Notes

CHROM. 3676

Three-dimensional model for the optimum column conditions in gas chromatography

As is well known, the concept of the height equivalent to a theoretical plate, HETP, has been introduced and extensively used in gas chromatography. KLINKENBERG AND SJENITZER[1] treated the HETP concept mathematically on the basis of Poisson distribution. VAN DEEMTER *et al.*[2] developed the famous kinetic theory. In addition, a new theory[3,4], in which the pressure drop along the column was taken into consideration, was formulated. This theory[4] predicts that HETP is related to the outlet carrier gas flowrate, u_o, by an equation of the following form:

$$H = \frac{B}{u_o} + C\sqrt{u_o}$$

This relationship was confirmed experimentally[5].

On the other hand, HETP varies remarkably with column temperature[6]. In the present report, the effect of column temperature and of carrier gas flowrate on HETP was studied and the relationship was found to be expressed suitably by a three-dimensional model.

Experimental

A Hitachi gas chromatograph, type KGL-2A, equipped with a thermal conductivity detector, was used. The column, 2 m in length and 0.4 cm in diameter, was a copper tube packed with a stationary phase of 10 % Apiezon grease L on 60–80 mesh Diasolid M. Helium gas flowrate was measured with a soap film meter and corrected for the saturated vapour pressure. The column inlet pressure was measured with a calibrated pressure gauge, and the outlet pressure was always kept at atmospheric pressure. The column temperature was maintained constant within *ca.* 0.1 %. The toluene sample was injected by means of a 10-μl syringe into the heated injection block which ensures rapid vaporization.

Results and discussion

The HETP varies with sample size. With decreasing sample size, from 5 μl to 0.6 μl, the plate number increased. The corresponding optimum column temperature also became higher with increasing sample size, as shown in Fig. 1. The plate number of the column was found to be almost independent of sample size, if one employs a sample smaller than 0.6 μl, as shown in Fig. 2. Thus the sample size was maintained at 0.4 μl throughout the experiment. Under such conditions the HETP can be considered to depend on only two variables, *i.e.*, column temperature and flowrate. Curve A in Fig. 3 shows the dependence of HETP on the absolute carrier gas flowrate

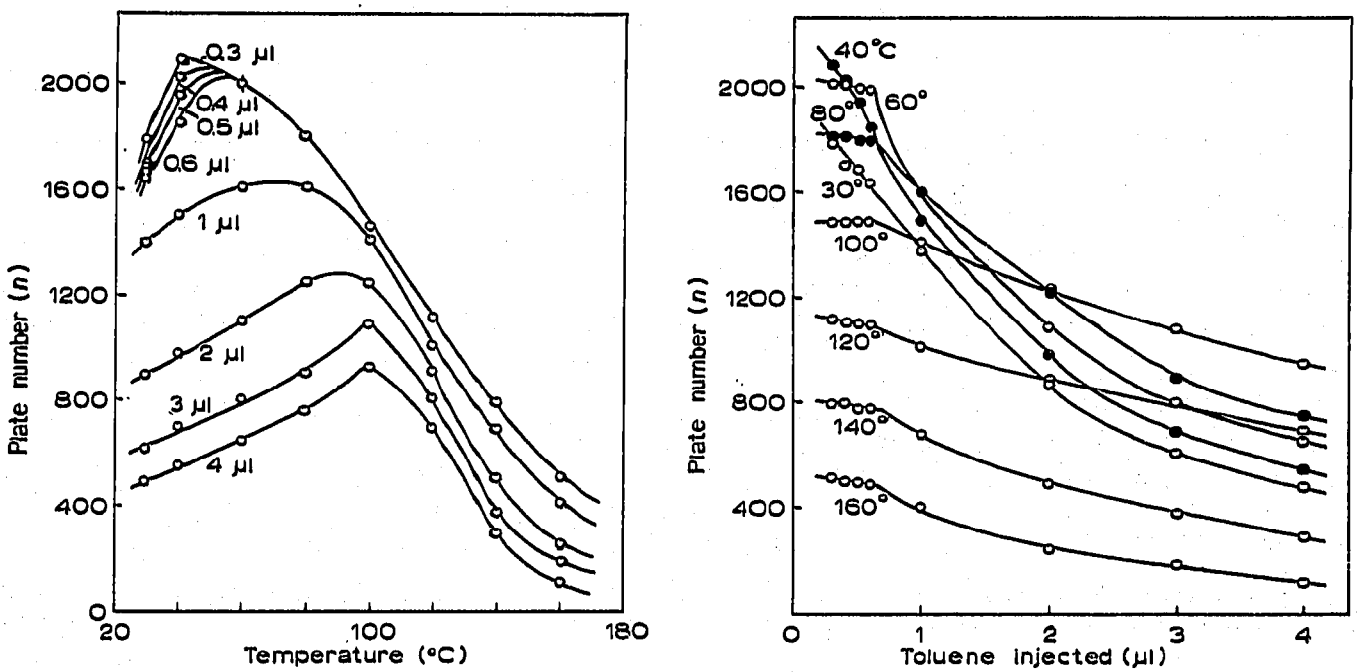

Fig. 1. Plot of plate number against column temperature. The figures show the sample amount injected. Sample: toluene. Column: 2 m long, 0.4 cm diam., 10% Apiezon grease L on 60–80 mesh Diasolid M.

Fig. 2. Variation of plate number with sample amount injected at various column temperatures.

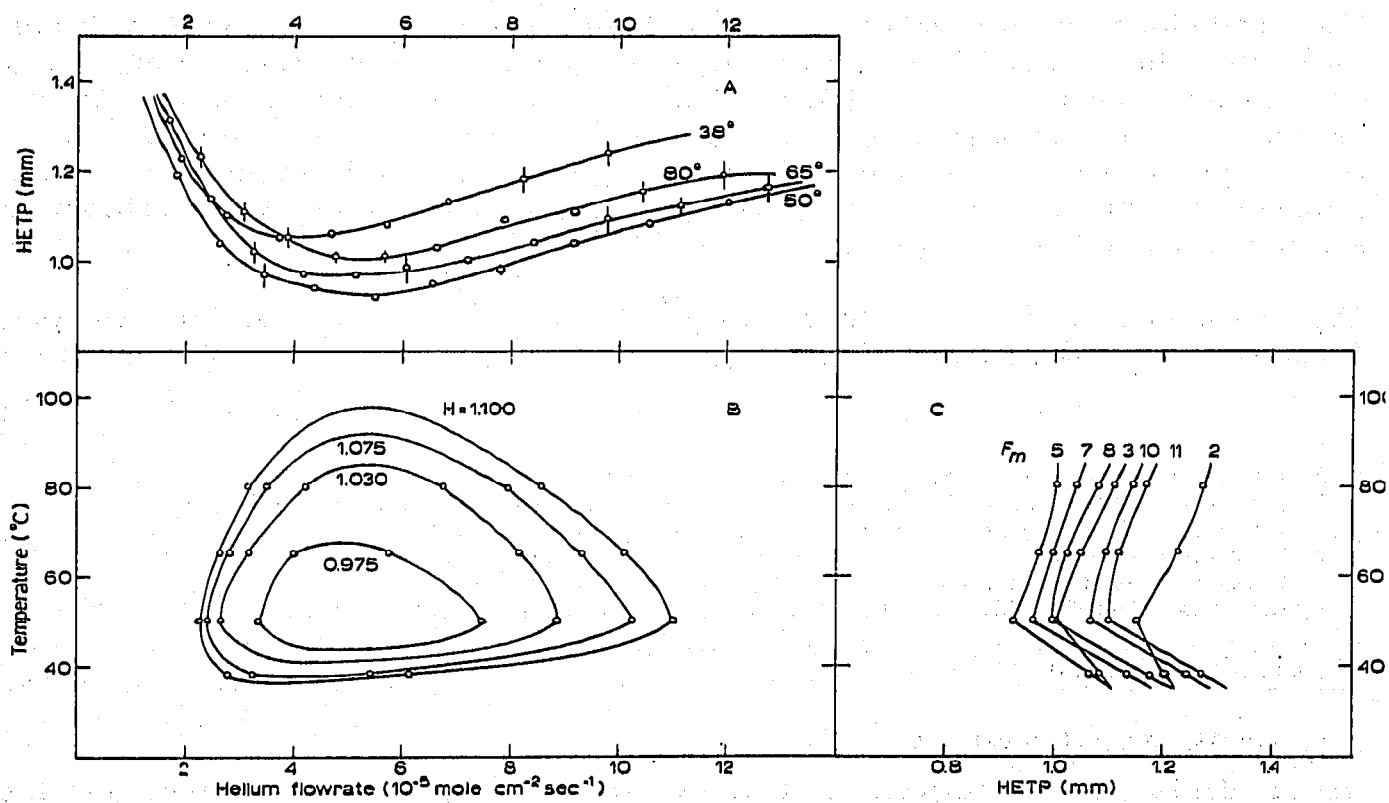

Fig. 3. The HETP plotted against absolute flowrate of the helium carrier gas at various column temperatures (curve A) and HETP plotted against column temperature at various absolute flowrates of carrier gas (curve C). Curve B shows the contour lines of HETP. The vertical lines in curve A denote the 95% confidence interval.

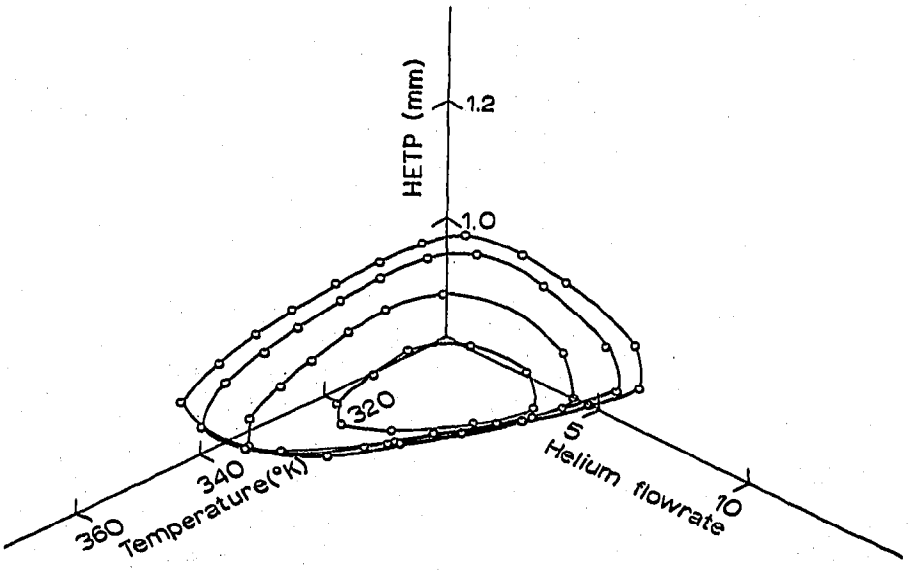

Fig. 4. Three dimensional model for the HETP expressed as the function of absolute carrier gas flowrate and column temperature (°K).

F_m at various temperatures. On increasing the column temperature from 50° to 80°, the H–F_m curve is shifted (lowered), and a minimum H-value was obtained at 50°, but at 38° the H–F_m curve is again shifted upwards and the column efficiency decreased. If we pick out the points that give the same value of HETP in curve A, Fig. 3 and plot them as a function of column temperature and flowrate, we can obtain the contour lines of HETP as shown in curve B, Fig. 3. The dependence of HETP on the column temperature is shown in curve C, Fig. 3. Illustrating these relations in oblique co-ordinates, we can obtain the conical diagram as shown in Fig. 4. With these diagrams one can obviously understand how the HETP depends on column temperature and the flowrate of carrier gas. The best condition of the column is given by the lowest point in Fig. 4.

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