Analysis and Identification of a Thingy using GC-MS
CHEM 4303
Analytical Separations

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December 9, 2018

Date Performed:	November 13, 2018
Date Completed:	November 27, 2018
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Abstract

Gas chromatography with a mass spectrometer was utilized for analysis.

1 Introduction

Gas chromatography (GC) is a separation technique that analyzes volatile compounds [1]. Consequently, this analysis can lead a lot of useful situations such as the determination of the purity of a compound or even quantifying individual components in mixtures [1]. In GC, the analyte is volatilized and carried through the column by the mobile phase, often called the carrier gas [2]. This carrier gas can either be He or H_2 [2]. These gases are often chosen as the carrier gas as they are chemically inert and would therefore not react with the analytes [1].

Polybrominated diphenyl ethers (PBDEs) are a class of halogenated compounds that are commonly used as flame retardants [3]. These compounds are an environmental health hazard as they have the potential to accumulate in the food chain [4]. In addition, 2,2',4,4'-Tetrabromodiphenyl ether, also known as BDE-47 [5], is a PBDE congener, and has been found to cause neurotoxic effects in adults [4]. Commonly used detection techniques for PBDEs are high-resolution mass spectrometry and low-resolution mass spectrometry (LRMS) [3].

LRMS is commonly done with selected ion monitoring (SIM) [3]. SIM increases the selectivity of mass spectrometry for analytes and reduces its response to everything else [2]. It does this by setting the mass spectrometer to detect just one or even a few m/z values [2]. One of the advantages of SIM is that the signal-to-noise ratio greatly increases as the mass spectrometer spends its entire analysis time measuring that particular ion, or a couple of ions [2].

Internal standards are used for a number of reasons, such as when sample loss is expected during the sample preparation step, or when the response of the quantity of sample detected differs at each analysis [2]. Essentially, an internal standard is a known quantity of a compound that differs from the analyte, and is added to the mixture containing the analyte to be detected [2].

Injectors are awesome!

The main objective of this entire experiment is to detect as well as quantify BDE-47 in a sample of fish oil. This is accomplished by making a method that utilizes a SIM.

2 Chemicals, Methods and Instrumentation

2.1 Chemicals

In the first week, BDE-47, PBB-77 and '2-HCH, all of each were at a concentration of $50\,\mu\mathrm{g/mL}$ in isooctane, were used as standard solutions. In the second week, fish oil (Exact Norwegian Cod Liver Oil), dichloromethane (emd, Lot 5Q160, CAS: 75-09-2) and PBB-77, at a concentration of $10\,\mu\mathrm{g/mL}$ in isooctane, were used as chemicals. Finally, in the last week, BDE-47, PBB-77 and '2-HCH, each at a concentration of $10\,\mu\mathrm{g/mL}$ in isooctane, were used. Throughout the entirety of the experiment, hexane (Caledon Laboratory Chemicals, CAS no. 110-54-3, LOT: 89001) and isooctane (OmniSolv, CAS: 540-84-1, LOT: 52054) were used.

2.2 Instrumentation

The separation and analysis of the entire experiment was performed on an Agilent 7890A GC, coupled with a 5975C inert XL EI/CI MSD with a triple axis detector. The dimensions of the column used was $30m \times 0.250mm \times 0.25\mu m$, by Agilent Technologies. The stationary phase was (5%-Phenyl)-methylpolysiloxane. Each analysis was performed with the injection mode at splitless, with He as the carrier gas, and the flow rate was set at $1\,\mathrm{mL/min}$. Do I need to state the pressure and volume and splitless?

2.3 Methods

3 Results and Discussion

3.1 Results

Table 1 shows some cool stuff, and table 2 shows the molecular weight of BDE-47, PBB-77 and ‘2-HCH. Table 3 shows the mass of fish oil that was weighed out for the experiment.

Table 1: Selected Ion Monitoring Parameters

Compound	Ions monitored (m/z)	Time window (min)
‘2-HCH	181, 219	0.5 - 19
BDE-47	326, 486	19 - 22.5
PBB-77	470, 310	22.5 - 24

Table 2: Molecular weight of BDE-47, PBB-77 and ‘2-HCH

Compound	Molecular weight (u)
BDE-47	486
PBB-77	470
‘2-HCH	291

Table 3: Mass of fish oil weighed out

Sample number	Mass (g)
1	1.1234
2	0.9062

3.2 Discussion

- maybe talk about the difference between internal standards and standard additions?
- maybe talk about LRMS?

4 References

References

(1) Vitha, M. F., *Chromatography: Principles and Instrumentation*; Wiley: Hoboken, New Jersey, 2017.

(2) Harris, D. C., *Quantitative chemical analysis*, 8th ed; W.H. Freeman and Co: New York, 2010.

(3) Björklund, J.; Tollbäck, P.; Östman, C. *Journal of Mass Spectrometry* **2003**, *38*, 394–400.

(4) Thomsen, C.; Småstuen Haug, L.; Leknes, H.; Lundanes, E.; Becher, G.; Lindström, G. *Chemosphere* **2002**, *46*, 641–648.

(5) Erratico, C. A.; Moffatt, S. C.; Bandiera, S. M. *Toxicological Sciences* **2011**, *123*, 37–47.

5 Appendix

5.1 Calculations

Calculating the response factor

$$\frac{\textit{Area of Analyte Signal}}{\textit{Concentration of Analyte}} = F\left(\frac{\textit{Area of Standard Signal}}{\textit{Concentration of Standard}}\right)$$
$$\frac{A_X}{[X]} = F\left(\frac{A_S}{[S]}\right) \tag{1}$$

Equation 1 was taken from [2], where $[X]$ and $[S]$ represent the concentrations of analyte and of the standard, respectively.

Calculating the capacity factor of chlorobenzene for figure 2 of the chromatogram

$$\begin{aligned} K &= \frac{t_r - t_m}{t_m} \\ K &= \frac{2.343 - 1.438}{1.438} \\ K &= \frac{0.905}{1.438} \\ K &= 0.629346 \approx 0.629 \end{aligned} \tag{2}$$

Equation 2 was taken from [2].

5.2 Chromatograms

There are ? sheets of GC-MS chromatograms.