Catalytic dehydration of alcohols

manual for experiment

Instructor dr Hanna Wilczura-Wachnik

University of Warsaw
Faculty of Chemistry

Organic Chemistry and Chemical Technology Division

Manual for experiment

Experiment aim

The main aim of this experiment is catalytic dehydration of chosen alcohol and study an influence of temperature, time and raw material dosage speed on the final products. In the experiment hexanol will be dehydrated using Al_2O_3 as a catalyst. The reaction products will be analyzed with gas-liquid chromatography using Hewlett-Packard GC 6890.

Installation description

A schematic diagram of device is presented in Figure 1. The reaction is performed at isothermal conditions in reactor (1) filled with catalysts (4,6g (5mL) Al_2O_3) Temperature inside the catalyst bed (2) is measured with thermocouple (3). Control unit (4) is a temperature regulator (involves on temperature programming). Alcohol is dosing with a stable rate into reactor with calibrated syringe using infusion pump (10). Reaction products passing through condenser (5) cool down and finally condensate in receiver (6). Element (8) is filled with “dry ice +acetone” in which all residual products are freezing out. Washer (9) filled with paraffin oil indicates gas flow. Air pump (12) doses air into reactor during catalyst regeneration. Inert gas (Ar in balloon 13) is used for reaction products removing from catalyst bed and at final step of catalyst regeneration for air removing.

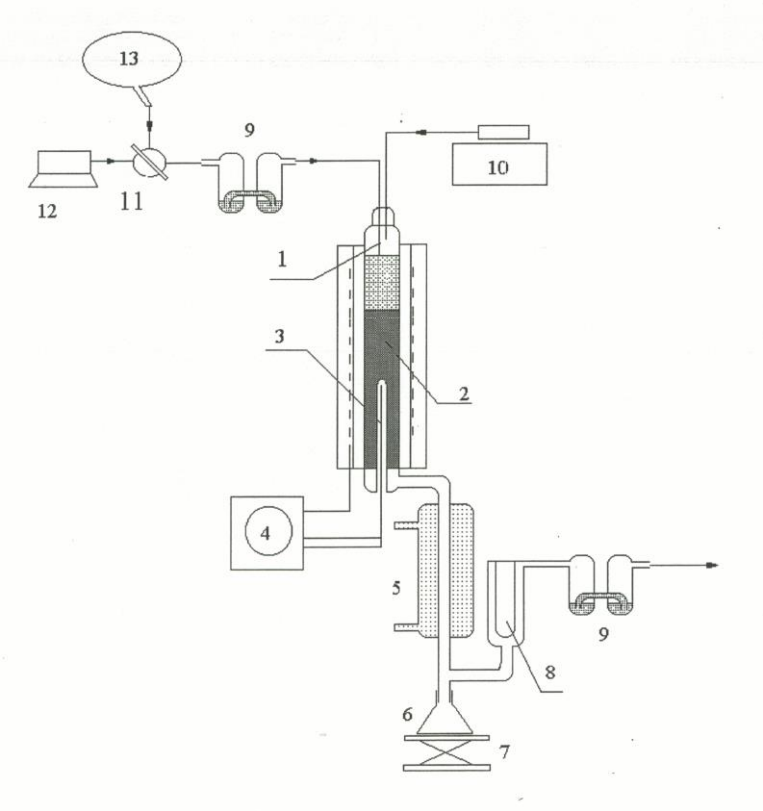

Figure 1. Installation scheme: 1- reactor; 2- catalyst bed; 3 – thermocouple; 4 – temperature control unit; 5 – Liebig condenser; 6 – receiver; 7 – elevator; 8 – “dry ice” container; 9 – washer; 10 – infusion pump; 11 – valve; 12 – air pump; 13 – balloon.

Experiment steps

1. Turn on air pump (12) and temperature control unit (4) set-up 600^{o}C for catalyst regeneration (1hour).
2. Weight products receiver (6).
3. Decrease temperature to value 250^{o}C – 350^{o}C according to teacher suggestion and purge catalyst bed with Argon.
4. Set dosing speed on suitable value (proposed by instructor).
5. Fill syringe with hexanol and place needle through membrane into reactor.
6. Connect receiver (6) with Liebig condenser (5).
7. Start dosing alcohol (pump “on”) and note time.
8. Reaction duration suggests instructor.
9. Time over, turn off dosing alcohol, take out needle.
10. Purge catalyst bed with Ar (balloon 13).
11. Disconnect receiver, weight it, separate water, weight dry products and make glc* of reaction products.

12. Obtained results note in suitable results sheet.
13. Calculate composition of reaction product using glc results.
14. Regenerate catalyst and eventually perform process under different conditions (change temperature, hexanol dosing speed,) according to points from 4 till 13.

The quantity of hexene-1 and hexanol in reaction products calculate according to the following equations (obtained in a separate calibration procedures):

for hexanol: y= 278,52x-4307,5 R^2 = 0,9992
where: y is the hexanol peak area
x is the hexanol quantity in miligrams in 1g of mixture

for hexane-1: y= 352,94x – 6897,6 R^2 = 09991
where: y is the hexane-1 peak area
x is the hexane-1 quantity in miligrams in 1g of mixture

* **glc basic principles are described below**

Gas-liquid chromatography is a form of column chromatography in which the absorbing medium is a liquid of low volatility, called the liquid phase, and this is dispersed over the surface of an inert solid support. The latter is usually a granular material which does not itself adsorb the components but which merely acts to hold the liquid phase in a stable dispersed form. A gas stream, known as a carrier gas, flows continuously through the column and the temperature of the system is controlled. When a small quantity of a volatile mixture is applied to the column the components are distributed between two phases and therefore moved more slowly than carrier gas stream, due to the retarding effect of the liquid phase. The column should be sufficiently long due to separating components as a result of differences in this retarding effect. When the separated components are eluted from the column a sensitive device, known as a detector, converts the concentration or mass substance contained in he exit gas stream into an equivalent electrical or other measurable signal (depending on detector type). This signal is usually measured by some continuous recording device to produce a chromatogram. Typical chromatogram consists of a number of peaks ach of which corresponds to a component of the

mixture. The position of each of these peaks is characteristic of the component to which it is due. These characteristic positions are measured in terms of the volume of carrier gas which has passed through the column between the time application of the sample to the column and the time of emergence of the component. This gas volume is called the retention volume, V_R , and is a fundamental gas chromatography property in qualitative analysis. The size of each peak (its area or height), is normally proportional to the amount of component causing it. This property is an important prerequisite for accurate quantitative analysis.

Experiment report should be prepared according to pattern attached to manual and contained:

- an aim of experiment,
- an experiment brief description
- glc results description and interpretation (including glc analysis parameters),
- a table containing obtained and calculated data,
- results discussion, and comments (including yield of reaction, sources of errors),
- Sankey diagram (if necessary prepared on basis of process mass flow balance) with a suitable scale and stream legends,
- conclusions (among, if the aim of experiment has been achieved),
- students remarks and suggestions if there are some (for example how to improve some steps of experiment).

A results sheet signed by instructor should be attached.