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

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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 detecting explosives [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, BDE-47, a PBDE congener, 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].

The main objective of this experiment is to make a method that uses a SIM for quantitative analysis of PBDEs by GC-EI-LRMS.

2 Chemicals, Methods and Instrumentation

2.1 Chemicals

Old: Benzaldehyde (Sigma Aldrich, Lot: 41696 PKV), nitrobenzene (Alfa Aesar, Lot: A06V040, 99%, CAS: 98-95-3), methanol (HPLC grade, Caledon, Lot: 103267, CAS: 108-90-7), salicylamide (Sigma Aldrich, Lot: S51885-279, CAS: 65-45-2), phenacetin (Sigma Aldrich, Lot 78C-0014), acetominophen (Sigma Aldrich, Lot: 32F-0073), caffeine (Sigma Aldrich, Lot: 0316B4), and an unknown pharmaceutical mixture was used in this experiment.

New: In the first week, something. In the second week, more of something. Finally, in the last week, the last of something.

2.2 Instrumentation

The separation and analysis of the entire experiment was performed on an Agilent 7890A GC, coupled with a 5975C inert XL EIICI MSD with a triple exis 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?

2.3 Methods

3 Results and Discussion

3.1 Results

Table 1 shows some cool stuff.

Table 1: Selected Ion Monitoring Parameters

Compound	Ions Monitored (m/z)	Time window (min)
‘2-HCH	180.9, 218.9, 181, 219	0.5 - 19
BDE-47	326, 486	19 - 22.5
PBB-77	470, 310	22.5 - 24 or 34

3.2 Discussion

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 Appendix

5.1 Calculations

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

$$\begin{aligned} K &= \frac{t_r - t_m}{t_m} \\ K &= \frac{2.343 - 1.438}{1.438} \\ K &= \frac{0.905}{1.438} \\ K &= 0.629346 \approx 0.629 \end{aligned} \tag{1}$$

Equation 1 was taken from [2].

5.2 Chromatograms

There are ? sheets of GC-MS chromatograms.