PREPARATION OF PURE CYCLOHEXANE BY A MOLECULAR DISTILLATION METHOD

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UDC 66.062.22 : 66.048.9

Together with existing methods of preparing pure compounds, such as close fractionation, crystallization, etc., zone refining and preparative chromatography have begun to be widely used in recent times.

J. Bullot [1] prepared cyclohexane of 99.91% purity by means of zone refining after passage through twelve zones.

However, for some forms of laboratory investigations, substances of higher purity are required, and moreover zone refining allows one to prepare only insignificant amounts of pure compounds after an extended time.

Below we describe the preparation of pure cyclohexane by a molecular distillation method, using a capillary [2].

The throughput of a capillary in the case of molecular or viscous flow is determined by the equation [3]:

$$F = \frac{K_t}{M^{1/2}},$$

where F is the throughput of the capillary in cm^3/sec; M is the molecular weight; and K_t is a constant which depends on the temperature, diameter and length of the capillary, and other parameters.

Thus, a fractionation should occur when vapors of a mixture are passed through a capillary [4].

A scheme of a set-up for purifying cyclohexane is shown in Fig. 1. The starting sample of cyclohexane is placed in flask 1, to which, on a ground joint, is fastened capillary 5 with an internal diameter equal approximately to 0.05-0.1 mm. The capillary in our work had a length of 200 mm and was cooled in the form of a spiral. At its end the capillary was terminated by a constriction 0.02-0.03 mm in diameter. Cooling of column 2 was effected by a cooling mixture which was set in the inner shell of the column. Cooling of flask 1 and receivers 3 and 4 was attained by immersing them in Dewar flask 6, which contained an acetone-dry ice cooling mixture.

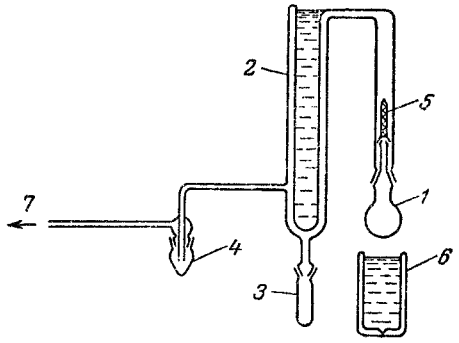

Fig. 1. Apparatus for purifying cyclohexane.

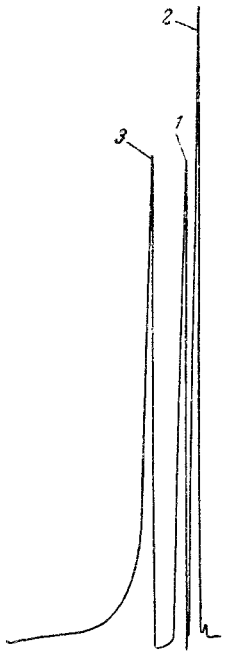

Fig. 2. Chromatogram of starting cyclohexane: cyclohexane (× 100,000); 2 and 3) impurities (×200).

All-Union Scientific Research Institute of Petroleum Chemistry. Translated from Khimiya i Tekhnologiya Topliv i Masel, No. 12, pp. 11-12, December, 1967.

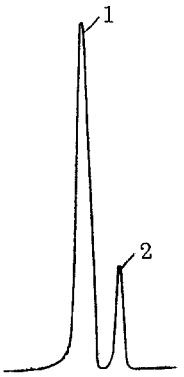

Fig. 3. Chromatogram of cyclohexane after purification: 1) cyclohexane (x 100,000); 2) impurity (x 200).

The whole assembly was pumped out to a pressure of $1 \cdot 10^{-1}$ mm Hg with a type VN-461 vacuum pump, 7 (not shown in figure).

Vapors of the sample being purified passed from flask 1 through capillary 5 into coolable column 2, on whose walls the cyclohexane vapors condensed for the most part, and the uncondensed vapors of cyclohexane and other compounds—impurities from column 2—went into receivers 3 and 4, in which they were condensed at the lower temperature.

The purity of the starting cyclohexane was 99.49%. Preliminary purification of the cyclohexane was carried out in the assembly without the capillary, cooling flask 1 to −30° and receivers 3 and 4 to −70°C. After the preliminary purification, in flask 1 there remained (about $1/3$ of the amount taken) cyclohexane of 99.7% purity.

Further purification was carried out in the same assembly with introduction of the capillary, 5; here flask 1 was at room temperature, the column temperature was held at −30°, and the temperature in receivers 3 and 4 was held at −70°C.

After 6 to 7 h of operation in our apparatus, cyclohexane of 99.97% purity was obtained (about 10% of the original sample).

In Figs. 2 and 3 we show chromatograms of the original cyclohexane and the purified material. From comparison of the chromatograms, it follows that one of the peaks corresponding to an impurity has disappeared completely, and the other has decreased sharply.

LITERATURE CITED

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