JACOBS UNIVERSITY

LAB COURSE ANALYTICS

Handout Fall 2011 – **Gas Chromatographie**

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SAFETY - Never open the column oven door when the GC is running . **HOT PARTS !**
When handling with chemicals , protect yourself wearing goggles and gloves.
WORK IN THE HOOD !

Experiments:

1A) Calculation of the retention index (Kovats Index) isothermal

1B) Influence of oven temperature

Equipment for both experiments :

Column : Rxi- 5ms, 15 m × 0.25 mm ID × 0.25 µm
Flame ionization detector
Carrier Gas : Helium

Substances

Sample A : C9, C10, C12, C14, C15,C16 n-alkanes 0.1 g/L in n-hexane

Procedure Experiment 1A :

Retention index in Gas Chromatography

By definition, saturated n-alkanes in all separation systems and under all separation conditions have exactly the retention index of C-number times 100.

For unbranched alkanes there is a linear relationship between the logarithm of retention time tR and the number of carbons.

In gas chromatography retention index system of n-alkanes as standards and a logarithmic interpolation is used.

The Kovats retention index is constant for the same temperature and column phase (important is the separation due to the boiling point – unpolar phase) Indepentdantly of the column layer thickness.

To calculate the retention index of an unknown compound you need the retention times of the Alkanes eluting before and after the substance.

The aim of this experiment is to see the influence of the oven temperature on the different chromatographic parameters under isothermal conditions.
We start with an oven temperature of 160 _C. The injection volume is 1 µL. Then, chromatograms at two different lower oven temperatures in the interval from 80 to 140 _C should be produced.

Task :

Once you obtain an chromatogram with six peaks baseline separated – calculate the retention index from the unknown alkane you will receive due to the following formula.

In Addition , the following plot should be prepared : log. t_R vs. C – Numb.x100

$$I = 100y\left(\frac{logt'Rx - logt'Rz}{logt'R(z+y) - logt'Rz}\right) + 100z$$

Example – compound eluting before – C5 – t`R_z = 2.0 min
- compound eluting after - C7 – t´R(z+y) = 2,85 min
Retention time unknown - t`R_x = 2.56 min

100z = 100 x 5 - Pentane
100y = 1oo x 2 - Heptane minus 2 carbons.

Result ?

Optimization of a separation of for different terpenes,one natural flavor, one synthetic flavor and a aromatic ketone by temperature gradient.

Substanzes : Thymol ,(-) Camphor , Cuminaldehyde , Resorcindimethylether (+) – Fenchol , Vaniline, 1-Phenyl-2-butanone

The separation of six different terpenes (0,1g / L in ethyl acetate or heptane) should be optimized by changing the temperature gradient until the peaks are almost baseline separated. Terpenes are naturylly occurring plant secondary metabolites that are frequently used in the perfume industry . Resorcindimethylether is a synthetic flavour to find in for expl.Mousses,puddings, jelly dishes, sweet sauces, chewing gum , etc.

Investigat the influence of the temperature ramp and choose an optimized temperature program.

Also think about , looking at the structure,in which order the coumpunds will elute (think about the different polarity of the compounds)

The student with the closest estimation will get bonus points.

To confirm , please measure all the compound separately.

The starting temperature of the column oven should be 60 deg. Celsius. – keeping for 1 min. for the solvent peak.

Maximum oven temperature – 300 degree Celsius.

Questions :

1. Depending on which physical – chemical property of the stationary phase the separation takes place.

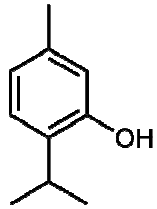
2. Explaine the terms
   a) FID
   b) MSD

3. Name at least four parameters influencing the separation of a substance. (HPLC and GC)

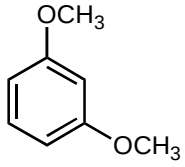
4. What substances can be analyzed by GC.

5. Is it possible to separate two substances with the same boiling point by GC.

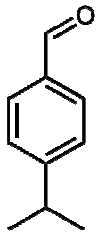
LIST OF Compunds



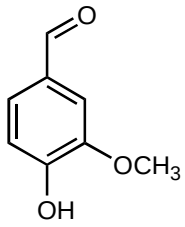
Thymol



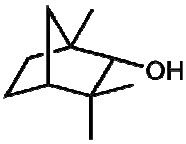
Resorcindimethylether



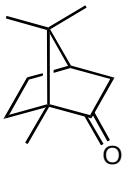
Cuminaldehyde



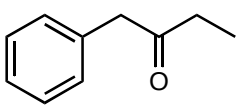
Vanillin



(+)-Fenchol



(-)-Camphor



1-Phenyl-2-butanone