THE ATOMIC WEIGHT OF LEAD
FROM SAMARSKITE

by

Arthur Lowell Davis, A. B.

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g. (15/6/42)
3-22-39 Rg. Herman
Schlundt

HISTORICAL.

In the disintegration of the radioactive elements, since each α -transformation involves the loss of an atom of helium and nothing else which is weighable, the atomic weight of the product should be just 3.99 less than that of the original substance, since 3.99 is the atomic weight of the helium evolved during the α -transformation. Thus if radium has an atomic weight of 225.97^{12} , radium emanation, the result of the loss of one α -particle, should have an atomic weight of 221.98; radium D, involving the loss of three more α -particles, should be 210.01; and radium G, yet another α -transformation, should be 206.02.

Fajans¹⁰ and Soddy²² have independently proposed a theory which indicates that some of the places in the periodic table, corresponding to high atomic weights, should perhaps include a number of elements, slightly differing in atomic weight but very similar in other properties. Thus, in the place we usually assign to lead, we should expect to find a mixture of ordinary lead, radium D, radium G and perhaps at least one other radioactive product from thorium and one from actinium. These different

substances, according to the hypothesis, should have identical spectra and be inseparable by chemical means, but coming from different sources they should have different atomic weights. The theory supposes that each a-transformation involves the loss of valence of two, and each b-transformation involves a gain in valence of one. The b-transformation involves no change in weight. Thus radium D, supposed to have an atomic weight of 210, after two b- and one a-transformations, returns again as radium G to the same place in the periodic system with an atomic weight of only 206. This is the place assigned to lead, which some suppose to be primarily radium G, the only one of the radium series possessed of a long life and not highly radioactive.

Soddy has arranged the consecutive disintegration series for uranium, actinium and thorium. The nature of the rays emitted is also given and from his theory the atomic weights and valence may also be shown. From the table we may see the elements that may be considered as isotopes of lead.

*The brief statements in regard to the theory of radioactive change are made by T.W. Richards and Max E. Lambert, Jour. Amer. Chem. Soc., 36, 1329 (1914).

DISINTEGRATION SERIES FOR URANIUM AND ACTINIUM.

DISINTEGRATION SERIES FOR THORIUM.

Richards and Lember¹⁰, with the greatest of care that is characteristic in the determination of atomic weights at Harvard, have determined the atomic weights of samples of lead of widely distributed origin, geographically. They found that all of the radioactive specimens possessed a lower atomic weight than that of ordinary lead determined under identical conditions and in one case that the deficiency amounted to as much as 0.75 of a unit. The radioactivity of the various samples was determined quantitatively but no simple relationship between the amount of radioactivity and the atomic weight was found. The ultraviolet spectrum of a typical radioactive sample was compared with that of ordinary lead in a quartz spectrograph, but no difference was found in the spectra of the specimens. They infer that radioactive lead contains an admixture of some substance different from ordinary lead, and very difficult to separate by chemical means. It has later been shown that product of one thousand and six fractional crystallizations of radioactive lead has the same atomic weight as the original material, thus proving conclusively that no separation is possible by chemical means. The results obtained were contrary to experience with a number of other elements, notably copper,

silver, iron, sodium and chlorine, each of which has a constant atomic weight, no matter what the geographical source may have been.

Soddy and Hyman²⁰ extracted the small quantity of lead present Ceylon thorite, a hydrated silicate of thorium, and determined the atomic weight relative to ordinary lead by a standard method. They obtain an atomic weight of 208.4 compared with 208.2 calculated from the percentage of thorium and uranium present. Out of some twenty-four determinations on the atomic weight of lead, referred to by Brauner⁴, seven give values between 206.8 and 206.95, six between 206.95 and 207.1, none between 207.1 and 207.35 and six between 207.35 and 207.6, showing quite considerable variation.

Hönigschmid and Mme. St. Horevitz^{13, 14, 15} have obtained samples of radio-lead from primary minerals in order to obtain specimens as free of ordinary lead as possible. Lead obtained from crystallized uranium oxide gave an atomic weight of 206.06 showing the practical absence of outward contamination. A sample from broggerite gave 206.08 and a portion of the same sample distilled gave a result of 206.066 showing very closely agreeing values.

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M. Curie⁴, on the examination of lead extracted from pitchblende, carnotite, yttrio-tantalite and monazite sand, found it to give an atomic weight of 206.64, 206.36, 206.34 and 207.08 for the respective samples.

Richards and Wadsworth⁵ on determining the atomic weight of a number of samples of radio-lead obtained essentially the same results as previous experimenters. Every sample used, except the ordinary lead, possessed radioactivity. In conclusion their results show that the most carefully prepared samples seem to give lower values, this being strong evidence that the higher results obtained were due merely to the accidental admixture of ordinary lead in varying proportions.

The foregoing results are summarized on the following page.

DETERMINATIONS OF ATOMIC WEIGHT OF LEAD OF RADIO-
ACTIVE ORIGIN.

Richards and Lambert¹⁰

North Carolina uraninite.....	206.40
Joachimsthal pitchblende.....	206.57
Colorado carnotite.....	206.59
Ceylonese thorianite.....	206.82
English pitchblende.....	206.86

Soddy and Hyman²³

Ceylon thorite.....	208.40
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Honigschmid and St. Horovitz¹⁴

Joachimsthal pitchblende.....	206.405
Uraninite, Morogoro, G. E. Africa...	206.059
Broggerite, Moss, Norway.....	206.083
Broggerite, (PbCl ₂ distilled)....	206.066

Curie, Maurice⁶

Yttrio-tantalite.....	206.34
Carnotite.....	206.36
Pitchblende.....	206.64
Monazite sand.....	207.08

Richards and Wadsworth²⁰

Australian carnotite.....	206.34
American carnotite.....	207.00
Norwegian cleveite, Moss.....	206.08
Norwegian broggerite, Langesund...	206.12

The object of this work is an extension of the investigations on the atomic weight of lead of radioactive origin by determining the value for lead from another source, samarskite.

The problem involves the separation of the lead from this mineral, its purification, and determination of its atomic weight by analyses of the lead chloride obtained.

Parallel experiments with ordinary lead are included, thus showing the relative values obtained under the same conditions.

Finally, in order to correlate the results with the theory of radioactive changes involved, the percentages of uranium and thorium in the mineral were determined.

ANALYSES OF SAMARSKITE. 7

	1.	2.	3.	4.	5.	6.
Na ₂ O...	-	-	-	-	.23	-
K ₂ O....	-	-	-	-	.39	-
MgO....	-	1.53	-	-	.11	-
CaO....	.55	-	-	-	5.38	-
MnO....	.75	-	-	-	.51	-
FeO....	10.90	11.74	14.61	14.30	4.83	8.98
Fe ₂ O ₃ ...	-	-	-	-	-	1.66
Al ₂ O ₃ ...	-	-	-	-	-	2.00
Y ₂ O ₃ ...	14.45	14.49	6.10	8.80	14.34	11.90
Er ₂ O ₃ ...	-	-	10.80	3.82		
La ₂ O ₃ ...	4.24	4.24	2.37	4.33	4.78	3.85
Ce ₂ O ₃ ...						
Pr ₂ O ₃ ...						
UO ₂	-	-	-	-	-	13.48
UO ₃	12.45	10.96	10.90	11.94	10.75	-
SiO ₂	-	-	.56	-	-	-
TiO ₂	-	-	-	1.08	-	-
SnO ₂	.08	.31	.16	.22	.10	-
Nb ₂ O ₅ ...	37.20	55.13	41.07	55.34	55.41	56.40
Ta ₂ O ₅ ...	18.60	-	14.36	-	-	-
H ₂ O....	1.12	.72	-	-	2.21	.20
Density	-	5.72	5.839	5.672	4.95	5.96
	100.35	99.12	100.93	99.83	99.04	98.57

	7.	8.	10.	9.	10.	11.	12.
Na ₂ O....	.24(✓Li ₂ O)	.28	.76	.62		.48	-
K ₂ O.....	.17	.21	.08	.08			
B ₂ O.....	-	-	.30	.64	.25	-	
MgO.....	-	.41	.13	.19	t	-	
CaO.....	.27	.51	4.30	3.79	.94	2.43	
MnO.....	.78	.69	.86	.79	t	-	
FeO.....	.32	11.15	4.40	4.08	7.36	5.40	
ZnO.....	.05	.17	-	-	-	-	
BaO.....	-	-	.38	.38	-	-	
PbO.....	.72	.15	.77	.98	-	-	
Fe ₃ O ₄ ...	8.77	2.13	-	-	-	-	
Al ₂ O ₃ ...	-	.19	.36	.45	.80	.80	
Y ₂ O ₃	6.41	7.83	9.07	8.33	6.65	9.50	
Er ₂ O ₃ ...	10.71	13.37			2.72		
Ce ₂ O ₃ ...	.54	.25	.89	1.90	3.82	4.05	
La ₂ O ₃ ...	1.80	.37			1.07		
Di ₂ O ₃ ...		1.56			.74		
SnO.....	.95	.79	.57	.15	t	-	
ZrO.....	2.29TiO ₂	1.03	.62	.79	2.17	-	
ThO.....	3.64	1.73	2.51	2.59	4.23	1.05	
UO ₂	-	-	9.66	10.82	4.35	8.70	
UO ₃	4.02	11.23	6.78	5.88	-	-	
WO ₃	2.25	1.41	-	-	1.90	-	
Ge ₂ O ₃ ...	-	.07	-	-	-	-	
SiO ₂	-	.12	1.82	2.39	-	-	
TiO ₂	-	.68	-	-	.60	1.42	
Nb ₂ O ₅ ...	27.77	32.82	38.83	46.44	33.80	43.60	
Ta ₂ O ₅ ...	27.03	11.18	10.70	1.81	26.88	11.15	
H ₂ O.....	1.58	1.22	6.54	7.61	.22	11.14	
	<u>100.31</u>	<u>100.75</u>	<u>100.33</u>	<u>100.21</u>	<u>98.98</u>	<u>99.24</u>	

11.

1. Samarskite from Mitchell County, North Carolina¹.
2. Samarskite from Mitchell County, North Carolina.²¹
3. Samarskite from Mitchell County, North Carolina.²²
4. Samarskite from Miask, Ural.²³
5. Samarskite from Berthier County, Quebec Canada.²⁴
6. Samarskite from Jones Fall, Baltimore, Md.
7. Samarskite from Devil's Head Mt., Colorado.²⁵
8. Samarskite from Miask, Ural.²⁶
9. Samarskite from Ödegardsletten, Norway.²⁷
10. Samarskite from Aslaktaket, Norway.²⁸
11. Samarskit aus dem Flussbett des Tschoroch, Kaukasus.²⁹

The analyses of samarskite indicate the complex nature of the mineral. It is regarded as a complex niobate and tantalate and contains a considerable amount of uranium with a small percentage of thorium. Most of the older analyses, it will be noted, failed to show either thorium or lead, but both of these elements are probably found in all samples of samarskite. In the earlier analyses of the mineral these constituents were doubtless separated with the rare earth elements.

Being a primary mineral deposited by magmatic intrusions, the lead present may be considered as the end-products of the uranium and thorium series rather than a secondary deposit.

SEPARATION OF RADIO-LEAD.

Samaraskite was readily decomposed and the lead separated by the following procedure:-

One and one-half times the amount of hydrofluoric acid necessary to combine with the bases present was added and the mixture evaporated nearly to dryness.* Dilute hydrofluoric acid was then added to the residue to take up soluble fluorides, the presence of the acid being necessary to prevent the hydrolysis of these fluorides. The mixture was filtered thru a rubber funnel and thoroly washed till free from acid, the filtrate and washings being collected in a platinum dish. The residue was transferred to a platinum dish with the smallest quantity of water possible, evaporated to dryness and then heated to fuming with concentrated sulphuric acid which has been found to be entirely free from lead. This mixture was cooled and the soluble sulphates extracted with hot water; the insoluble sulphates boiled with sodium hydroxide, the mixture treated with nitric acid, diluted, filtered and evaporated to dryness. This residue was taken up with water, acidified with hydrochloric acid and the lead precipitated by saturation with well scrubbed hydrogen sulphide. The freshly precipitated lead sulphide was boiled with strong

* In platinum dishes.

hydrochloric acid and the solution evaporated to dryness. From 1100 grams of samarskite, fourteen grams of the crude lead chloride was obtained.

TABLE OF SEPARATION.

Treat 80 mesh samarskite with 1 1/2 times H_2F_2 necessary to combine with bases present. Evap. nearly to dryness, add a little H_2F_2 , filter thru a rubber funnel. Wash thoroly.

F. Soluble fluorides of Nb, Ta, W and Sn.	R. Insoluble fluorides, Pb, Ba, U, Th, Ce and Y earths. Wash from filter into platinum dish. Evap. to dryness. Heat to fuming with H_2SO_4 . Cool. Boil with water. Filter.
---	---

F. Sulphates U, Th, Ce and Y earths. Set aside.	R. Sulphates Ba, Ra, Pb. Boil with NaOH. Treat with HNO_3 . Dil., filter, evap. to dryness. Dis. in water, add HCl, saturate with scrubbed H_2S . Ppt. PbS . While freshly precipitated, boil with HCl, evap. sol. to dryness. Product crude $PbCl_2$.
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PURIFICATION OF LEAD SALTS.

The impure lead chloride of radioactive origin was dissolved in water, saturated with hydrogen sulphide and the sulphide formed dissolved in nitric acid; the sulphate formed being dissolved, the lead precipitated as the sulphide and treated with nitric acid as before.

The lead was recrystallized as the nitrate four times, converted to the chloride and as such submitted to three more crystallizations. Baxter and Grover² have shown this procedure to be efficient and very satisfactory in their preparation of samples for the determination of the atomic weight of ordinary lead. All filtrations were made thru a platinum sponge and the latter recrystallizations were carried on entirely in platinum.

In order to obtain a sample of ordinary lead, galena, from Commerce Oklahoma, was treated with nitric acid. The nitrate obtained was recrystallized six times as such, converted to the chloride and recrystallized twice. In all cases the final products of crystallization were kept in a vacuum desiccator over fused potassium hydroxide.

Apparatus for Fusion of Lead Chloride.

Arthur L. Davis.

ANALYSIS OF LEAD CHLORIDE.

The method and procedure is almost in detail the same as used by Baxter and Grover.²

After the preliminary drying in a vacuum desiccator the final drying is accomplished by fusion in an atmosphere of thoroly dried hydrogen chloride and slowly replacing the hydrogen chloride by mean of a stream of carefully scrubbed and dried air.

The apparatus consisted of five drying tubes containing concentrated sulphuric acid for the final drying of the hydrogen chloride generated by dropping concentrated sulphuric acid into the purest concentrated hydrochloric acid; two drying tubes containing silver nitrate solution for the removal of hydrogen sulphide from the air, one containing solid potassium hydroxide for the removal of carbon dioxide and two tubes containing concentrated sulphuric acid for the final drying of the air; connected to the so-called bottling apparatus. All connections were fused or ground glass joints.

A platinum boat, previously heated to constant weight and weighed in a weighing bottle, was filled with the dry lead chloride and placed in the combustion tube with the weighing bottle

in place. The apparatus was completely filled with hydrogen chloride by the passage of a gentle stream of the dried gas and then the chloride was gradually heated till a perfect state of fusion was reached, under which condition it was allowed to remain for ten minutes. The apparatus was allowed to cool very slowly, still with the passage of hydrogen chloride, When perfectly cool the hydrogen chloride was replaced by a gentle stream of scrubbed dried air. Without disconnecting the apparatus, the boat was placed in the weighing bottle and then the bottle removed to a desiccator. The weighing of the bottle, boat and fused chloride was then made.

Solution of the fused chloride was effected by attaching a platinum wire to the boat and suspending in 500 c.c. of double distilled water, slightly acidified with nitric acid, contained in a liter Erlenmeyer flask, heated by means of a hot plate. When the solution was complete, the boat was thoroly washed with hot water and removed. The precipitation of the chloride was made by the addition of a very slight excess of fiftieth normal silver nitrate solution. After standing several days in a dark place, with occasional agitation, the silver chloride was collected on a platinum sponge crucible. The silver chloride was dried

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at 235 for sixteen hours, cooled and weighed.

The calculations are based on the atomic weights of silver and chlorine as 107.88 and 35.457 respectively.

2) were corrections made for the solubility of AgCl?

ANALYSES OF LEAD CHLORIDE.

Number of analysis.	Weight of PbCl ₂ .	Weight of AgCl.	Factor. PbCl ₂ : 2AgCl.	Atomic weight.
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Ordinary lead.

1.	0.94411*	0.97264	0.97066	207.354
2.	1.24607	1.28454	0.97010	207.186

207.27

Radioactive lead.

3.	1.06795	1.10199	0.96683	206.246
4.	0.73514	0.76011	0.96722	206.363

206.30

* Weights reduced to vacuo.

DETERMINATION OF THE URANIUM AND THORIUM CONTENT OF SAMARSKITE.

A very good method of separation ¹⁰ of the elements present in a complex niobate and tantalate was found but since a fusion with acid potassium sulphate is made, it was hardly deemed applicable under the circumstances with the possible convenient use of hydrofluoric acid.

By the use of the following procedure very satisfactory results were obtained:

Treat 1 g. 200 mesh samarskite with considerable excess H_2F_4 . Evaporate nearly to dryness. Add a little dilute H_2F_4 . Filter thru rubber funnel into platinum dish.

F. R. Insoluble fluorides. Wash into platinum Dish; evaporate to dryness; heat to fuming card. with H_2SO_4 . Cool, boil with water, filter.

F. Sulphates of U, Th, Ce and Y earths. Add NH_4Cl , NH_4OH , boil. Let stand, filter.	R. Dish- card.
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F. P. Dissolve in HCl ; dilute; add slight excess ammonium oxalate to boiling solution. Allow to stand twelve hours. Filter.

F. Evaporate to dryness with excess of HNO_3 . Take up with water; add NH_4Cl and NH_4OH .

P. Wash from filter and evap. to dryness with HNO_3 . Dissolve in water; boil with excess $Na_2S_2O_8$. Let stand, filter.

F. P. Dry and Dish- ignite. Weigh card. as U_2O_5 .

F. P. Dry and Dish- ignite. Weigh card. as ThO_2 .

Following out the analysis in the above manner
 duplicates gave very closely agreeing results.

U_2O_5 12.14 12.29 Ave. 12.21 per cent.

ThO_2 1.12 .94 1.03 per cent.

Atomic
 Percentages $U = 91.7\% ; Th = 8.3\%$

Radioactive
 Percentages $\overline{U = 97\%} \quad ; Th = 3\%$

CONCLUSION.

It has been conclusively shown by Boltwood that lead is the end-product of the disintegration of uranium. The lead-uranium ratio in primary uranium minerals is remarkably constant. Then lead from this source should have an atomic weight of 206, considering the loss of five helium atoms from radium of an atomic weight of 226.

Soddy and Hyman found that the atomic weight of lead from a primary thorium mineral, containing a trace of uranium, to be 208.4 compared with the theoretical value of 208.2, considering that the radio-lead from this source to be the result of the loss of six atoms of helium from thorium of an atomic weight of 232.4. Richards and Lambert found the atomic weight of lead from thorianite, a thorium mineral from Ceylon, to be 206.82, whereas if the lead was a result of the decay of thorium the weight would at least be greater than 207.2. M. Curie found that lead from monazite sand, remarkably free from uranium, gave a result of 207.08.

Holmes and Lawson¹² have made a number of analyses of uranium-thorium minerals for uranium, thorium and lead content. Previous analyses which

contained the sought for data were also collected. They found in all uranium-thorium minerals of the same age, that the lead-uranium ratio was remarkably constant and varied sympathetically with the geologic age of different antiquities. The lead-thorium ratio in uranium-thorium and thorium minerals was of the greatest degree of variability. The evidence of this and atomic weight determinations points to the stability of radium C and the instability of thorium E.

Should actinium lead be a stable product with an atomic weight of 207, the lead extracted from a thorium-free uranium mineral should have an atomic weight of 206.08 and 206.36 if the atomic weight of actinium lead is 210. But if actinium-lead is relatively short lived, considering about eight per cent of uranium being transformed to the actinium series, the high degree of stability of the uranium lead would make it certain that its atomic weight would be unappreciably affected by the presence of actinium lead, even if it has an atomic weight of 210. Honigschmid found that the lead coming from crystallized uraninite from Morogoro, C. E. Africa, gave a value of 206.04 compared with 206.36 if stable actinium lead of an atomic weight of 210

was present.

Lead from samarskite gave an atomic weight of 206.30. Considering the lead as the end-product of the uranium series only, with an atomic weight 206, perhaps the lead consisted of a mixture of three parts of radio-lead to one of ordinary lead.

As an end-product of the uranium-thorium series, calculating from the per cent of uranium and thorium present in the original mineral, a weight of 206.06 would be obtained, thus showing the possibility of an admixture of eleven parts of radio-lead to three of ordinary lead being present in this case.

If eight per cent of uranium is transformed along the actinium series and stable actinium lead of atomic weight of 210 was the result, a value of 206.36 would be obtained, which agrees rather closely with the results obtained.

If radio-lead from a uranium-thorium mineral was the end-product of the uranium-actinium-thorium series, a value of 206.364 should have been obtained.

The atomic weight of the lead is proportional to the percent of the parent element and inversely to its rate of decay. The theoretical value for the atomic weight is calculated by multiplying the percent of U by 206 and the percent of Th by 208.4 and .36 since Th decays .36 as rapidly as U, and taking the average.

The results of this investigation, although relative, are fairly absolute, inasmuch as the value for the atomic weight of ordinary lead agrees very closely with that of Baxter and his colleague.

Finally, I wish to express my thanks to Dr. Herman Schlundt, Professor of Physical Chemistry, University of Missouri, for the suggestion of the problem and his valued supervision of the work.

Chemical Laboratory,
University of Missouri,
Columbia.
May 1918.

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