Gas-chromatography/mass spectrometry (GC-MS) Interpretation of EI spectra

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CCIC MSP

Mass Spec Summer Workshop

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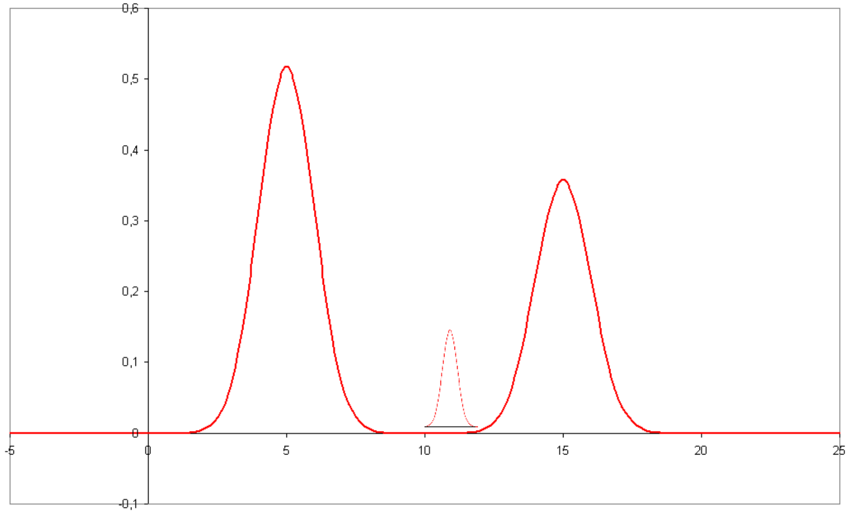
Gas Chromatography

Important goals in chromatography

- Achieve the best separation
    - Little band-broadening – narrow chromatogram peaks – efficient column
- “Dynamic range”
    - Separate and detect the “small” in the close vicinity of the “big”
- Reproducibility
    - Stable peak positions, retention times



Even with the best column ***diffusion*** always plays a role!

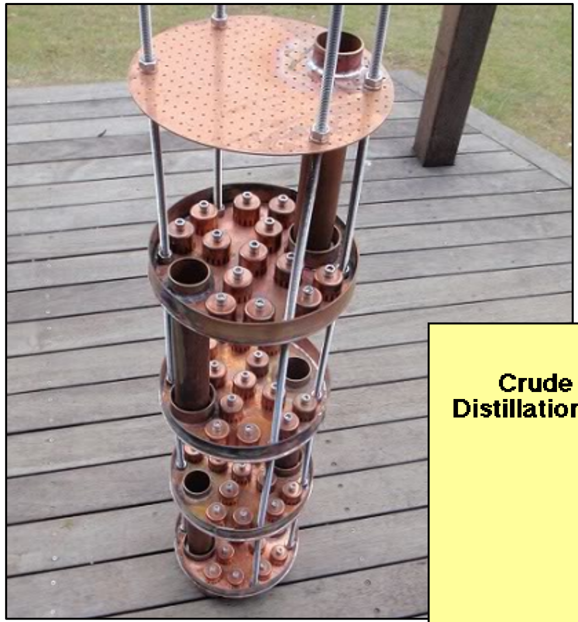
Parameters to control diffusion

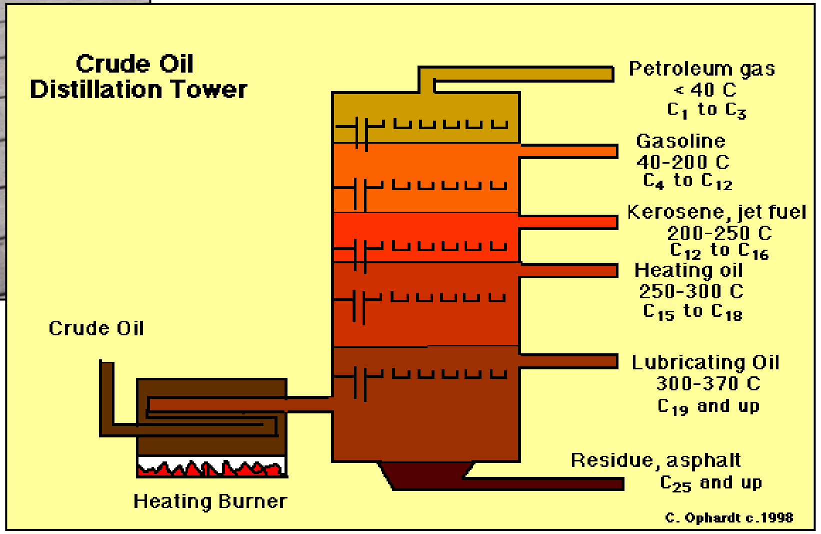
Particle size (Column type)

Flow rate

The Van Deemter Equation

$$HETP = A + (B/u) + (C_s + C_m) \times u$$





HETP = height equivalent to a theoretical plate

-a measure of the resolving power of the column

H = L/N

The Van Deemter Equation

$$HETP = A + (B/u) + (C_s + C_m) \times u$$

•HETP = height equivalent to a theoretical plate, a measure of the resolving power of the column [m] (Height = Length/number of plates=L/N)

•A = Eddy-diffusion parameter, related to channeling through a non-ideal packing [m]

•B = diffusion coefficient of the eluting particles in the longitudinal direction, resulting in dispersion [$m^2\ s^{-1}$]

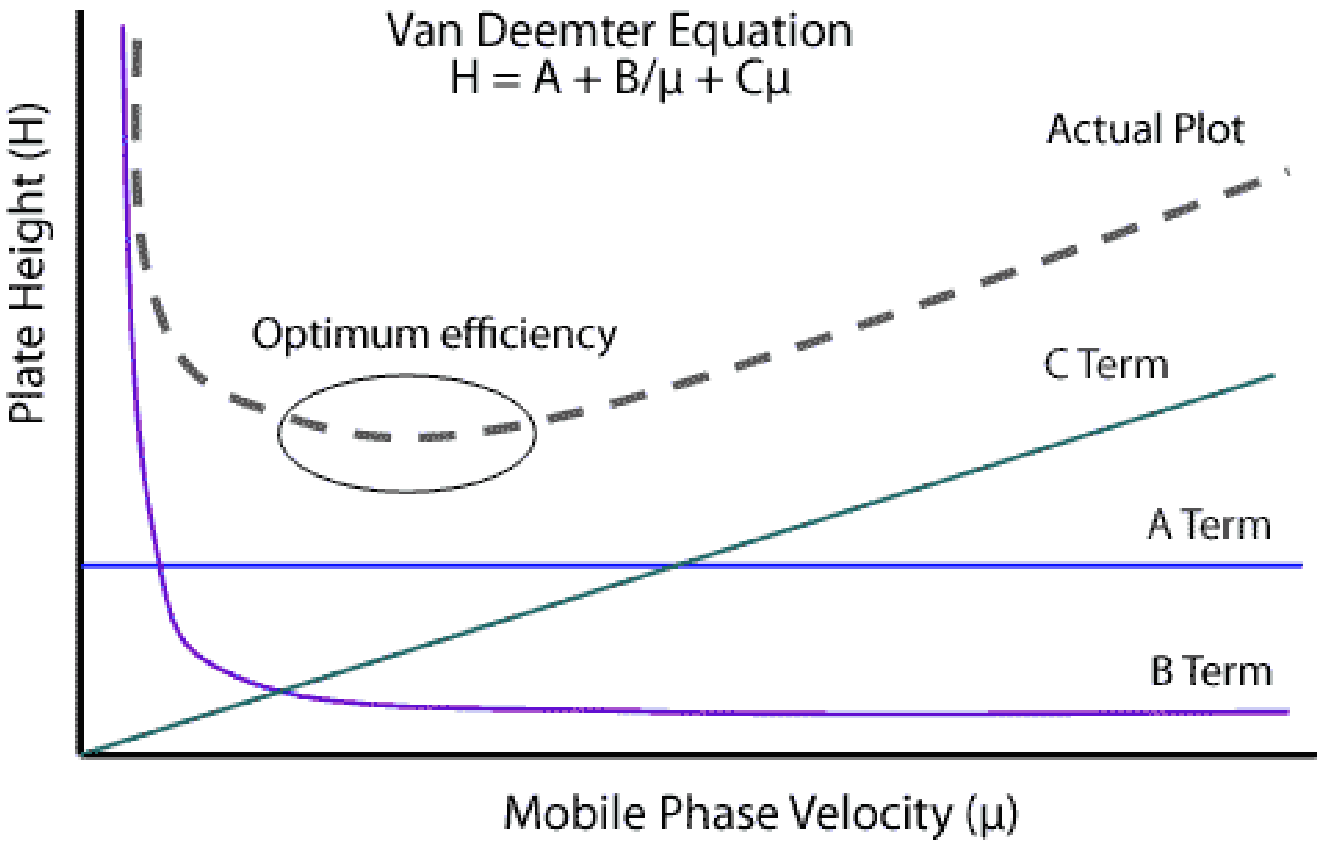
•C = Resistance to mass transfer coefficient of the analyte between mobile [m] and stationary phase [s]

•u = Linear Velocity [$m\ s^{-1}$]

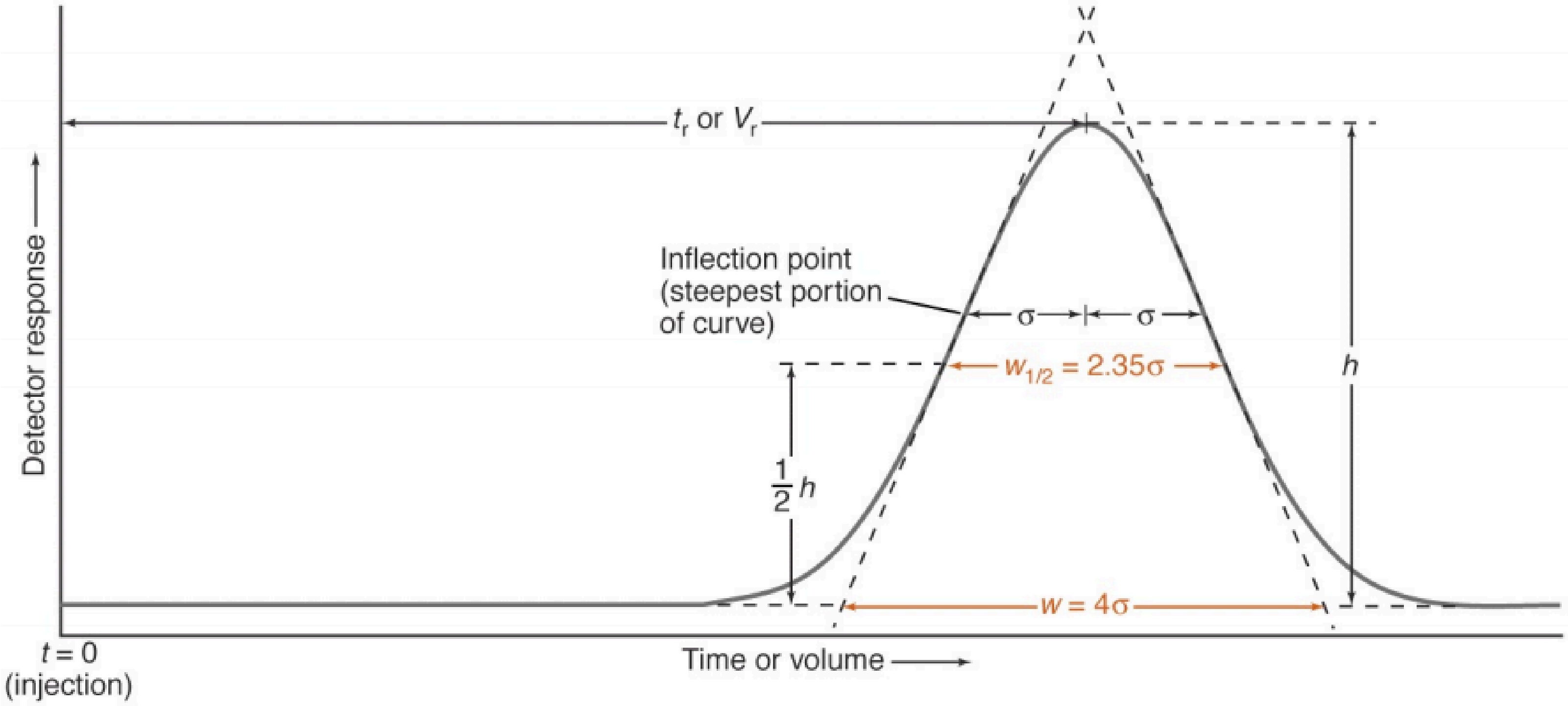
Original paper: Van Deemter JJ, Zuiderweg FJ and Klinkenberg A (**1956**). "Longitudinal diffusion and resistance to mass transfer as causes of non ideality in chromatography". *Chem. Eng. Sc.* 5: 271–289

Youtube: https://www.youtube.com/watch?v=8i_4-OMCANE

The Van Deemter Curve



Resolution



Solute moving through a column spreads into a Gaussian shape with standard deviation σ. Common Measures of breadth are:

1) The width w½ measured at half-height
2) The width w at the baseline between tangents drawn to the steepest parts of the peak (inflection points).

Derive Van Deemter for Resolution

HETP = L/N

N = L/N → substitute H = σ^2/L

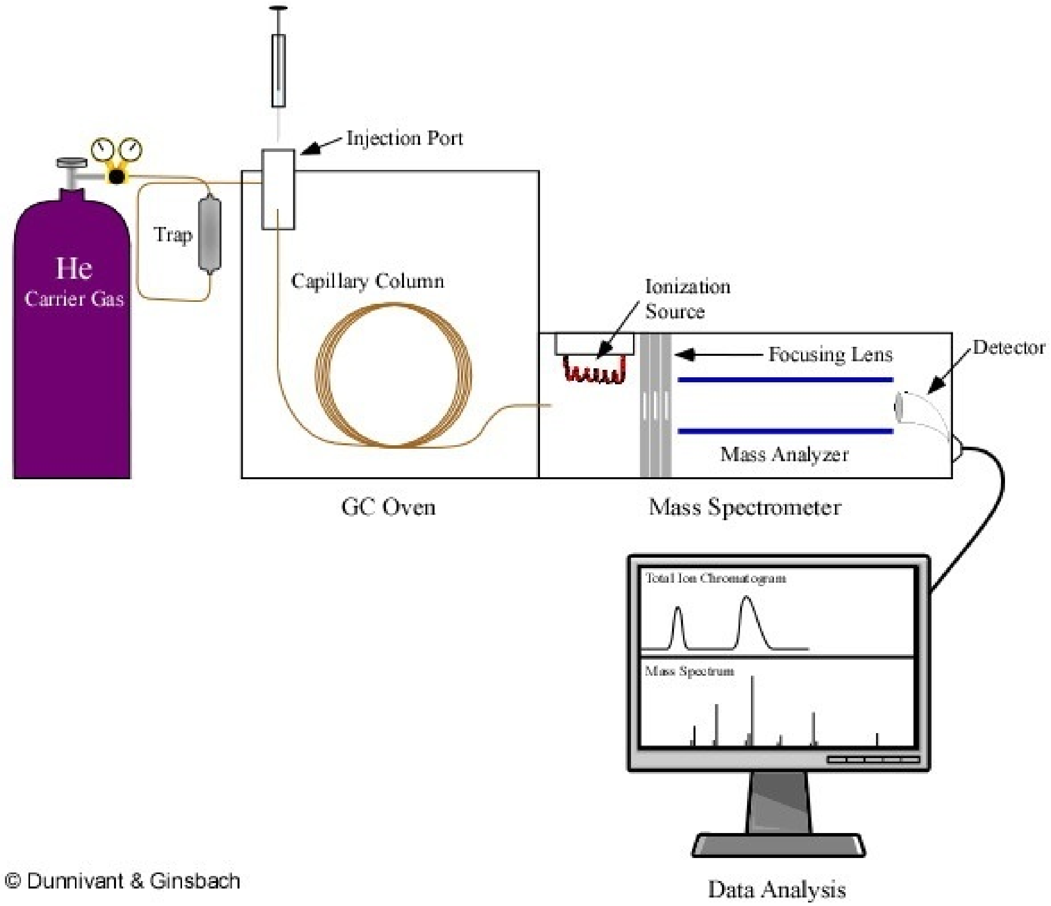
N = L^2/σ^2 → convert length to time (utility)

N = $(t_r)^2/(\sigma)^2$ → relate σ to $w_{1/2}$ (2.35σ) and *w* (4σ)

N = $16t_r^2/w^2$

N = $5.55t_r^2/w_{1/2}^2$

GC coupled to Mass Spectrometry (MS)



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<u>Autosampler</u>

<u>MS Analyzer:</u>
-Quadrupole
-Ion trap
-Time of flight (TOF)
-Orbitrap

<u>GC oven</u>
-GC Column

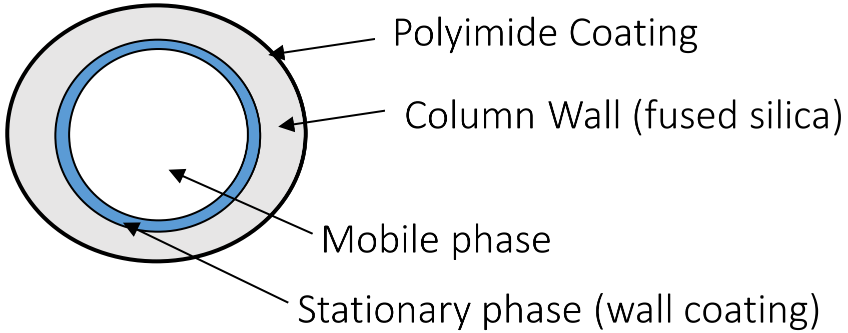
<u>GC controller</u>

GC Columns

- Two Common Formats
  - Packed columns (most common with bonded liquid coating)
  - Open tubular (typically long columns with small diameters)

Packed Columns

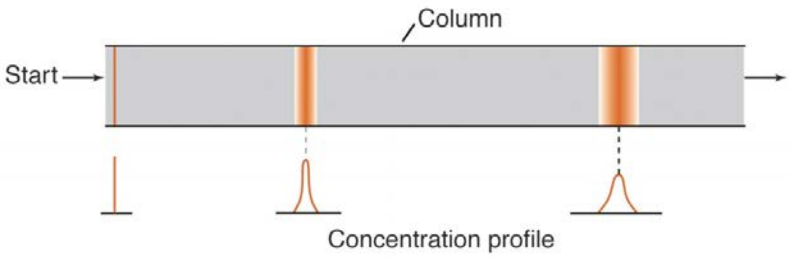
Open Tubular (end on, cross section view)

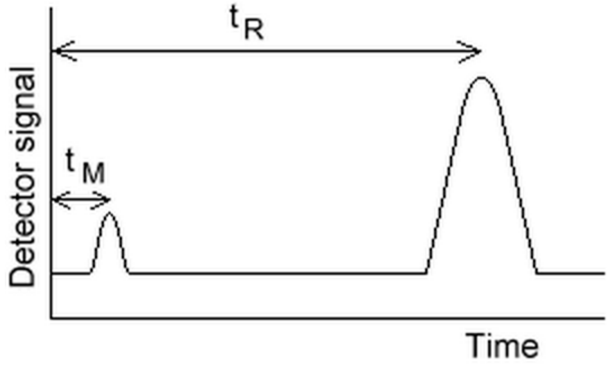


GC Columns

- Advantages of Open Tubular Columns
  - Best resolution (negligible A term, small C term in Van Deemter Equation)
  - More robust
  - Better sensitivity with many detectors (due to less band broadening vs. lower mass through column)
- Column Selection
  - High resolution (thin film, 0.25 mm diameter, 60 m) vs. higher capacity (thick film, 0.53 mm diameter)
  - Stationary phase based on polarity

GC Stationary Phase





Retention factor (k’) describes the migration of the analyte on the column

$$k' = (t_R - t_M)/t_M$$

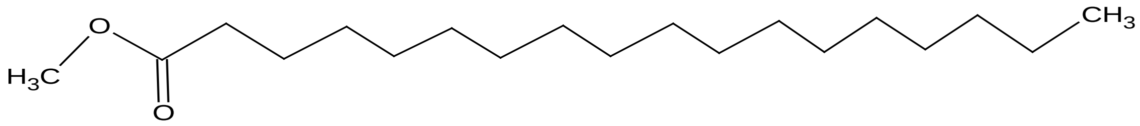
Selectivity factor (α) describes the separation of 2 species, A and B, on the column

$$\alpha = k'_B/k'_A$$

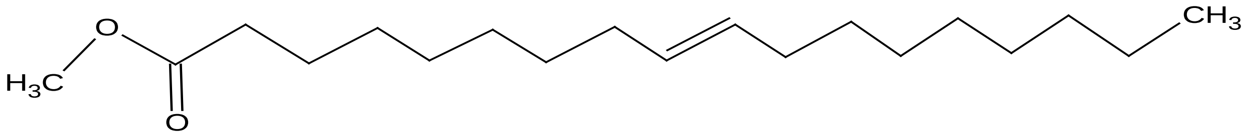
$$\text{Resolution (R)} = \underbrace{\tfrac{1}{4}\sqrt{N}}_{\text{efficiency}}\ \underbrace{(\alpha-1/\alpha)}_{\text{selectivity}}\ \underbrace{(k'/k'+1)}_{\text{retention}}$$

GC Adjustments

- k is adjusted by changing temperature (higher T means smaller k)
- Selection of stationary phase affects k and α values
  - The α values are adjusted by changing column (will work if there is a difference in solute polarity)
    - example: separation of saturated and unsaturated fatty acid methyl esters (FAMEs).



C18:0 FAME bp = 369°C

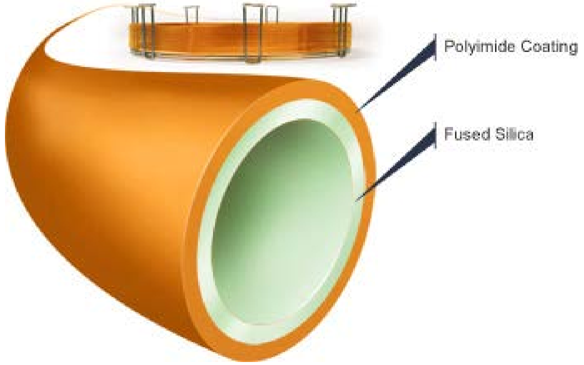


C18:1 FAME bp = 352°C

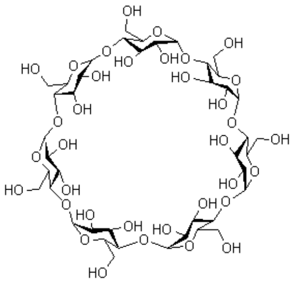
    - Retention of C18:0 and C18:1 FAMEs on RTX-5MS columns is very similar (due to similar boiling points)
    - Retention on more polar columns (RTX-50MS) is greater for the more polar unsaturated FAMEs
- Main concerns of stationary phase are: polarity, functional groups, maximum operating temperature, and column bleed (loss of stationary phase)
- More polar columns suffer from lower maximum temperatures and greater column bleed-**derivatize?**
- Changing carrier gas has no effect on retention

GC Column Options

Type	Functional Groups	Polarity
RTX-1MS	100% dimethyl	Non-polar
RTX-5MS	5% diphenyl/95% dimethyl	Low polarity
RTX-50MS	Phenyl methyl	More polar
Stabliwax-MS	PEG	High polarity



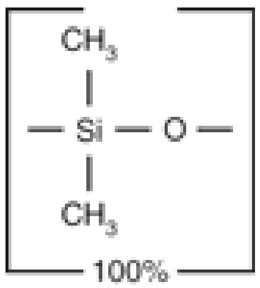
Chirality Columns



β-cyclodextrin

GC Column Options

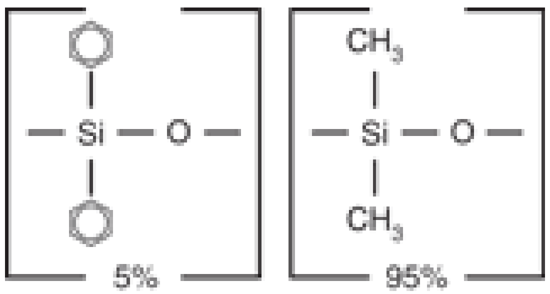
RTX-1MS = NonPolar



Saturated Hydrocarbons
Olefinic Hydrocarbons
Aromatic Hydrocarbons
Halocarbons
Mercaptans
Sulfides
CS2

Long lifetime and very low bleed at high operating temperatures.
Temperature range: -60 °C to 350 °C

RTX-5MS = Low Polarity

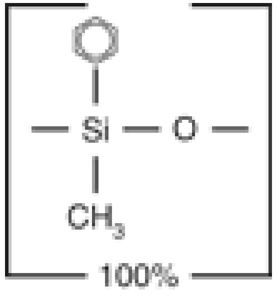


Ethers
Ketones
Aldehydes
Esters
Tertiary amines
Nitro compounds without α-H atoms
Nitrile compounds without α-H atoms

Column specifically tested for low-bleed performance.
Temperature range: -60 °C to 350 °C.

GC Column Options

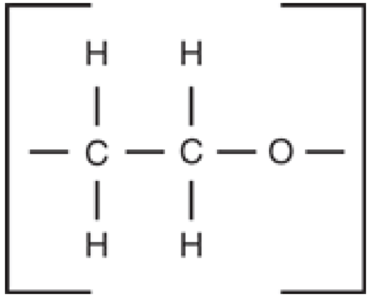
RTX-50MS = Medium Polarity



Alcohols
Carboxylic acids
Phenols
Primary and secondary amines
Oximes
Nitro compounds without α-H atoms
Nitrile compounds without α-H atoms

Low bleed
Temperature range: 40 °C to 320 °C.

stabliwax-5MS = High Polarity

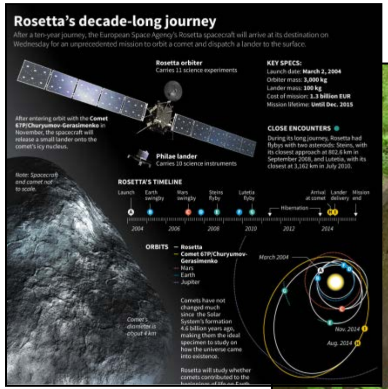


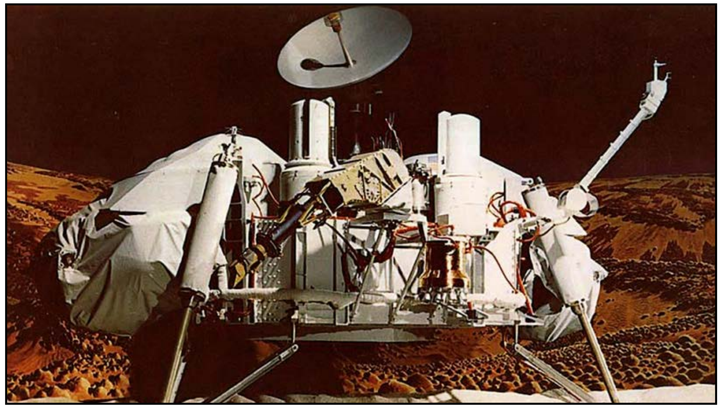
Polyhydroxyalcohols
Amino alcohols
Hydroxy acids
Polyprotic acids
Polyphenols

Low bleed but rugged enough to with stand repeated cycles without retention time shifting.
Sensitive to water and oxidation.
Temperature range: 40 °C to 250/260 °C.

GC-MS Applications

- Environmental monitoring
- Food, beverage, flavor, and fragrance analysis
- Forensic and criminal cases → **confirmation test**
- Biological and pesticides detection
- Fermentation control
- Security and chemical warfare agent detection
- Astro chemistry and Geo chemical research
- Medicine and Pharmaceutical Applications
- Petrochemical and hydrocarbon analysis
- Clinical toxicology
- Energy and fuel applications
- Industrial use
- Academics

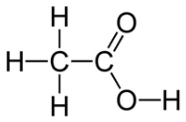




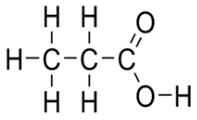
http://www.omicsonline.org/open-access/gc-ms-technique-and-its-analytical-applications-in-science-and-technology-2155-9872.1000222.pdf

GC-MS Projects in the MS&P

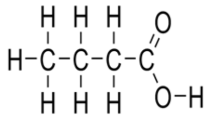
https://www.youtube.com/watch?v=7yxWBOPNkJ8



Acetic acid (acetate)

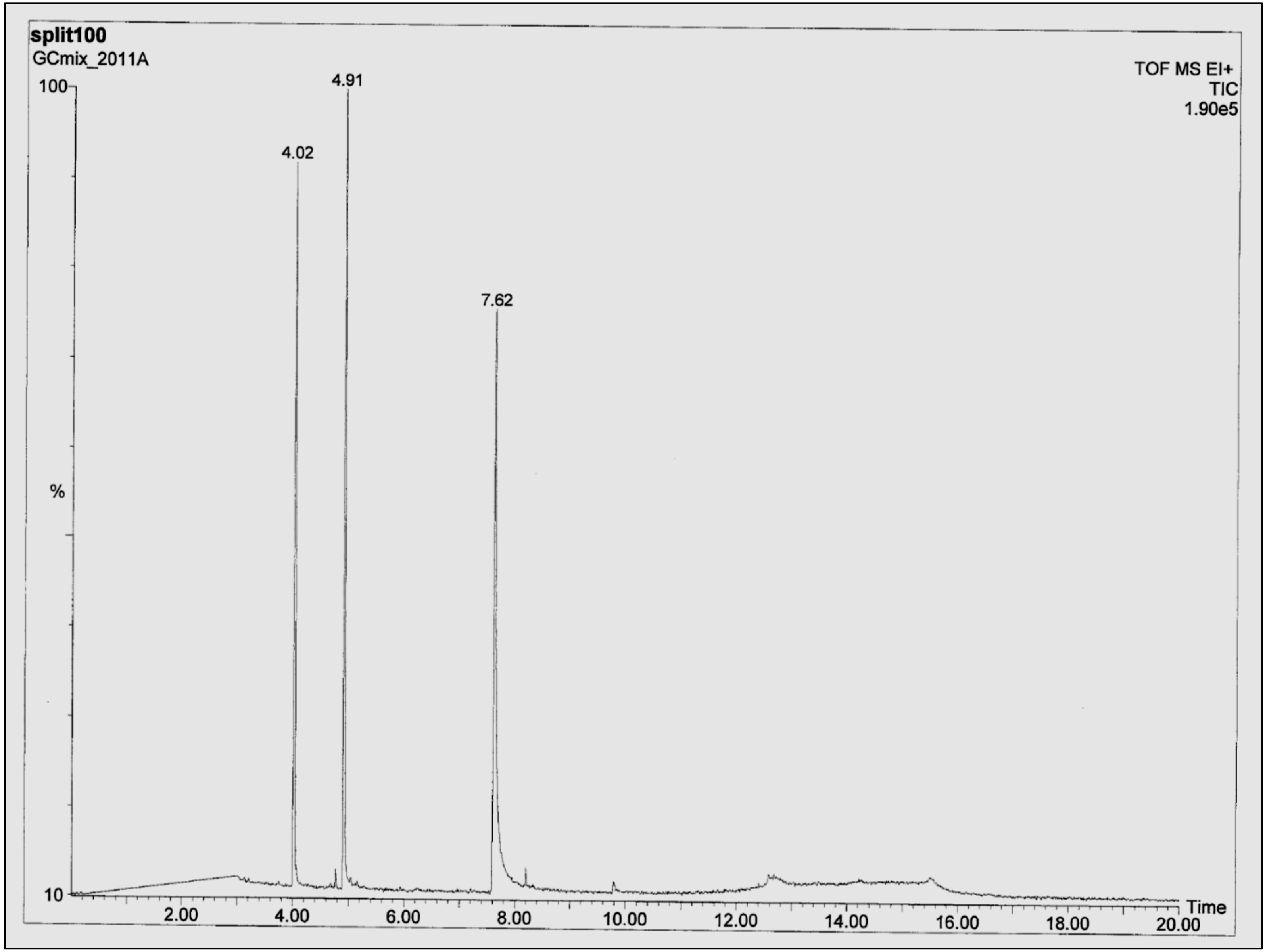


Propionic acid (propionate)

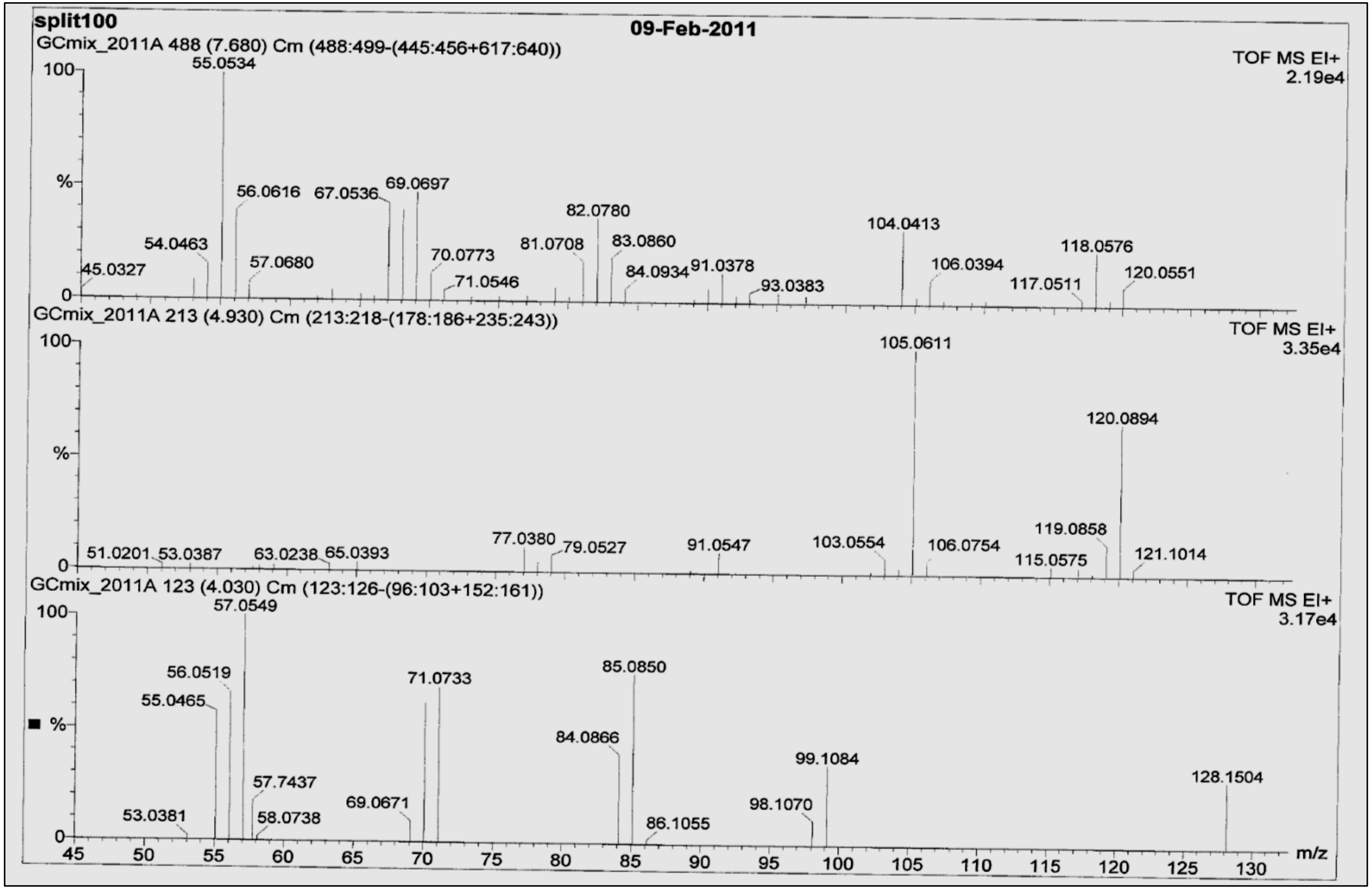


Butyric acid (butyrate)

GC-Chromatogram



EI Spectrum



split100
09-Feb-2011
GCmix_2011A 488 (7.680) Cm (488:499-(445:456+617:640))
TOF MS EI+
2.19e4
100
%
0
55.0534
56.0616
67.0536
69.0697
54.0463
45.0327
57.0680
70.0773
71.0546
82.0780
81.0708
83.0860
84.0934
91.0378
93.0383
104.0413
106.0394
118.0576
117.0511
120.0551
GCmix_2011A 213 (4.930) Cm (213:218-(178:186+235:243))
TOF MS EI+
3.35e4
105.0611
120.0894
51.0201
53.0387
63.0238
65.0393
77.0380
79.0527
91.0547
103.0554
106.0754
119.0858
115.0575
121.1014
GCmix_2011A 123 (4.030) Cm (123:126-(96:103+152:161))
TOF MS EI+
3.17e4
57.0549
56.0519
55.0465
71.0733
85.0850
84.0866
99.1084
128.1504
57.7437
69.0671
53.0381
58.0738
98.1070
86.1055
45 50 55 60 65 70 75 80 85 90 95 100 105 110 115 120 125 130
m/z

The Nitrogen Rule

- Compounds* that contain ***even*** number of N atoms have ***even*** number of *nominal* molecular weight
- Compounds* that contain ***odd*** number of N atoms have ***odd*** number of *nominal* molecular weight

But what about *singly protonated* molecules and *accurate* molecular weights??

*** Common organic compounds**

Ion Stabilities

- ***Even electron ions are more stable*** **than odd electron (radical) ions**

How about protonated molecules: even electron or not?

And how about ions formed by electron impact (EI) ionization?

Selected Isotope Ratios and 13C Contributions

Accurate elemental masses:

C: 12.000000
H: 1.007825
O: 15.9949
N: 14.003
Cl: 34.9688
Br: 78.9183
S: 31.9720

Table 2.1. *Natural isotopic abundances of common elements.*[a]

Element	A Mass	A Per cent	A + 1 Mass	A + 1 Per cent	A + 2 Mass	A + 2 Per cent	Element type
H	1	100	2	0.015			"A"
C	12	100	13	1.1[b]			"A + 1"
N	14	100	15	0.37			"A + 1"
O	16	100	17	0.04	18	0.20	"A + 2"
F	19	100					"A"
Si	28	100	29	5.1	30	3.4	"A + 2"
P	31	100					"A"
S	32	100	33	0.80	34	4.4	"A + 2"
Cl	35	100			37	32.5	"A + 2"
Br	79	100			81	98.0	"A + 2"
I	127	100					"A"

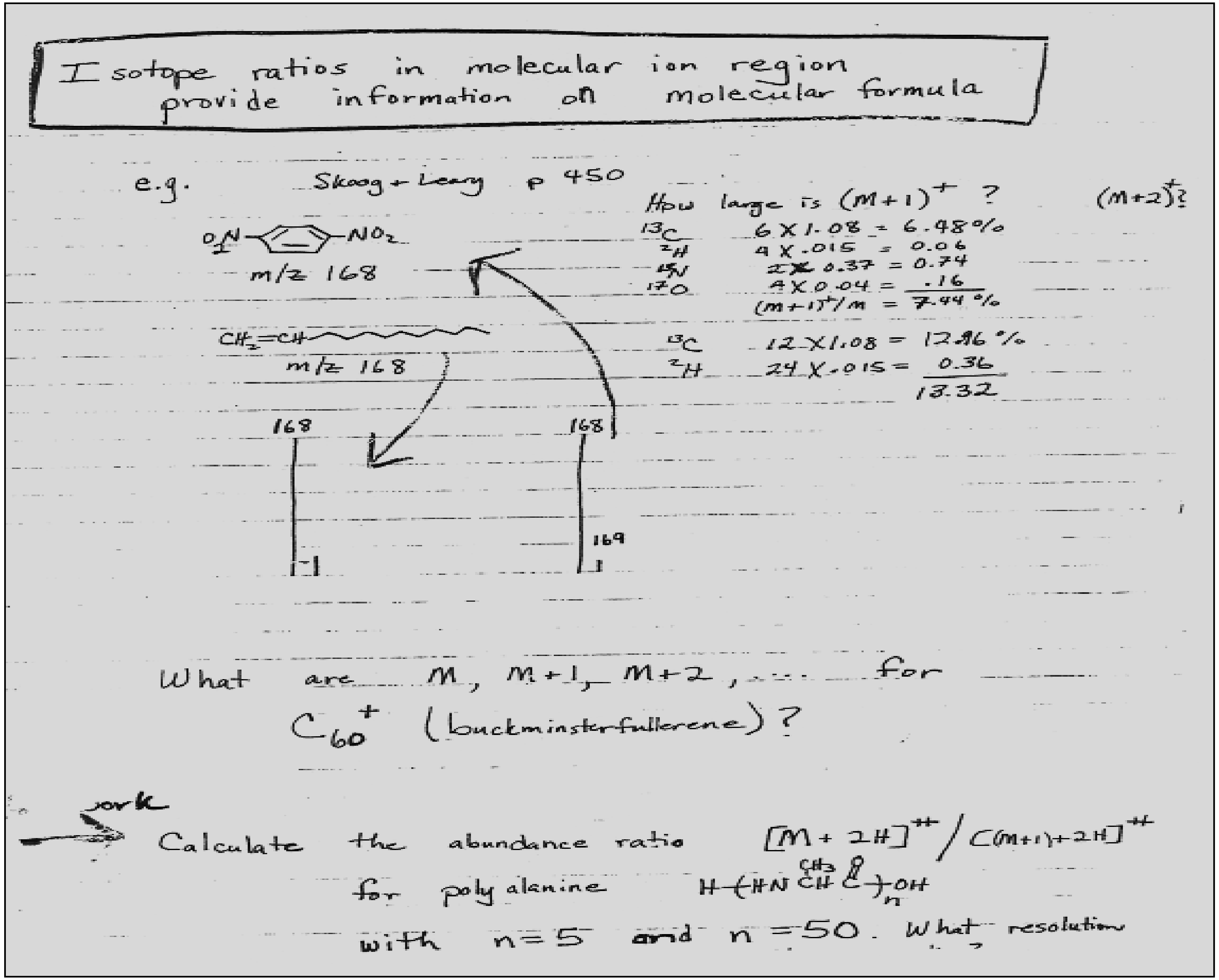
[a] Wapstra and Gove (1971).
[b] 1.1 ±0.02, depending on source.

Table 2.2. *Isotopic contributions for carbon and hydrogen.*
If the abundance of the peak A is 100 (after correction for isotopic contributions to it), then its isotopic contributions will be:

	(A + 1)	(A + 2)		(A + 1)	(A + 2)	(A + 3)
C_1	1.1	0.00	C_{16}	18	1.5	0.1
C_2	2.2	0.01	C_{17}	19	1.7	0.1
C_3	3.3	0.04	C_{18}	20	1.9	0.1
C_4	4.4	0.07	C_{19}	21	2.1	0.1
C_5	5.5	0.12	C_{20}	22	2.3	0.2
C_6	6.6	0.18	C_{22}	24	2.8	0.2
C_7	7.7	0.25	C_{24}	26	3.3	0.3
C_8	8.8	0.34	C_{26}	29	3.9	0.3
C_9	9.9	0.44	C_{28}	31	4.5	0.4
C_{10}	11.0	0.54	C_{30}	33	5.2	0.5
C_{11}	12.1	0.67	C_{35}	39	7.2	0.9
C_{12}	13.2	0.80	C_{40}	44	9.4	1.3
C_{13}	14.3	0.94	C_{50}	55	15	1.3
C_{14}	15.4	1.1	C_{60}	66	21	4.6
C_{15}	16.5	1.3	C_{100}	110	60	22

For each additional element present, add *per atom:*
(A + 1): N, 0.37; O, 0.04; Si, 5.1; S, 0.80.
(A + 2): O, 0.20; Si, 3.4; S, 4.4; Cl, 32.5; Br, 98.0.
Typical values for (A + 4): C_{25}, 0.02; C_{40}, 0.13; C_{100}, 5.7.

Use of Isotope Ratios to Distinguish Structures



Isotope ratios in molecular ion region provide information on molecular formula

e.g. Skoog + Leary p 450

$O_2N-C_6H_4-NO_2$

m/z 168

$CH_2=CH$~~~~~~~~

m/z 168

How large is $(M+1)^+$? $(M+2)^+$?

^{13}C $6 \times 1.08 = 6.48\%$

^{2}H $4 \times .015 = 0.06$

^{15}N $2 \times 0.37 = 0.74$

^{17}O $4 \times 0.04 = .16$

$(M+1)^+/M = 7.44\%$

^{13}C $12 \times 1.08 = 12.96\%$

^{2}H $24 \times .015 = 0.36$

13.32

168 | 168, 169

What are M, M+1, M+2, ... for C_{60}^+ (buckminsterfullerene)?

work → Calculate the abundance ratio $[M+2H]^{++}/[(M+1)+2H]^{++}$ for polyalanine $H(HNCH(CH_3)CO)_nOH$ with n = 5 and n = 50. What resolution

Increasing ^{13}C contribution in polyalanines

Page: 1

[Theoretical Ion Distribution]
Molecular Formula : C15 H29 O6 N5
(m/z 375.2118, MW 375.4252, U.S. 4.0)
Base Peak : 375.2118, Averaged MW : 375.4263(a), 375.4269(w)

m/z	INT.	
375.2118	100.0000	**
376.2146	19.1839	************
377.2168	2.9467	**
378.2192	0.3302	
379.2215	0.0310	
380.2238	0.0025	
381.2261	0.0002	

n = 5

[Theoretical Ion Distribution]
Molecular Formula : C90 H129 O26 N25
(m/z 1975.9541, MW 1977.1662, U.S. 39.0)
Base Peak : 1976.9570, Averaged MW : 1977.1722(a), 1977.1729(w)

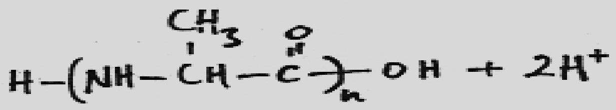
m/z	INT.	
1975.9541	89.1183	***
1976.9570	100.0000	**
1977.9597	60.2390	************************************
1978.9624	25.6258	***************
1979.9651	8.5827	*****
1980.9677	2.3982	*
1981.9702	0.5794	
1982.9727	0.1240	
1983.9753	0.0239	
1984.9778	0.0042	
1985.9802	0.0007	
1986.9827	0.0001	

n = 25

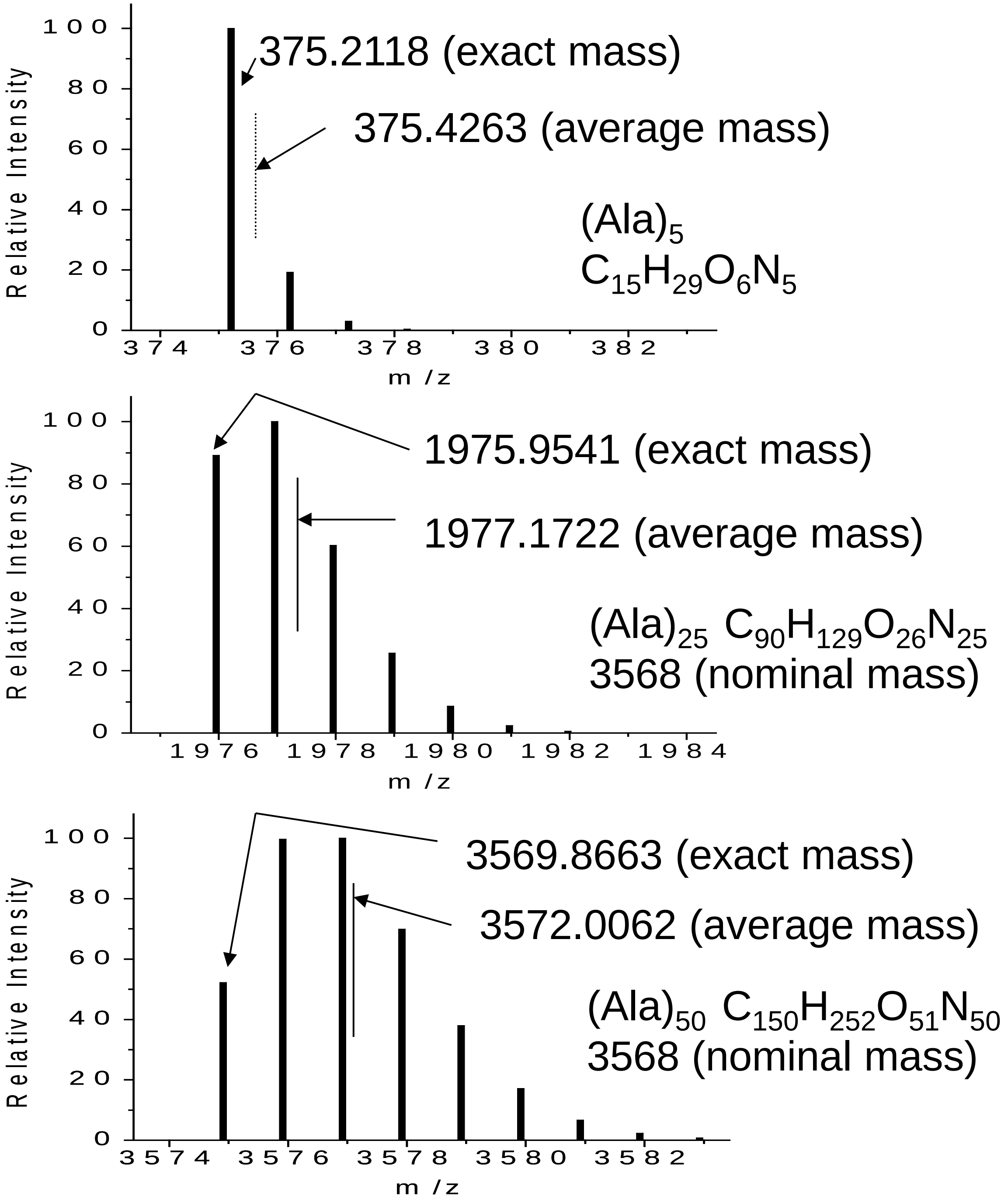
[Theoretical Ion Distribution]
Molecular Formula : C150 H257 O51 N50
(m/z 3574.9054, MW 3576.9950, U.S. 47.5)
Base Peak : 3576.9109, Averaged MW : 3577.0061(a), 3577.0067(w)

m/z	INT.	
3574.9054	52.1735	*******************************
3575.9082	99.6516	**
3576.9109	100.0000	**
3577.9136	69.8217	**
3578.9163	37.9680	***********************
3579.9189	17.0842	**********
3580.9214	6.6049	****
3581.9240	2.2507	*
3582.9265	0.6886	
3583.9290	0.1917	
3584.9315	0.0491	
3585.9340	0.0117	
3586.9364	0.0026	
3587.9389	0.0005	
3588.9413	0.0001	

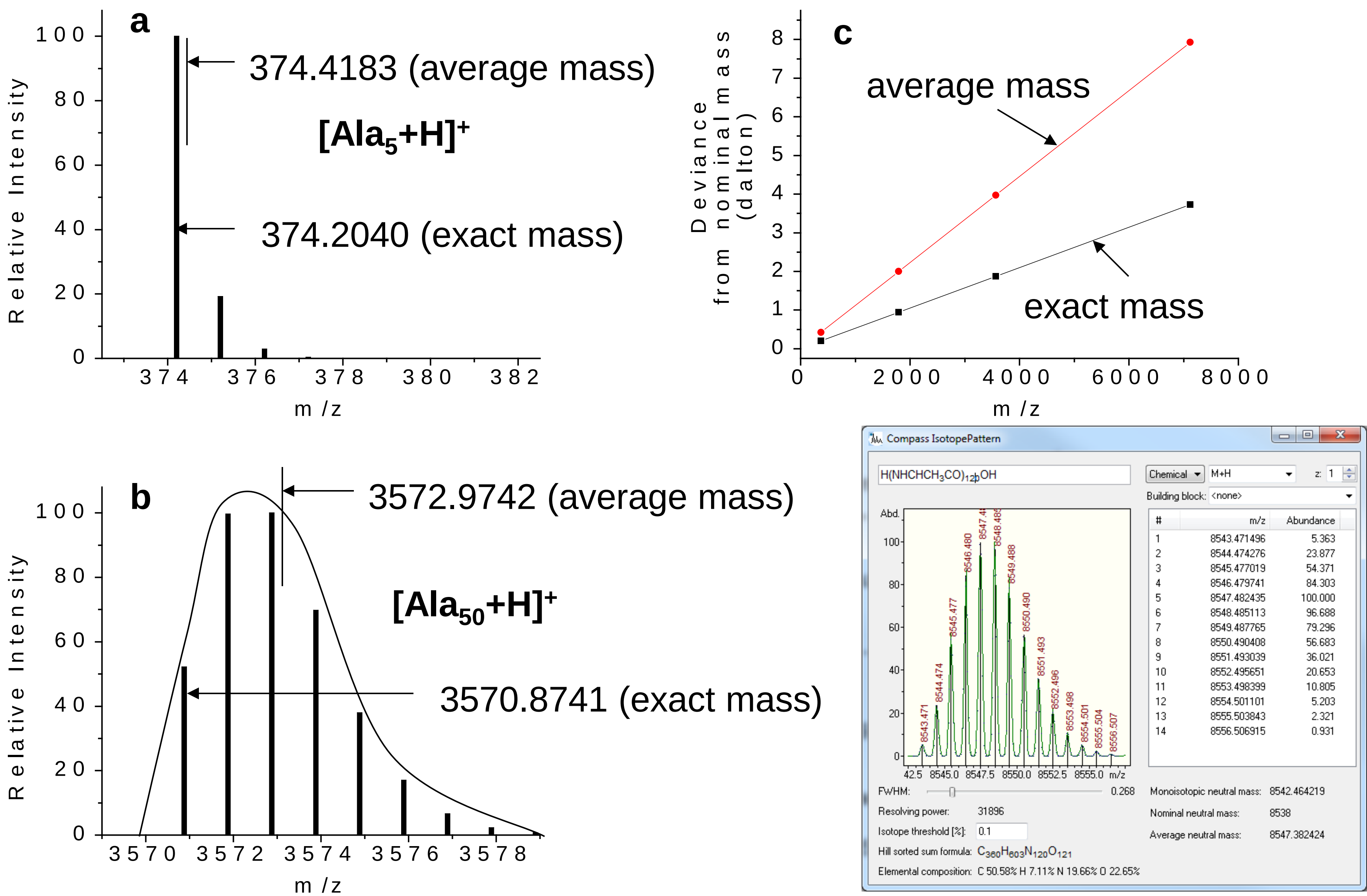
n = 50



Increasing ^{13}C contribution in polyalanines

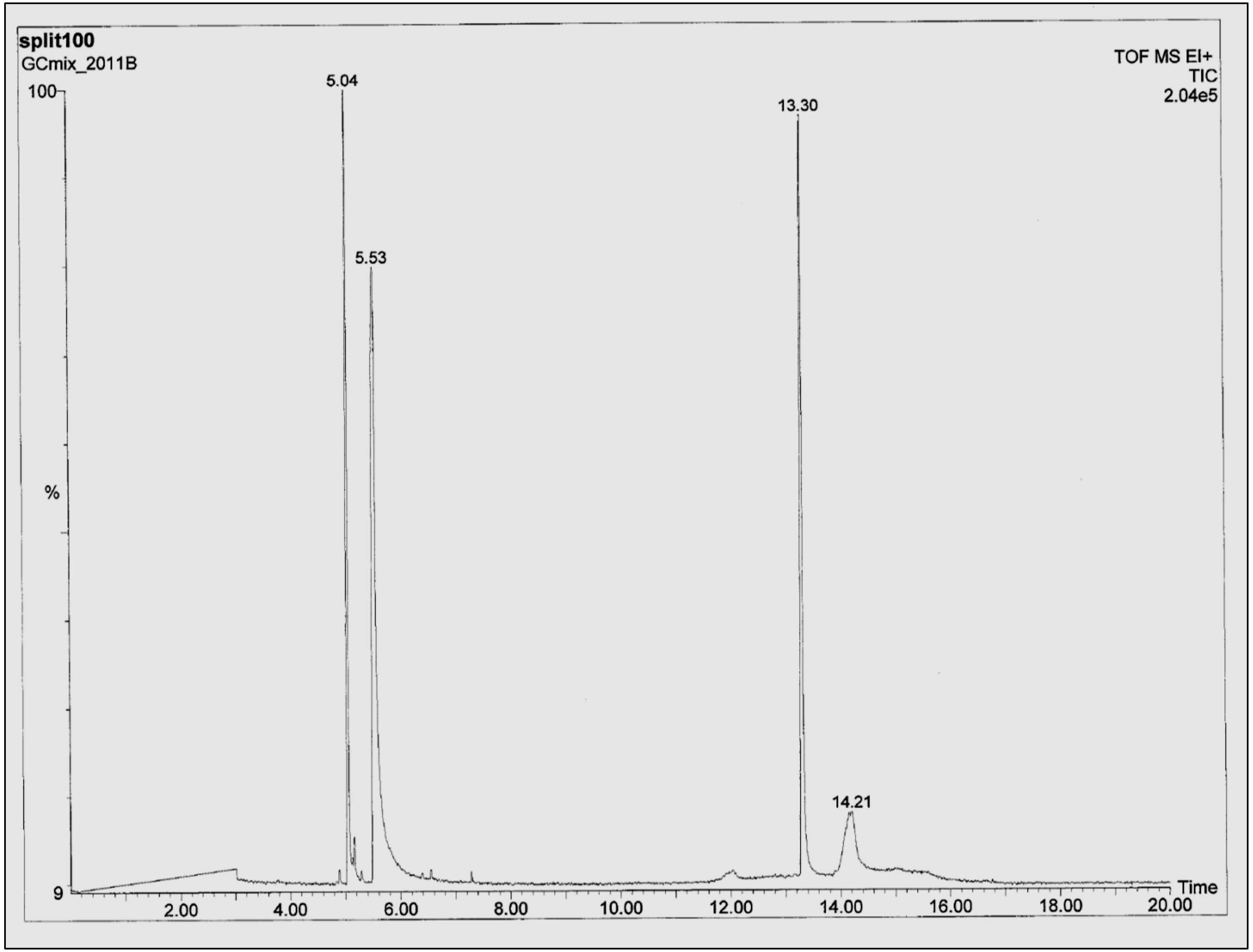


Increasing ^{13}C contribution in polyalanines

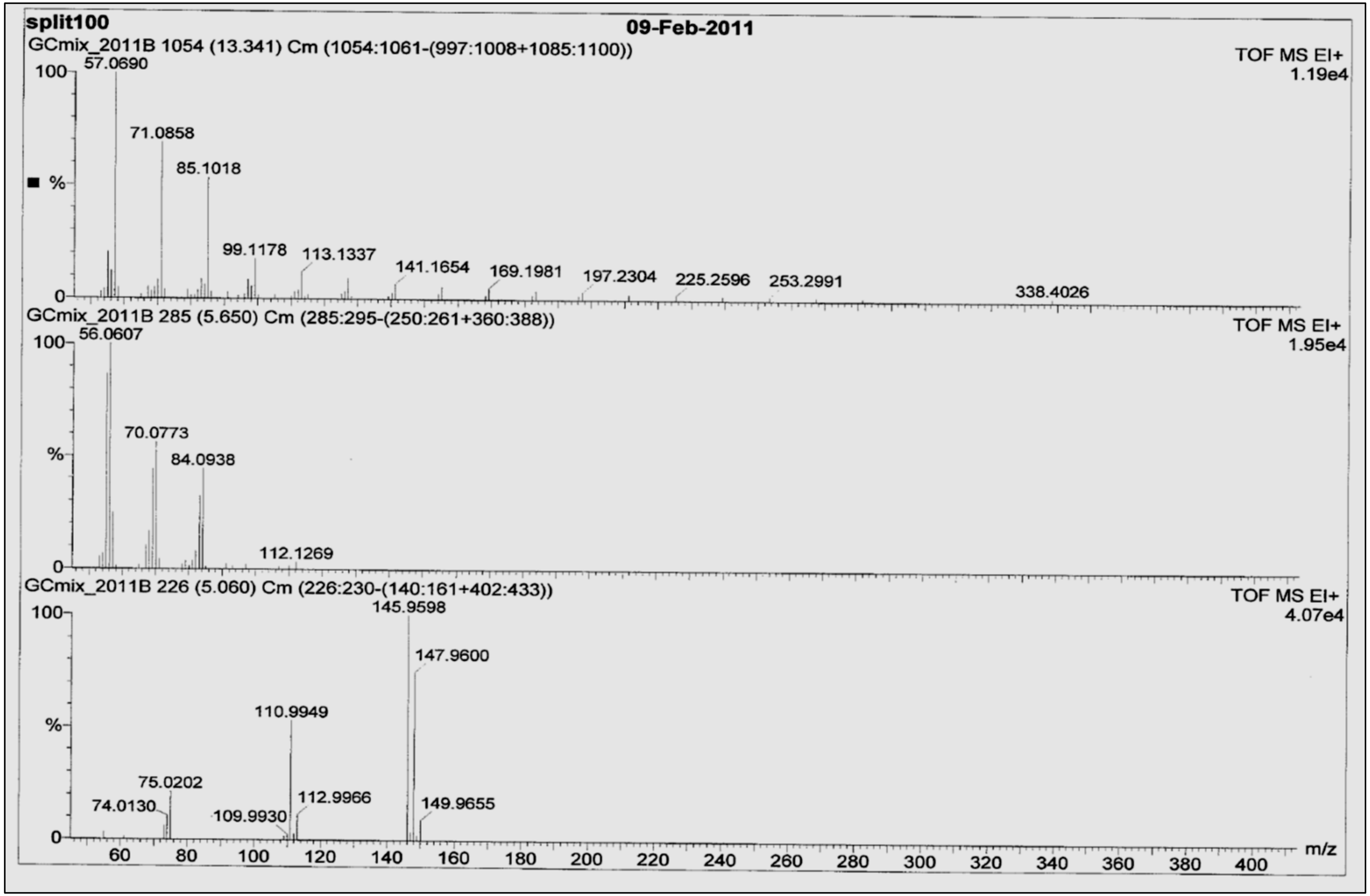


[Ala$_{120}$+H]

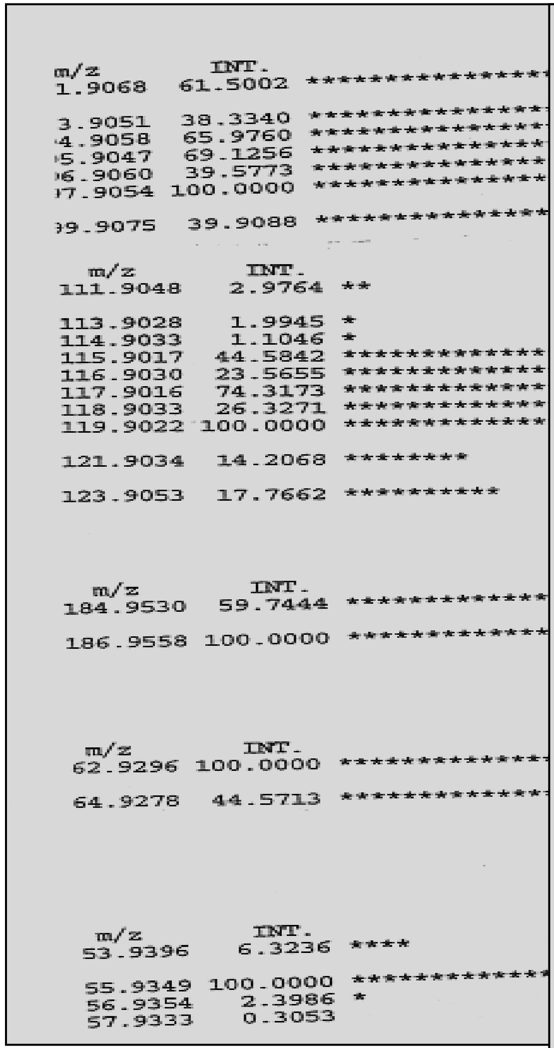
GC Chromatogram- an example for you



EI Spectrum- give the top and bottom a try



Characteristic Isotope Distribution of Selected Transition Metal Elements



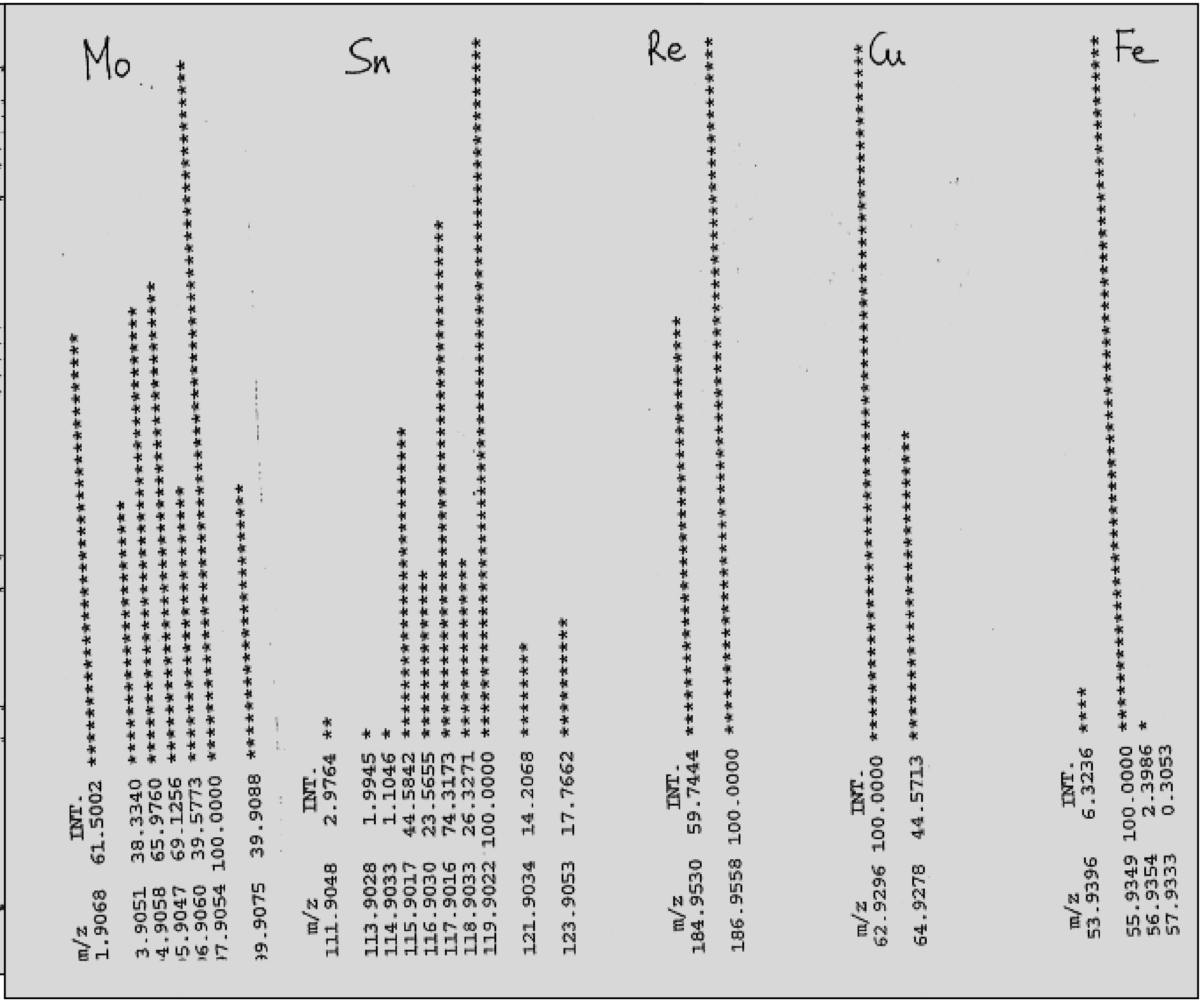
m/z	INT.	
1.9068	61.5002	
3.9051	38.3340	
4.9058	65.9760	
5.9047	69.1256	
6.9060	39.5773	
7.9054	100.0000	
9.9075	39.9088	

m/z	INT.	
111.9048	2.9764	**
113.9028	1.9945	*
114.9033	1.1046	*
115.9017	44.5842	
116.9030	23.5655	
117.9016	74.3173	
118.9033	26.3271	
119.9022	100.0000	
121.9034	14.2068	********
123.9053	17.7662	**********

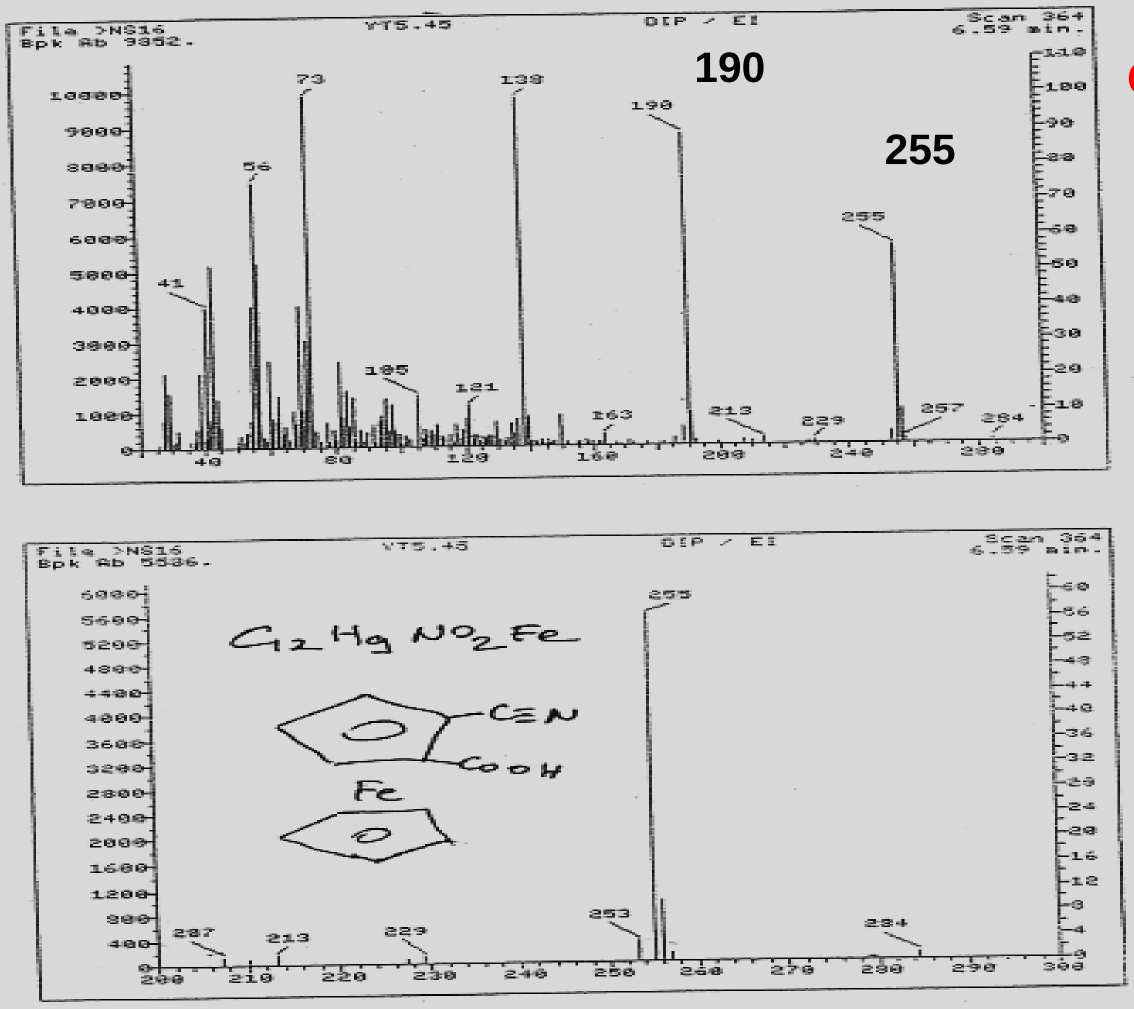
m/z	INT.	
184.9530	59.7444	
186.9558	100.0000	

m/z	INT.	
62.9296	100.0000	
64.9278	44.5713	

m/z	INT.	
53.9396	6.3236	****
55.9349	100.0000	
56.9354	2.3986	*
57.9333	0.3053	



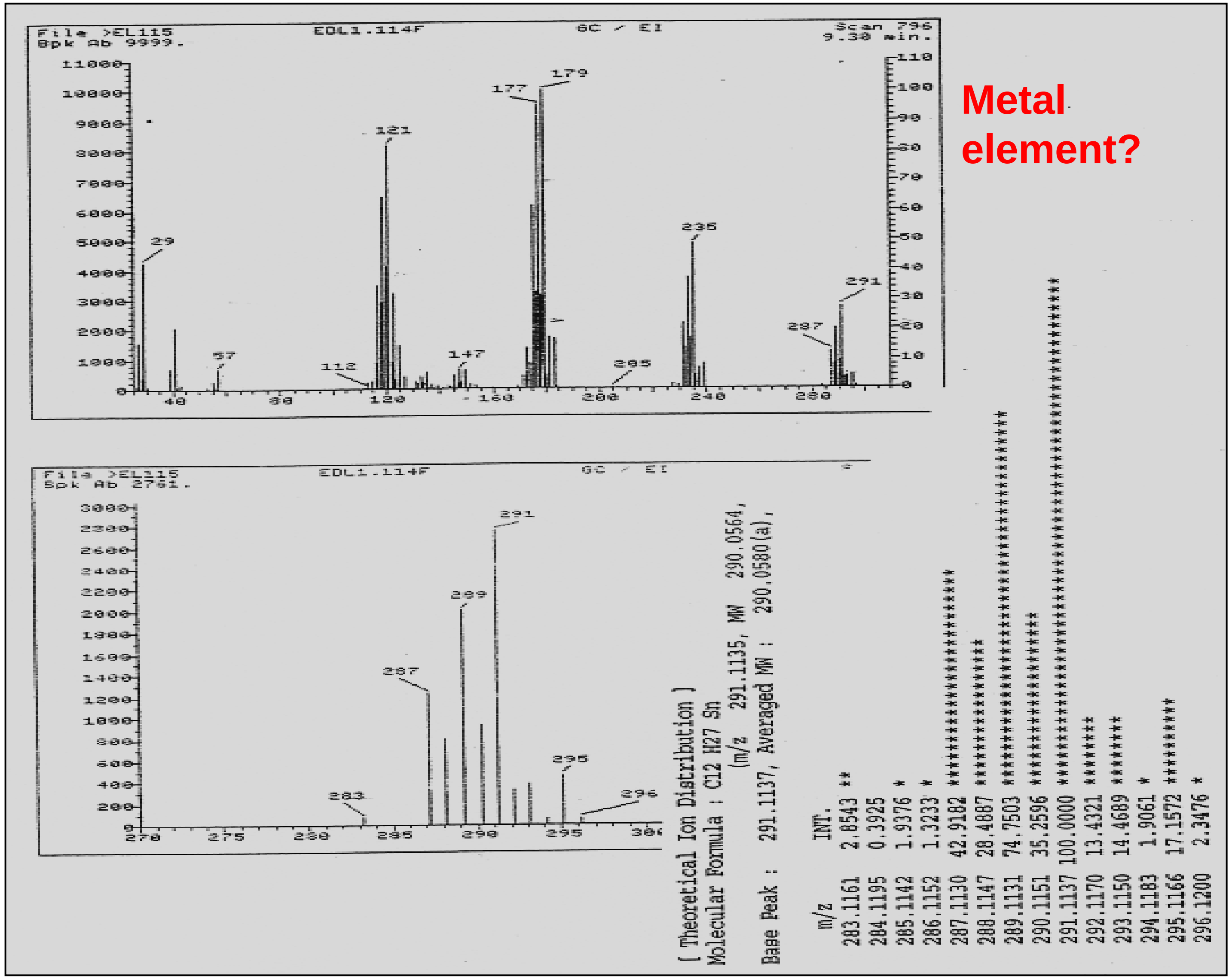
Origin of m/z 190?

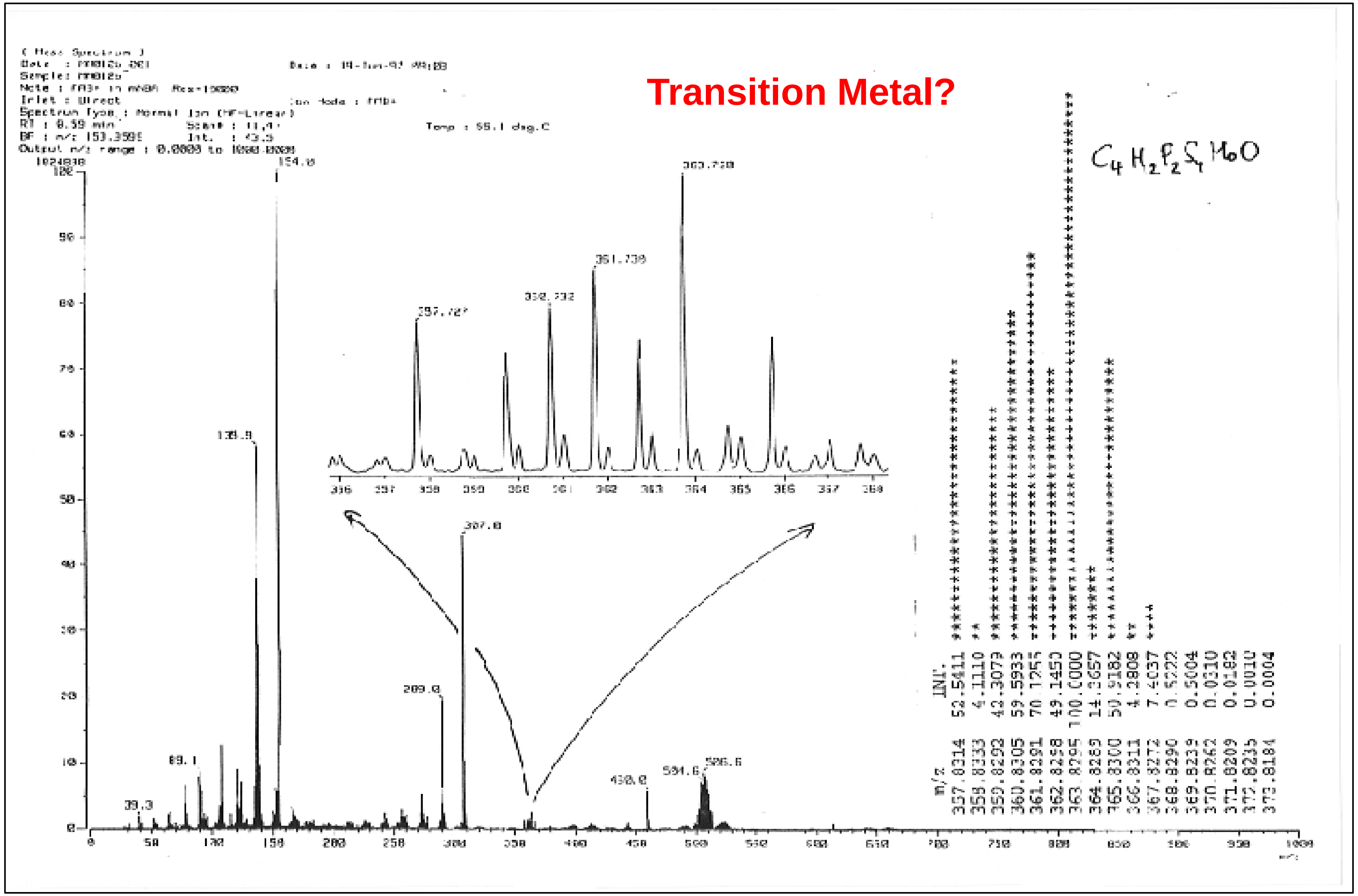


Theoretical Ion Distribution)
Molecular Formula : C12 H9 N O2 Fe
(m/z 254.9983, MW 255.0560, U.S. 9.5)
Base Peak : 254.9983, Averaged MW : 255.0564(a), 255.0580(w)

m/z	INT.	
253.0029	6.3184	****
254.0062	0.8799	*
254.9983	100.0000	**
256.0011	16.3163	**********
257.0025	1.9336	*
258.0044	0.1643	
259.0063	0.0110	
260.0082	0.0006	

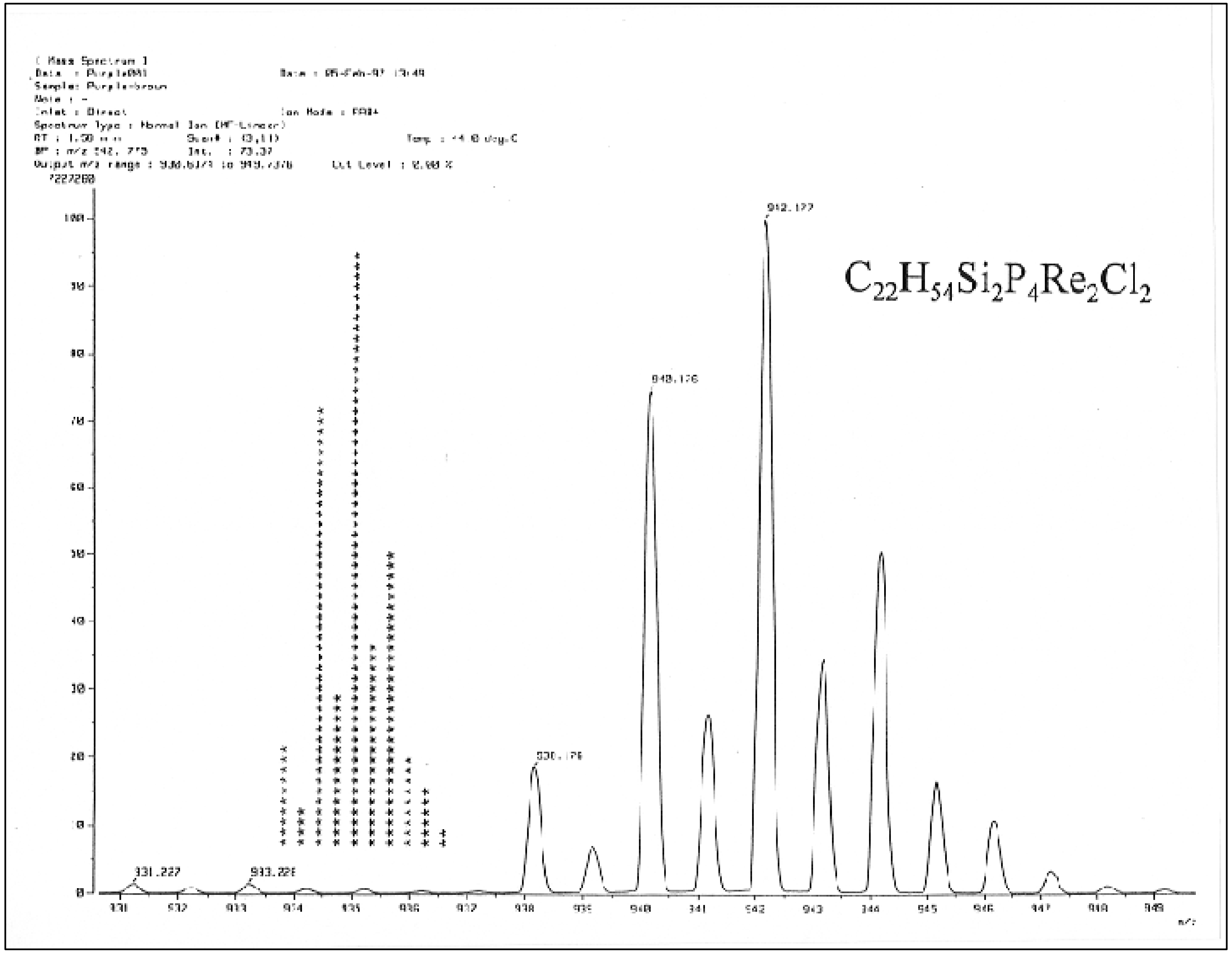
Metal element?



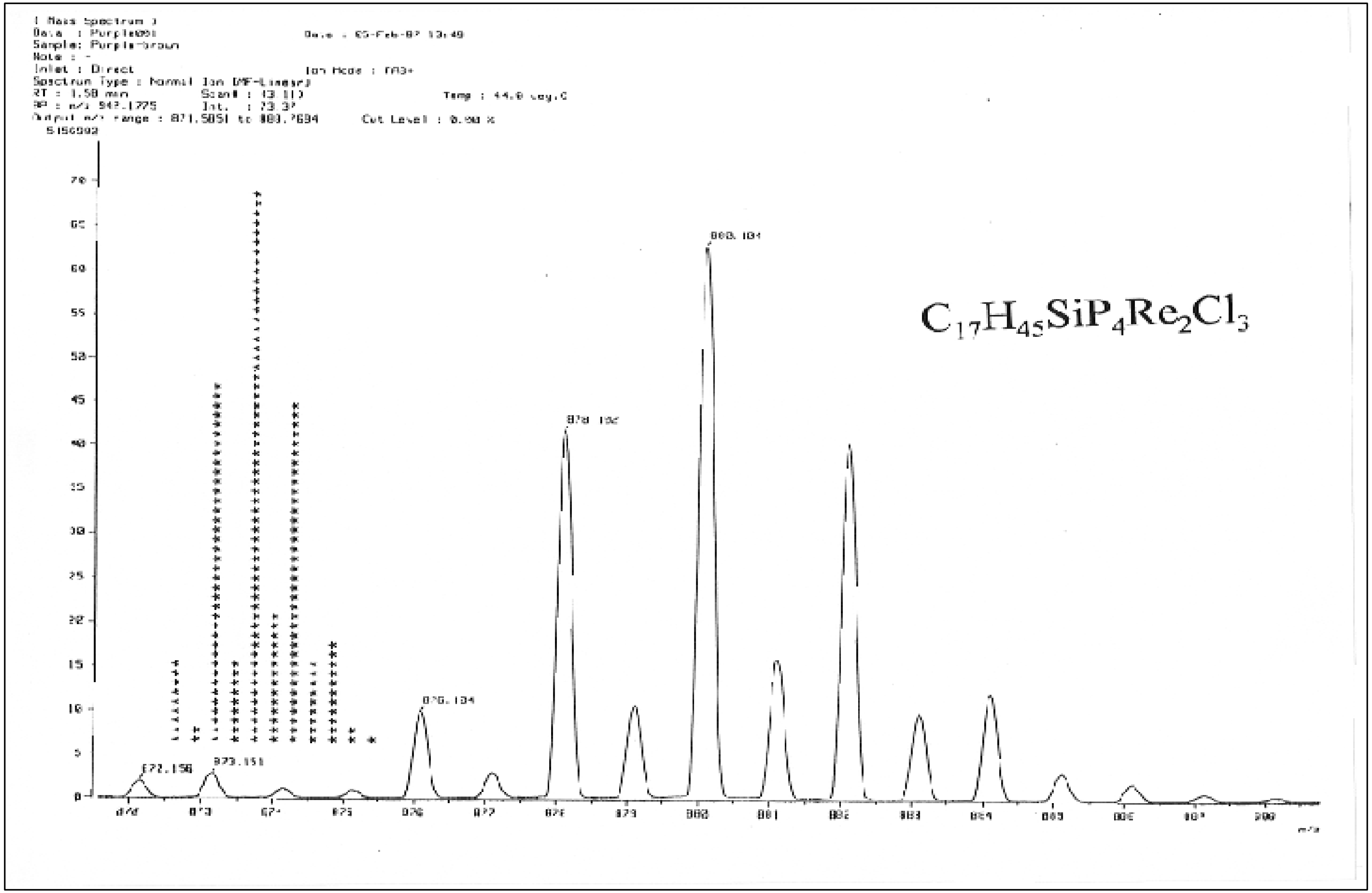


Transition Metal?
154.0
139.9
307.0
289.0
69.1
39.3
430.0
504.6
506.6
357.727
359.732
361.730
363.728
m/z INT.
357.8314 52.5411
358.8333 4.1110
359.8292 42.3079
360.8305 59.5933
361.8291 70.1255
362.8258 49.1450
363.8295 100.0000
364.8265 14.3657
365.8300 50.9182
366.8311 4.3808
367.8272 7.4037
368.8290 0.5222
369.8239 0.5004
370.8262 0.0310
371.8209 0.0182
372.8235 0.0010
373.8184 0.0004

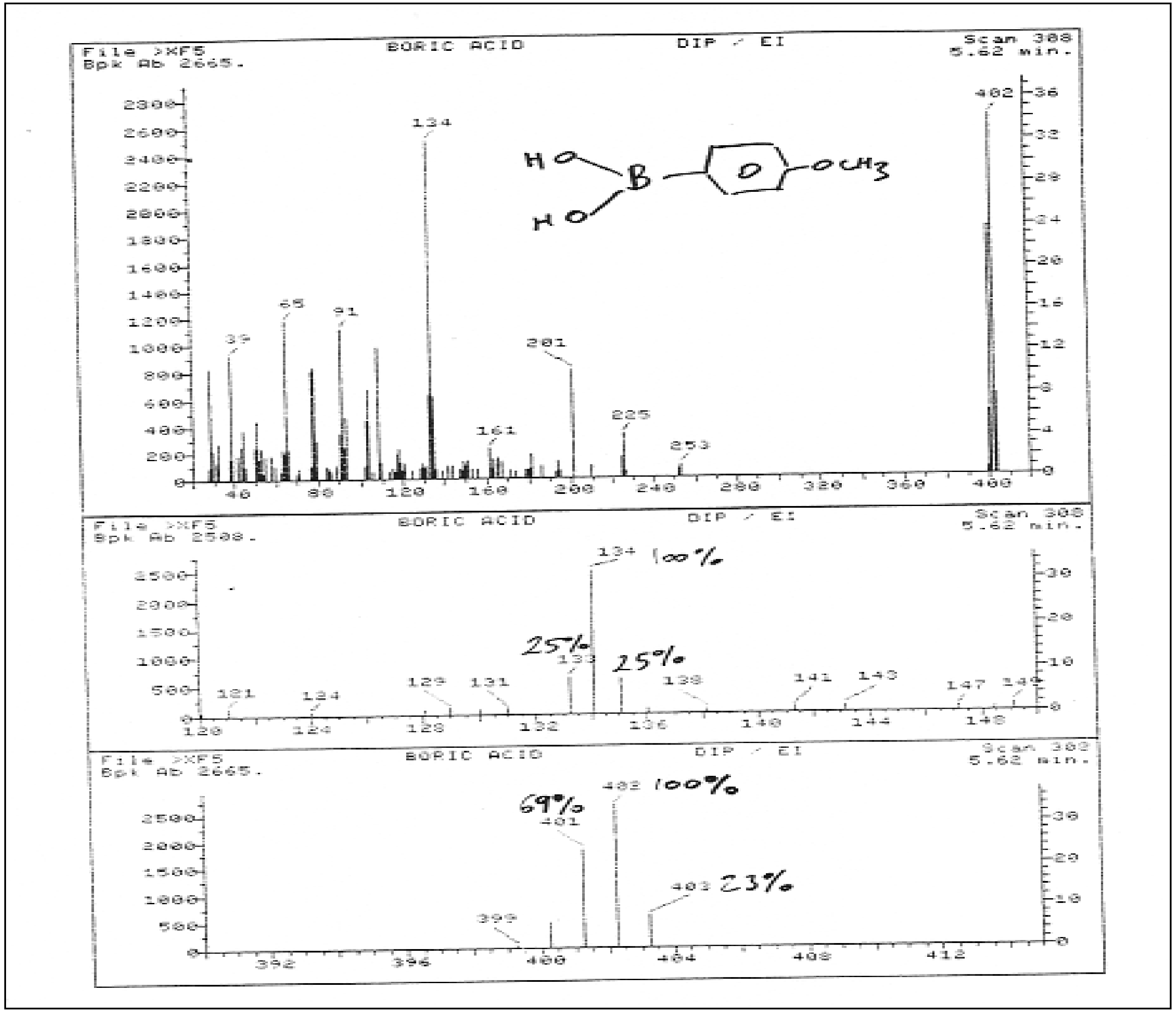
Isotope Ratios to Identify Chemical Compositions…No Accurate Mass!



Isotope Ratios to Identify Chemical Compositions…No Accurate Mass!



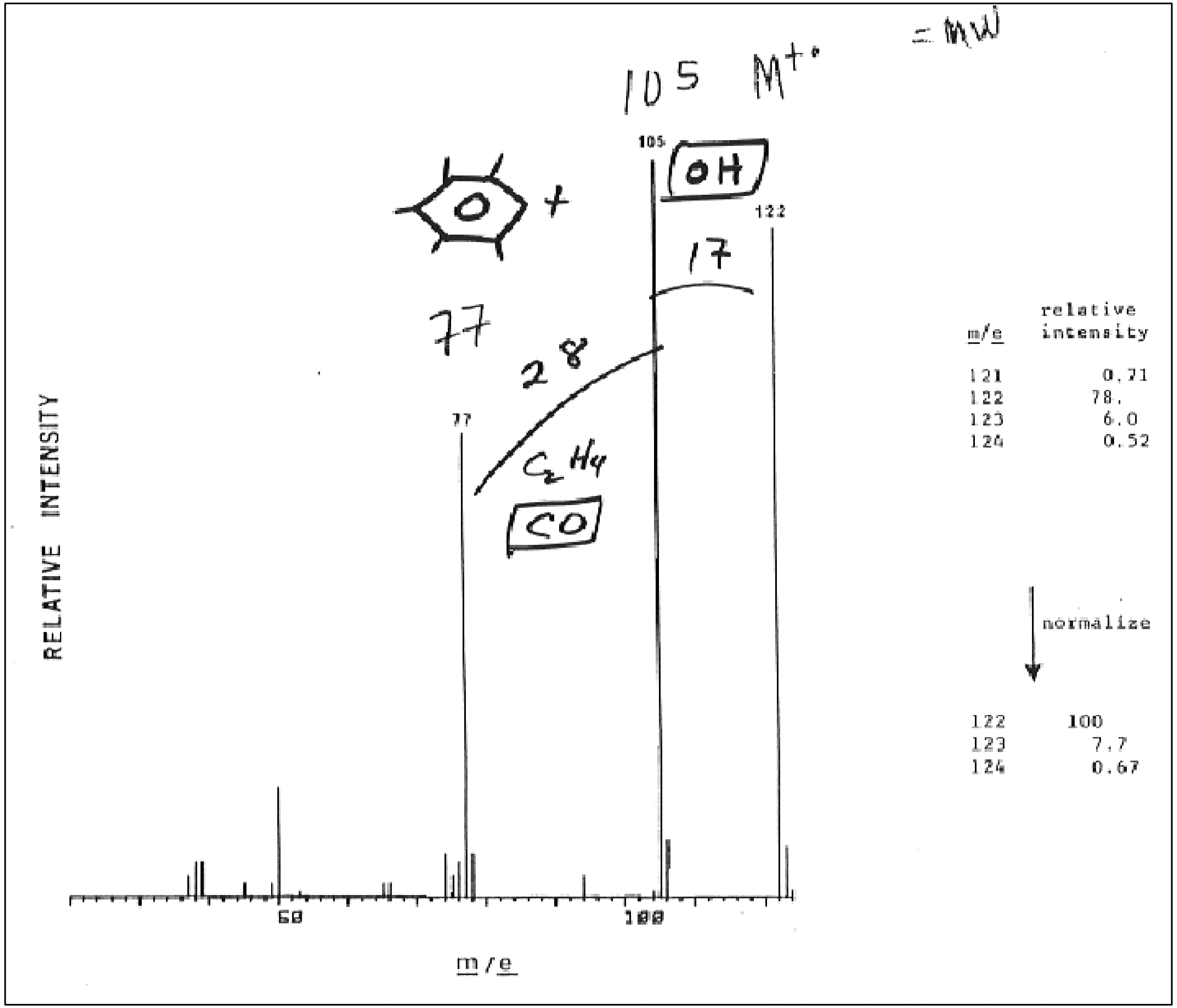
Isotope Ratios to Identify Chemical Compositions…No Accurate Mass!



^{10}B: 25%
^{11}B: 100%

What Happened?

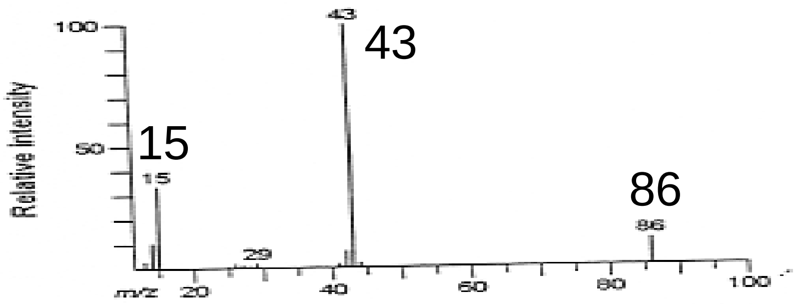
Renormalized Isotope Distribution in the $M^{+\cdot}$ Region



Try these!!

Unknown 5.2

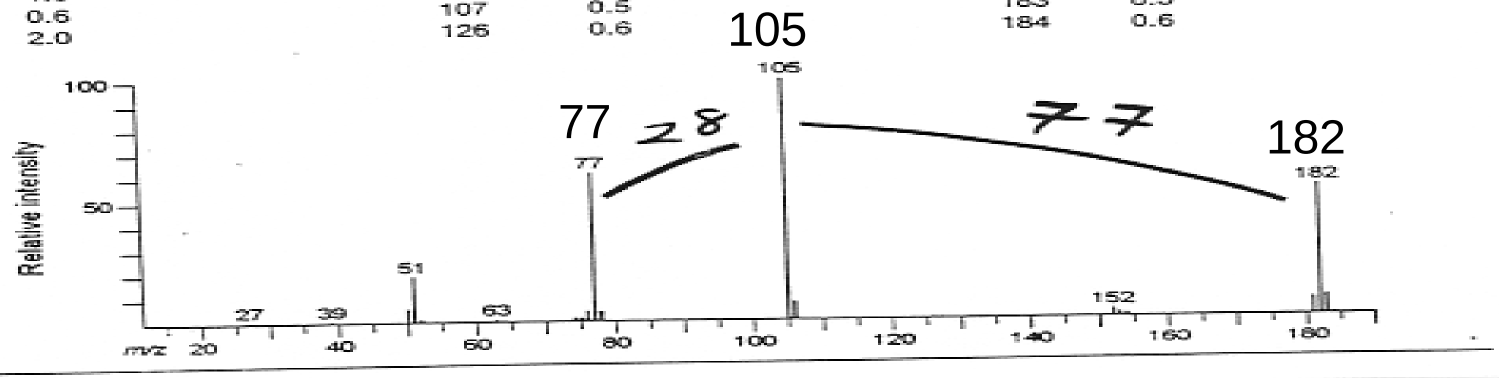
m/z	Int.
15	34.
16	0.5
27	1.4
28	1.0
29	2.0
41	1.8
42	7.2
43	100.
44	2.1
45	0.2
86	11.
87	0.4



Unknowns 5.3 and 5.4 are the spectra of larger molecules producing only a few prominent peaks. In evaluating the *m/z* 50 and 51 peaks of Unknown 5.3, don't forget that the importance of a peak decreases with decreasing mass, as well as with decreasing abundance.

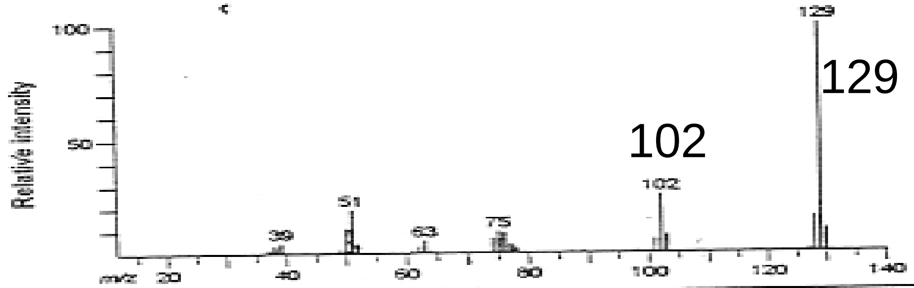
Unknown 5.3

m/z	Int.	*m/z*	Int.	*m/z*	Int.
38	0.4	75	1.7	127	0.4
39	1.1	76	4.3	151	1.1
50	6.2	77	62.	152	3.4
51	19.	78	4.2	153	1.8
52	1.4	104	0.4	154	1.4
53	0.3	105	100.	181	7.4
63	1.3	106	7.8	182	55.
64	0.6	107	0.5	183	8.3
74	2.0	126	0.6	184	0.6

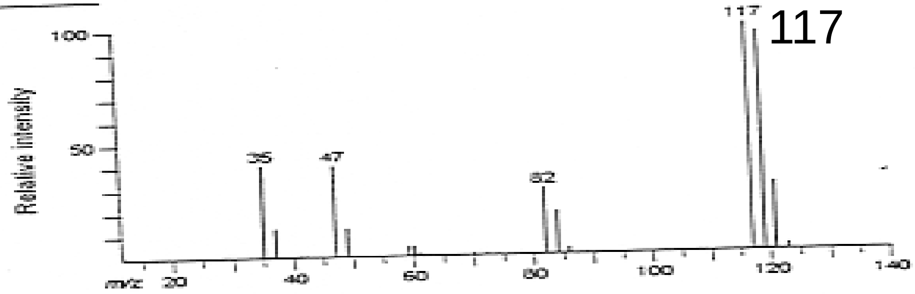


…and these!!

m/z	Int.	m/z	Int.	m/z	Int.
38	3.7	62	3.2	88	0.3
39	4.8	63	5.8	98	1.2
40	0.6	64	1.5	99	0.9
43	0.4	64.5	3.9	100	0.9
49	2.0	65	0.8	101	5.6
49.5	0.6	74	7.1	102	24.
50	12.	75	9.9	103	7.6
50.5	0.4	76	9.0	104	0.6
51	19.	77	3.8	127	1.8
51.5	1.0	78	2.5	128	16.
52	4.2	79	0.5	129	100.
53	0.4	81	0.4	130	10.
		87	0.9	131	0.5



m/z	Int.
35	41.
37	13.
41	1.2
42	0.8
47	40.
48	0.5
49	13.
58.5	4.7
59.5	4.5
60.5	1.4
70	1.4
72	0.9
82	29.
83	0.3
84	19.
85	0.2
86	2.9
117	100.
118	1.0
119	96.
120	1.0
121	30.
122	0.3
123	3.1



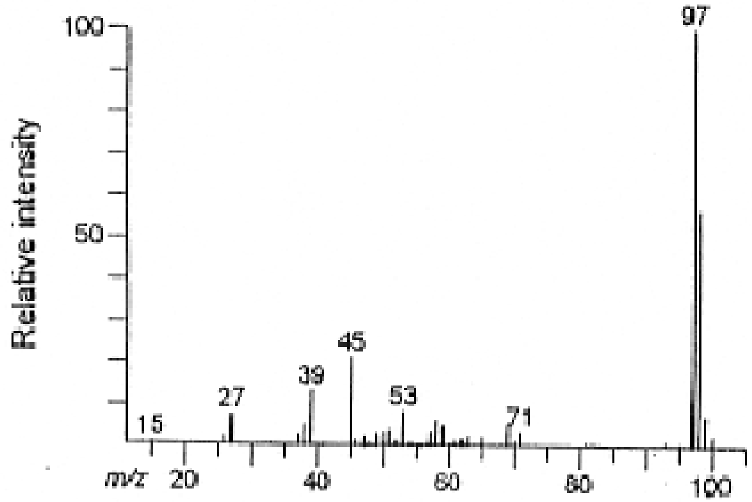
Halogen?

…and this!!

Unknown 4.3

m/z	Int.	*m/z*	Int.	*m/z*	Int.
15	1.1	52	1.4	70	1.3
26	3.3	53	8.7	71	3.8
27	8.0	54	0.3	72	0.5
37	4.1	57	3.9	81	0.9
38	4.9	58	6.6	82	1.0
39	13.	59	5.1	83	0.5
40	0.4	60	0.7	95	1.0
45	21.	61	1.6	96	0.6
46	0.9	62	1.9	97	100.
47	1.9	63	3.0	98	56.
48	0.8	64	0.5	99	7.6
49	3.1	65	2.4	100	2.4
50	3.6	68	0.7		
51	4.0	69	6.2		

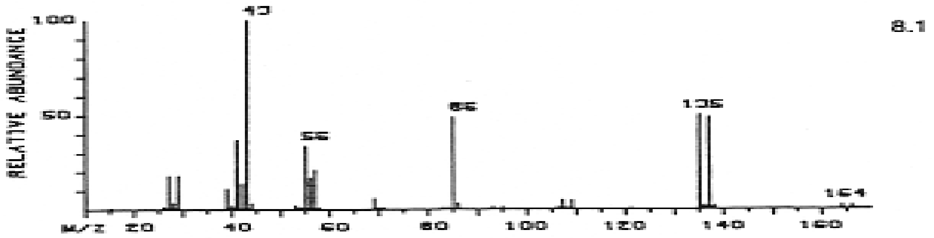
Use Isotope Distribution in the $M^{+\cdot}$ Region



…and these!!

Unknown 8.1. What is unusual about this ion series?

m/z	Rel. abund.	m/z	Rel. abund.	m/z	Rel. abund.
15	0.6	56	17.	106	0.6
27	18.	57	21.	107	5.0
28	3.5	58	0.9	108	0.7
29	18.	69	6.0	109	4.7
30	0.4	70	0.5	121	0.5
39	11.	71	0.5	123	0.4
40	2.1	83	0.7	135	50.
41	37.	84	0.6	136	2.2
42	14.	85	49.	137	49.
43	100.	86	3.2	138	2.1
44	3.4	93	1.2	164	2.2
55	34.	95	1.2	166	2.2

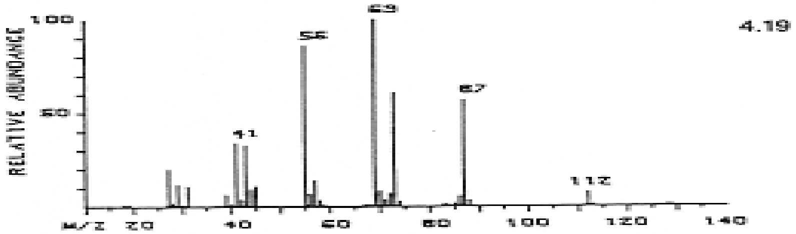


Unknown 4.19 involves some important points that have been covered previously. Be sure you understand its solution before proceeding. The utility of the *m/z* 96.5 peak formed by metastable ion decomposition will be discussed in Section 6.4.

Unknown 4.19

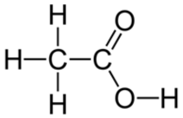
m/z	Rel. abund.	m/z	Rel. abund.	m/z	Rel. abund.
15	0.7	45	11.	84	0.8
18	1.1	46	0.2	85	1.7
19	0.9	55	86.	86	5.6
27	20.	56	6.6	87	57.
28	2.0	57	14.	88	3.4
29	12.	58	3.6	96.5	0.1
30	0.6	59	1.0	112*	7.4
31	11.	69	100.	113	1.0
39	6.4	70	8.4	127	0.3
40	1.0	71	3.7	128	0.5
41	34.	72	7.1	129*	0.9
42	4.0	73	61.	130	0.1
43	33.	74	2.8		
44	9.2	83	1.6		

*The elemental compositions of the *m/z* 112 and 129 ions are C_8H_{16} and $C_8H_{17}O$, respectively, by high-resolution mass spectrometry.

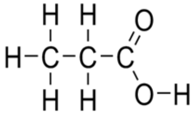


GC-MS Projects in the MS&P

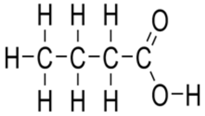
https://www.youtube.com/watch?v=7yxWBOPNkJ8



Acetic acid (acetate)

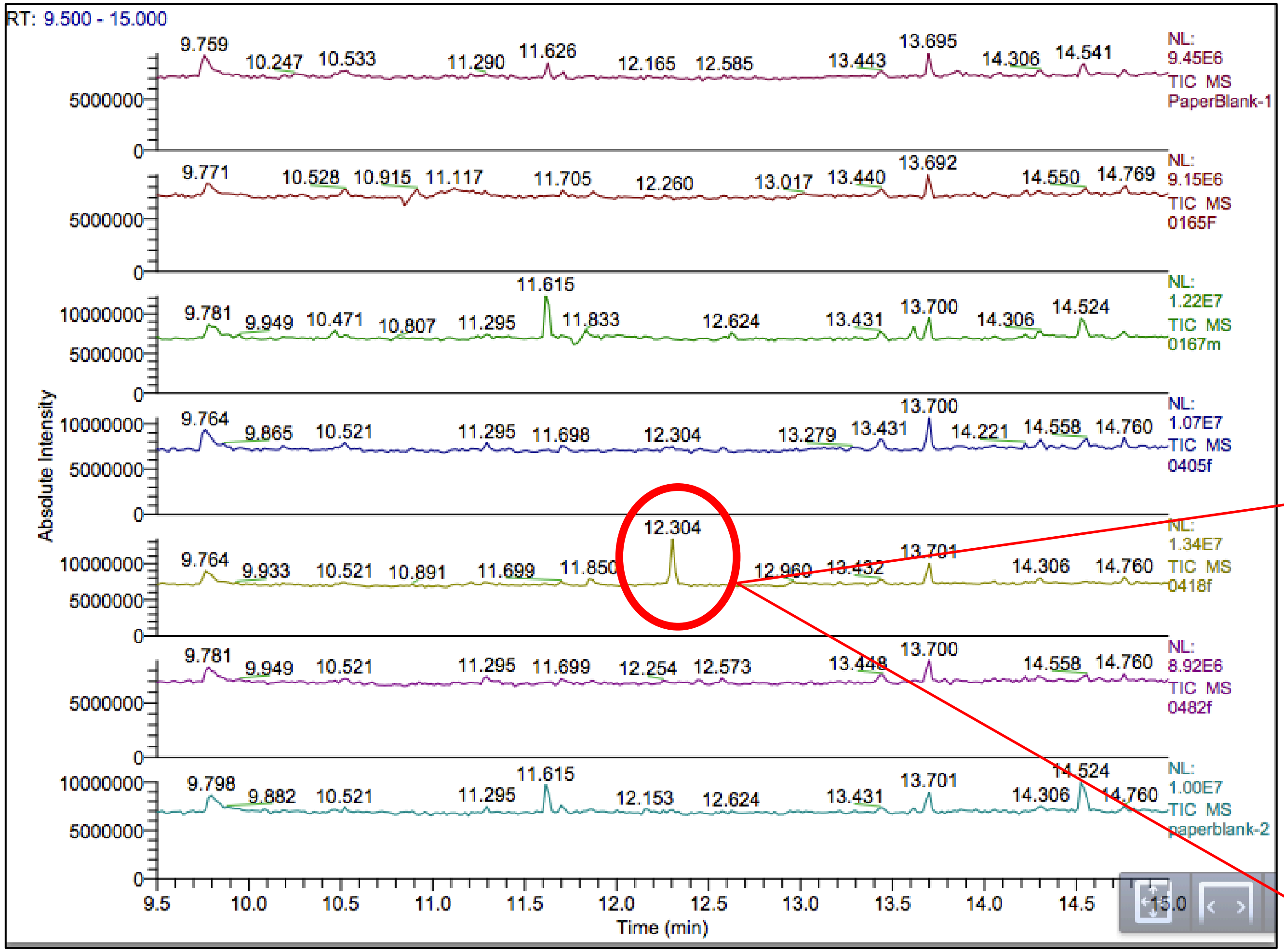


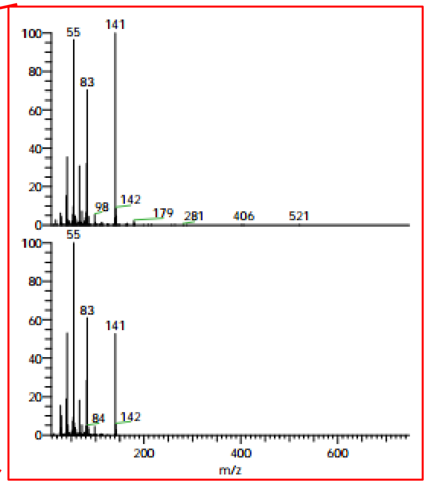
Propionic acid (propionate)



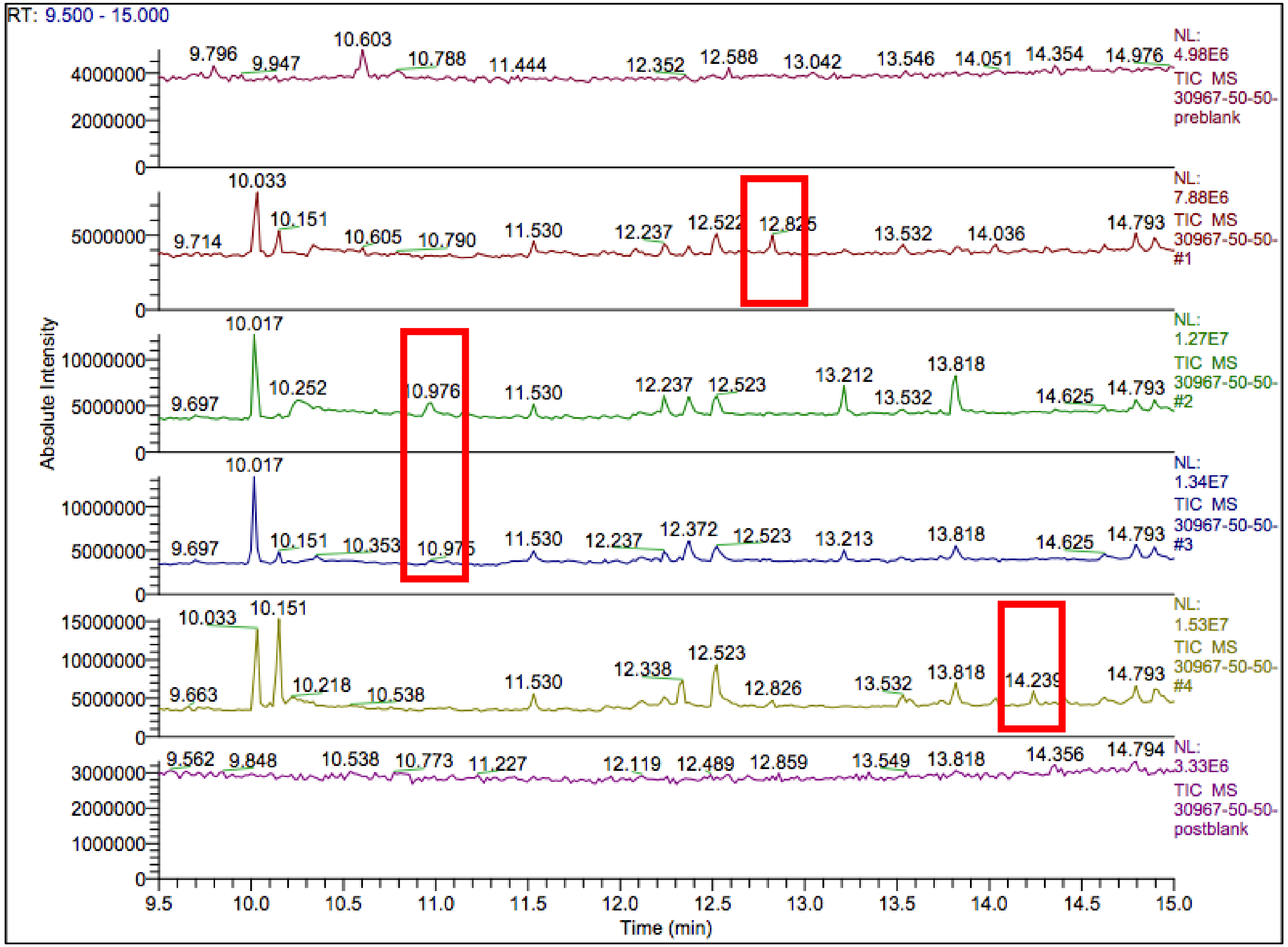
Butyric acid (butyrate)

Untargeted GC-MS Analysis of Spider Silk Droppings
-the hunt for hormones

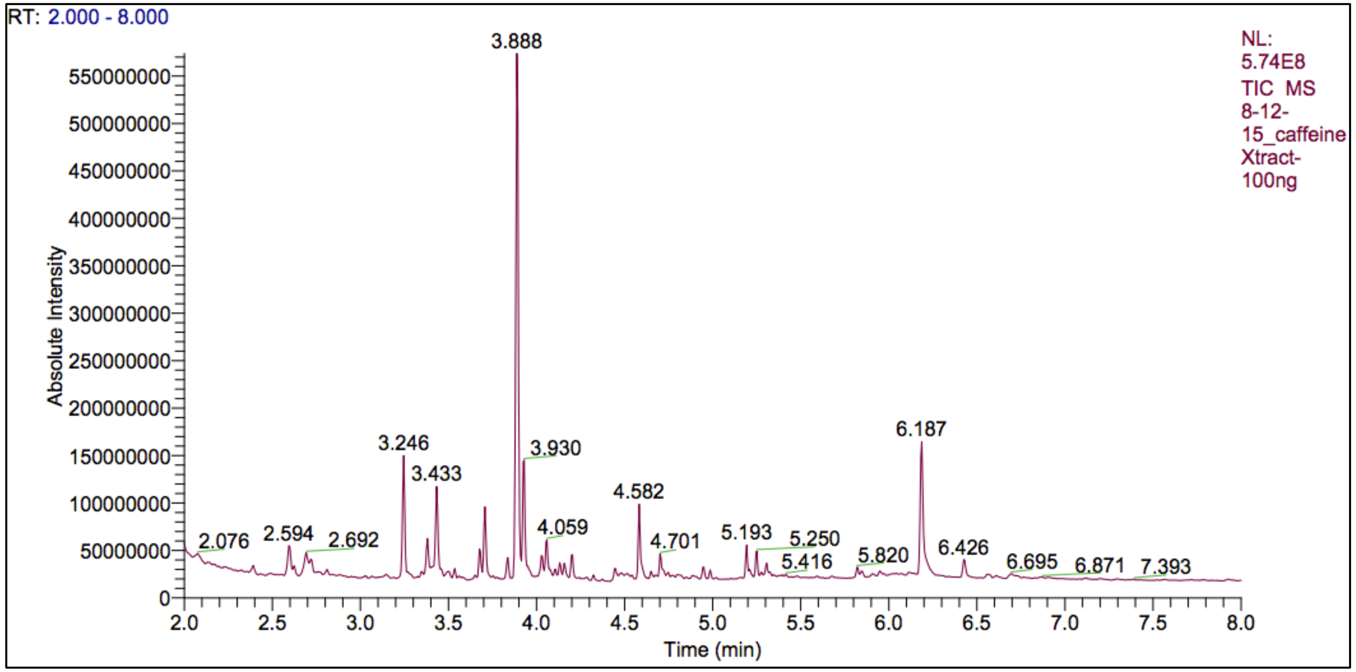


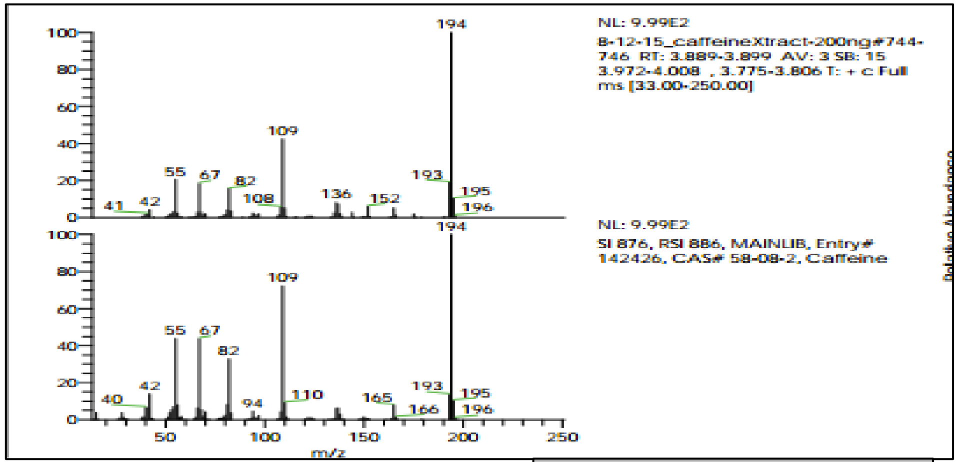


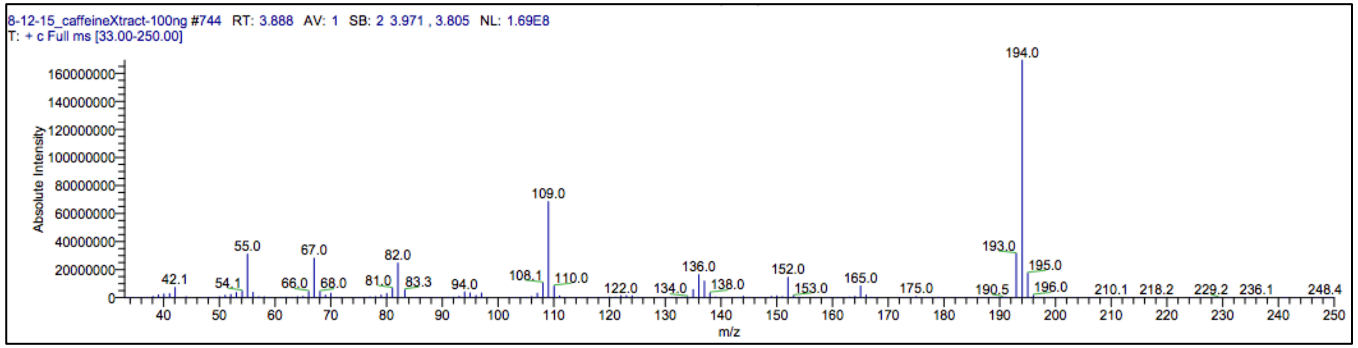
Untargeted GC-MS Analysis of Plants Exposed to Crude Oil



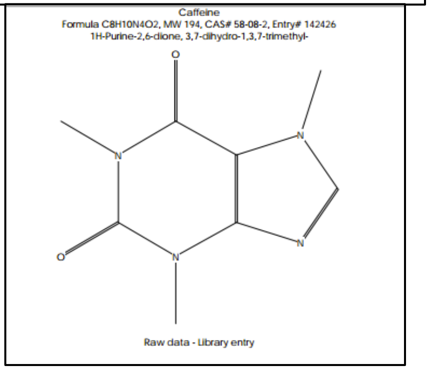
Targeted GC-MS Analysis of Caffeine in Urine-Full Scan



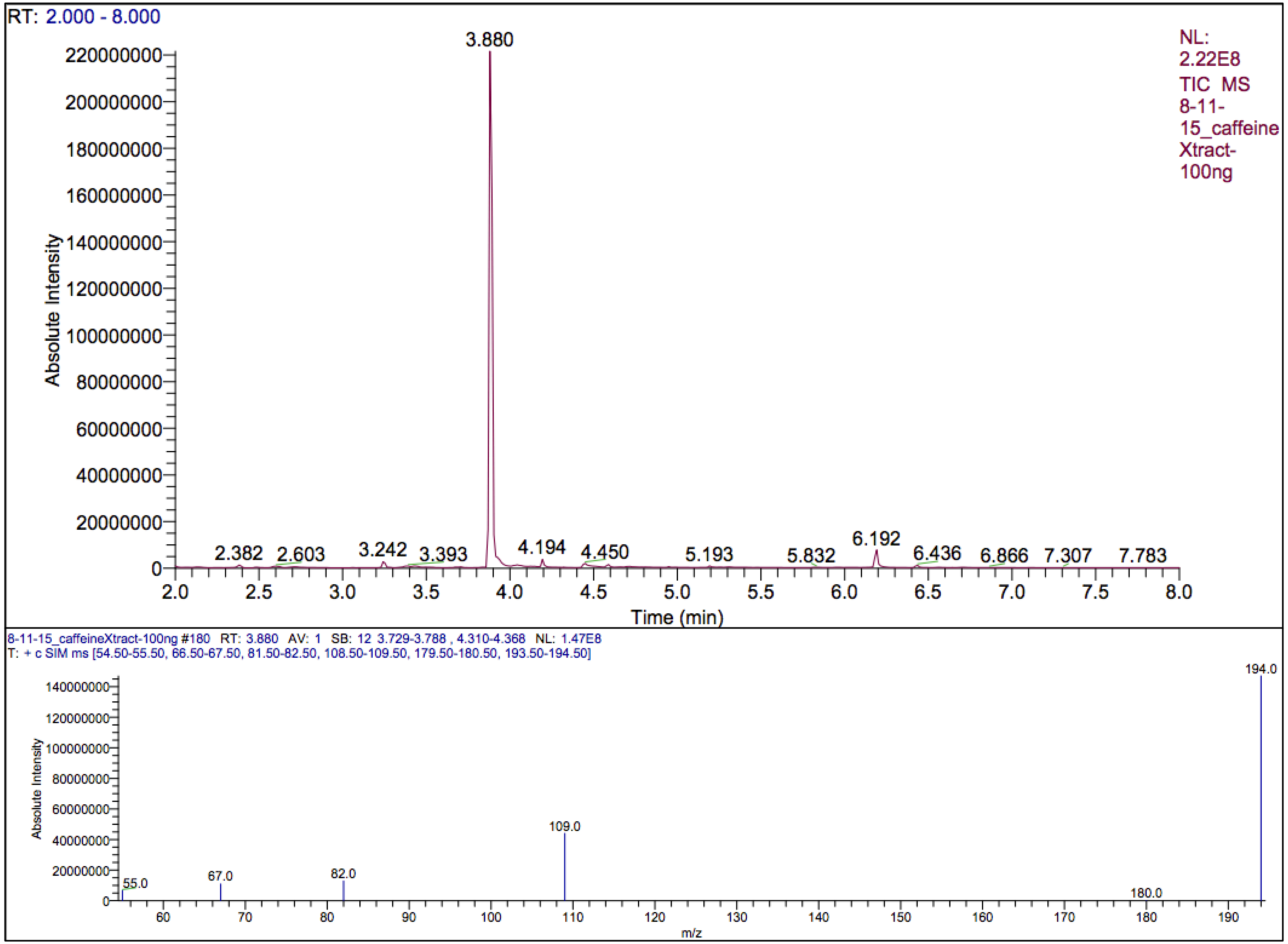




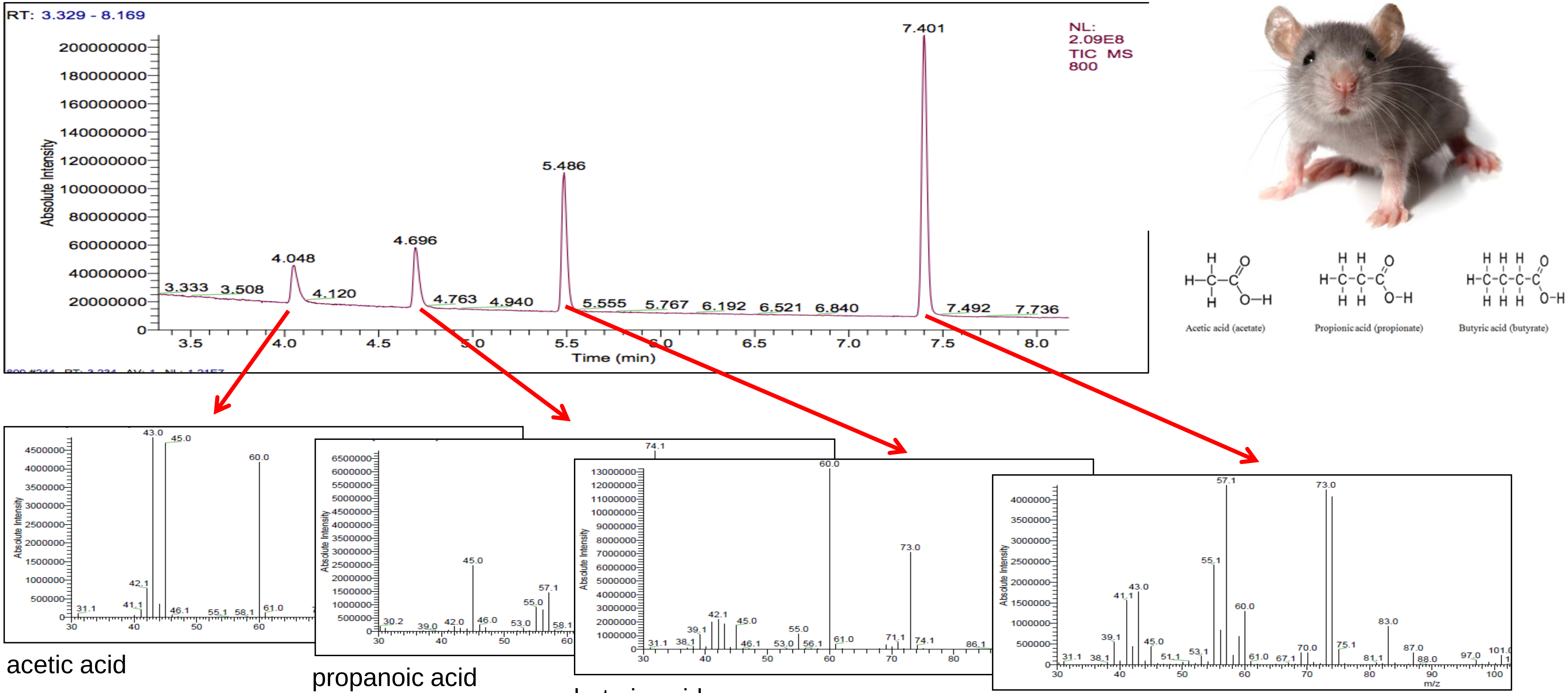
Hit	SI	RSI	Prob	Name	Library Name
1	876	886	95.38	Caffeine	MAINLIB
2	719	733	2.44	1,4-Dimethyl-4,5,7,8-tetrahydroimidazo-[4,5-	MAINLIB
3	670	874	0.50	2-Fluorobenzylamine, N,N-dibutyl	MAINLIB
4	663	681	0.38	2-(1,3-Dimethyl-2,6-dioxo-1,2,3,6-tetrahydro	MAINLIB
5	656	707	0.29	6-Amino-1,3-dimethyl-1,6-dihydro-pyrrolo[3,	MAINLIB
6	633	640	0.10	Proxyphylline	MAINLIB
7	624	637	0.07	1(2H)-Pyridinecarboxaldehyde, 3,4-dihydro-	MAINLIB
8	622	631	0.07	2H-Pyrazol-3-ol, 5-(2,5-dimethylthiophen-3-y	MAINLIB
9	621	629	0.06	2-(1,3-Dimethyl-2,6-dioxo-1,2,3,6-tetrahydro	MAINLIB
10	620	630	0.06	Valerolactimether, (nb)-O-[(diethylboryloxy	MAINLIB



Targeted GC-MS Analysis of Caffeine in Urine-SIM



Targeted GC-MS Analysis of SCFAs (Short Chain Fatty Acids) in Mouse Feces



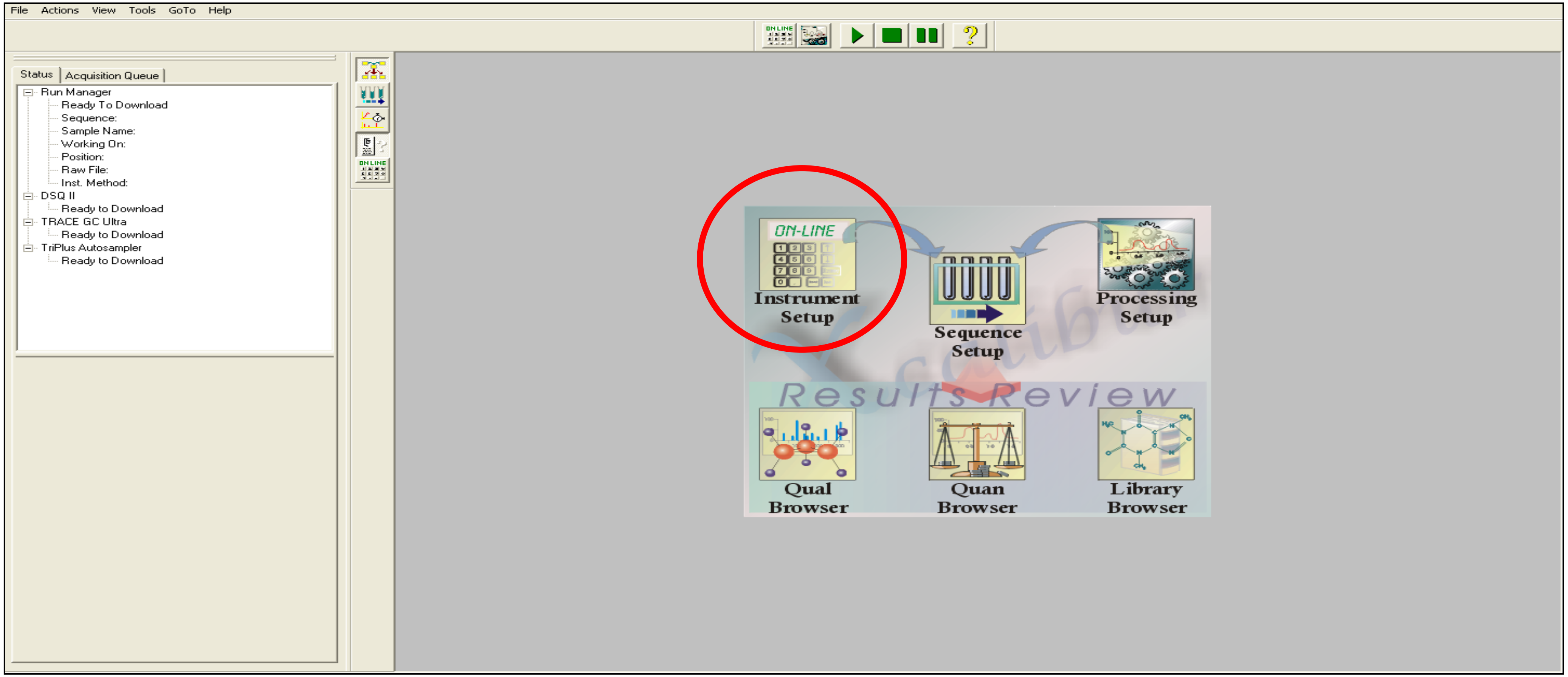
acetic acid

propanoic acid

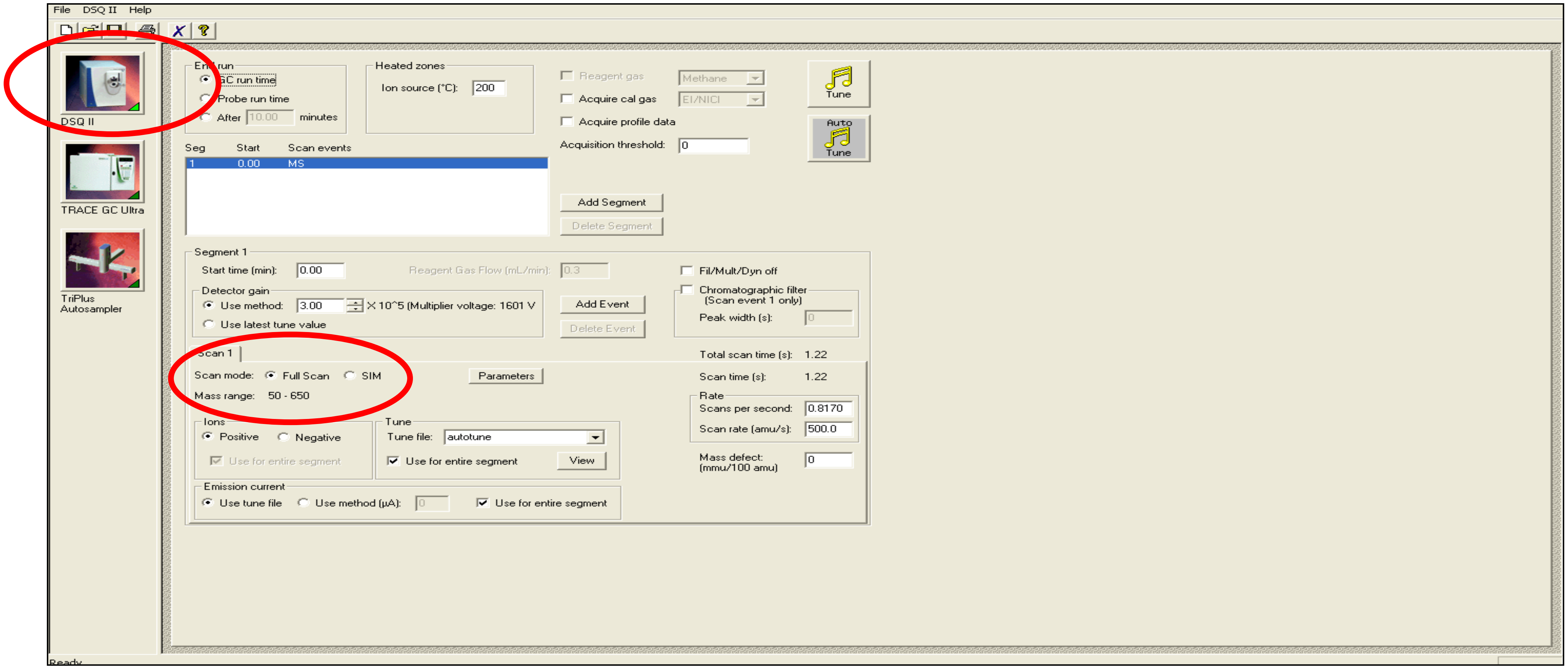
butyric acid

4-methyl valeric acid

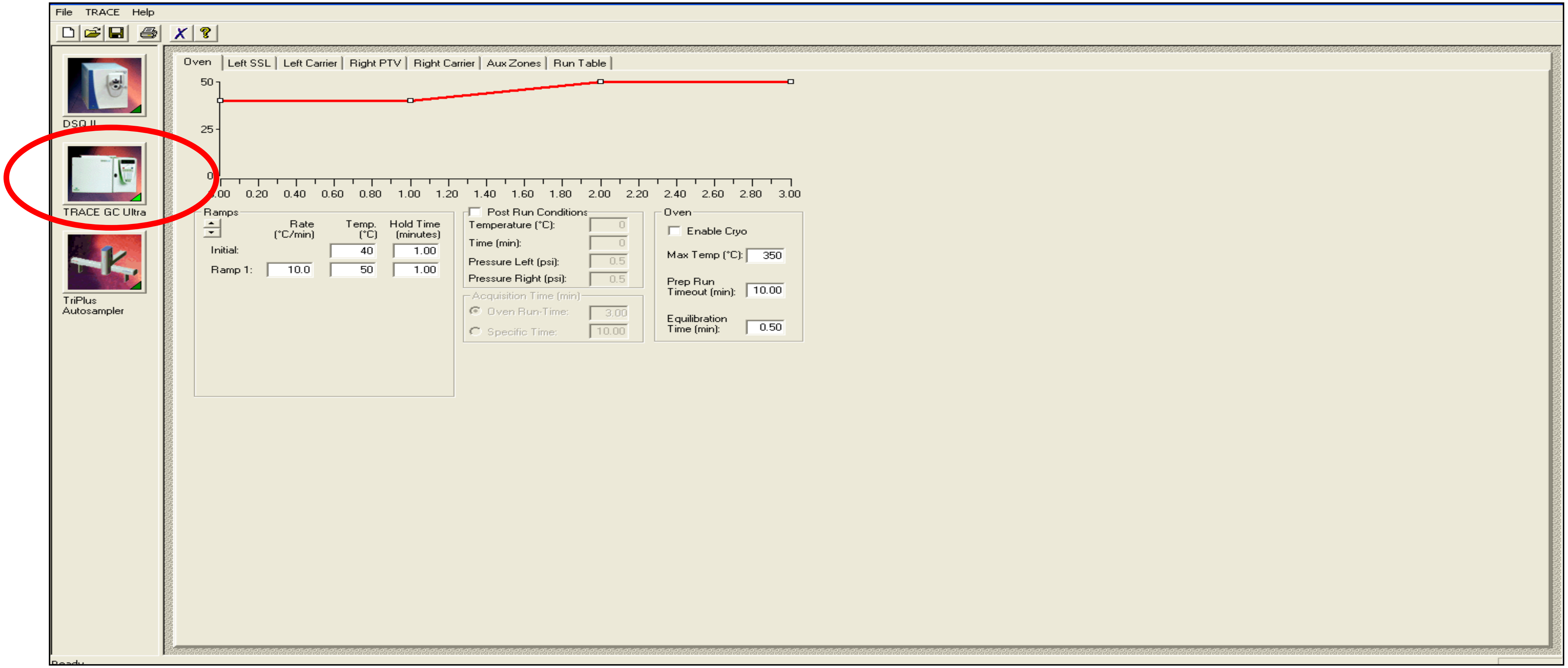
Quantitative GC-MS Analysis with Xcalibur



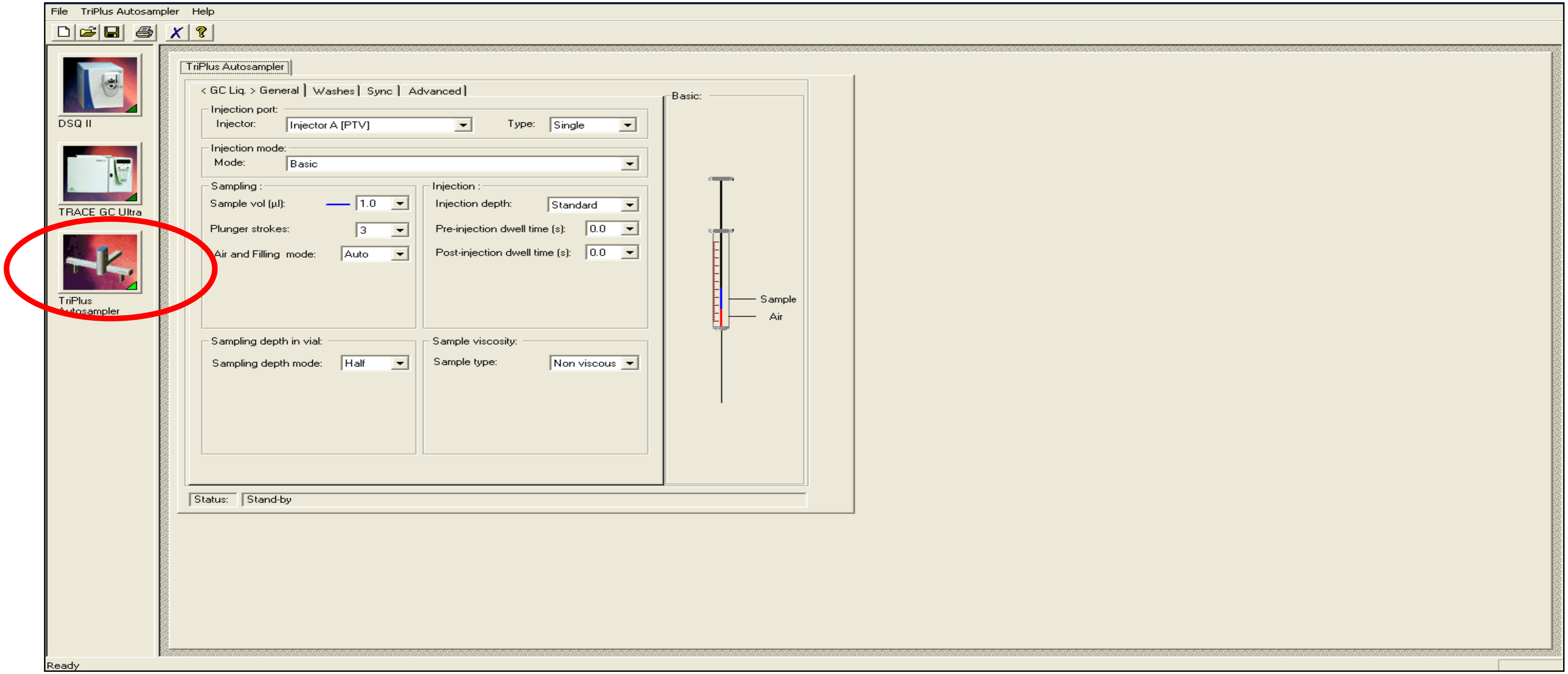
Xcalibur Method Setup-DSQ II



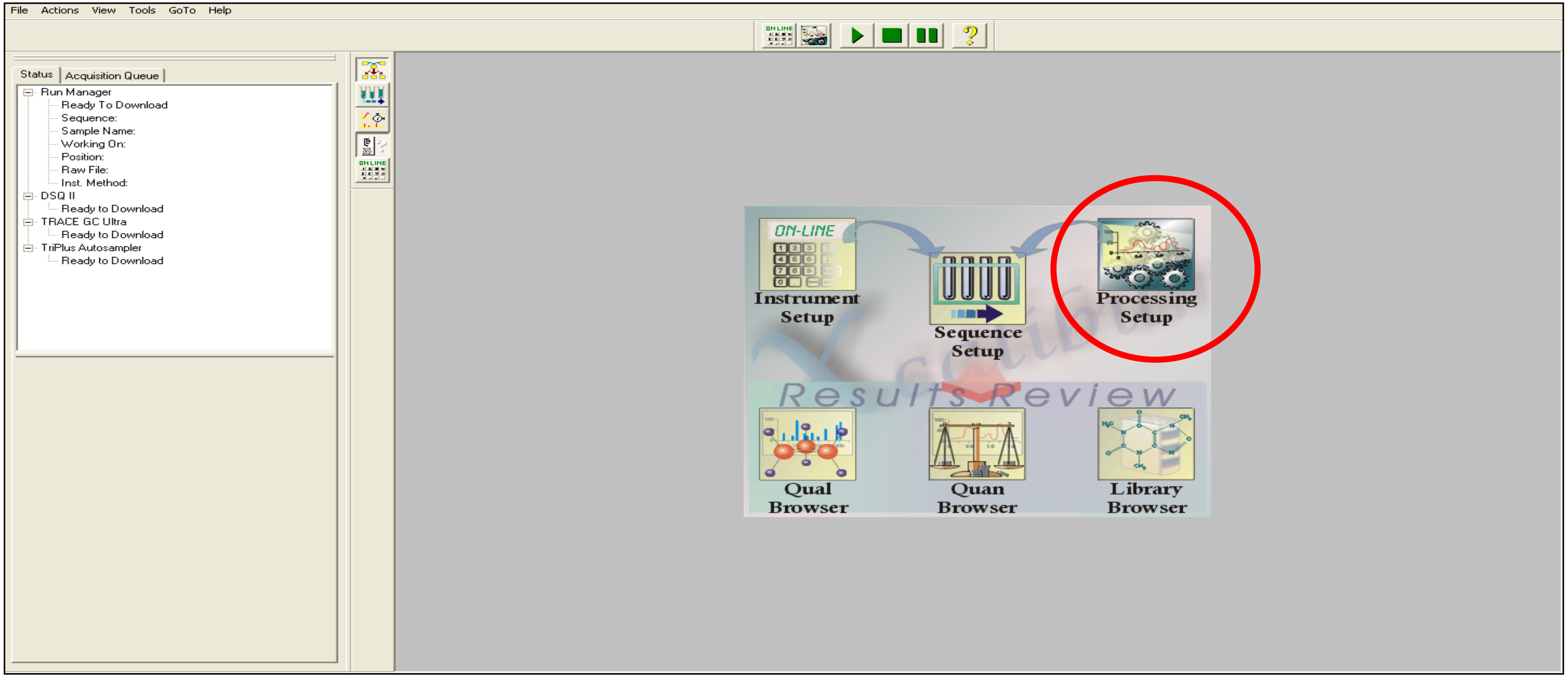
Xcalibur Method Setup-Trace GC Ultra



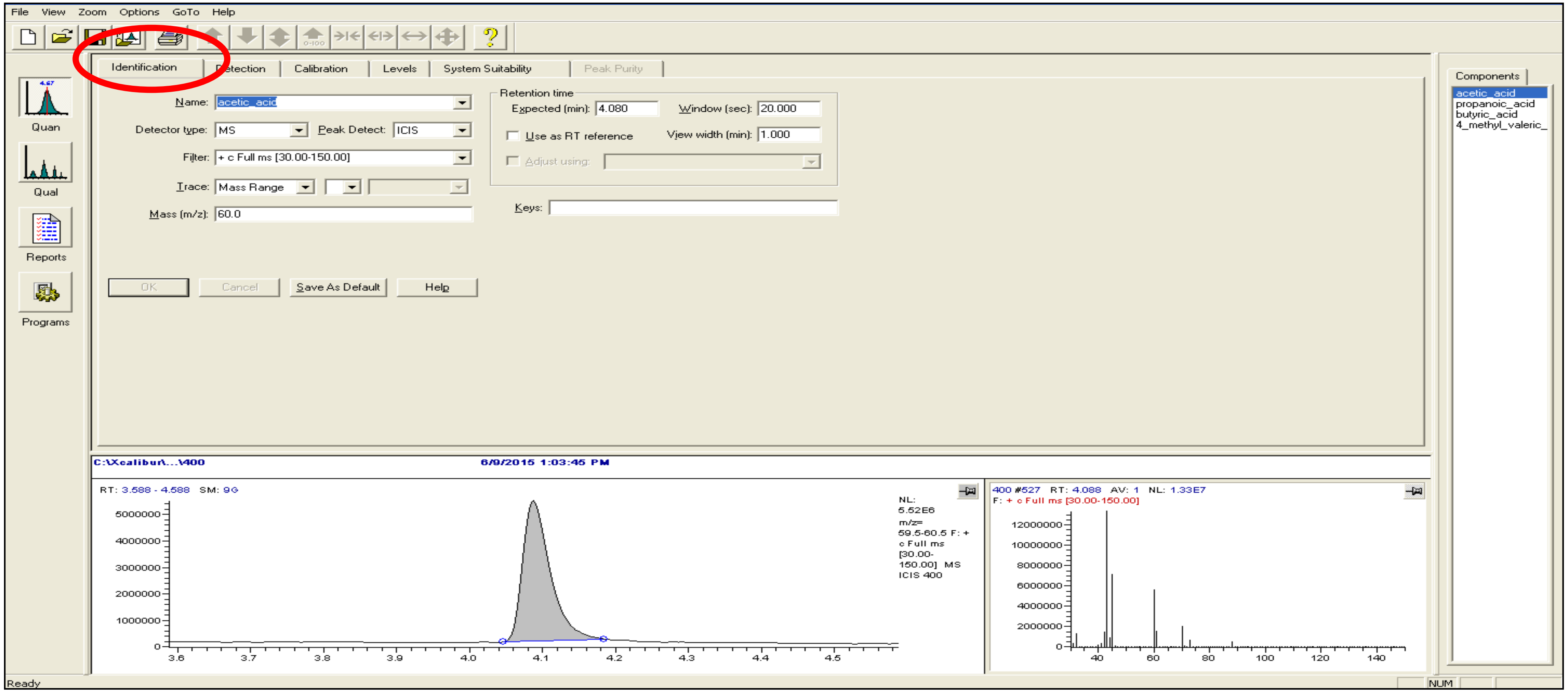
Xcalibur Method Setup-Autosampler



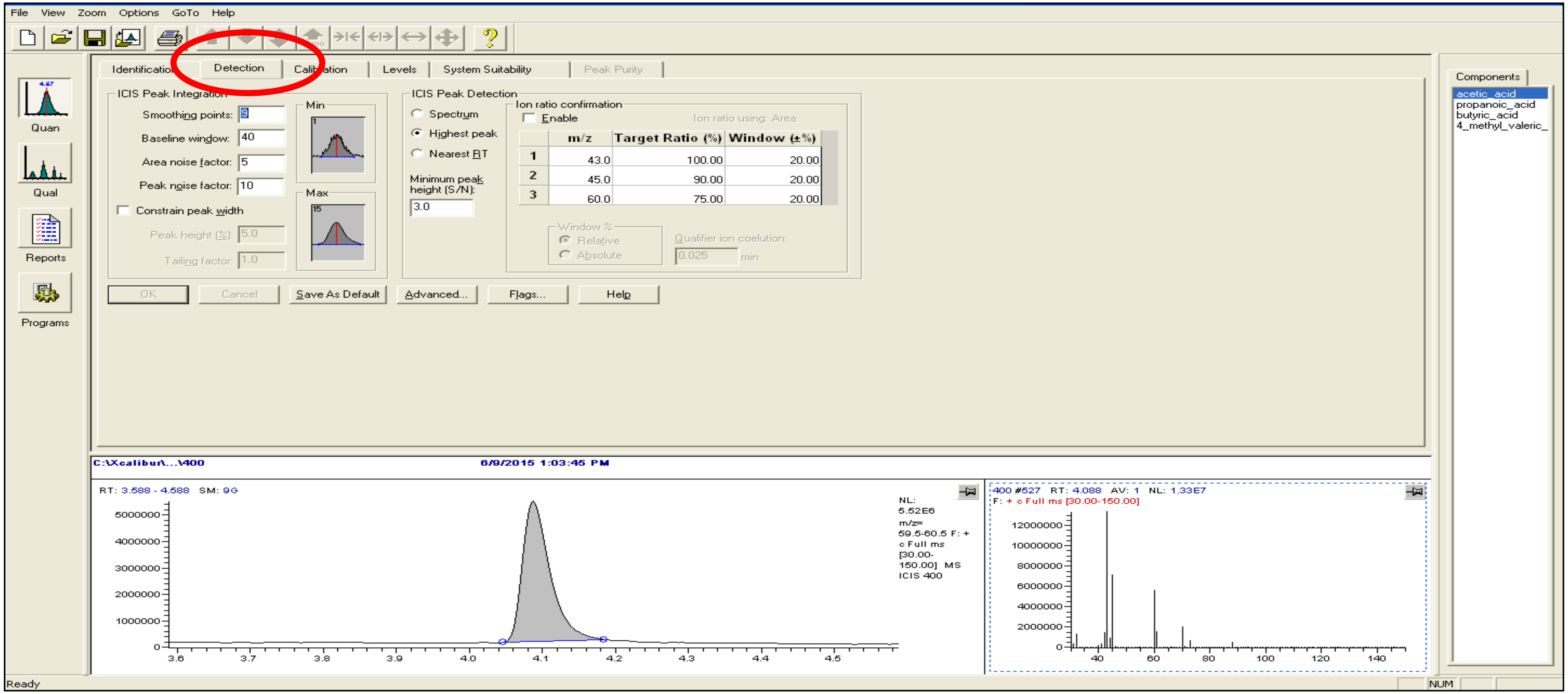
Xcalibur Processing Method Setup



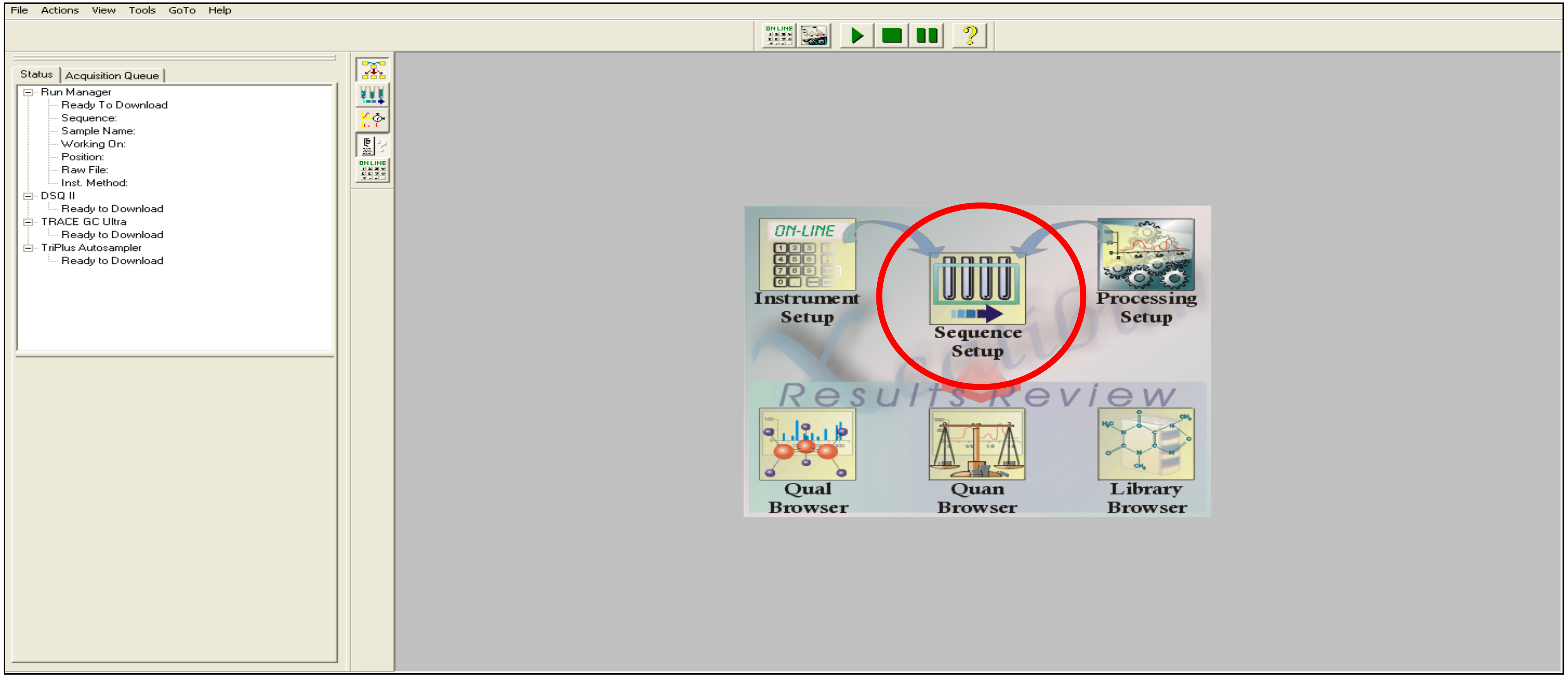
Xcalibur Processing Method Setup-Identification



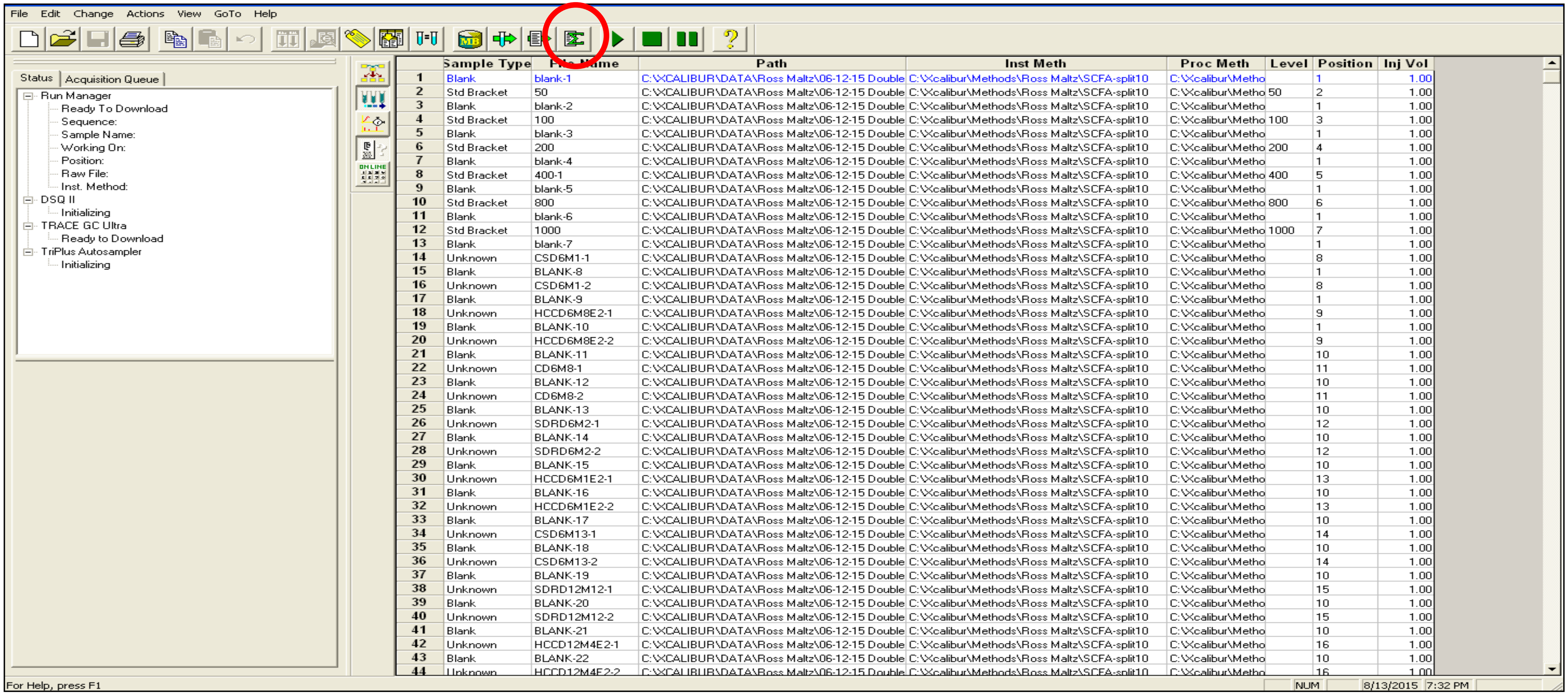
Xcalibur Processing Method Setup-Detection



Xcalibur Processing Data



Xcalibur Processing Data



	Sample Type	File Name	Path	Inst Meth	Proc Meth	Level	Position	Inj Vol
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2	Std Bracket	50	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho	50	2	1.00
3	Blank	blank-2	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		1	1.00
4	Std Bracket	100	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho	100	3	1.00
5	Blank	blank-3	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		1	1.00
6	Std Bracket	200	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho	200	4	1.00
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11	Blank	blank-6	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		1	1.00
12	Std Bracket	1000	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho	1000	7	1.00
13	Blank	blank-7	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		1	1.00
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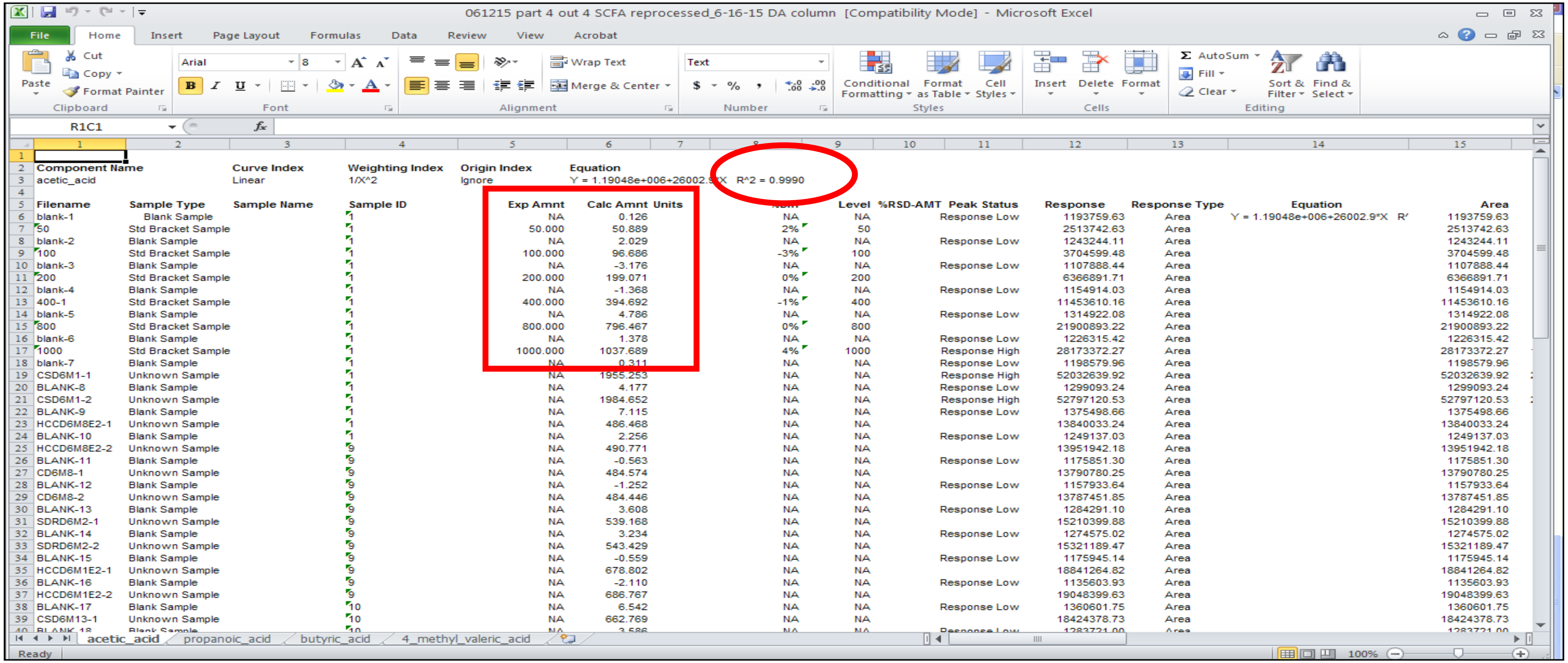
19	Blank	BLANK-10	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		1	1.00
20	Unknown	HCCD6M8E2-2	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		9	1.00
21	Blank	BLANK-11	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		10	1.00
22	Unknown	CD6M8-1	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		11	1.00
23	Blank	BLANK-12	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		10	1.00
24	Unknown	CD6M8-2	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		11	1.00
25	Blank	BLANK-13	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		10	1.00
26	Unknown	SDRD6M2-1	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		12	1.00
27	Blank	BLANK-14	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		10	1.00
28	Unknown	SDRD6M2-2	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		12	1.00
29	Blank	BLANK-15	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		10	1.00
30	Unknown	HCCD6M1E2-1	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		13	1.00
31	Blank	BLANK-16	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		10	1.00
32	Unknown	HCCD6M1E2-2	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		13	1.00
33	Blank	BLANK-17	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		10	1.00
34	Unknown	CSD6M13-1	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		14	1.00
35	Blank	BLANK-18	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		10	1.00
36	Unknown	CSD6M13-2	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		14	1.00
37	Blank	BLANK-19	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		10	1.00
38	Unknown	SDRD12M12-1	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		15	1.00
39	Blank	BLANK-20	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		10	1.00
40	Unknown	SDRD12M12-2	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		15	1.00
41	Blank	BLANK-21	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		10	1.00
42	Unknown	HCCD12M4E2-1	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		16	1.00
43	Blank	BLANK-22	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		10	1.00
44	Unknown	HCCD12M4E2-2	C:\XCALIBUR\DATA\Ross Maltz\06-12-15 Double	C:\Xcalibur\Methods\Ross Maltz\SCFA-split10	C:\Xcalibur\Metho		16	1.00

Xcalibur Exported Results



061215 part 4 out 4 SCFA reprocessed_6-16-15 DA column [Compatibility Mode] - Microsoft Excel

Component Name	Curve Index	Weighting Index	Origin Index	Equation	
acetic_acid	Linear	1/X^2	Ignore	Y = 1.19048e+006+26002.9*X	R^2 = 0.9990

Filename	Sample Type	Sample Name	Sample ID	Exp Amnt	Calc Amnt	Units	[illegible]	Level	%RSD-AMT	Peak Status	Response	Response Type	Equation	Area
blank-1	Blank Sample		1	NA	0.126		NA	NA		Response Low	1193759.63	Area	Y = 1.19048e+006+26002.9*X R'	1193759.63
50	Std Bracket Sample		1	50.000	50.889		2%	50			2513742.63	Area		2513742.63
blank-2	Blank Sample		1	NA	2.029		NA	NA		Response Low	1243244.11	Area		1243244.11
100	Std Bracket Sample		1	100.000	96.686		-3%	100			3704599.48	Area		3704599.48
blank-3	Blank Sample		1	NA	-3.176		NA	NA		Response Low	1107888.44	Area		1107888.44
200	Std Bracket Sample		1	200.000	199.071		0%	200			6366891.71	Area		6366891.71
blank-4	Blank Sample		1	NA	-1.368		NA	NA		Response Low	1154914.03	Area		1154914.03
400-1	Std Bracket Sample		1	400.000	394.692		-1%	400			11453610.16	Area		11453610.16
blank-5	Blank Sample		1	NA	4.786		NA	NA		Response Low	1314922.08	Area		1314922.08
800	Std Bracket Sample		1	800.000	796.467		0%	800			21900893.22	Area		21900893.22
blank-6	Blank Sample		1	NA	1.378		NA	NA		Response Low	1226315.42	Area		1226315.42
1000	Std Bracket Sample		1	1000.000	1037.689		4%	1000		Response High	28173372.27	Area		28173372.27
blank-7	Blank Sample		1	NA	0.311		NA	NA		Response Low	1198579.96	Area		1198579.96
CSD6M1-1	Unknown Sample		1	NA	1955.253		NA	NA		Response High	52032639.92	Area		52032639.92
BLANK-8	Blank Sample		1	NA	4.177		NA	NA		Response Low	1299093.24	Area		1299093.24
CSD6M1-2	Unknown Sample		1	NA	1984.652		NA	NA		Response High	52797120.53	Area		52797120.53
BLANK-9	Blank Sample		1	NA	7.115		NA	NA		Response Low	1375498.66	Area		1375498.66
HCCD6M8E2-1	Unknown Sample		1	NA	486.468		NA	NA			13840033.24	Area		13840033.24
BLANK-10	Blank Sample		1	NA	2.256		NA	NA		Response Low	1249137.03	Area		1249137.03
HCCD6M8E2-2	Unknown Sample		9	NA	490.771		NA	NA			13951942.18	Area		13951942.18
BLANK-11	Blank Sample		9	NA	-0.563		NA	NA		Response Low	1175851.30	Area		1175851.30
CD6M8-1	Unknown Sample		9	NA	484.574		NA	NA			13790780.25	Area		13790780.25
BLANK-12	Blank Sample		9	NA	-1.252		NA	NA		Response Low	1157933.64	Area		1157933.64
CD6M8-2	Unknown Sample		9	NA	484.446		NA	NA			13787451.85	Area		13787451.85
BLANK-13	Blank Sample		9	NA	3.608		NA	NA		Response Low	1284291.10	Area		1284291.10
SDRD6M2-1	Unknown Sample		9	NA	539.168		NA	NA			15210399.88	Area		15210399.88
BLANK-14	Blank Sample		9	NA	3.234		NA	NA		Response Low	1274575.02	Area		1274575.02
SDRD6M2-2	Unknown Sample		9	NA	543.429		NA	NA			15321189.47	Area		15321189.47
BLANK-15	Blank Sample		9	NA	-0.559		NA	NA		Response Low	1175945.14	Area		1175945.14
HCCD6M1E2-1	Unknown Sample		9	NA	678.802		NA	NA			18841264.82	Area		18841264.82
BLANK-16	Blank Sample		9	NA	-2.110		NA	NA		Response Low	1135603.93	Area		1135603.93
HCCD6M1E2-2	Unknown Sample		9	NA	686.767		NA	NA			19048399.63	Area		19048399.63
BLANK-17	Blank Sample		10	NA	6.542		NA	NA		Response Low	1360601.75	Area		1360601.75
CSD6M13-1	Unknown Sample		10	NA	662.769		NA	NA			18424378.73	Area		18424378.73

acetic_acid | propanoic_acid | butyric_acid | 4_methyl_valeric_acid

Make Discoveries