: Positive for EDTA
Sample JC5: Negative for EDTA
Sample JC6: Positive for EDTA
Sample JC8: Negative for EDTA

Ex 446
(600)

Memo

To: Madeline A. Montgomery, Marc A. LeBeau
From: Cynthia L. Morris-Kukoski
Date: February 26, 2007
Subject: Competency EDTA

Madeline Montgomery successfully completed her competency test for the EDTA in Bloodstains SOP. She 100% correctly identified which blood samples contained EDTA and which ones did not.

Ex 446
(601)

competency test

Date: 2/20/2007

To: LEBEAU, MARC A. (LD) (FBI)

From: Jay A. Clark JAC

RE: Competency Test

Congratulations, you have successfully completed your competency exam for the Analysis of EDTA in Blood Stains.

Please see the attached Key for the correct answers, and the procedure for the preparation of the test samples.

Ex 446
(602)

competency test key

Date: 2/15/2007

RE: Competency Test Key

Competency Tests for Analysis of EDTA in Blood Stains results are as follows

JC1 – non-preserved blood

JC2 – EDTA preserved blood

JC3 - EDTA preserved blood

JC4 - EDTA preserved blood

JC5 – non-preserved blood

JC6 - EDTA preserved blood

JC7 – non-preserved blood

JC8 – non-preserved blood

JC9 - EDTA preserved blood

JC10 – non-preserved blood

Prepared By: Jay A. Clark

Date: 2-15-07

Ex 446
(603)

Preparation of Competency Tests for "Analysis of EDTA in Blood Stains" SOP:

1. Select a sterile cotton-tip swab and label it with a unique identifier.

2. Place 10 µL of blood (EDTA-preserved or non-EDTA-preserved) onto the cotton tip swab. Document if the swab was prepared with blood containing EDTA.

3. Repeat with appropriate number of swabs.

4. Allow all swabs to dry overnight. Blindly distribute to test participants.

Ex 446
(604)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for JASON D BREWER 0731-0000

Current Staff Qualification 00003847

Discipline	Sub Discipline	Process
Controlled Substance	Drug Analysis	Sample Prep.,Wet Chem. & Inst. Analysis

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
8/4/2005 12:00:00AM		9/23/2007 11:59:58PM	000000000000223

Satisfactory Associated Proficiency Test 0000016253

Identifier 05-15C
Staff Position
Test Source External
Source Unit

Supplier Collaborative Testing Services
Preparer Name
Verifier Name
Validator Name

Other Comments CTS 05-502 satisfactory completion

Distribution Date	8/11/2005 12:00:00AM	LT-PTPM Notification of Test	9/8/2005 12:00:00AM
Due Date	9/23/2005 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	9/8/2005 12:00:00AM	UC Designee Final Eval	
Testee Feedback	11/1/2005 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000016950

'entifier 06-17D
.aff Position
Test Source External
Source Unit

Supplier Collaborative Testing Services
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date	8/10/2006 12:00:00AM	LT-PTPM Notification of Test	9/12/2006 12:00:00AM
Due Date	9/22/2006 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	9/12/2006 12:00:00AM	UC Designee Final Eval	
Testee Feedback	12/5/2006 12:00:00AM	LD-PTPM Eval	

Ex 446
(605)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

\TU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for JASON D BREWER 0731-0000

Current Staff Qualification 00003833

Discipline	Sub Discipline	Process
Trace Analysis (Chem.)	Bank Dyes	Sample Prep.,Wet Chem. & Inst. Analysis

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
5/10/2005 12:00:00AM		7/1/2007 11:59:58PM	000000000000260

Satisfactory Associated Proficiency Test 0000016251

Identifier	05-16C	Supplier	
Staff Position		Preparer Name	Deborah Wang
Test Source	Internal	Verifier Name	Robert Mothershead
Source Unit	Chemistry	Validator Name	Deborah Wang

Other Comments Test set #4- satisfactory completion

Distribution Date	10/27/2005 12:00:00AM	LT-PTPM Notification of Test	12/5/2005 12:00:00AM
Due Date	12/12/2005 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	12/5/2005 12:00:00AM	UC Designee Final Eval	
Testee Feedback	12/6/2005 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000016529

ntifier	06-25C	Supplier	
ff Position		Preparer Name	Pamela Reynolds
Test Source	Internal	Verifier Name	Eileen Waninger
Source Unit	Chemistry Unit	Validator Name	Jason Brewer

Other Comments

Distribution Date	5/18/2006 12:00:00AM	LT-PTPM Notification of Test	5/31/2006 12:00:00AM
Due Date	6/30/2006 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	5/31/2006 12:00:00AM	UC Designee Final Eval	
Testee Feedback	6/16/2006 12:00:00AM	LD-PTPM Eval	

Ex 446
(606)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MARC A LEBEAU 0731-0000

Current Staff Qualification 00003859

Discipline	Sub Discipline	Process
Toxicology	Quantitative - Drug Analysis	Interpretation of Results

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/1999 12:00:00AM		6/29/2007 12:00:00AM	000000000000739

Satisfactory Associated Proficiency Test 0000016277

Identifier	05-5B	Supplier	College of American Pathologists
Staff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments FTC-A- satisfactoy completion

Distribution Date	3/17/2005 12:00:00AM	LT-PTPM Notification of Test	5/5/2005 12:00:00AM
Due Date	5/5/2005 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	5/5/2005 12:00:00AM	UC Designee Final Eval	
Testee Feedback	8/5/2005 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000016524

ntifier	06-3B	Supplier	College of American Pathologists
taff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	3/16/2006 12:00:00AM	LT-PTPM Notification of Test	5/3/2006 12:00:00AM
Due Date	5/8/2006 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	5/3/2006 12:00:00AM	UC Designee Final Eval	
Testee Feedback	6/20/2006 12:00:00AM	LD-PTPM Eval	

Ex 446
(607)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MARC A LEBEAU 0731-0000

Expired Staff Qualification 00001122

Discipline	Sub Discipline	Process	
Toxicology	Quantitative - Drug Analysis	Complete Examination & Analysis	
Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/1999 12:00:00AM	4/30/2002 11:59:59PM	11/5/2004 11:59:59PM	000000000000276

Satisfactory Associated Proficiency Test 0000007619

Identifier	99-18A	Supplier	CAP
Staff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	11/1/1999 12:00:00AM	LT-PTPM Notification of Test	5/1/2000 12:00:00AM
Due Date	12/16/1999 12:00:00AM	LD-PTPM Review Notified	
PT Complete Date	12/10/1999 12:00:00AM	UC Designee Final Eval	
Testee Feedback	5/3/2000 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000008062

ntifier	00-17	Supplier	College of American Pathologists
ff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	11/16/2000 12:00:00AM	LT-PTPM Notification of Test	5/2/2001 12:00:00AM
Due Date	12/21/2000 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	12/26/2000 12:00:00AM	UC Designee Final Eval	
Testee Feedback	5/7/2001 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000007835

Identifier	01-15	Supplier	CAP
Staff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	8/13/2001 12:00:00AM	LT-PTPM Notification of Test	12/31/2001 12:00:00AM
Due Date	9/25/2001 12:00:00AM	LD-PTPM Review Notified	12/31/2001 12:00:00AM
PT Complete Date	9/25/2001 12:00:00AM	UC Designee Final Eval	12/31/2001 12:00:00AM
Testee Feedback	12/31/2001 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000008029

Identifier	02-2A	Supplier	College of American Pathologists
Staff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	
Test Source	External	Verifier Name	
rce Unit		Validator Name	

ner Comments

Ex 446
(608)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MARC A LEBEAU 0731-0000

Distribution Date	3/18/2002 12:00:00AM	LT-PTPM Notification of Test	7/16/2002 12:00:00AM
Due Date	4/30/2002 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	4/30/2002 12:00:00AM	UC Designee Final Eval	
Testee Feedback	7/16/2002 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000009115

Identifier	03-27A	Supplier	College of American Pathologists
Staff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments See attached memos

Distribution Date	10/27/2003 12:00:00AM	LT-PTPM Notification of Test	1/15/2004 12:00:00AM
Due Date	11/7/2003 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	12/9/2003 12:00:00AM	UC Designee Final Eval	
Testee Feedback	2/10/2004 12:00:00AM	LD-PTPM Eval	

Ex 446
(609)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
TU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MARC A LEBEAU 0731-0000

Current Staff Qualification 00003858

Discipline	Sub Discipline	Process
Toxicology	Qualitative - Drug Analysis	Interpretation of Results

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/1999 12:00:00AM		5/9/2007 11:59:58PM	000000000000738

Satisfactory Associated Proficiency Test 0000016276

Identifier	05-5B	Supplier	College of American Pathologists
Staff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments FTC-A- satisfactory completion

Distribution Date	3/17/2005 12:00:00AM	LT-PTPM Notification of Test	5/5/2005 12:00:00AM
Due Date	5/5/2005 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	5/5/2005 12:00:00AM	UC Designee Final Eval	
Testee Feedback	8/5/2005 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000016522

ntifier	06-3B	Supplier	College of American Pathologists
aff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	3/16/2006 12:00:00AM	LT-PTPM Notification of Test	5/3/2006 12:00:00AM
Due Date	5/8/2006 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	5/3/2006 12:00:00AM	UC Designee Final Eval	
Testee Feedback	6/20/2006 12:00:00AM	LD-PTPM Eval	

Ex 446
(610)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MARC A LEBEAU 0731-0000

Expired Staff Qualification 00001116

Discipline	Sub Discipline	Process
Toxicology	Qualitative - Drug Analysis	Complete Examination & Analysis

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/1999 12:00:00AM	4/30/2002 11:59:59PM	11/5/2004 11:59:59PM	000000000000274

Satisfactory Associated Proficiency Test 0000007602

Identifier	99-4	Supplier	CAP
Staff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	3/17/1999 12:00:00AM	LT-PTPM Notification of Test	3/17/1999 12:00:00AM
Due Date	3/29/1999 12:00:00AM	LD-PTPM Review Notified	7/13/1999 12:00:00AM
PT Complete Date	7/2/1999 12:00:00AM	UC Designee Final Eval	8/10/1999 12:00:00AM
Testee Feedback	8/4/1999 12:00:00AM	LD-PTPM Eval	3/15/2000 12:00:00AM

Satisfactory Associated Proficiency Test 0000007719

entifier	00-6	Supplier	CAP
aff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	6/7/2000 12:00:00AM	LT-PTPM Notification of Test	8/30/2000 12:00:00AM
Due Date	6/19/2000 12:00:00AM	LD-PTPM Review Notified	8/30/2000 12:00:00AM
PT Complete Date	6/19/2000 12:00:00AM	UC Designee Final Eval	8/29/2000 12:00:00AM
Testee Feedback	8/30/2000 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000007834

Identifier	01-15	Supplier	CAP
Staff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	8/13/2001 12:00:00AM	LT-PTPM Notification of Test	12/31/2001 12:00:00AM
Due Date	9/25/2001 12:00:00AM	LD-PTPM Review Notified	12/31/2001 12:00:00AM
PT Complete Date	9/25/2001 12:00:00AM	UC Designee Final Eval	12/31/2001 12:00:00AM
Testee Feedback	12/31/2001 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000008028

Identifier	02-2A	Supplier	College of American Pathologists
Staff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	
Test Source	External	Verifier Name	
urce Unit		Validator Name	

ther Comments

Ex 446
(611)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MARC A LEBEAU 0731-0000

Distribution Date	3/18/2002 12:00:00AM	LT-PTPM Notification of Test	7/16/2002 12:00:00AM
Due Date	4/30/2002 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	4/30/2002 12:00:00AM	UC Designee Final Eval	
Testee Feedback	7/16/2002 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000009114

Identifier	03-27A	Supplier	College of American Pathologists
Staff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments See attached memos

Distribution Date	10/27/2003 12:00:00AM	LT-PTPM Notification of Test	1/15/2004 12:00:00AM
Due Date	11/7/2003 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	12/9/2003 12:00:00AM	UC Designee Final Eval	
Testee Feedback	2/10/2004 12:00:00AM	LD-PTPM Eval	

Ex 446
(612)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MARC A LEBEAU 0731-0000

Current Staff Qualification 00003836

Discipline	Sub Discipline	Process
Toxicology	Blood Alcohol	Interpretation of Results

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/2004 12:00:00AM		9/2/2007 11:59:58PM	000000000000736

Satisfactory Associated Proficiency Test 0000016250

Identifier	05-27A	Supplier	College of American Pathologists
Staff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments CAP AL1-C- satisfactory completion

Distribution Date	10/5/2005 12:00:00AM	LT-PTPM Notification of Test	10/14/2005 12:00:00AM
Due Date	10/20/2005 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	10/14/2005 12:00:00AM	UC Designee Final Eval	
Testee Feedback	12/12/2005 12:00:00AM	LD-PTPM Eval	

Discontinued Associated Proficiency Test 0000016532

·entifier	06-14A	Supplier	College of American Pathologists
·ff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	6/19/2006 12:00:00AM	LT-PTPM Notification of Test	
Due Date	6/30/2006 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date		UC Designee Final Eval	
Testee Feedback		LD-PTPM Eval	

In Progress - Pending Results Associated Proficiency Test 0000016948

Identifier	06-13A	Supplier	Quality Forenics
Staff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	7/21/2006 12:00:00AM	LT-PTPM Notification of Test	7/31/2006 12:00:00AM
Due Date	9/1/2006 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	7/21/2006 12:00:00AM	UC Designee Final Eval	
Testee Feedback		LD-PTPM Eval	

Ex 446
(613)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MARC A LEBEAU 0731-0000

Expired Staff Qualification 00001112

Discipline	Sub Discipline	Process
Toxicology	Blood Alcohol	Complete Examination & Analysis

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/1999 12:00:00AM	10/20/2004 12:00:00AM	10/20/2004 11:59:59PM	000000000000272

Satisfactory Associated Proficiency Test 0000007600

Identifier 99-7
Staff Position SUPERVISORY CHEMIST-UNIT CHIEF
Test Source External
Source Unit Chemistry Unit

Supplier CAP
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date	6/15/1999 12:00:00AM	LT-PTPM Notification of Test	6/15/1999 12:00:00AM
Due Date	6/25/1999 12:00:00AM	LD-PTPM Review Notified	6/24/1999 12:00:00AM
PT Complete Date	6/22/1999 12:00:00AM	UC Designee Final Eval	8/10/1999 12:00:00AM
Testee Feedback	8/4/1999 12:00:00AM	LD-PTPM Eval	3/15/2000 12:00:00AM

Satisfactory Associated Proficiency Test 0000008061

ntifier 00-12
ff Position SUPERVISORY CHEMIST-UNIT CHIEF
Test Source External
Source Unit

Supplier College of American Pathologists
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date	9/19/2000 12:00:00AM	LT-PTPM Notification of Test	12/4/2000 12:00:00AM
Due Date	9/29/2000 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	9/29/2000 12:00:00AM	UC Designee Final Eval	
Testee Feedback	12/18/2000 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000007836

Identifier 01-16A
Staff Position SUPERVISORY CHEMIST-UNIT CHIEF
Test Source External
Source Unit Chemistry Unit

Supplier CAP
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date	9/18/2001 12:00:00AM	LT-PTPM Notification of Test	2/18/2002 12:00:00AM
Due Date	9/27/2001 12:00:00AM	LD-PTPM Review Notified	2/18/2002 12:00:00AM
PT Complete Date	9/24/2001 12:00:00AM	UC Designee Final Eval	2/6/2002 12:00:00AM
Testee Feedback	2/18/2002 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000008439

Identifier 02-18B
Staff Position SUPERVISORY CHEMIST-UNIT CHIEF
Test Source External
rce Unit

Supplier College of American Pathologists
Preparer Name
Verifier Name
Validator Name

ther Comments

Ex 446
(614)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MARC A LEBEAU 0731-0000

Distribution Date	10/18/2002 12:00:00PM	LT-PTPM Notification of Test	10/25/2002 11:00:00AM
Due Date	10/25/2002 11:59:58PM	LD-PTPM Review Notified	
PT Complete Date	10/25/2002 11:00:00AM	UC Designee Final Eval	
Testee Feedback	11/26/2002 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000009113

Identifier	03-25A	Supplier	College of American Pathologists
Staff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	10/15/2003 12:00:00AM	LT-PTPM Notification of Test	10/21/2003 12:00:00AM
Due Date	10/28/2003 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	10/21/2003 12:00:00AM	UC Designee Final Eval	
Testee Feedback	11/24/2003 12:00:00AM	LD-PTPM Eval	

Ex 446
(615)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MARC A LEBEAU 0731-0000

Expired Staff Qualification 00000795

Discipline	Sub Discipline	Process	
Controlled Substance	Drug Analysis	Complete Examination & Analysis	
Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/1999 12:00:00AM			000000000000224

Satisfactory Associated Proficiency Test 0000007589

Identifier	99-5A	Supplier	CTS
Staff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	5/18/1999 12:00:00AM	LT-PTPM Notification of Test	7/13/1999 12:00:00AM
Due Date	7/8/1999 12:00:00AM	LD-PTPM Review Notified	7/13/1999 12:00:00AM
PT Complete Date	7/2/1999 12:00:00AM	UC Designee Final Eval	8/10/1999 12:00:00AM
Testee Feedback	8/4/1999 12:00:00AM	LD-PTPM Eval	3/15/2000 12:00:00AM

Satisfactory Associated Proficiency Test 0000007712

'entifier	00-3B	Supplier	CTS
.aff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	5/3/2000 12:00:00AM	LT-PTPM Notification of Test	8/7/2000 12:00:00AM
Due Date	6/28/2000 12:00:00AM	LD-PTPM Review Notified	8/7/2000 12:00:00AM
PT Complete Date	6/13/2000 12:00:00AM	UC Designee Final Eval	7/28/2000 12:00:00AM
Testee Feedback	8/7/2000 12:00:00AM	LD-PTPM Eval	

Ex 446
(616)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MARC A LEBEAU 0731-0000

Expired Staff Qualification 00001090

Discipline	Sub Discipline	Process	
Trace Analysis (Chem.)	Bank Dyes	Complete Examination & Analysis	
Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/1999 12:00:00AM		12/6/2002 11:59:59PM	000000000000261

Satisfactory Associated Proficiency Test 0000006809

Identifier	99-3B	Supplier	
Staff Position	SUPERVISORY CHEMIST-UNIT CHIEF	Preparer Name	Waninger, Eileen Marie
Test Source	Internal	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	3/16/1999 12:00:00AM	LT-PTPM Notification of Test	5/17/1999 12:00:00AM
Due Date	4/27/1999 12:00:00AM	LD-PTPM Review Notified	5/17/1999 12:00:00AM
PT Complete Date	4/19/1999 12:00:00AM	UC Designee Final Eval	5/4/1999 12:00:00AM
Testee Feedback	4/27/1999 12:00:00AM	LD-PTPM Eval	3/15/2000 12:00:00AM

Ex 446
(617)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Expired Staff Qualification 00001118

Discipline	Sub Discipline	Process
Toxicology	Quantitative - Drug Analysis	Sample Prep.,Wet Chem. & Inst. Analysis

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/1999 12:00:00AM	12/16/1999 12:00:00AM	5/25/2002 11:59:58PM	000000000000275

Satisfactory Associated Proficiency Test 0000007617

Identifier	99-18A	Supplier	CAP
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	11/1/1999 12:00:00AM	LT-PTPM Notification of Test	5/1/2000 12:00:00AM
Due Date	12/16/1999 12:00:00AM	LD-PTPM Review Notified	
PT Complete Date	12/10/1999 12:00:00AM	UC Designee Final Eval	
Testee Feedback	5/2/2000 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000008071

ntifier	00-17	Supplier	College of American Pathologists
ff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	11/6/2000 12:00:00AM	LT-PTPM Notification of Test	5/7/2001 12:00:00AM
Due Date	12/21/2000 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	12/26/2000 12:00:00AM	UC Designee Final Eval	
Testee Feedback	5/14/2001 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000007778

Identifier	01-3	Supplier	CAP
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	3/16/2001 12:00:00AM	LT-PTPM Notification of Test	6/5/2001 12:00:00AM
Due Date	4/26/2001 12:00:00AM	LD-PTPM Review Notified	6/5/2001 12:00:00AM
PT Complete Date	4/20/2001 12:00:00AM	UC Designee Final Eval	6/4/2001 12:00:00AM
Testee Feedback	6/4/2001 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000008042

Identifier	02-4	Supplier	College of American Pathologists
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
rce Unit		Validator Name	

Other Comments

Ex 446
(618)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Distribution Date	3/25/2002 12:00:00AM	LT-PTPM Notification of Test	5/2/2002 12:00:00AM
Due Date	5/24/2002 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	5/2/2002 12:00:00AM	UC Designee Final Eval	
Testee Feedback	11/4/2002 12:00:00AM	LD-PTPM Eval	

Ex 446
(619)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Current Staff Qualification 00001284

Discipline	Sub Discipline	Process	
Toxicology	Quantitative - Drug Analysis	Complete Examination & Analysis	
Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
7/1/2002 12:00:00AM	5/14/2003 11:59:59PM	12/17/2007 11:59:58PM	000000000000276

Satisfactory Associated Proficiency Test 0000008822

Identifier 02-12A
Staff Position CHEMIST-FORENSIC EXAMINER
Test Source External
Source Unit

Supplier College of American Pathologists
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date	7/2/2002 12:00:00AM	LT-PTPM Notification of Test	8/16/2002 12:00:00AM
Due Date	7/15/2002 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	7/9/2002 12:00:00AM	UC Designee Final Eval	
Testee Feedback	11/15/2002 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000009121

ntifier 03-4A
taff Position CHEMIST-FORENSIC EXAMINER
Test Source External
Source Unit

Supplier College of American Pathologists
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date	3/26/2003 12:00:00AM	LT-PTPM Notification of Test	4/25/2003 12:00:00AM
Due Date	5/14/2003 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	4/25/2003 12:00:00AM	UC Designee Final Eval	
Testee Feedback	7/29/2003 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000015820

Identifier 04-20A
Staff Position CHEMIST-FORENSIC EXAMINER
Test Source External
Source Unit

Supplier Quality Forenics
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date	10/26/2004 12:00:00AM	LT-PTPM Notification of Test	1/18/2005 12:00:00AM
Due Date	12/16/2004 12:00:00AM	LD-PTPM Review Notified	
PT Complete Date	11/9/2004 12:00:00AM	UC Designee Final Eval	
Testee Feedback		LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000016639

Identifier 05-23B
Staff Position
Test Source External
rce Unit

Supplier Quality Forenics
Preparer Name
Verifier Name
Validator Name

Other Comments

Ex 446
(620)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Distribution Date	11/7/2005 12:00:00AM	LT-PTPM Notification of Test	12/14/2005 12:00:00AM
Due Date	12/16/2005 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	12/14/2005 12:00:00AM	UC Designee Final Eval	
Testee Feedback	12/14/2006 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000016528

Identifier	06-3A	Supplier	College of American Pathologists
Staff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	3/22/2006 12:00:00AM	LT-PTPM Notification of Test	4/13/2006 12:00:00AM
Due Date	5/8/2006 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	4/13/2006 12:00:00AM	UC Designee Final Eval	
Testee Feedback	6/16/2006 12:00:00AM	LD-PTPM Eval	

Ex 446
(621)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Current Staff Qualification 00001283

Discipline	Sub Discipline	Process	
Toxicology	Qualitative - Drug Analysis	Complete Examination & Analysis	
Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
7/1/2002 12:00:00AM	5/14/2003 11:59:59PM	12/15/2007 11:59:58PM	000000000000274

Satisfactory Associated Proficiency Test 0000008821

Identifier	02-12A	Supplier	College of American Pathologists
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	7/2/2002 12:00:00AM	LT-PTPM Notification of Test	8/16/2002 12:00:00AM
Due Date	7/15/2002 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	7/9/2002 12:00:00AM	UC Designee Final Eval	
Testee Feedback	11/15/2002 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000009120

ntifier	03-4A	Supplier	Collaborative Testing Services
aff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	3/26/2003 12:00:00AM	LT-PTPM Notification of Test	4/25/2003 12:00:00AM
Due Date	5/14/2003 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	4/25/2003 12:00:00AM	UC Designee Final Eval	
Testee Feedback	7/29/2003 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000015819

Identifier	04-20A	Supplier	Quality Forenics
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	10/26/2004 12:00:00AM	LT-PTPM Notification of Test	1/18/2005 12:00:00AM
Due Date	12/16/2004 12:00:00AM	LD-PTPM Review Notified	
PT Complete Date	11/9/2004 12:00:00AM	UC Designee Final Eval	
Testee Feedback	1/18/2005 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000016638

Identifier	05-23B	Supplier	Quality Forenics
Staff Position		Preparer Name	
Test Source	External	Verifier Name	
urce Unit		Validator Name	

Other Comments

Ex 446
(622)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Distribution Date	11/7/2005 12:00:00AM	LT-PTPM Notification of Test	12/14/2005 12:00:00AM
Due Date	12/14/2005 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	12/14/2005 12:00:00AM	UC Designee Final Eval	
Testee Feedback	12/14/2006 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000016527

Identifier	06-3A	Supplier	College of American Pathologists
Staff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	3/22/2006 12:00:00AM	LT-PTPM Notification of Test	4/13/2006 12:00:00AM
Due Date	5/8/2006 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	4/13/2006 12:00:00AM	UC Designee Final Eval	
Testee Feedback	6/16/2006 12:00:00AM	LD-PTPM Eval	

Ex 446
(623)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Expired Staff Qualification 00001113

Discipline	Sub Discipline	Process
Toxicology	Qualitative - Drug Analysis	Sample Prep.,Wet Chem. & Inst. Analysis

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/1999 12:00:00AM	5/24/2002 11:59:59PM	5/25/2002 11:59:58PM	000000000000273

Satisfactory Associated Proficiency Test 0000007603

Identifier 99-4
Staff Position CHEMIST-FORENSIC EXAMINER
Test Source External
Source Unit Chemistry Unit

Supplier CAP
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date	3/17/1999 12:00:00AM	LT-PTPM Notification of Test	3/17/1999 12:00:00AM
Due Date	3/29/1999 12:00:00AM	LD-PTPM Review Notified	7/13/1999 12:00:00AM
PT Complete Date	7/2/1999 12:00:00AM	UC Designee Final Eval	8/10/1999 12:00:00AM
Testee Feedback	8/4/1999 12:00:00AM	LD-PTPM Eval	3/15/2000 12:00:00AM

Satisfactory Associated Proficiency Test 0000007720

ntifier 00-6
Staff Position CHEMIST-FORENSIC EXAMINER
Test Source External
Source Unit Chemistry Unit

Supplier CAP
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date	6/7/2000 12:00:00AM	LT-PTPM Notification of Test	8/30/2000 12:00:00AM
Due Date	6/19/2000 12:00:00AM	LD-PTPM Review Notified	8/30/2000 12:00:00AM
PT Complete Date	6/19/2000 12:00:00AM	UC Designee Final Eval	8/29/2000 12:00:00AM
Testee Feedback	8/30/2000 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000007777

Identifier 01-3
Staff Position CHEMIST-FORENSIC EXAMINER
Test Source External
Source Unit Chemistry Unit

Supplier CAP
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date	3/16/2001 12:00:00AM	LT-PTPM Notification of Test	6/5/2001 12:00:00AM
Due Date	3/29/2001 12:00:00AM	LD-PTPM Review Notified	6/5/2001 12:00:00AM
PT Complete Date	3/28/2001 12:00:00AM	UC Designee Final Eval	6/4/2001 12:00:00AM
Testee Feedback	6/4/2001 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000008035

Identifier 02-4
Staff Position CHEMIST-FORENSIC EXAMINER
Test Source External
irce Unit

Supplier College of American Pathologists
Preparer Name
Verifier Name
Validator Name

Other Comments

Ex 446
(624)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Distribution Date	3/25/2002 12:00:00AM	LT-PTPM Notification of Test	5/2/2002 12:00:00AM
Due Date	5/24/2002 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	5/2/2002 12:00:00AM	UC Designee Final Eval	
Testee Feedback	11/4/2002 12:00:00AM	LD-PTPM Eval	

Ex 446
(625)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Current Staff Qualification 00001282

Discipline	Sub Discipline	Process	
Toxicology	Blood Alcohol	Complete Examination & Analysis	
Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
7/1/2002 12:00:00AM	7/7/2003 11:59:59PM	12/2/2007 11:59:58PM	000000000000272

Satisfactory Associated Proficiency Test 0000008294

Identifier 02-12A
Staff Position CHEMIST-FORENSIC EXAMINER
Test Source External
Source Unit

Supplier College of American Pathologists
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date 7/2/2002 12:00:00AM
Due Date 7/15/2002 11:59:59PM
PT Complete Date 7/9/2002 12:00:00AM
Testee Feedback

LT-PTPM Notification of Test 7/15/2002 12:00:00PM
LD-PTPM Review Notified
UC Designee Final Eval
LD-PTPM Eval

Satisfactory Associated Proficiency Test 0000009119

ntifier 03-9A
Staff Position CHEMIST-FORENSIC EXAMINER
Test Source External
Source Unit

Supplier College of American Pathologists
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date 6/23/2003 12:00:00AM
Due Date 7/7/2003 11:59:59PM
PT Complete Date 7/1/2003 12:00:00AM
Testee Feedback 7/29/2003 12:00:00AM

LT-PTPM Notification of Test 7/1/2003 12:00:00AM
LD-PTPM Review Notified
UC Designee Final Eval
LD-PTPM Eval

Satisfactory Associated Proficiency Test 0000013982

Identifier 04-2A
Staff Position CHEMIST-FORENSIC EXAMINER
Test Source External
Source Unit

Supplier College of American Pathologists
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date 3/9/2004 12:00:00AM
Due Date 3/23/2004 11:59:59PM
PT Complete Date 3/17/2004 12:00:00AM
Testee Feedback 4/12/2004 12:00:00AM

LT-PTPM Notification of Test 3/17/2004 12:00:00AM
LD-PTPM Review Notified
UC Designee Final Eval
LD-PTPM Eval

Satisfactory Associated Proficiency Test 0000016182

Identifier 05-3A
Staff Position
Test Source External
rce Unit

Supplier College of American Pathologists
Preparer Name
Verifier Name
Validator Name

Other Comments CAP #6451701/AL1-A: satisfactory completion

Ex 446
(626)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Distribution Date	3/8/2005 12:00:00AM	LT-PTPM Notification of Test	3/11/2005 12:00:00AM
Due Date	3/22/2005 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	3/11/2005 12:00:00AM	UC Designee Final Eval	
Testee Feedback	7/18/2005 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000016931

Identifier	06-22A	Supplier	Collaborative Testing Services
Staff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	10/10/2006 12:00:00AM	LT-PTPM Notification of Test	11/20/2006 12:00:00AM
Due Date	12/1/2006 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	11/20/2006 12:00:00AM	UC Designee Final Eval	
Testee Feedback	2/1/2007 12:00:00AM	LD-PTPM Eval	

Ex 446
(627)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Current Staff Qualification 00000799

Discipline	Sub Discipline	Process
Toxicology	Blood Alcohol	Sample Prep.,Wet Chem. & Inst. Analysis

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/1999 12:00:00AM			000000000000225

Satisfactory Associated Proficiency Test 0000007601

Identifier	99-7	Supplier	CAP
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	6/15/1999 12:00:00AM	LT-PTPM Notification of Test	6/15/1999 12:00:00AM
Due Date	6/25/1999 12:00:00AM	LD-PTPM Review Notified	6/24/1999 12:00:00AM
PT Complete Date	6/22/1999 12:00:00AM	UC Designee Final Eval	8/10/1999 12:00:00AM
Testee Feedback	8/4/1999 12:00:00AM	LD-PTPM Eval	3/15/2000 12:00:00AM

Satisfactory Associated Proficiency Test 0000007730

ntifier	00-12	Supplier	CAP
ff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	9/19/2000 12:00:00AM	LT-PTPM Notification of Test	12/18/2000 12:00:00AM
Due Date	9/29/2000 12:00:00AM	LD-PTPM Review Notified	12/18/2000 12:00:00AM
PT Complete Date	9/29/2000 12:00:00AM	UC Designee Final Eval	12/4/2000 12:00:00AM
Testee Feedback	12/18/2000 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000007785

Identifier	01-6B	Supplier	CAP
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	5/14/2001 12:00:00AM	LT-PTPM Notification of Test	7/2/2001 12:00:00AM
Due Date	5/23/2001 12:00:00AM	LD-PTPM Review Notified	7/2/2001 12:00:00AM
PT Complete Date	5/21/2001 12:00:00AM	UC Designee Final Eval	7/2/2001 12:00:00AM
Testee Feedback	7/2/2001 12:00:00AM	LD-PTPM Eval	

Ex 446
(628)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Current Staff Qualification 00001424

Discipline	Sub Discipline	Process
Controlled Substance	Drug Analysis	Complete Examination & Analysis

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
7/1/2002 12:00:00AM	9/10/2003 11:59:59PM	9/23/2007 11:59:58PM	000000000000224

Satisfactory Associated Proficiency Test 0000009122

Identifier	03-12A	Supplier	Collaborative Testing Services
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	7/30/2003 12:00:00AM	LT-PTPM Notification of Test	8/13/2003 12:00:00AM
Due Date	9/10/2003 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	8/13/2003 12:00:00AM	UC Designee Final Eval	
Testee Feedback	12/2/2003 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000014000

ntifier	04-13A	Supplier	Collaborative Testing Services
aff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	7/15/2004 12:00:00AM	LT-PTPM Notification of Test	10/26/2004 12:00:00AM
Due Date	8/26/2004 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	10/26/2004 12:00:00AM	UC Designee Final Eval	
Testee Feedback	10/26/2004 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000016205

Identifier	05-15A	Supplier	Collaborative Testing Services
Staff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments CTS 05-502- satisfactory completion

Distribution Date	8/11/2005 12:00:00AM	LT-PTPM Notification of Test	9/12/2005 12:00:00AM
Due Date	9/23/2005 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	9/12/2005 12:00:00AM	UC Designee Final Eval	
Testee Feedback	11/1/2005 12:00:00AM	LD-PTPM Eval	

Discontinued Associated Proficiency Test 0000016946

Identifier	06-3A	Supplier	Collaborative Testing Services
Staff Position		Preparer Name	
Test Source	External	Verifier Name	
irce Unit		Validator Name	

Other Comments Mistaken entry

Ex 446
(629)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Distribution Date	3/22/2006 12:00:00AM	LT-PTPM Notification of Test	
Due Date	5/8/2006 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date		UC Designee Final Eval	
Testee Feedback		LD-PTPM Eval	

Satisfactory Associated Proficiency Test: 0000016951

Identifier	06-17A	Supplier	Collaborative Testing Services
Staff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	8/10/2006 12:00:00AM	LT-PTPM Notification of Test	9/14/2006 12:00:00AM
Due Date	9/22/2006 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	9/14/2006 12:00:00AM	UC Designee Final Eval	
Testee Feedback	12/4/2006 12:00:00AM	LD-PTPM Eval	

Ex 446
(630)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:

ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for MADELINE A MONTGOMERY 0731-0000

Current Staff Qualification 00000789

Discipline	Sub Discipline	Process
Controlled Substance	Drug Analysis	Sample Prep.,Wet Chem. & Inst. Analysis

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/1999 12:00:00AM			000000000000223

Satisfactory Associated Proficiency Test 0000007614

Identifier	99-14B	Supplier	CTS
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	10/1/1999 12:00:00AM	LT-PTPM Notification of Test	2/29/2000 12:00:00AM
Due Date	11/19/1999 12:00:00AM	LD-PTPM Review Notified	2/29/2000 12:00:00AM
PT Complete Date	11/4/1999 12:00:00AM	UC Designee Final Eval	2/28/2000 12:00:00AM
Testee Feedback	2/29/2000 12:00:00AM	LD-PTPM Eval	3/15/2000 12:00:00AM

Satisfactory Associated Proficiency Test 0000007771

ntifier	00-11C	Supplier	CTS
aff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	8/31/2000 12:00:00AM	LT-PTPM Notification of Test	12/18/2000 12:00:00AM
Due Date	10/30/2000 12:00:00AM	LD-PTPM Review Notified	12/18/2000 12:00:00AM
PT Complete Date	10/16/2000 12:00:00AM	UC Designee Final Eval	12/18/2000 12:00:00AM
Testee Feedback	12/18/2000 12:00:00AM	LD-PTPM Eval	

Ex 446
(631)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for EILEEN MARIE WANINGER 0731-0000

Current Staff Qualification 00000796

Discipline	Sub Discipline	Process
Controlled Substance	Drug Analysis	Complete Examination & Analysis

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/1999 12:00:00AM	10/8/2004 12:00:00AM	9/23/2007 11:59:58PM	000000000000224

Satisfactory Associated Proficiency Test 0000007593

Identifier 99-5E
Staff Position CHEMIST-FORENSIC EXAMINER
Test Source External
Source Unit Chemistry Unit

Supplier CTS
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date	5/18/1999 12:00:00AM	LT-PTPM Notification of Test	7/13/1999 12:00:00AM
Due Date	7/8/1999 12:00:00AM	LD-PTPM Review Notified	7/13/1999 12:00:00AM
PT Complete Date	6/29/1999 12:00:00AM	UC Designee Final Eval	8/10/1999 12:00:00AM
Testee Feedback	8/4/1999 12:00:00AM	LD-PTPM Eval	3/15/2000 12:00:00AM

Satisfactory Associated Proficiency Test 0000007714

ntifier 00-3D
aff Position CHEMIST-FORENSIC EXAMINER
Test Source External
Source Unit Chemistry Unit

Supplier CTS
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date	5/3/2000 12:00:00AM	LT-PTPM Notification of Test	8/7/2000 12:00:00AM
Due Date	6/28/2000 12:00:00AM	LD-PTPM Review Notified	8/7/2000 12:00:00AM
PT Complete Date	6/20/2000 12:00:00AM	UC Designee Final Eval	7/28/2000 12:00:00AM
Testee Feedback	8/7/2000 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000007781

Identifier 01-5A
Staff Position CHEMIST-FORENSIC EXAMINER
Test Source External
Source Unit Chemistry Unit

Supplier CTS
Preparer Name
Verifier Name
Validator Name

Other Comments

Distribution Date	3/21/2001 12:00:00AM	LT-PTPM Notification of Test	6/12/2001 12:00:00AM
Due Date	5/17/2001 12:00:00AM	LD-PTPM Review Notified	6/12/2001 12:00:00AM
PT Complete Date	4/10/2001 12:00:00AM	UC Designee Final Eval	6/11/2001 12:00:00AM
Testee Feedback	6/11/2001 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000008020

Identifier 02-1C
Staff Position CHEMIST-FORENSIC EXAMINER
Test Source External
rce Unit

Supplier Collaborative Testing Services
Preparer Name
Verifier Name
Validator Name

Other Comments

Ex 446
(632)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for EILEEN MARIE WANINGER 0731-0000

Distribution Date	3/8/2002 12:00:00AM	LT-PTPM Notification of Test	7/1/2002 12:00:00AM
Due Date	4/26/2002 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	3/15/2002 12:00:00AM	UC Designee Final Eval	
Testee Feedback	7/1/2002 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000009146

Identifier	03-1D	Supplier	Collaborative Testing Services
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	3/17/2003 12:00:00AM	LT-PTPM Notification of Test	4/15/2003 12:00:00AM
Due Date	4/29/2003 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	4/15/2003 12:00:00AM	UC Designee Final Eval	
Testee Feedback	9/3/2003 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000009550

Identifier	RE031007012	Supplier	Re-Examination
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

ier Comments LD-QAM required re-examination for 2003. Controlled Substances

Distribution Date	11/19/2003 12:00:00AM	LT-PTPM Notification of Test	
Due Date	12/31/2003 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	12/2/2003 12:00:00AM	UC Designee Final Eval	
Testee Feedback		LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000014003

Identifier	04-13D	Supplier	Collaborative Testing Services
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments

Distribution Date	7/15/2004 12:00:00AM	LT-PTPM Notification of Test	10/26/2004 12:00:00AM
Due Date	8/26/2004 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	7/27/2004 12:00:00AM	UC Designee Final Eval	
Testee Feedback	10/26/2004 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000016208

Identifier	05-15D	Supplier	Collaborative Testing Services
Staff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	

Other Comments CTS 05-502-satisfactory completion

Distribution Date	8/10/2005 12:00:00AM	LT-PTPM Notification of Test	8/24/2005 12:00:00AM
Due Date	9/22/2005 11:59:59PM	LD-PTPM Review Notified	
Complete Date	8/24/2005 12:00:00AM	UC Designee Final Eval	
estee Feedback	11/1/2005 12:00:00AM	LD-PTPM Eval	

Ex 446
(633)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for EILEEN MARIE WANINGER 0731-0000

Satisfactory Associated Proficiency Test 0000016949

Identifier	06-17C	Supplier	Collaborative Testing Services
Staff Position		Preparer Name	
Test Source	External	Verifier Name	
Source Unit		Validator Name	
Other Comments			
Distribution Date	8/10/2006 12:00:00AM	LT-PTPM Notification of Test	8/10/2006 12:00:00AM
Due Date	9/22/2006 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	9/7/2006 12:00:00AM	UC Designee Final Eval	
Testee Feedback	12/4/2006 12:00:00AM	LD-PTPM Eval	

Ex 446
(634)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for EILEEN MARIE WANINGER 0731-0000

Current Staff Qualification 00001091

Discipline	Sub Discipline	Process
Trace Analysis (Chem.)	Bank Dyes	Complete Examination & Analysis

Qualifications Date	Most Recent Proficiency Test	Expiration Date without a new PT	QualTypeLinkID
1/1/1999 12:00:00AM	10/23/2003 11:59:59PM	8/21/2007 11:59:59PM	000000000000261

Satisfactory Associated Proficiency Test 0000006817

Identifier	99-3F	Supplier	
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	Wang, Deborah
Test Source	Internal	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	3/16/1999 12:00:00AM	LT-PTPM Notification of Test	5/17/1999 12:00:00AM
Due Date	4/27/1999 12:00:00AM	LD-PTPM Review Notified	5/17/1999 12:00:00AM
PT Complete Date	3/26/1999 12:00:00AM	UC Designee Final Eval	5/4/1999 12:00:00AM
Testee Feedback	4/27/1999 12:00:00AM	LD-PTPM Eval	3/15/2000 12:00:00AM

Discontinued Associated Proficiency Test 0000007166

ntifier	00-8C	Supplier	
aff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	Cook, Pamela C.
Test Source	Internal	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments Test 00-8 was withdrawn and a new test was reissued.

Distribution Date	7/10/2000 12:00:00AM	LT-PTPM Notification of Test	
Due Date	8/21/2000 12:00:00AM	LD-PTPM Review Notified	
PT Complete Date		UC Designee Final Eval	
Testee Feedback		LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000007338

Identifier	00-14D	Supplier	
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	Cook, Pamela C.
Test Source	Internal	Verifier Name	
Source Unit	Chemistry Unit	Validator Name	

Other Comments

Distribution Date	9/26/2000 12:00:00AM	LT-PTPM Notification of Test	12/18/2000 12:00:00AM
Due Date	11/9/2000 12:00:00AM	LD-PTPM Review Notified	12/18/2000 12:00:00AM
PT Complete Date	9/28/2000 12:00:00AM	UC Designee Final Eval	12/18/2000 12:00:00AM
Testee Feedback	12/18/2000 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000007357

Identifier	01-11C	Supplier	
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	Wang, Deborah
Test Source	Internal	Verifier Name	
rce Unit	Chemistry Unit	Validator Name	

Other Comments

Ex 446
(635)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for EILEEN MARIE WANINGER 0731-0000

Distribution Date	7/19/2001 12:00:00AM	LT-PTPM Notification of Test	9/25/2001 12:00:00AM
Due Date	8/30/2001 12:00:00AM	LD-PTPM Review Notified	9/25/2001 12:00:00AM
PT Complete Date	7/31/2001 12:00:00AM	UC Designee Final Eval	9/25/2001 12:00:00AM
Testee Feedback	9/25/2001 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000008426

Identifier	02-16B	Supplier	
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	DEBORAH WANG
Test Source	Internal	Verifier Name	MICHAEL P RICKENBACH
Source Unit	D7-LABORATORY DIVISION	Validator Name	DEBORAH WANG

Other Comments

Distribution Date	9/26/2002 5:00:00PM	LT-PTPM Notification of Test	10/25/2002 9:00:00AM
Due Date	11/8/2002 12:00:00AM	LD-PTPM Review Notified	
PT Complete Date	10/24/2002 12:00:00PM	UC Designee Final Eval	
Testee Feedback	12/9/2002 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000009147

Identifier	03-13C	Supplier	
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	DEBORAH WANG
Test Source	Internal	Verifier Name	MICHAEL P RICKENBACH
Source Unit	D7-CHEMISTRY	Validator Name	DEBORAH WANG

er Comments

Distribution Date	9/11/2003 12:00:00AM	LT-PTPM Notification of Test	10/3/2003 12:00:00AM
Due Date	10/23/2003 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	10/3/2003 12:00:00AM	UC Designee Final Eval	
Testee Feedback	10/27/2003 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000013997

Identifier	04-8C	Supplier	
Staff Position	CHEMIST-FORENSIC EXAMINER	Preparer Name	DEBORAH WANG
Test Source	Internal	Verifier Name	ROBERT F MOTHERSHEAD
Source Unit	CU	Validator Name	DEBORAH WANG

Other Comments

Distribution Date	6/23/2004 12:00:00AM	LT-PTPM Notification of Test	6/30/2004 12:00:00AM
Due Date	8/4/2004 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	6/30/2004 12:00:00AM	UC Designee Final Eval	
Testee Feedback	6/30/2004 12:00:00AM	LD-PTPM Eval	

Satisfactory Associated Proficiency Test 0000016185

Identifier	05-10C	Supplier	
Staff Position		Preparer Name	DEBORAH WANG
Test Source	Internal	Verifier Name	ROBERT MOTHERSHEAD
Source Unit	CHEMISTRY	Validator Name	DEBORAH WANG

Other Comments TEST SET #1- satisfactory completion

Distribution Date	5/18/2005 12:00:00AM	LT-PTPM Notification of Test	5/18/2005 12:00:00AM
Due Date	6/29/2005 11:59:59PM	LD-PTPM Review Notified	
Complete Date	5/18/2005 12:00:00AM	UC Designee Final Eval	
estee Feedback	8/11/2005 12:00:00AM	LD-PTPM Eval	

Ex 446
(636)

Detailed Qualifications and Proficiency Test Report

Date: 2/28/2007

Report Description:
ATU Personnel use this report to view detailed qualifications and proficiency test for selected staff.

Detailed Qualifications and Proficiency Test Report for EILEEN MARIE WANINGER 0731-0000

Satisfactory Associated Proficiency Test 0000016530

Identifier	06-25B	Supplier	
Staff Position		Preparer Name	Pamela Reynolds
Test Source	Internal	Verifier Name	Eileen Waninger
Source Unit	Chemistry	Validator Name	Jason Brewer
Other Comments			
Distribution Date	5/18/2006 12:00:00AM	LT-PTPM Notification of Test	5/26/2006 12:00:00AM
Due Date	6/30/2006 11:59:59PM	LD-PTPM Review Notified	
PT Complete Date	5/26/2006 12:00:00AM	UC Designee Final Eval	
Testee Feedback	6/15/2006 12:00:00AM	LD-PTPM Eval	

Ex 446
(637)