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Determination of a Liquid–Vapor Phase Diagram

An experiment for determining the liquid-vapor phase diagram for the benzene-toluene system by GLC has been developed as a modification of a similar experiment using refractive indices.[1] The apparatus consists of a simple distillation setup and a GLC. The experiment accomplishes two things: (1) It gives the student some "feeling" for the meaning of a phase diagram besides the usual eutectics so often studied; (2) The student learns to use and understand a GLC system. The experiment can be carried out in 4 hr of laboratory time when the students work in pairs and there is a GLC unit for each three or four pairs of students.

A distillation is started with 50 ml of a 50/50 by volume mixture of benzene and toluence in the pot. Ten milliliters of distillate are collected using some care to avoid evaporation of the distillate during collection. A few drops of distillate are then collected in a separate vial. The temperature of this fraction is recorded. The whole apparatus is allowed to cool to below the boiling point of the benzene and a few drops of the residue in the flask are placed in another capped vial. This procedure is repeated after every 10 cc of distillate is collected until four samples of distillate and four residue samples are obtained. Each of these samples is analyzed by GLC. The boiling points of the benzene and toluene used are then obtained separately using the same apparatus.

The GLC apparatus used by our students was a Fisher Gulph Partitioner and a Speedomax H Recorder. An 8-ft, $^1/_4$ in. o.d. copper column, filled with 25% carbowax-1500 on 80–100 mesh chromosorb, was used. The runs were made at 15 psig helium pressure and 75°C column temperature. There are other columns and GLC's equally useful for this benzene-toluene separation. To calibrate, the ratio of the peak height of benzene to the peak height of toluene plus the peak height of benzene is determined from the GLC analysis for three mixtures of known composition. (A calibration obtained by a student is shown in Fig. 1.) The mole fractions of the distillates and residues are then determined from the corresponding peak height ratios and the calibration curve. The mole fractions are then plotted versus the corresponding temperatures to give the phase diagram (Fig. 2).

[1] This experiment is a modification of the one described by Daniels, F., Williams, J. W., Bender, P., Alberty, R. A., and Cornwell, C. D., "Experimental Physical Chemistry," (6th *ed.*), McGraw-Hill Book Co., Inc., **1962,** p. 54 ff.

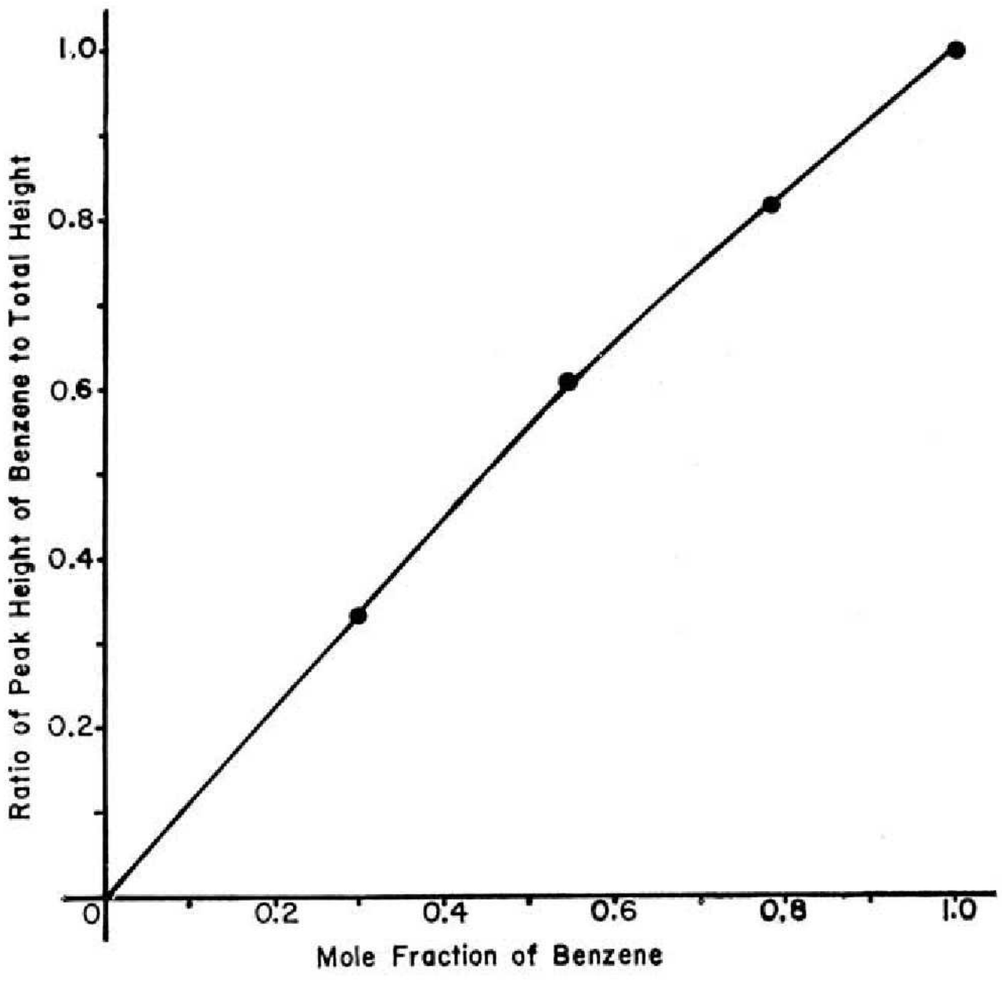

Figure 1. GLC calibration curve used to obtain mole fraction from peak height ratio.

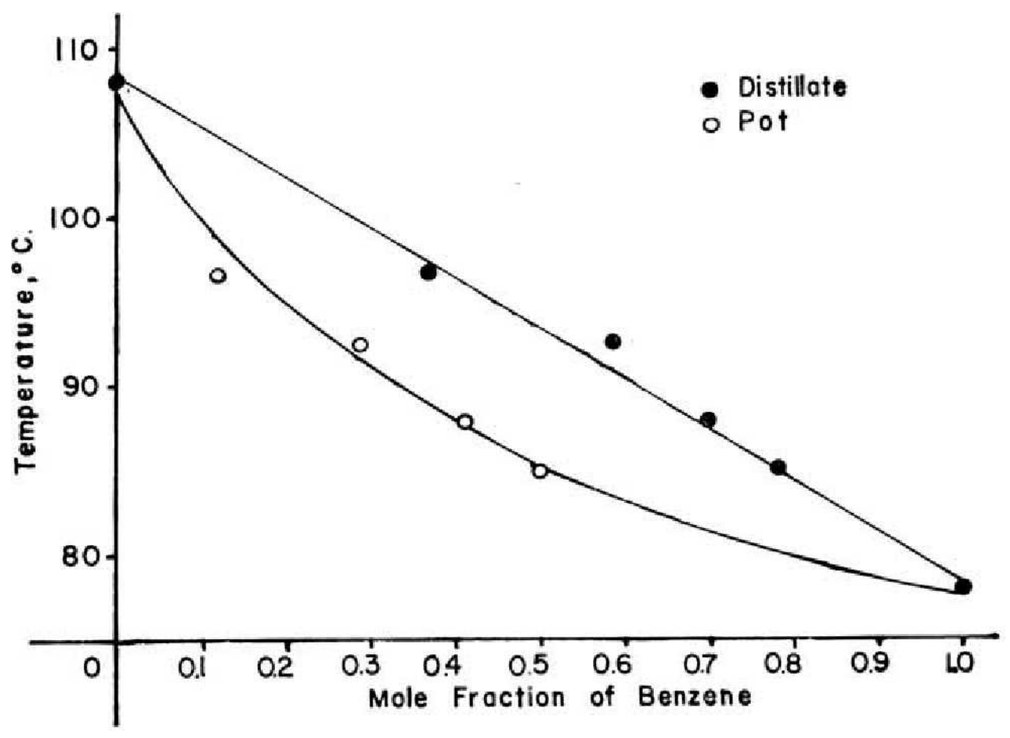

Figure 2. A liquid-vapor phase diagram for the benzene-toluene system as determined by the method of this note.