[CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF HARVARD COLLEGE.]

A REVISION OF THE ATOMIC WEIGHT OF IRON.

[FOURTH PAPER.]

THE ATOMIC WEIGHT OF METEORIC IRON.

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Received January 18, 1911.

So far as we know atomic weight investigations have always in the past been carried out with material of terrestrial origin. While there is no reason to believe that an extra-terrestrial element differs from the corresponding terrestrial element in any way, a comparison is at any rate worth while.[1] As sensitive a means as any other for detecting a difference between the two sorts of material lies in the determination of the atomic weight, and since iron is one of the few elements available in sufficient amount for such a comparison, at the close of the research upon the atomic weight of iron described in the preceding paper the experiments were continued with meteoric material.

Through the kindness of Professor John Eliot Wolff, Curator of the Mineralogical Museum of Harvard University, we were fortunate enough to secure a considerable quantity of the "Cumpas" meteorite weighing 63 pounds, found in 1903 near Cumpas, Sonora, Mexico. The meteorite shows well-marked Widmanstättian figures on etching with nitric acid. The sample given us contained a little over 88 per cent. of iron, the chief impurity being nickel.

In general the separation of the iron from the accompanying impurities and the determination of the atomic weight by analysis of ferrous bromide followed the course outlined in the preceding paper. The original turnings of meteorite were dissolved in hydrochloric acid, and an insoluble residue which remained was removed by filtration. Upon saturating the solution with hydrogen sulfide, apparently no precipitate except sulfur was formed. The solution was then made nearly neutral by the addition of ammonia, and was again saturated with hydrogen sulfide. The resulting small amount of black precipitate, chiefly ferrous sulfide, was collected upon a filter and rejected. Next an excess of ammonia was added, hydrogen sulfide was passed in until precipitation was complete, and the precipitated sulfides were thoroughly washed by decantation. Upon treating the sulfides with 4 per cent. hydrochloric acid solution the greater part of the nickel and cobalt sulfides remained insoluble. After filtration the process of precipitating the ferrous sulfide and redissolving in dilute hydrochloric acid was repeated. The second residue of nickel and cobalt sulfides was so small that it seemed advisable to re-

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[1] Such a comparison was suggested to one of us nearly ten years ago by Prof. T. W. Richards, and independently through him more recently by Sir Henry Roscoe.

move the remainder of the nickel and cobalt by precipitating the iron as ferric hydroxide in the presence of large amounts of ammonium salts and ammonia. First the ferrous chloride solution was oxidized by boiling with nitric acid, and then, after dilution, it was poured into a large volume of redistilled ammonia. The mother liquor of this precipitation, when evaporated to small bulk and treated with ammonium sulfide, was found to contain nickel, hence the precipitate of ferric hydroxide, after being washed by decantation, was dissolved in nitric acid and reprecipitated with ammonia as before. In all, nine such precipitations were necessary to eliminate the nickel and cobalt completely. The ninth precipitate of ferric hydroxide was dissolved in redistilled sulfuric acid and was converted into ferrous bromide exactly as described in the preceding paper with Sample C, by electrolytic reduction and crystallization as ferrous sulfate, electrolytic deposition as metal from oxalate solution, solution in nitric acid and crystallization as ferric nitrate, ignition to oxide, reduction in hydrogen, and solution in concentrated hydrobromic acid solution and crystallization as ferrous bromide. On account of the limited quantity of material, in the crystallizations of the ferric nitrate and the ferrous bromide the mother liquors were worked up by the usual process of fractional crystallization. During the purification of this material no difference in behavior could be discerned from that of terrestrial iron.

The ferrous bromide was prepared for analysis by dehydration and fusion in a current of dry nitrogen and hydrobromic acid gases, in a quartz boat contained in a quartz tube. After being weighed it was dissolved in acidulated water and oxidized to the ferric state by dichromate. Finally the bromine was determined by titration against silver and the silver bromide was estimated gravimetrically. The details of the procedure were exactly as described in the preceding paper. Sufficient material for only five analyses was finally obtained.

ATOMIC WEIGHT OF IRON.

Ag = 107.880. Br = 79.916.

Series I. $FeBr_2 : 2Ag$

Number of analysis	Weight of $FeBr_2$ in vacuum. Grams.	Weight of Ag. in vacuum. Grams.	Weight of Ag added or subtracted. Gram.	Corrected weight of Ag in vacuum. Grams.	Ratio $FeBr_2 : 2Ag$.	Atomic weight of iron.
1	3.95460	3.95621	0.00010	3.95631	0.999568	55.835
2	4.66954	4.67147	0.00030	4.67177	0.999523	55.825
3	4.75335	4.75530	0.00020	4.75550	0.999548	55.831
4	6.95582	6.95894	—0.00040	6.95854	0.999609	55.844
5	3.20762	3.20864	0.00040	3.20904	0.999557	55.833
				Average,	0.999561	55.834
				Average, rejecting Nos. 2 and 3,	0.999578	55.837

Series II. $FeBr_2 : 2AgBr$.

Number of analysis.	Weight of $FeBr_2$ in vacuum. Grams.	Weight of Ag in vacuum. Grams.	Loss on fusion. Gram.	Corrected weight of AgBr in vacuum. Grams.	Ratio $FeBr_2 : 2\,Ag$.	Atomic weight of iron.
6	3.95460	6.88729	0.00009	6.88720	0.574196	55.831
7	4.66954	8.13307	0.00025	8.13282	0.574160	55.818
8	4.75335	8.27870	0.00015	8.27855	0.574177	55.824
9	6.95582	12.11361	0.00032	12.11329	0.574230	55.844
10	3.20762	5.58655	0.00023	5.58632	0.574192	55.830
				Average,	0.574191	55.829
				Average, rejecting Nos. 7 and 8,	0.574206	55.835

While the average of the two series, 55.832, is slightly lower than the outcome of the work described in the preceding paper, 55.838, the difference corresponds to only one part in ten thousand in the atomic weight of iron and to less than one part in thirty thousand in the molecular weight of ferrous bromide, the quantity actually determined. It is to be noted that analyses 4 and 9, in which the largest amount of material was used, gave a result even higher than the average of the preceding series. In the four lowest analyses 2 and 7, and 3 and 8, a modification was introduced in the method of analysis, which consisted in adding 98 instead of 99 per cent. of the dichromate necessary to oxidize the ferrous salt. In one of these analyses the fused silver bromide, instead of being yellow, was perceptibly darker than the pure substance, possibly owing to slight reduction of the silver salts by the unoxidized ferrous salt. The ratio of silver to silver bromide in these four experiments is distinctly lower than the value to be expected, 0.574453.[1]

		Ag : AgBr.
Analyses	1 and 6	0.574444
	2 and 7	0.574434
	3 and 8	0.574436
	4 and 9	0.574455
	5 and 10	0.574446

In the preceding paper the conclusion was reached that 99 per cent. of the dichromate theoretically necessary is sufficient to avoid error from reduction. If the four experiments giving the lowest ratio of silver to silver bromide, 2 and 7 and 3 and 8, are omitted, the averages of the two series become 55.837 and 55.835 respectively, which agree as closely with the final result of the preceding investigation as can be expected. Even the average of the four experiments in question differs from that of the others by only one part in twenty thousand in the molecular weight of ferrous bromide. At any rate there seems to be no evidence of dissimilarity between this specimen of meteoric iron and the ordinary metal. We intend to investigate this problem farther.

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[1] Baxter, *Proc. Am. Acad.*, **42**, 210 (1906); THIS JOURNAL, **28**, 1332.

We are particularly indebted to the Carnegie Institution of Washington for pecuniary assistance in carrying out this investigation and also to the Cyrus M. Warren Fund for Research in Harvard University for indispensable platinum vessels.

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THE EFFICIENCY OF CALCIUM BROMIDE, ZINC BROMIDE AND ZINC CHLORIDE AS DRYING AGENTS.

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Received January 18, 1911.

The problem of the drying of hydrobromic acid gas is one which has frequently been met in this laboratory in the preparation of anhydrous metallic bromides for analysis. Until recently phosphorus pentoxide was used as the final drying agent, but experiments by Baxter and Hines[1] in the drying of hydrochloric acid gas with phosphorus pentoxide have shown that the pentoxide is attacked by the acid gas with the formation of volatil phosphorus compounds, and that therefore there is danger in using this drying agent for hydrobromic acid also. Since hydrobromic acid gas is decomposed by concentrated sulfuric acid, fused metallic bromides seemed to be the most suitable substances for the desiccation of this gas. Hence experiments were carried out with two anhydrous bromides which can easily be prepared in a state of sufficient purity, with the purpose of determining their efficiency as drying agents.

Since phosphorus pentoxide was used to absorb the residual moisture in the gas which had been exposed to the anhydrous bromides, it was not possible to employ hydrobromic acid as the gas to be dried. Instead air was used for this purpose, since the nature of the gas is of no consequence, provided the gas does not combine with the drying agent.

The method was as follows: A measured amount of moist air was passed through a tube containing a layer of crushed fused bromide, and then through a weighed phosphorus pentoxide tube. From the gain in weight of the pentoxide tube and the volume of air passed through the system, the pressure of the aqueous vapor in the air which was in equilibrium with the fused bromide could be determined.

The calcium bromide was made from very pure calcium carbonate which had been precipitated from a solution of the nitrate, and a solution of hydrobromic acid which had been synthesized from pure bromine and hydrogen. The bromine had been twice distilled from a bromide so that it must have been free from chlorine. The slightly acid solution of the bromide was evaporated in a platinum dish, and the residue was

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[1] THIS JOURNAL, **28**, 779 (1906).