Experiment 22: GC of gasolines

SYNOPSIS Tetraethylead will be determined in a mixture of alkanes. **<u>This form of lead is particularly toxic. All experiments should be carefully vented.</u>** The doped tetraethyllead can be separated from the other components by gas chromatography using a packed column and a flame photometric detector.

READING 331 in Critical Review. Attached is an article detailing GC to separate tetralkyl compounds for subsequent analysis.

Instrument

Shimadzu GC-8A1F gas chromatograph

Operating Conditions

Glass column 5 mm o.d., 2.6 mm i.d., 5.4 m long, OV-101 stationary phase
Column Temperature 50 °C
Injection Temperature 120 °C
FID detector at 400 °C
3 µL injection
N_2 carrier gas at 60 mL/min Pressuge 0.6 bp/cm^2
H_2 fuel gas at 50 mL/min 1.9
Air oxidant gas at 500 mL/min 1.2

ignition: H_2 at 0.9 pb/cm^2
Air at 0.1 to 0.2 bp/cm^2

Programmed Temperatures

50 °C initial + 10°C/min up to no more than 200 °C

Reagents and Solutions

CS_2 as a matrix or carrier for the alkanes and tetraethyllead.
Heptane
Hexane
Octane
Tetraethyllead
CAUTION: this latter should be handled only with the hood due to its toxcity.

Mixtures:

5 mL CS_2
5 mL CS_2 + 10 µL heptane
5 mL CS_2 + 10 µL hexane
5 mL CS_2 + 10 µL octane

5 mL CS_2 + 10 µL tetraethyllead
5 mL CS_2 + 5 µL tetraethyllead
5 mL CS_2 + 2 µL tetraethyllead
5 mL CS_2 + 10 µL heptane + 10 µL hexane + 10 µL octane + 10 µL tetraethyllead

Experiment

Obtain a GC separately for each solutions.

Report In addition to materials and methods:

1. Tabulate the retention times of the individual species, of their peak width, and their resolution from their nearest neighbors.
2. Plot the log adjusted retention time of the alkanes against their carbon number. Is the plot linear? Does the slope change when you changed flow rate or temperature?
3. Using the plot in 2 calculate the apparent carbon number of tetraethyllead, assuming that temperature has remained constant. (If you do a temperature programming you can not do this.)
4. Is there a discernable trend in the peak widths for the species with retention time?
5. What is the effect of varying the flow rate or temperature on the retention times and on the peak widths.
6. What is the resolution of heptane and hexane? Is it reasonable? What is meant by reasonable resolution?
7. Calculate H for your column. What is the *meaning* of H?
8. How does the definition for resolution in chromatography related to definitions we use for LOD and LOQ in population statistics?
9. Was the temperature of the column maintained constant during the analysis of the tetraethyllead + alkane mixture? Why or why not?
10. Why are you instructed not to take the temperature of the column any higher than 200 ^{o}C?
11. Show a calibration plot for tetraethyllead. What is the LOD?
12. What controls the LOD in the FID detector? That is, what is the background signal likely to be due to?
13. How does your LOD compare to that obtained by Lobinski (0.1 pg/mL)? The density of tetraethyllead is 1.653 g/mL.
14. Why is tetraethyllead so toxic? How does the mechanism of toxicity to humans relate to the mechanism by which it is found in wines?