

dently identical with the quinone obtained by Skita, m. p. 142°. Needles melting at 142–144° were obtained by crystallization from methanol.

**Silver Oxide Oxidation of Octahydrophenanthrenehydroquinone.**—A mixture of 0.42 g. of hydroquinone (m. p. 133–137°), 1 g. of silver oxide, and 1 g. of magnesium sulfate in absolute ether was shaken for one-half hour and filtered. The quinone proved to be soluble with difficulty in ether and was therefore extracted from the solids with benzene. Most of the benzene was evaporated and the product (0.338 g., m. p. about 137°) precipitated from the remaining solvent with hexane. Red needles melting at 142° were obtained by crystallization from acetone, which proved to be the best solvent for the purpose.

**Reductive Acetylation of Octahydrophenanthraquinone.**—Two drops of triethylamine was added to 0.1 g. of quinone with 0.1 g. of zinc dust in 2 cc. of acetic anhydride. The mixture was allowed to stand for ten minutes, heated to boiling, filtered, and the residue washed with hot acetic acid. Water was added to the boiling filtrate and washings to the cloud point. On cooling, the solution deposited 0.134 g. of white needles melting at 162–163°.

**Acetylation of the Hydroquinone.**—Addition of a drop of triethylamine to some of the hydroquinone (m. p. 135–137°) with a pinch of zinc dust in acetic anhydride caused slight warming. The mixture was allowed to stand a few hours and boiled for five minutes. The mixture was worked up in the same way as the product from the reductive acetylation of the quinone. The white needles melted at 161.5–163°. The mixed m. p. with the diacetate from the quinone was 162–163°.

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### Atomic Weight of Lead from a Second Sample of Pitchblende, Great Bear Lake, N. W. T., Canada

BY JOHN PUTNAM MARBLE

In a continuation of the geochemical studies on the pitchblende ores of Great Bear Lake, the atomic weight of the lead in a second sample has been determined. This sample, from the 800-foot section of the No. 2 vein, Eldorado mine, Labine Point, Great Bear Lake, N. W. T., Canada, was received from H. S. Spence, Esq., of the Canada Department of Mines and Resources, for whose coöperation we are deeply grateful. The material is of the pitchblende-silica type ore, came from depth (below 100 feet), and carried no visible alteration products. Microscopic studies show a very small amount of galena, estimated at a fraction of one per cent.

The lead-uranium ratio uncorrected for "com-

mon lead," with an autoradiograph showing little evidence of alteration<sup>1</sup> and a complete analysis<sup>2</sup> have been previously published. While the results of this present work are admittedly incomplete, it was thought worth while to put them on record, as they are in virtual agreement with those of the first sample<sup>3</sup> and the carrying out of further atomic weight determinations, or preferably of the determination of the isotope ratios of this sample, have necessarily had to be deferred. Since the pitchblende is virtually thorium-free, the atomic weight indicates the approximate correction to be made for "common lead," which cannot be done for thorium-rich minerals.

The preparation of the material for analysis, the extraction and purification of the lead, and the method of analysis were essentially the same as in the case of the first sample of Great Bear Lake lead.<sup>3</sup> The work was done in the T. Jefferson Coolidge Memorial Laboratory of Chemistry at Harvard University, by kind permission of Dr. Gregory P. Baxter, for whose interest and assistance we are deeply grateful. The results of the analysis are as follows

Vac. wt. PbCl <sub>2</sub>	1.56541 g.
Vac. wt. Ag	1.21935 g.
Wt. Ag added in solution	0.00010 g.
Vac. wt. Ag ⇌ PbCl <sub>2</sub>	1.21945 g.
Ratio PbCl <sub>2</sub> /2Ag	1.28370
Atomic weight Pb	206.057
(Ag = 107.880; Cl = 35.457)	

This value agrees so closely with that of the work on the earlier sample (Pb = 206.054), that we may conclude that the lead in the Great Bear Lake pitchblende ore shows no appreciable variation in isotopic composition in different parts of the deposit, and also that a correction of about 4 per cent. should be applied to the "uncorrected" lead-uranium ratio of the second analyzed sample, as was done for the first. Further work may confirm these conclusions.

(1) J. P. Marble, *Am. Mineralogist*, **22**, 564 (1937).

(2) J. P. Marble, *ibid.*, **24**, 272 (1939).

(3) J. P. Marble, *THIS JOURNAL*, **56**, 854 (1934).

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### Saponins and Sapogenins. XX. Some Color Reactions of Triterpenoid Sapogenins

BY C. R. NOLLER, R. A. SMITH, G. H. HARRIS AND J. W. WALKER

While attempting to prepare the acid chlorides of some triterpenoid acids by means of thionyl