

on Whatman No. 1 paper impregnated with 0.5 *M* KCl solution, dried, and developed 25–35 cm with *n*-butanol–HCl (98:2 v/v), water-saturated, are reported in Table I. Revelation was achieved by spraying the dried paper with modified Dragendorff reagent.

The major alkaloids, in the several root samples investigated, are III and V; the major quaternary base is VI. Compound II is choline. None of these alkaloids is nicotine and this compound has not been observed in root and leaf samples investigated.

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### Gas chromatographic analysis in fractional distillation of multi-component systems

The course of fractional distillation of a multi-component system is commonly followed by noting the changes in boiling points and/or refractive indices. These techniques are not completely adequate in those cases in which the boiling points are very close and the differences in refractive indices are very small or non-existent. Gas chromatography offers a very useful and indispensable tool for following fractional distillations of this type. Gas chromatographic analysis of each fraction as the distillation proceeds permits one to make a more judicious choice in effecting the best separation of the components and therefore obtaining a maximum amount of each component in high purity.

In this note, the utilization of gas chromatography in the purification of 2,2,4,6,6-pentamethylheptane (PMH) by fractional distillation is described.

#### *Experimental*

PMH was prepared by catalytic hydrogenation of commercially available triisobutylene<sup>1</sup>. The crude PMH, after washing and drying, was charged to a total condensation, intermittent take-off-type distillation column. The packed section, 1 in. by 4 ft., was packed with Podbielniak Heli-Pak No. 2117 stainless steel packing. The column and take-off valve were designed so that the distillate did not come in contact

with stopcock grease at any point. The distillation was carried out at 18 mm pressure. A chaser, 200 g of eicosane, was added. After proper equilibration of the column, fractions of 25 ml or less were taken. The take-off valve was operated by a solenoid and a 30-sec cycle timer arrangement so that the valve was open 5 % of every 30-sec cycle. Refractive indices and chromatograms were obtained on each fraction.

The chromatograms were obtained on a 26-ft. column. The packing consisted of Dow-Corning high vacuum silicone grease, 20 % by weight, on 30-60 Chromosorb. The conditions for the chromatograms are given in Fig. 1.

### Results and discussions

WHITMORE *et al.*<sup>2</sup> reported that triisobutylene is composed of four components: (a) 35 % 2,2,4,6,6-pentamethylheptene-3, (b) 55 % 2-neopentyl-4,4-dimethylpentene-1, (c) 5 % 2,4,4,6,6-pentamethylheptene-1, and (d) 5 % 2,4,4,6,6-pentamethylheptene-2. Hydrogenation of this mixture would yield two saturated hydrocarbons: 2,2,4,6,6-pentamethylheptane (PMH) and 2,2,4,4,6-pentamethylheptane. The purification of PMH, obtained by hydrogenating triisobutylene, is mainly a problem of separating the PMH from 2,2,4,4,6-pentamethylheptane. A chromatogram of the crude PMH is shown in Fig. 1. This purification was accomplished by careful fractional distillation of the crude PMH at reduced pressure.

Gas chromatography served as a valuable analytical aid in following the course of the distillation. The gas chromatograms provided the information necessary to decide which fractions were pure PMH. The chromatograms of the first six fractions showed that impurities, more volatile than PMH, were present. The refractive indices and boiling points for fractions 2 through 6 indicated that these fractions were essentially pure PMH. A representative chromatogram of those obtained for each of the first six fractions is shown in the chromatogram for fraction 2 in Fig. 1.

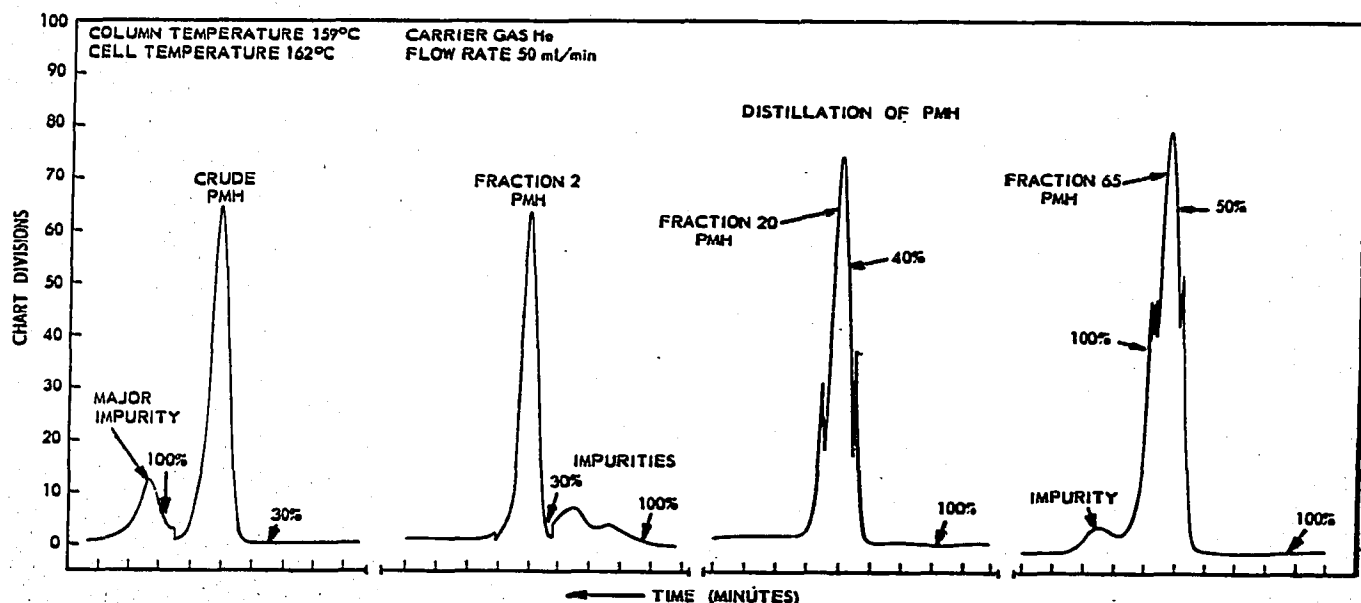


Fig. 1. Representative chromatograms. The relative responses (sensitivities) are given in percentages along the chromatograms.

The impurities, of course, decreased in amount with each fraction through fraction 6 and were not present starting with fraction 7. The chromatogram for fraction 20, Fig. 1, is representative of those obtained for each of the fractions 7 through 63. These fractions represent the pure PMH obtained by the fractional distillation of the crude material. Starting with fraction 64, a small peak was present immediately after the PMH peak, even though there was no apparent change in the boiling point or refractive index. This showed that the higher boiling impurity, 2,2,4,4,6-pentamethylheptane, was present, and the amount increased with each succeeding fraction. The chromatogram for fraction 65 is representative of the chromatograms for these fractions, with the higher boiling impurity increasing with each fraction. The utilization of gas chromatography in this particular case provided the necessary information as to which fractions were pure PMH, and, therefore, a maximum amount of pure PMH was obtained.

Gas chromatography offers a valuable analytical tool in following the course of the fractional distillation of multi-component systems. In those cases where differences in boiling points are small and differences in refractive indices are non-existent, gas chromatography is virtually indispensable.

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### **Use of 2-methylpiperazine diformamide in gas chromatography**

In the course of a research project being carried out in this laboratory, an experimental quantity of 2-methylpiperazine diformamide was prepared from 2-methylpiperazine and formic acid. This compound proved to be very polar and showed good thermal stability. It distilled at 200° at about 5 mm pressure without decomposition and melted at about 36°.

Because of its high polarity, low melting point, and thermal stability, this compound appeared to be a logical choice as a stationary phase in gas chromatography as a substitute for dimethylformamide or dimethylsulfolane, especially under