you-try-it-10.xlsx

ver. 9/7/2018

For use with:

Brian M. Tissue, *Basics of Analytical Chemistry and Chemical Equilibria,* (John Wiley: New York, 2013).

http://www.achem.org

Worksheets in this file

notes	This page with background information.
10.A TLC	Calculating R_F to identify components in a mixture.
10.B quantitation	Determining unknown concentrations with an internal standard.
10.C resolution	Resolution of chromatographic peaks.
10.D mass-spec	Fingerprint analysis of mass spectra.

Background

Refer to Chapter 10 in the text for equations and explanations.

Each worksheet has instructions in the blue shaded box.

For step-by-step help see you-try-it-10guide.pdf.

You-Try-It 10.A **TLC**

Table 10.A.1 lists the spot positions for a standard solution and Table 10.A.2 lists results for an unknown mixture.

1. Calculate R_F for each spot in the reference mixture.
2. Calculate R_F for each spot in the unknown mixture and identify analytes.

Table 10.A.1 Reference

spot	analyte	migration distance (cm)	R_F
1	unknown	0.2	
2	unknown	0.4	
3	tyrosine	0.6	
4	lysine	1.1	
5	glycine	2.0	
6	alanine/tryptophan	2.6	
7	methionine	3.0	
8	phenylanlanine	3.5	
9	valine	3.8	
10	isoleucine	4.3	
11	leucine	4.5	
12	proline	5.3	
	solvent front	6.2	

Table 10.A.2 Unknown mixture

spot	migration distance (cm)	R_F	analyte
1	0.4		
2	1.1		
3	3.4		
4	4.9		
5	5.2		
6	6.0		
	7.1		solvent front

data adapted from: Poole, C. F.; Poole, S. K. *Analytical Chemistry* , **1989**, *61* , 12571.

You-Try-It 10.B Quantitation with Internal Standards

Table 10.B.1 lists the retention times and integrated areas for a standard mixture.
Table 10.B.2 and 10.B.3 lists the retention times and integrated areas for two unknown mixtures.

1. Use the peak area data in Table 10.B.1 to determine sensitivity factors for each analyte. The standard mixture contained 2000 ppm of each analyte in 40:60 ethanol-water solvent.
2. Use the sensitivity factors and the data in Tables 10.B.2 and 10.B.3 to determine the concentration of each analyte. The internal standard is spiked in at 2000 ppm.

Table 10.B.1

Peak	Retention Time (min)	identity	Concentration (ppm)	Peak Area (µV * s)	Relative Sensitivity
1	1.51	methanol	2000	2370	
2	1.79	ethanol	40%	949746	
3	1.97	2-propanol	2000	3853	
4	2.46	1-propanol	2000	4073	
5	3.15	ethyl acetate	2000	1424	
6	3.34	2-methyl-1-propanol	2000	4291	
7	3.98	1-butanol (IS)	2000	3628	
8	5.45	3-methyl-1-butanol	2000	4428	
9	5.51	2-methyl-1-butanol	2000	3812	

Table 10.B.2 Gas chromatographic data for a bourbon whiskey spiked with 2000-ppm IS.

Peak	Retention Time (min)	identity	Peak Area (µV * s)	Relative Sensitivity	Concentration (ppm)
1	1.54	methanol	83.5		
2	1.85	ethanol	1943410	---	---
3		2-propanol	ND		
4	2.49	1-propanol	131.9		
5		ethyl acetate	213.8		
6		2-methyl-1-propanol	832.5		
7		1-butanol (IS)	5469.7		
8		3-methyl-1-butanol	2724.7		
9	1.97	2-methyl-1-butanol	1122.2		

Table 10.B.3 Gas chromatographic data for an Irish whiskey spiked with 2000-ppm IS.

Peak	Retention Time (min)	identity	Peak Area (µV * s)	Relative Sensitivity	Concentration (ppm)
1	1.56	methanol	83.5		
2	1.85	ethanol	1680398	---	---
3	---	2-propanol	ND		
4	2.5	1-propanol	231.8		
5	3.17	ethyl acetate	47.1		
6	3.37	2-methyl-1-propanol	103.6		
7	3.99	1-butanol (IS)	5267.7		
8	5.46	3-methyl-1-butanol	323		
9	5.52	2-methyl-1-butanol	119.9		

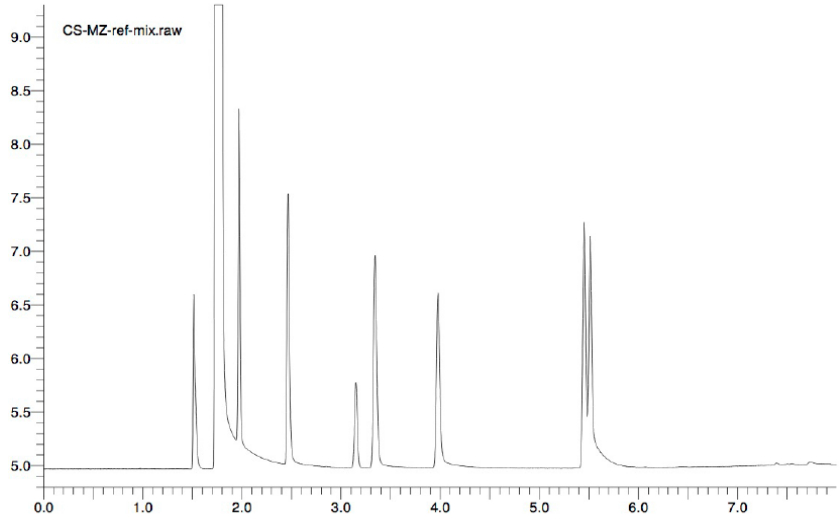


Figure 10.B.1. Chromatogram of 2000-ppm standard solution.

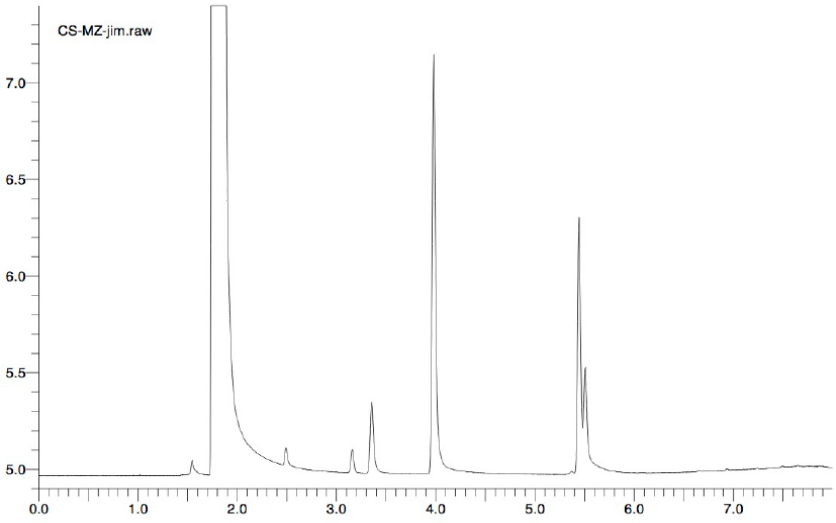


Figure 10.B.2. Chromatogram of a whiskey spiked with 2000-ppm internal standard.

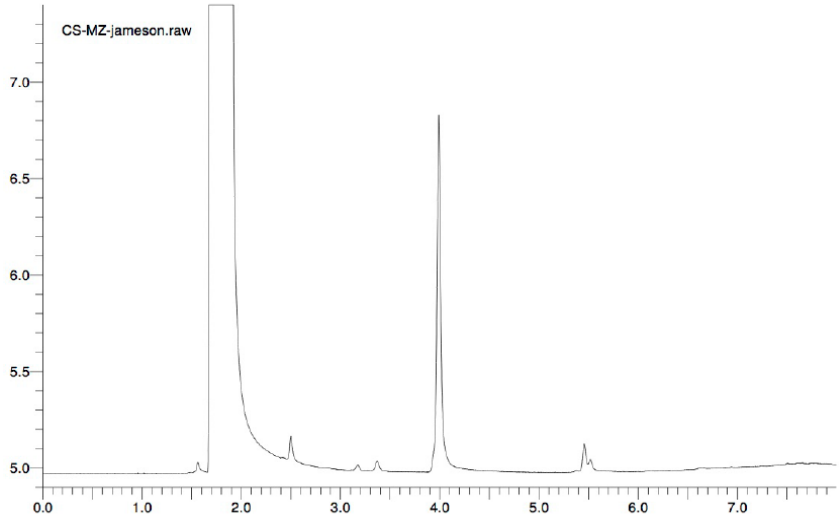


Figure 10.B.2. Chromatogram of a whiskey spiked with 2000-ppm internal standard.

You-Try-It 10.C **Chromatographic Resolution**

Tables 10.C.1 and 10.C.2 list retention times and peak widths for the chromatograms below each table.

1. Calculate the resolution of the close pairs in each chromatogram.

Table 10.C.1 GC peak parameters for a column with *N* = 1500.

Peak	Retention Time (min)	Base Width (min)	*R*
1	1.4	0.24	
2	1.8	0.26	
3	3	0.45	
4	3.4	0.48	
5	4.5	0.52	
6	4.9	0.52	

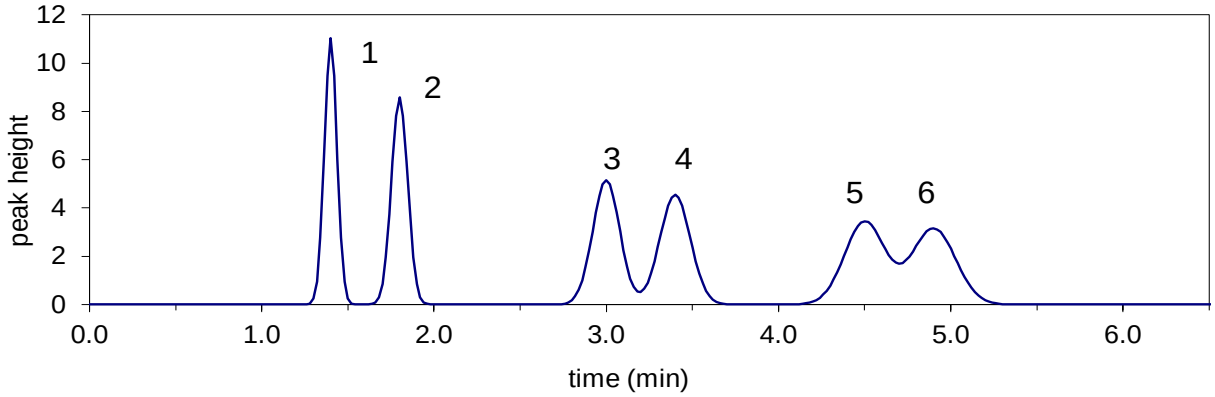
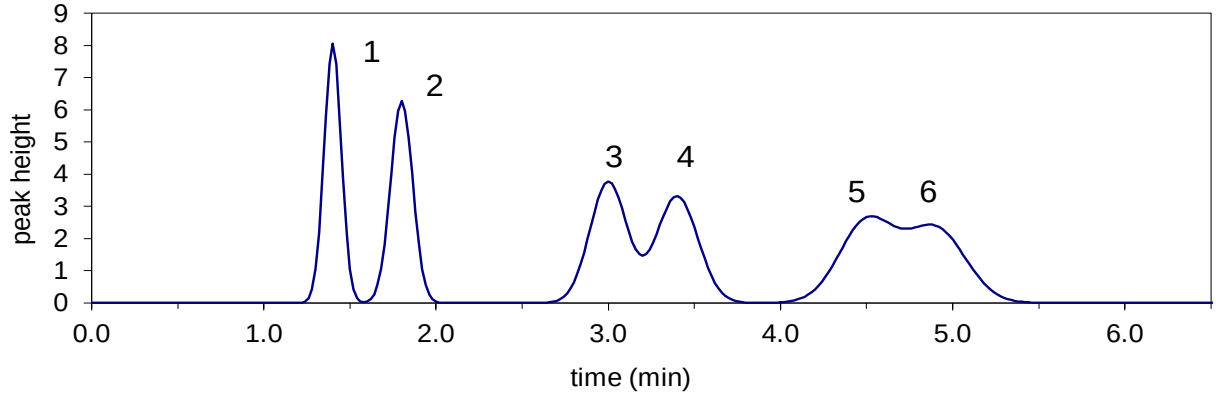


Table 10.C.2 GC peak parameters for a column with *N* = 800.

Peak	Retention Time (min)	Base Width (min)	*R*
1	1.4	0.26	
2	1.8	0.28	
3	3	0.5	
4	3.4	0.51	
5	4.5	0.7	
6	4.9	0.7	

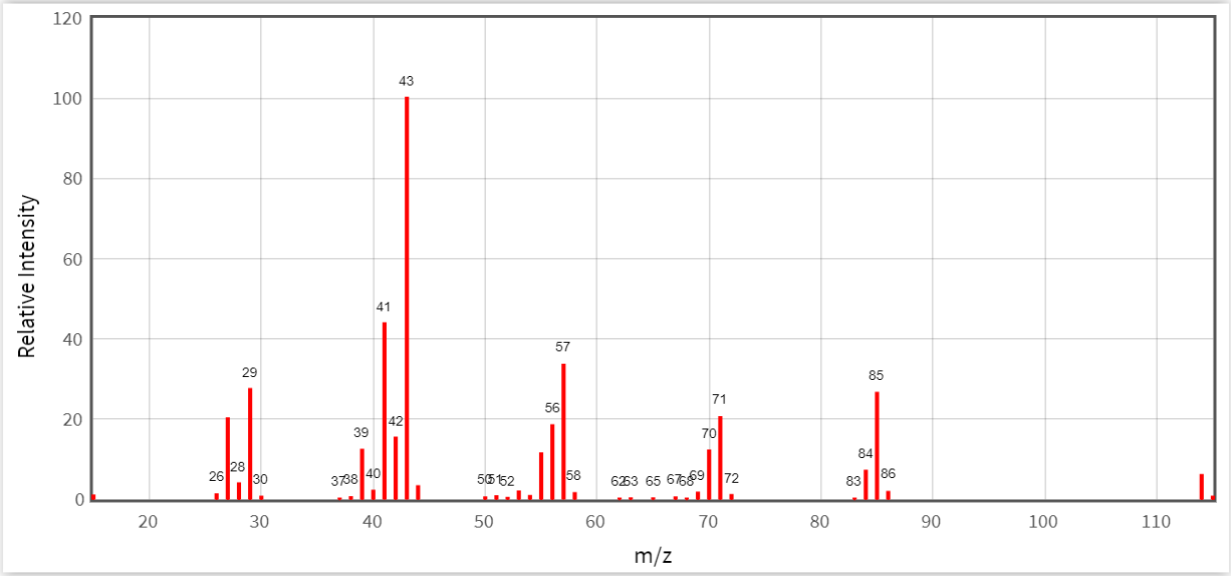


You-Try-It 10.D **Mass Spectrometry**

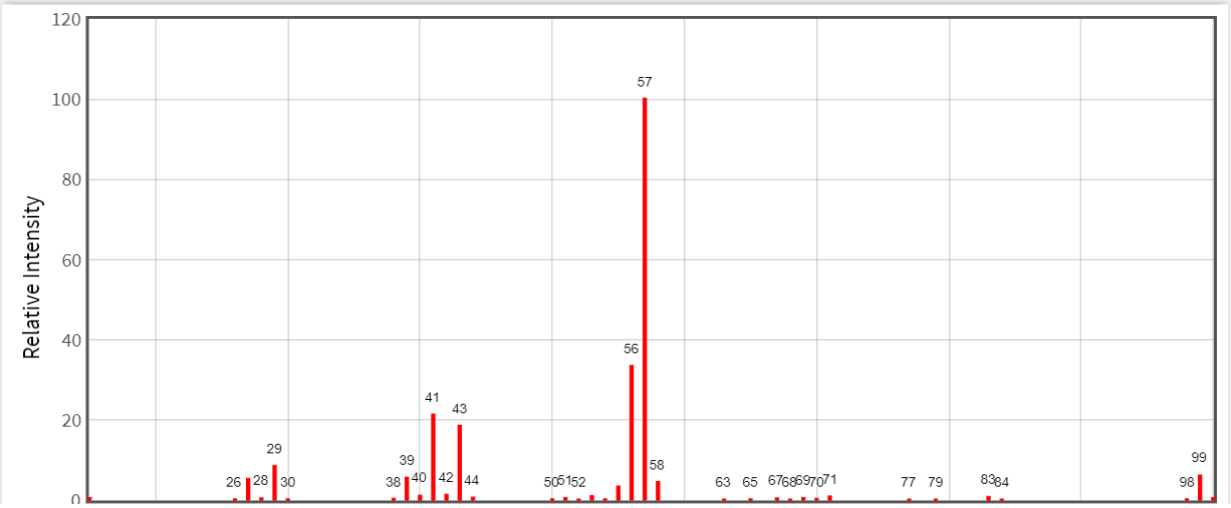
This worksheet contains mass spectra of several analytes.

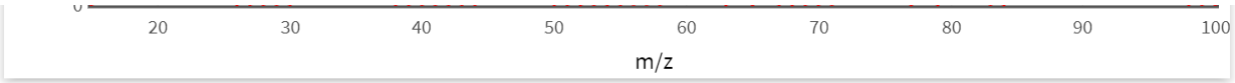
1. Predict the mass of the molecular ion for each analyte.
   Comment on the intensity of this peak in the mass spectrum.
2. Predict the chemical formula for the three most abundant peaks in each mass spectrum.
3. Calculate the expected ratio for the following peaks.
   benzene 78 and 79
   chlorobenzene 112 and 114
   benzoic acid 122, 123, and 124

		molecular ion mass:	C	H
octane	C_8H_{18}	Da		

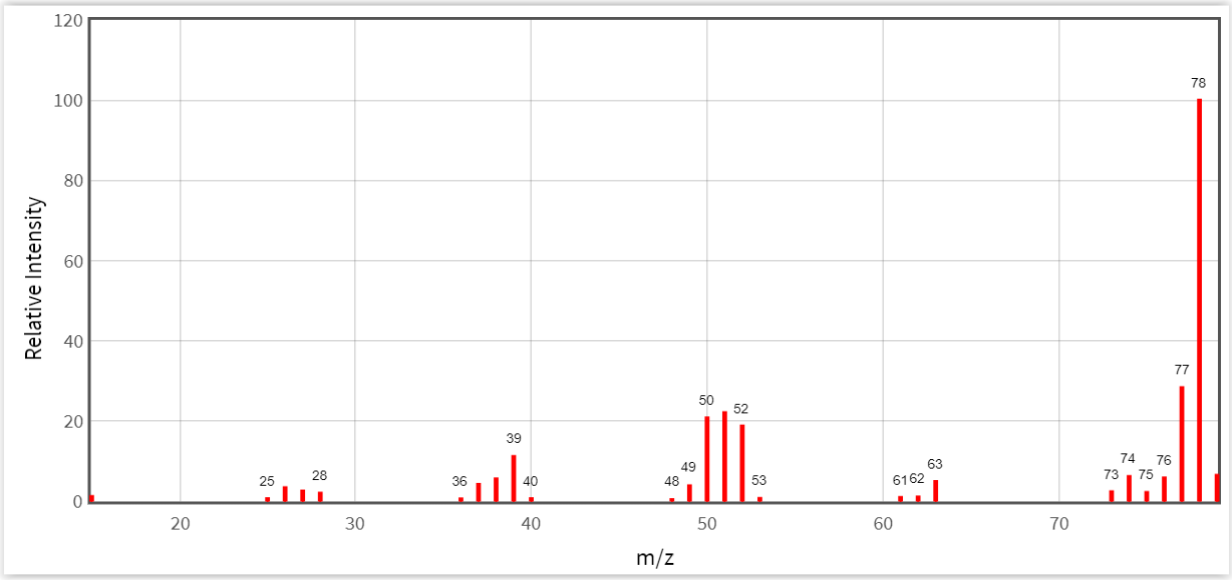


		molecular ion mass:	C	H
isooctane (2,2,4-trimethylpentane)	C_8H_{18}	Da		

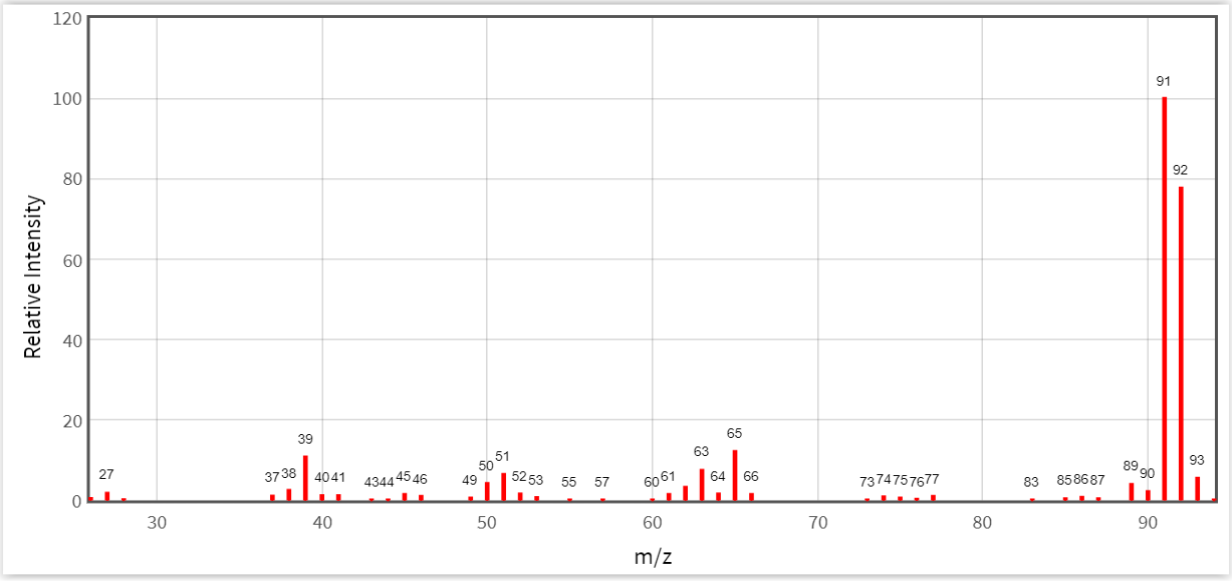




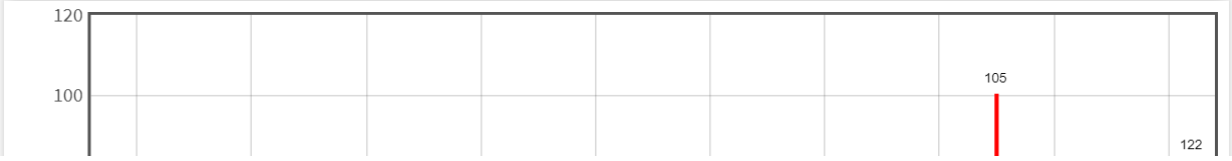
		molecular ion mass:	C	H
benzene	C_6H_6	Da		

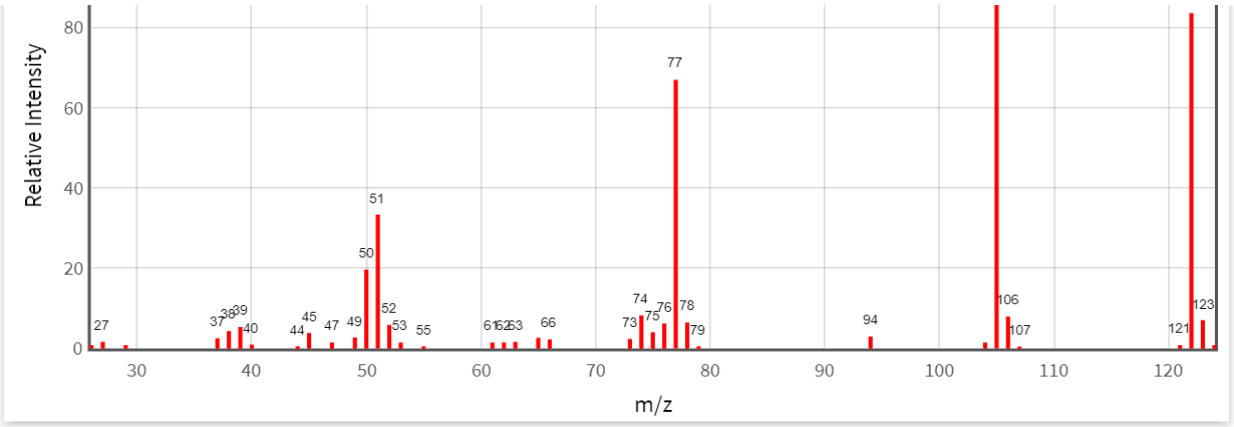


		molecular ion mass:	C	H
toluene	C_7H_8	Da		

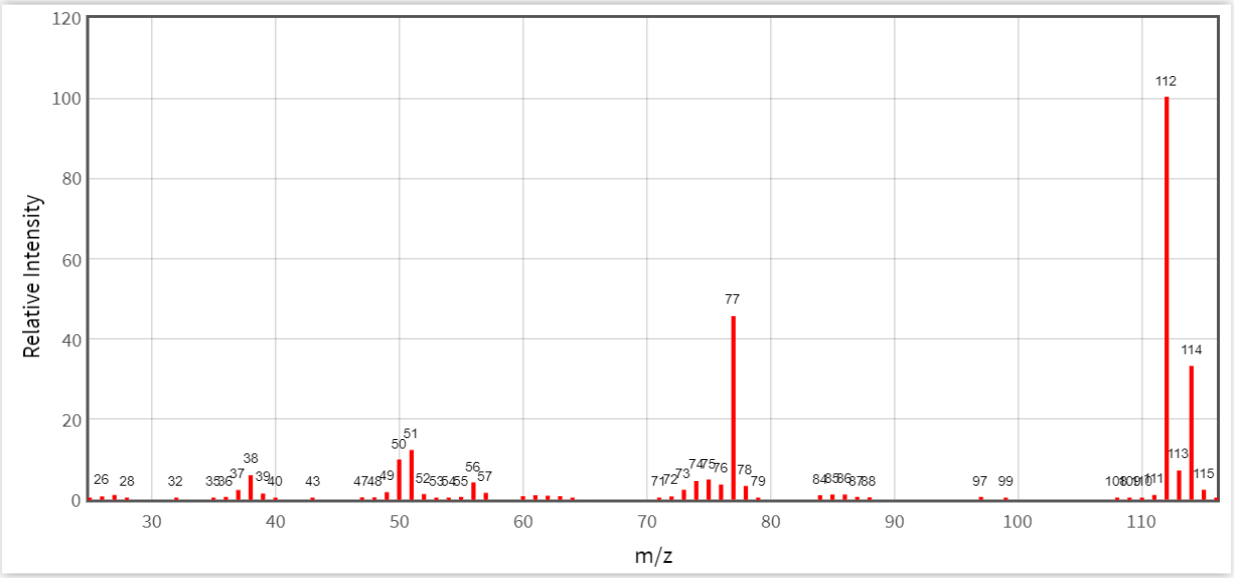


		molecular ion mass:	C	H	O
benzoic acid	$C_7H_6O_2$	Da			

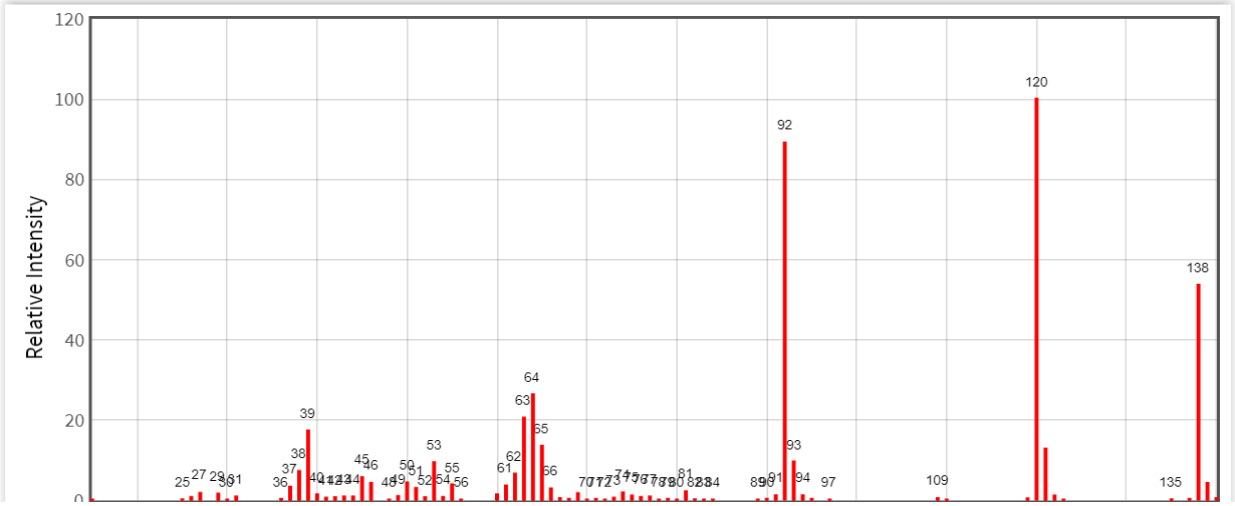




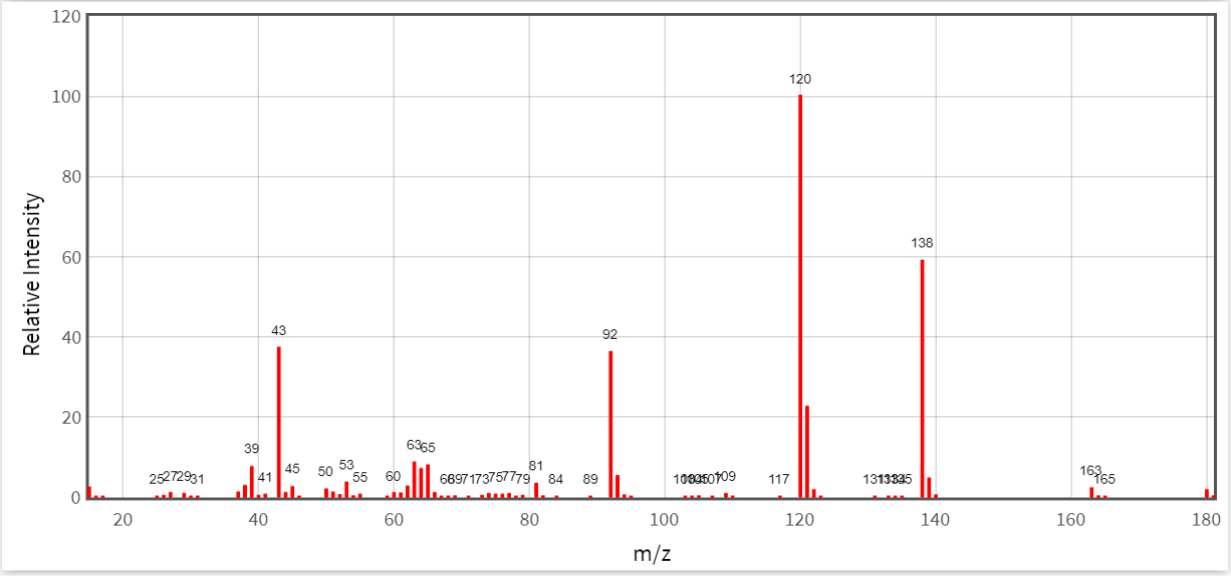
molecular ion mass:	C	H	Cl
chlorobenzene C_6H_5Cl	Da		



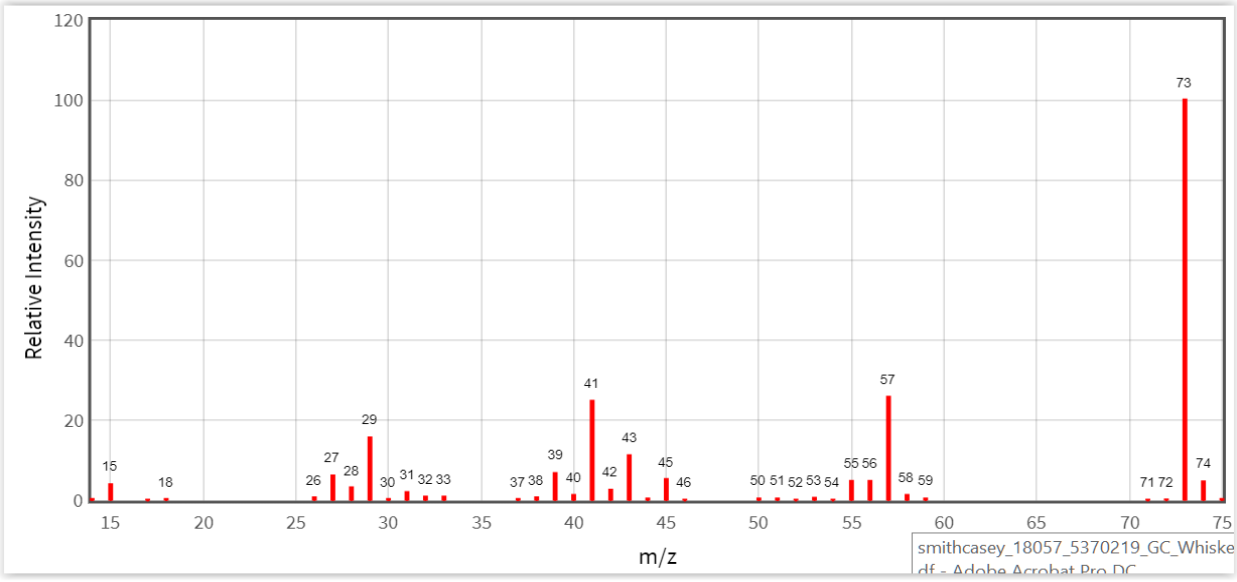
molecular ion mass:	C	H	O
salicylic acid $C_7H_6O_3$ (2-Hydroxybenzoic acid)	Da		



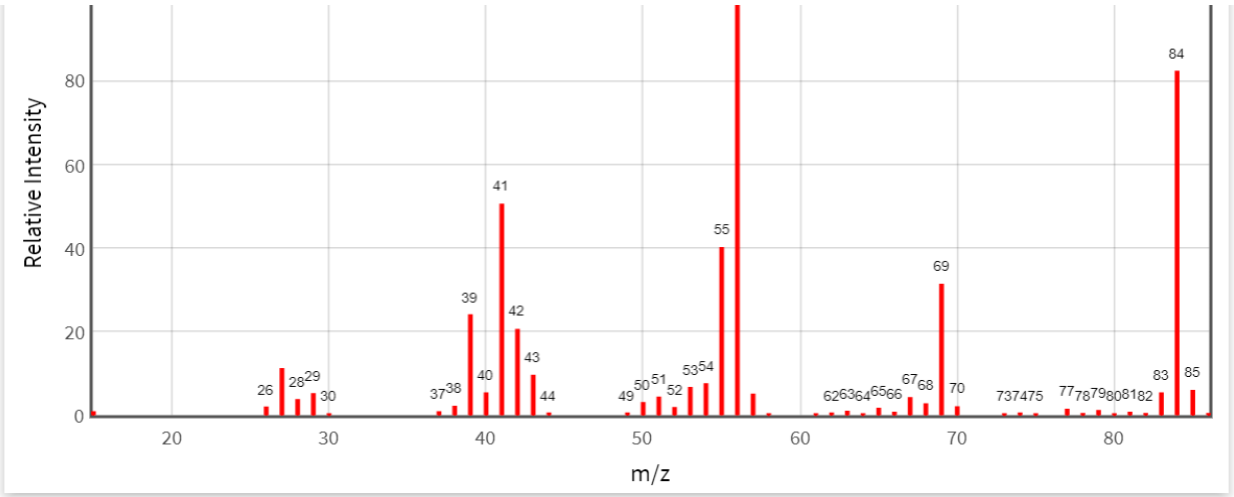
acetylsalicylic acid $C_9H_8O_4$ | molecular ion mass: Da | C | H | O



Methyl t-butyl ether (MTBE) $C_5H_{12}O$ | molecular ion mass: Da | C | H | O



cyclohexane C_6H_{12} | molecular ion mass: Da | C | H | O


Relative Intensity
m/z
0
20
40
60
80
20
30
40
50
60
70
80
26
28
29
30
37
38
39
40
41
42
43
44
49
50
51
52
53
54
55
62
63
64
65
66
67
68
69
70
73
74
75
77
78
79
80
81
82
83
84
85

NIST Mass Spec Data Center, S.E. Stein, director, "Mass Spectra" in
NIST Chemistry WebBook, NIST Standard Reference Database Number 69,
Eds. P.J. Linstrom and W.G. Mallard,
National Institute of Standards and Technology, Gaithersburg MD, 20899,
doi:10.18434/T4D303, (retrieved July 31, 2018).