The yield of the metal from the pentachloride is poor as compared to the iodide.

The pentachloride can be prepared readily at 550°, by reaction of the oxide with phosgene, according to the equation $Pa_2O_5 + 5COCl_2 \longrightarrow 2PaCl_5 + 5CO_2\uparrow$. Under these conditions the oxide is volatilized quantitatively; the pentachloride sublimes in beautiful transparent nearly colorless long needles, reaching a length of 1 cm. and more, if sublimed slowly. The chloride melts at 301° at atmospheric pressure to a pale yellow liquid, but sublimes appreciably below its melting point. The substance is ideally suited for an atomic weight determination of protactinium, which is under way.

The present experiments are of a preliminary nature; M. Agruss and the writer are planning to prepare larger amounts of the metal and study its properties quantitatively.

We take this opportunity to express our sincere gratitude to Mr. Hiram J. Halle of New York City, who made this work possible by a generous financial grant. We are very much indebted to Dr. Gustav Egloff for placing at our disposal for this work the facilities of the Research Laboratories of the Universal Oil Products Company. It is also a pleasure to acknowledge the assistance of M. Agruss and L. Borst.

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RECEIVED SEPTEMBER 10, 1934

THE EFFECT OF FRACTIONAL CRYSTALLIZATION ON THE ISOTOPIC COMPOSITION OF WATER

Sir:

In a recent communication Professor G. Bruni [THIS JOURNAL, **56,** 2013 (1934)] has discussed the possibility of separating the isotopic species present in ordinary water by fractional crystallization. His conclusion, based on experiment, is that "*if* a fractionation takes place it amounts to less than $^1/_{10,000}$ ..... when the original mass is reduced to $^1/_{14,300}$." The figure probably refers to change in specific gravity.

I have recrystallized distilled water by freezing half the amount taken and advancing the fractions as was done by Richards and Hall [*ibid.*, **39,** 531 (1917)]. The water was thoroughly stirred during the two hours required for freezing. Samples were further purified and specific gravities were taken in the way already described [E. S. Gilfillan, Jr., THIS JOURNAL, **56,** 406 (1934)]. Blank runs were made between each two determinations; the average deviation of these from the mean was 0.1 p. p. m. The results are given in Table I.

TABLE I

Crystns.	10	10	8	12	8	12
Sp.—gr., 1, p. p. m.	1.2	1.0	0.9	1.1	0.7	1.3

The average effect per crystallization is 0.10 p. p. m. The effect was also determined for 6% sodium chloride solutions and found to be 0.5 ± 0.3 p. p. m.

The experiments of La Mer, Eichelberger and Urey [THIS JOURNAL, **56,** 248 (1934)] permit an approximate thermodynamic calculation of the effect to be expected due to concentration of deuterium. The following assumptions are made: (1) In mixtures of protium and deuterium oxides the molecular species H^1H^2O, H^1H^2O, and H^2H^2O, are present in mobile equilibrium. (2) The equilibrium constant $K_c = [H^1H^2O]^2/[H^1H^1O][H^2H^2O] = 4$. (3) These species form "perfect solutions" in the sense of Lewis [G. N. Lewis and M. Randall, "Thermodynamics," McGraw-Hill Book Co., 1923, p. 221] in both liquid and solid phases. (4) The heats of fusion of the three kinds of water are the same. (5) The freezing point of deuterium oxide is 3.82°. (6) The specific gravity of ordinary water is 22.2 p. p. m. higher than that of H^1H^1O. The first two assumptions have been made by Lewis and Cornish [THIS JOURNAL, **55,** 2616 (1933)]. The result is that one crystallization should change the specific gravity of ordinary water by 0.22 ± 0.01 p. p. m. This indicates that fractional crystallization concentrates O^{16} with respect to O^{18} and that the freezing point of $H^1H^1O^{18}$ is $-0.1 \pm 0.05°$.

From each of the measurements of La Mer, Eichelburger and Urey the freezing point of H^1H^2O can be calculated. The values found are 2.20, 2.21 and 2.28, average 2.23°. From this value one can calculate the freezing point of the water studied by Washburn, Smith and Frandsen [*Bur. Standards J. Research*, **11,** 453 (1933)] to be 0.053° in agreement with the value found.

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PUBLICATION NO. 338 FROM THE
RESEARCH LABORATORY OF PHYSICAL CHEMISTRY
OF THE MASSACHUSETTS INSTITUTE OF TECHNOLOGY
CAMBRIDGE, MASS.
RECEIVED SEPTEMBER 12, 1934