NEOPENTYL DEUTERIDE

Sir:

Since an intensive study of neopentane and its derivatives is being made in this Laboratory, the importance of preparing and studying neopentyl deuteride is obvious.

A quantity of neopentylmagnesium chloride [Whitmore and Fleming, THIS JOURNAL, **55**, 4161 (1933)] was prepared and this material was divided into two equal portions. One portion was allowed to react with 2.5 cc. of distilled water to give neopentane. The other portion was allowed to react with 2.5 cc. of heavy water (sp. gr., 1.0735) and 6.9 g. (yield, 78%) of a mixture of neopentane and neopentyl deuteride was obtained. The reactions were carried out in the same apparatus in exactly the same manner, the only difference being the substitution of heavy water for ordinary water in the second case.

A 4.5-cc. sample of the neopentane produced in this manner was used to obtain its Raman spectrum. This material yielded a spectrum which was identical with that obtained earlier [Rank, *J. Chem. Physics*, **1**, 572 (1933)] for pure neopentane. A similar sample of neopentane and neopentyl deuteride yielded a spectrum which clearly showed the presence of the deuteride. The carbon deuterium vibration is shown by the appearance of a line at approximately $\Delta\bar{\nu} = 2150$ cm.$^{-1}$ shift from the exciting line, which is in accord with the predictions of simple theory. The spectrum clearly shows that the introduction of the deuterium atom into the neopentane molecule partially removes the vibrational degeneracy of this molecule.

The following physical constants were then determined on the neopentane and the neopentane-neopentyl deuteride mixture, using the same apparatus and technique for both.

Melting Point.—The melting points were determined simultaneously and it was observed that the neopentane melted at −21 to −20° and the neopentane-neopentyl deuteride mixture at −22 to −21°. Since these two samples were prepared in precisely the same way as to reagents, conditions and treatment, it appears that the neopentyl deuteride causes a lowering of the melting point of the mixture of about 1°. The purest neopentane that we have had showed a freezing point of −19.5° [Whitmore and Fleming, THIS JOURNAL, **55**, 3803 (1933)].

Refractive Index.—Using a Valentine refractometer of the Abbe type (manufactured by the Industro-Scientific Co., Colmar, Penna.; calibration checked by Bureau of Standards) at −7.4° (room temperature, −7.4°), the refractive index of the neopentane was observed to be 1.35375 ± 0.00005 and that of the neopentyl deuteride-neopentane mixture to be 1.35360 ± 0.00005.

Boiling Point.—The difference in the two boiling points at 740 mm. was approximately 0.2°, the deuteride having the lower boiling point.

Preparation of pure neopentyl deuteride is in progress, upon which accurate physical constants will be obtained. Extensive studies will be made on this unique compound.

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THE ISOTOPIC FRACTIONATION OF WATER BY DISTILLATION

Sir:

In work with heavy water, particularly in exchange of experiments, distillations are frequently desirable either to separate water from substances dissolved in it, or to purify the separated water. It is perhaps worth while to call attention to the serious effect on the results which may arise from isotopic fractionation during distillation. This effect will not be found, of course, where the entire quantity of water distilled is collected, nor when a truly representative sample is obtained by discarding exactly equivalent amounts of the first and last runnings. Moreover, the work of Washburn [Washburn, Smith and Frandsen, *Bur. Stand. J. Res.*, **11**, 453–462 (1933)], and Lewis [Lewis and Cornish, THIS JOURNAL, **55**, 2616 (1934)], indicates that the effects to be expected on samples approaching ordinary water in composition will be very small in ordinary apparatus at atmospheric pressure. That such effects are far from negligible in dealing with more highly enriched samples is shown by the following results. (1) A sample of 125 cc. volume containing about 3% of the heavier isotope was very carefully purified and then distilled slowly at atmospheric pressure from an ordinary flask surmounted by a simple Vigreux column about 10 cm. long connected to a quartz condenser. The vertical part of the condenser before the bend was about 20 cm. long. Three fractions of the distillate