Summary

The conductance of solutions of trimethyl tin chloride in nitrobenzene and acetone on addition of small amounts of pyridine has been measured. A marked increase takes place in conductance on addition of pyridine, and this effect is the greater, the smaller the amount of pyridine already present.

The conductance of solutions of trimethyl tin chloride in mixtures of 0–100% of acetone and alcohol and nitrobenzene and pyridine has been measured. The initial conductance in nitrobenzene is extremely low, indicating that trimethyl tin chloride is practically un-ionized in this solvent. The initial conductance in acetone is markedly higher than in nitrobenzene. While the conductance in acetone on addition of alcohol is increased, this increase is much less marked than it is on addition of pyridine.

The results are in agreement with the view that trimethyl tin chloride itself is not a true electrolyte and that its electrolytic properties in solution are due to the formation of compounds of the oxonium and ammonium type, the trimethyl tin group transferring from chlorine to tetravalent oxygen or pentavalent nitrogen, respectively.

WORCESTER, MASSACHUSETTS

[CONTRIBUTION FROM THE WOLCOTT GIBBS MEMORIAL LABORATORY, HARVARD UNIVERSITY]

THE ATOMIC WEIGHT OF LEAD FROM THE BELGIAN CONGO

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The fundamental relation of the isotopes of lead to the process of radioactive disintegration gives an especial interest to the atomic weights of these isotopes. The difference in the geological histories of uranium deposits in different parts of the world makes it especially desirable that the quantitative relations of the lead in deposits from as many widely separated localities as possible should be examined. Within the past few years, as is well known, a new source of uranium and radium material has been found in the Belgian Congo. Accordingly, we have investigated the atomic weight of lead derived from this source.

The metal employed was obtained from a mixture of several minerals existing in the center of Africa. These all doubtless originated in a common primary deposit of uraninite, of which an analysis has been published.[2a] Among the decomposition products of this primary mineral, Schoep[2b] has discovered several secondary minerals containing lead, namely, curite ($2PbO.5UO_3.4H_2O$), kasolite ($3PbO.3UO_3.3SiO_2.4H_2O$), dewindtite ($4PbO.8UO_3.3P_2O_5.12H_2O$) and stasite ($4PbO.8UO_3.3P_2O_5.12H_2O$). In all these

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[2a] Steinkuhler, *Bull. soc. chim. Belg.*, **32**, 233 (1923).

[2b] Schoep, *Compt. rend.*, **173**, 1186, 1476 (1921); **174**, 623, 875 (1922).

minerals, the lead was probably chiefly derived from the uraninite—the end-product of a large amount of uranium which had undergone disintegration. The specimen upon which we worked was from a mixture of all these minerals, and was obtained through the kindness of Professor W. Mund, of Louvain, Belgium. Monsieur Clérin, chief chemist of the Hoboken plant of the Compagnie minière du Katanga, has since been so kind as to give us larger amounts of the same material; to both we express our hearty thanks. This lead apparently may be available in quantity, and may therefore be used for a great variety of large scale experiments on the comparison of isotopes. For this reason, especially, a determination of its atomic weight is of value. A qualitative test by means of the gold-leaf electroscope showed appreciable radioactivity.

The method of determination adopted in the present research was essentially the same as that used by Baxter and his assistants[3] in the study of the atomic weight of ordinary lead, and by others in this Laboratory for the determination of the atomic weight of uranium lead.[4] For this reason a very brief account suffices.

Purification of Materials

The methods employed for the purification of nitric acid, water and the several gases used in the work needed no variations from the usual procedure. The silver we owe to the kindness of Dr. Norris F. Hall, who prepared a large quantity of the metal in a very pure state for coulometric work several years ago.[5]

The preparation of the lead deserves more detailed treatment. For comparison with the radioactive sample and for practice, a small sample of ordinary lead chloride was prepared from C. P. lead nitrate (once recrystallized) by precipitation in dilute solution by hydrochloric acid. The chloride was recrystallized twice from slightly acidified water. Although not very thoroughly purified, it was pure enough to serve as a basis for practice analyses and to show that the details of the analytical process were well in hand. This was designated as Specimen A.

The radioactive metal (which had been already purified considerably by commercial methods) was dissolved in pure nitric acid, the solution filtered and evaporated, and the lead nitrate recrystallized thrice, the last of these recrystallizations being conducted in quartz dishes. The solution of the purest nitrate was filtered again through a Gooch-Monroe platinum crucible, and subsequently lead chloride was precipitated from it by pure hydrogen chloride. After two recrystallizations (using purest water and quartz dishes) the recrystallized lead chloride was carefully dried for anal-

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[3] Baxter and others, THIS JOURNAL, **30**, 187 (1908); **37**, 1020, 1027 (1915).

[4] Richards and others, *ibid.*, **36**, 1329 (1914); **38**, 2613 (1916); **39**, 531 (1917). *Smithsonian Report* for **1918**, p. 205, Pub. No. 2557.

[5] Richards and Hall, THIS JOURNAL, **39**, 531 (1917); **38**, 2045 (1916).

ysis; it constituted Sample B. From the mother liquors of this preparation, the residual chloride was precipitated by concentration and by addition of hydrochloric acid, and was recrystallized. It was then converted into the nitrate by repeated evaporation with the purest nitric acid, and the nitrate was recrystallized thrice from very acid solutions. Two more recrystallizations as nitrate from the purest water and filtration through a Gooch-Monroe crucible provided a very pure solution of the nitrate, which was precipitated by a solution of the purest hydrochloric acid. Three recrystallizations of the lead chloride from the purest water acidified with a few drops of hydrochloric acid (with centrifugal draining, as usual) yielded a preparation C. This specimen was doubtless purer than Specimen B, having received much more careful treatment and many more recrystallizations. The several samples were kept dry in quartz dishes in desiccators containing solid caustic alkali.

Method of Analysis

Seven analyses were made of these several samples. The method may be very briefly outlined.

The lead chloride was fused in a platinum boat in a current of pure hydrogen chloride. This gas having been displaced by pure nitrogen during the cooling, the product was finally enclosed in a weighing bottle in a current of pure dry air by means of the well-known bottling apparatus usually employed in such work. In every case the fused lead chloride was entirely colorless and transparent; but upon dissolving it in warm water slightly acidified with nitric acid in a suitable Erlenmeyer flask, traces of insoluble black residue were found floating on the surface of the water, especially in Specimens A and B. This impurity was collected on a tared Gooch-Monroe crucible, and weighed, and the weight was duly subtracted from the weight of the chloride. The average weight

TABLE I

DATA AND RESULTS

Analysis	Corrected wt. fused $PbCl_2$ in vacuum	Corrected wt. equivalent Ag in vacuum	Ratio $PbCl_2$/Ag	Atomic weight
	Sample A.	Ordinary lead		
1	5.70194	4.42331	1.28906	207.21
2	4.65819	3.61405	1.28891	207.18
3	4.87664	3.78388	1.28879	207.15
				Av. 207.18
	Sample B.	Congo lead (preliminary analyses)		
4	3.38089	2.63325	1.28392	206.10
5	4.21302	3.28093	1.28409	206.14
				Av. 206.12
	Sample C.	Congo lead (final analyses)		
6	3.66388	2.85263	1.28439	206.20
7	4.30262	3.34997	1.28438	206.20
				Av. 206.20

The data are calculated for Ag = 107.88 and Cl = 35.458.

of the contamination, the nature of which was not determined, was (counting all determinations) less than 0.2 mg.. and in the last two (the most important) determinations, less than 0.1 mg. The platinum boat was weighed before and after each analysis and its average loss of weight found to be only 0.07 mg.

The clear solution of the lead chloride used occupied a volume of about 1.5 liters; it was precipitated with a suitable weighed amount of purest silver which had been dissolved, with the usual precautions, and the end-point was adjusted with the help of a few cubic centimeters of very dilute standard solutions of silver nitrate and hydrochloric acid by means of a nephelometer.

The rough preliminary analyses of ordinary lead chloride (Nos. 1, 2 and 3) for which no great accuracy is claimed, nevertheless yielded results (in average 207.18) sufficiently concordant with themselves and with the accepted value of the atomic weight of lead, 207.20, for the present purpose. The two following analyses upon the Congo lead (Nos. 4 and 5) likewise suffered from minor defects and were made with material only roughly purified. On the other hand, the final analyses (Nos. 6 and 7) were made with material far more carefully purified, and every step of each analysis was satisfactory. The loss in weight of the platinum boat and the weights of black residue were so small as to be practically negligible, and the end-point test in the nephelometer was unusually satisfactory. Further analyses seemed to be unnecessary, since these left nothing to be desired.

Evidently, lead from the uranium deposits in the Congo has an atomic weight exactly one unit less than the atomic weight of ordinary lead, and perhaps only 0.14 unit higher than that of pure uranium lead. Apparently, then, we may infer (assuming that the material contains nothing but a mixture of uranium lead and ordinary lead) that about 88% of the material under investigation consisted of uranium lead, and only 12% of ordinary lead. This is a far lower percentage of ordinary lead than that existing in the similar metal from Australia. The result leaves little room for doubt that the lead which forms an integral part of the four new radioactive minerals was really derived chiefly from uranium lead. The uranium lead from the disintegration must afterwards have combined chemically with much of the residual undisintegrated uranium. Clearly, these minerals must have been formed long after the original deposit of uraninite crystallized in its bed. In this case, as in others, a careful study of the atomic weight of the mixed isotopes in relation to the coexistent minerals thus affords an interesting clue concerning the geological history of the region.

In conclusion we are glad to express our indebtedness to the Education Foundation of the Commission for Relief in Belgium and to the Carnegie Institution of Washington for pecuniary assistance.

Summary

The atomic weight of a sample of radioactive lead obtained from a mixture of minerals found in radium ore from the Belgian Congo was deter-

mined as 206.20, as compared with a control sample of ordinary lead which gave the value 207.18. Evidently, therefore, the lead in these minerals consisted chiefly of uranium lead; and the minerals must have been formed long after the original deposit of uraninite.

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NOTES

A New Light Filter.—As far back as 1904 I observed accidentally that a solution of ordinary chrome alum of a proper concentration and thickness of layer is admirably adapted for detecting the potassium flame when it is masked by the presence of an excess of sodium light. It was found later by Mr. William D. Pardoe, a graduate student working in my laboratory, that it was possible to prepare a light filter with a solution of chrome alum which absorbs *completely* not only sodium light but that of lithium, strontium, calcium and barium. Viewed through such a filter the potassium flame is seen to rise from the glowing platinum loop in the form of a long, crimson, very bright streamer when a relatively large amount of this element is present, and less so, but always perceptible, as the quantity diminishes. Under the circumstances the flames of rubidium and cesium are also visible, but since these elements occur only in a few localities and in the minutest amounts, they are not apt to mislead one.

The filter is as sensitive as it is reliable and *will keep indefinitely*. One which has been in use off and on for fifteen years is as efficient today as it was when first prepared. In some cases, it has proved even more reliable than a Browning direct vision spectroscope. Such a filter is very handy in testing for potassium in the residues obtained by the evaporation of mineral and other waters, and in examining mineral silicates and silicate rocks for this element. Insoluble siliceous materials should be finely powdered, mixed with (a) pure gypsum or (b) four parts of pure calcium carbonate and one part of resublimed ammonium chloride, and the mixture made into a thick paste with water. A bit of the paste is then collected in a loop on the end of a platinum wire, brought into the fusion zone of the Bunsen burner, and the flame viewed through the filter.

To prepare a number of these filters (they will be found very useful in the laboratory for qualitative analysis), dissolve 310 g. of crystallized chrome alum in a large flask in a liter of water by gentle heating, cool and filter the solution and fill with it glass bottles of square prismatic form. Those used by me are 10 cm. high from bottom to shoulder, and the lateral edge measures 4.7 cm. Ordinary wooden corks will serve as stoppers. The filter is best held upright, very close to the eye, and about 5 or 6 cm. from the flame of the burner.

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