GAS CHROMATOGRAPHY (GC)

THE PURPOSE OF THE EXERCISE

The purpose of the exercise is to learn about the influence of various factors, such as

* polarity of the separated substances
* boiling point of separated substances
* stationary phase polarity
* column operating temperature
* column length
* carrier gas flow rate

on the efficiency of chromatographic separation.

DESRIPTION

The software enables the simulation of the process of separating a mixture of organic compounds by gas chromatography using a liquid stationary phase. The separated mixture may contain from 1 to 5 ingredients selected from a set of 37 substances from the group of aliphatic and aromatic hydrocarbons, halohydrocarbons, aldehydes, ketones, alcohols, esters, ethers and nitriles. The composition of the separated mixture is chosen freely (1 ÷ 5 ingredients) or randomly (3 ingredients in the mixture).
The selected mixture can be separated on the following stationary phases:

1. Silicone DC-200
2. N-decyl phthalate
3. Di (mrtyloxyl) sebacate
4. Poly (ethylene glycol)at the selected wavelength.

The length of the column can be changed in the range from 1 to 5 m and the carrier gas flow rate in the range from 0 to 60 mL min.
The chromatographic process can be carried out under isothermal conditions (i.e. at a constant temperature in the range of 25 to 250 ° C) or with programming temperature mode. In the latter case, the initial and final temperature, temperature gradient, i.e. the rate of increase in ° C / min should be determined, as well

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as the so-called isothermal period, i.e. the time after which the temperature increase is to occur. The initial temperature is maintained during the isothermal period.

The program screen consists of four parts: the main menu (at the top of the screen) containing the basic program options, the part in which the chromatogram is drawn, and two windows at the bottom of the screen. The left window displays the components of the separated mixture and their retention times, while the right window displays the type of stationary phase and other parameters of the chromatographic process.

After starting the program, the main menu and the Parameters option are active (highlighted). All main menu options have a collapsible menu. sub-menu.

Move the mouse cursor (arrow) to the name of the given option or menu command and press the left mouse button activates this option. Return from the sub-menu to the main menu is done by moving the mouse cursor out of the sub-menu area and pressing the left button.

Selecting the options:Temperature, Column length, Gas flow and Programming Temperature opens the input window. This window displays the current value of a given parameter, which can be changed by the user and accepted by pressing ENTER. Pressing the ESC key, despite changing the parameter value, closes the window and maintains the current parameter value.

The implementation of the **Stationary Phase** option opens the next sub-menu with the names of the stationary phases available in the program. Selecting a given phase is done by moving the highlight to its name and pressing the ENTER key.

Activating the Main menu functions option causes the sub-menu to expand with the following commands and options:

Start - starting the chromatographic process and plotting the chromatogram.

Simultaneously with the chromatogram, a graph of temperature changes is drawn. The chromatogram plotting is completed when the "pen" returns to the baseline after the last of the mixture peaks has been drawn. Drawing stops when the analysis time exceeds 40 minutes (on-screen).

Randomization - random selection of 3 components of the separated mixture.

Operator choice - opens a window with a list of all compounds available in the program database. The selection is done by moving the highlight to the compound name and pressing ENTER. Termination occurs after pressing the ESC key or automatically, after selecting the fifth subsequent ingredient of the mixture.

PROCEDURE

1. In the Functions / Free selection option, select two components of the mixture: an aliphatic hydrocarbon and alcohol with similar boiling points. The column operating temperature should be 10 °C lower than the boiling point of the mixture components. Column length 2 or 3 m. Perform chromatograms on polar and non-polar stationary phase: DC-200 silicone and polyethylene glycol. Note the retention times of ingredients obtained.

In the study, explain and justify the behavior of the components based on their properties and the properties of the stationary phases used.

2. Select the following alcohols: methyl, ethyl, n-propyl, n-butyl and n-amyl (n-pentanol). Use a 3 m long column as the stationary phase Silicon DC-200. Choose other conditions, i.e. temperature and flow rate, so as to achieve complete separation of all components of the mixture, i.e. that the calculated resolution is not less than 1.50. Note the retention times. Keeping the other parameters unchanged, choose such a temperature program to reduce the differences in retention times of the highest boiling ingredients and thus shorten the time of chromatogram preparation. Record selected parameters and retention times obtained.

3. Draw the ternary mixture in the Functions / Randomization option. Select the appropriate stationary phase and other operating parameters so as to achieve complete separation of the mixture (resolution not less than 1.50) in the shortest possible time. Note the finally selected parameters and the retention times obtained component of the mixture.

LITERATURE

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2. D. Harvey, *Modern Analytical Chemistry*
3. Douglas A. Skoog, Donald M. West, F. James Holler, Stanley R. Crouch, *Fundamentals of Analytical Chemistry*
4. Douglas A. Skoog, F. James Holler, Stanley R. Crouch, *Principles of Instrumental Analysis*
5. B. Sivasankar, *Instrumental Methods of Analysis*