

# Separation of *n*-Propylbenzene and Each of the Three Methyleneethylbenzenes from Petroleum

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THIS REPORT describes the isolation of the following four C<sub>9</sub> alkylbenzenes from the representative petroleum of the API Research Project 6 (4): *n*-propylbenzene at 159.22° C.; 1-methyl-3-ethylbenzene at 161.30° C.; 1-methyl-4-ethylbenzene at 161.99° C.; and 1-methyl-2-ethylbenzene at 165.15° C. Because this work was completed some years ago, and is just now being reported for the record, only the barest details are given—the separations involved could today be accomplished much more easily.

## PROCEDURE FOR SEPARATION

The aromatic material of the range 159° to 166° C. was separated from the paraffin and cycloparaffin material by azeotropic distillation of appropriately narrow boiling fractions of the original petroleum.

By systematic regular distillation, the aromatic material was separated into the following portions: a concentrate of *n*-propylbenzene, a concentrate mixture of 1-methyl-3-ethylbenzene and 1-methyl-4-ethylbenzene, and a concentrate mixture of 1-methyl-2-ethylbenzene and 1,3,5-trimethylbenzene.

The "best" sample of *n*-propylbenzene was obtained from its concentrate by crystallization with centrifuging using dichlorodifluoromethane as a solvent.

Extended regular distillation of the mixture of 1-methyl-3-ethylbenzene and 1-methyl-4-ethylbenzene concentrated the former compound in the front end of the distillate. The best sample of 1-methyl-3-ethylbenzene was separated from this material by crystallization with centrifuging using ethane as a solvent. Continued regular distillation of the remaining material of this mixture concentrated the 1-methyl-4-ethylbenzene near the tail end of the distillate. The best sample of 1-methyl-4-ethylbenzene was separated from this material by crystallization with centrifuging using ethane as a solvent.

The mixture of 1,3,5-trimethylbenzene and 1-methyl-2-ethylbenzene was distilled azeotropically to give a concentrate of the latter compound in the tail end of the distillate. The best sample of 1-methyl-2-ethylbenzene was separated from this material by crystallization with centrifuging using ethane as a solvent. No work was done on the remaining

material, as a best sample of 1,3,5-trimethylbenzene had been isolated (3).

The freezing points and purities of the four compounds separated are given in Table I. The amounts estimated to be in the representative petroleum of the API Research Project 6 are given in the last column of Table I.

Table I. Freezing Points, Purities, and Amounts in the Petroleum

| Compound                             | Freezing Point in Air at 1 Atm., ° C. | Purity of Sample Mole % |              | Estd. Amount in Petrol., <sup>a</sup> Vol. % |
|--------------------------------------|---------------------------------------|-------------------------|--------------|--|
|                                      |                                       | <sup>b</sup>            | <sup>c</sup> |  |
| <i>n</i> -Propylbenzene <sup>d</sup> | -100.07                               | 98                      | ..           | 0.09   |
| 1-Methyl-3-ethylbenzene <sup>d</sup> | -96.21                                | 98                      | 99           | 0.17   |
| 1-Methyl-4-ethylbenzene <sup>d</sup> | -64.07                                | 94                      | 94           | 0.06   |
| 1-Methyl-2-ethylbenzene <sup>d</sup> | -89.36                                | 91                      | 89           | 0.09   |

<sup>a</sup> See also Forziati and Rossini (2).

<sup>b</sup> Determined from measurements of freezing points.

<sup>c</sup> Determined spectrographically from measurements made in Research Laboratory, Sun Oil Co.

<sup>d</sup> Values for boiling point, in ° C. at 760 mm. Hg. refractive index ( $n_D^{25}$ ) and density (g./ml.) at 25° C., respectively, differed from values given by the API Research Project 44 (1), as follows: *n*-propylbenzene, 0.00° C., 0.0001, 0.0001 g./ml.; 1-methyl-3-ethylbenzene, -0.24° C., -0.0002, -0.0010 g./ml.; 1-methyl-4-ethylbenzene, -0.51° C., -0.0004, -0.0017 g./ml.; 1-methyl-2-ethylbenzene, 0.08° C., 0.0008, 0.0020 g./ml.

## LITERATURE CITED

- (1) American Petroleum Institute Research Project 44, Carnegie Institute of Technology, Pittsburgh 13, Pa.
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- (3) Mair, B. J., Schick Tanz, S. T., *Ibid.*, **11**, 665 (1933).
- (4) Rossini, F. D., Mair, B. J., Streiff, A. J., "Hydrocarbons from Petroleum," Reinhold, New York, 1953.

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