Ag = 107.880	Ag = 107.870	Ag = 107.860
P = 31.023	P = 31.020	P = 31.017

On any basis the rounded off value 31.02 is as exact as the experimental work warrants.

Summary of Results.

1. Methods are described for the preparation of pure phosphorus trichloride and its analysis.
2. The molecular weight of phosphorus trichloride referred to silver 107.880 is found to be 137.389 whence phosphorus has the atomic weight 31.018.
3. Using the same value for silver, the average result of the analyses of silver phosphate, phosphorus tribromide and phosphorus trichloride is 31.028, while the average of the more reliable tribromide and trichloride analyses is 31.023.

We are particularly indebted to the Carnegie Institution of Washington for generous pecuniary assistance in carrying out this investigation.

CAMBRIDGE, MASS., July 1, 1912.

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF HARVARD COLLEGE.]

A REVISION OF THE ATOMIC WEIGHT OF IRON.[1]

[FIFTH PAPER.]

THE ANALYSIS OF FERRIC OXIDE.

BY GREGORY PAUL BAXTER AND CHARLES RUGLAS HOOVER.

Received October 17, 1912.

The atomic weight of iron has already been subjected to three separate investigations in this laboratory. Richards and Baxter[2] first analyzed ferric oxide by reduction in hydrogen, and obtained the result 55.88, a value much lower than the one in general use at that time, 56.02. The problem was further investigated by Baxter[3] by the analysis of ferrous bromide, and the result yielded by material known to be slightly impure, 55.845 (Ag = 107.880), agrees very closely with that obtained by Baxter, Thorvaldson, and Cobb[4] in a very recent investigation with much purer material, 55.838. Furthermore, Baxter and Thorvaldson[5] found that meteoric material is identical, so far as could be told by the analysis of ferrous bromide, with material of terrestrial origin.

Thus, although the analysis of ferrous bromide supports the verdict of the oxide analysis in yielding a low value for the atomic weight of iron,

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[1] Reprinted from Original Communications, Eighth International Congress of Applied Chemistry, Vol. II, p. 37 (1912).

[2] *Proc. Am. Acad.*, **35**, 253 (1900); *Z. anorg. Chem.*, **23**, 245.

[3] *Ibid.*, **39**, 245 (1903); **38**, 232.

[4] THIS JOURNAL, **33**, 319 (1911); *Z. anorg. Chem.*, **70**, 325.

[5] *Ibid.*, **33**, 337 (1911); **70**, 348.

yet the difference between the results of the two methods, 0.04, is much too large to be allowed to rest without further investigation. The recent availability of fused quartz apparatus seemed to offer distinct possibilities of improvement in the original oxide analysis, especially in the reduction of the oxide, so that the decision was made to repeat the analysis of ferric oxide with all possible precautions in the light of experience gained since the earlier work.

In outline the operations consisted in preparing ferric oxide by igniting carefully purified ferric oxide in a current of pure air. The oxide was next weighed, and then, after reduction in a current of very pure hydrogen, the weight of the residual metal was found. The problems involved were, therefore, first, the purification of pure ferric salt; second, the preparation of ferric oxide free from higher or lower oxides and occluded gases; third, the complete reduction of the oxide.

Purification of Reagents.

Water.—In the earlier stages of the purification of the iron salts, the ordinary distilled water was employed. In the later steps this water was twice redistilled, first from an alkalin permanganate solution, then, after the addition of a trace of sulfuric acid, through a block tin condenser, in an apparatus free from rubber or cork. Usually the distillate was collected in platinum vessels.

Ammonia.—The best commercial ammonia was distilled into the purest water in an apparatus constructed entirely of glass.

Nitric Acid.—C. P. concentrated acid was distilled from a glass still through a platinum condenser, with rejection of the first two-thirds of the distillate. A platinum receiver also was employed.

Hydrochloric Acid.—C. P. concentrated acid was diluted with an equal volume of water and distilled through a glass condenser with rejection of the first half of the distillate.

Sulfuric Acid.—C. P. concentrated sulfuric acid was distilled from a small non-tubulated glass retort into a Jena glass flask. Here also the first half of the distillate was rejected.

Oxalic Acid and Ammonium Oxalate.—The best commercial substances were three times crystallized in Jena vessels with centrifugal drainage and washing.

Air.—Air was freed from organic matter by being passed over red hot copper oxide in a hard glass tube heated electrically with "Nichrome" ribbon. Then it was passed through a train of Emmerling towers, containing in turn beads drenched with silver nitrate solution, solid potassium hydroxide which had been fused with a small proportion of permanganate, and beads drenched with concentrated sulfuric acid, to which a trace of potassium dichromate had been added. Finally the air was dried in a long tube containing phosphorus pentoxide which had been resublimed in air. The apparatus was constructed wholly of glass with fused or ground joints throughout. The joints were either dry or lubricated with sulfuric acid, except in the case of a two-way stopcock beyond the phosphorus pentoxide tube, which was lubricated with Ramsay grease. Since ferric oxide quickly came to constant weight when ignited in this air, it was obviously sufficiently pure for the purpose.

Oxygen.—Oxygen prepared by the Linde Air Products Company was purified in the same apparatus as that used for purifying air. This gas contained about 97% of oxygen. Apparently a large percentage of the impurity was argon.[1]

Hydrogen.—At first, because of convenience, hydrogen was generated by the action of water on "hydrone." It was scrubbed with water in a tower filled with moist glass wool and was then dried with fused potassium hydroxide and finally phosphorus pentoxide. This gas proved to be unsatisfactory, for when it was passed through a hot quartz tube for some time, a black deposit which appeared to be carbon was formed and iron reduced from the oxide in this gas showed a continuous gain in weight, owing apparently to absorption of carbon. Since it seemed probable that this hydrogen contained hydrocarbons, an electrolytic method of generating the gas was substituted. The generator contained the system, zinc amalgam—dilute hydrochloric acid—platinized platinum, and has already been shown to give very pure hydrogen.[2] Two such generators give a current of hydrogen sufficiently rapid for most purposes and by applying an external electromotive force, the evolution of gas can be made much more rapid. After purification by scrubbing with dilute potassium hydroxide solution and drying by freshly fused potassium hydroxide and resublimed phosphorus pentoxide, this hydrogen gave no evidence of impurity of any sort. For some time after the charging of the generators with fresh acid, the gas undoubtedly contains air originally dissolved in the acid, but in the course of time this must be gradually swept out by the continuous flow of minute bubbles of hydrogen. The apparatus was constructed entirely of glass, with ground stoppers, except for two very short rubber connections which were used to join the generators to the purifying train. The ground joints were lubricated with Ramsay grease.

Purification of Ferric Oxide.

Two specimens of ferric nitrate were purified for conversion into ferric oxide. Sample A was of terrestrial, Sample B of meteoric origin.

Sample A was prepared from the same specimen of pure iron, manufactured by the American Rolling Mills Co., that was used in the previous investigation upon ferrous bromide. This material contains sulfur

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[1] Morey, This Journal, **34**, 491 (1912).

[2] Cooke and Richards, *Proc. Am. Acad.*, **23**, 149 (1887).

0.019%, phosphorus 0.003%, carbon 0.018%, copper 0.05%, and traces of silicon and manganese, less than one-tenth of 1% in all. A block of this iron was washed with ether, alcohol and water, and was etched with pure nitric acid to remove surface contamination. It was then placed in a large platinum dish and treated with several successive portions of 30% nitric acid, until about 100 grams had dissolved. In order to remove a small amount of insoluble material, chiefly carbon and basic nitrate, the solution, which still contained a large excess of nitric acid, was filtered by suction through a carefully prepared asbestos mat in a platinum funnel into a quartz flask. Next, the solution was transferred to a large platinum dish and evaporated to crystallization on an electric stove in order to avoid introduction of sulfur compounds from a gas flame. The crystals were freed as far as possible from mother liquor by centrifugal drainage in an apparatus in which the material could come in contact only with platinum,[1] and were rinsed in the centrifuge with concentrated nitric acid. The crystals were then recrystallized four times from concentrated nitric acid, with all the foregoing precautions. The mother liquor of the first crystallization was evaporated until a second crop of crystals could be obtained and this second crop was recrystallized three times from the successive mother liquors of the first set of crystals. The two final crops of crystals were converted into a mixture of oxide and basic nitrate by heating them in small portions in a platinum dish on an electric stove. The best material is designated Sample A_1, that recovered from the mother liquors Sample A_2.

In order to show that mere crystallization of the nitrate is sufficient to eliminate the predominating metallic impurities, about 100 grams of ferric nitrate were rendered impure with 1 gram each of copper, nickel, aluminum, and manganese nitrates, and the mixture was recrystallized. After two crystallizations the crystals were found free from copper, nickel and manganese by tests with ammonia, dimethylglyoxime, and alkalin fusion, respectively, and, while the second crop of crystals contained a trace of aluminum, the third crop gave no indication of the latter element when treated with an excess of sodium hydroxide and the filtrate tested with ammonium chloride.

The purification of the meteoric material, owing to the larger proportion of impurity, was necessarily more difficult and prolonged. A new portion (80 grams) of the "Cumpas" meteorite, found near Cumpas, Senora, Mexico, in 1903, was very kindly given to us by Professor John Eliot Wolff, Curator of the Harvard Mineralogical Museum. This meteorite contains about 88% of iron, the remainder being chiefly nickel. Although the method used by Baxter and Thorvaldson[2] for the elimination

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[1] Baxter, THIS JOURNAL, 30, 286 (1908).

[2] *Loc. cit.*

of the nickel was a tedious one, yet comparison on a small scale with other methods failed to show any marked advantage of any one of the latter, so that the process used in the earlier work was adhered to.

After the metal had been cleansed with ether and alcohol and etched with nitric acid, it was dissolved in redistilled hydrochloric acid, and an insoluble residue, consisting largely of graphite, was removed by filtration. The solution was next diluted considerably and saturated with hydrogen sulfide, which had been thoroughly scrubbed with water. The precipitate produced in this way, chiefly sulfur, was removed, and after nearly neutralizing the free acid with ammonia, the solution was again saturated with hydrogen sulfide. The black precipitate, which contained little but ferrous sulfide, was discarded. A large excess of ammonia was then added and hydrogen sulfide in excess passed in. The precipitated sulfides were washed several times with water containing ammonium chloride made from distilled reagents, and were transferred to a large washed filter where they were extracted with 2% hydrochloric acid. Since the solution still contained considerable nickel after two repetitions of this process, the remainder of the nickel was eliminated by precipitating the iron as ferric hydroxide. The solution of ferrous chloride was oxidized with nitric acid and after dilution to nearly ten liters was poured into a large excess of redistilled ammonia in equal volume. The precipitate was washed by decantation three times and was dissolved in redistilled nitric acid. This process was repeated six times before nickel could not be detected by means of ammonium sulfide in the mother liquor after concentration to small volume. Even in the seventh filtrate dimethylglyoxime showed traces of nickel to be present, but these traces were undoubtedly eliminated in the subsequent processes.

The seventh precipitate of ferric hydroxide was dissolved in an excess of redistilled sulfuric acid with the assistance of a small amount of nitric acid, and the sulfate was freed from nitric acid by evaporation and heating. The ferric sulfate was next electrolytically reduced to ferrous sulfate in a large platinum dish, which served as cathode, while a spiral of heavy platinum wire formed the anode. The solution was originally so concentrated and the solubility of the ferrous sulfate was so much reduced by the sulfuric acid originally present together with that formed in the reduction, that, on cooling the solution after the passage of a heavy current for some time, ferrous sulfate crystallized out. Electrolysis with intermittent cooling and evaporation was continued until the greater portion of the iron was obtained in the form of ferrous sulfate. This ferrous sulfate was once recrystallized from aqueous solution, with centrifugal drainage. Iron was next deposited in the metallic state by electrolysis in ammonium oxalate solution. A warm concentrated ammonium oxalate solution was nearly saturated with ferrous oxalate by adding ferrous sulfate solution and the solution was electrolyzed hot with a high current density in a large platinum dish which served as cathode. From time to time recrystallized oxalic acid was added in order partially to provide for loss at the anode. Occasionally the metallic deposit was thoroughly washed with water and dissolved in redistilled nitric acid. During the electrolysis there was absolutely no evidence of the deposition of manganese dioxide on the anode. When all the material had been thus electrolyzed, the accumulated solution of ferric nitrate was filtered, evaporated and crystallized six times exactly in the manner previously described. The nitrate in turn was ignited to oxide and basic nitrate. Since crystallization of the nitrate alone would probably have sufficed to eliminate the impurities, there can be little doubt, in light of the protracted series of operations to which the meteoric material was subjected, that the final product was sufficiently freed from known impurities.

Preparation of the Ferric Oxide for Weighing.—The ignition of the ferric oxide in air and its subsequent reduction in hydrogen were carried on in a quartz tube which formed part of a bottling apparatus[1] by means of which the platinum boat containing the material could be transferred to a weighing bottle in a current of dry gas without the slightest exposure to moisture. The tube was electrically heated by a removable mica sleeve wound with "Nichrome" resistance ribbon, while the control of temperature was secured through external resistance. By this method a temperature of 1200° can readily be secured. But at that temperature the life of the coil is precarious, and, furthermore, not only was the outside of the quartz tube attacked where it came in contact with the mica, but the platinum boat showed signs of adhering to the quartz. Hence in actual practice the temperature was not allowed to exceed about 1100°. That the temperature was actually as high as this was shown by the fact that silver melted readily at temperatures below the maximum employed. Even under these conditions the outside of the quartz tube suffered where it came in contact with the mica so that occasionally its surface was renewed by fire polishing in an oxyhydrogen flame.

Since, if ferric oxide is reduced in contact with platinum, the metals alloy to so considerable an extent that it is a very difficult matter subsequently to remove the iron, the boat was entirely lined with thin platinum foil. Although, of course, the iron alloyed with the lining of the boat, this lining was replaced with one of new platinum foil in each determination. In this way the boat was entirely prevented from attack in all but a few cases where a small amount of oxide accidentally found its way between the boat and lining and occasioned some difficulty.

The boat was prepared for an experiment by scouring with sea sand, igniting in a blast flame, and boiling with hydrochloric acid, the opera-

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[1] Richards and Parker, *Proc. Am. Acad.*, **32**, 59 (1896); *Z. anorg. Chem.*, **13**, 85.

tions being repeated several times. Next, the lining was constructed of new platinum foil and boiled with hydrochloric acid several times more. Then the boat was ignited in the quartz tube for several hours in a current of pure, dry air and after it had been allowed to cool was transferred by means of the bottling apparatus to the weighing bottle in which it was eventually weighed by comparison with a similar counterpoise.

Owing to accidental contamination of the boat with iron in one of the earlier experiments, in the next analysis, after thorough cleansing, the boat was weighed after ignition both in air and in hydrogen. A loss of 0.2 milligram was found in this way. In subsequent experiments with the same boat, the difference in weight after ignition in air and in hydrogen gradually diminished to about 0.1 milligram but never entirely disappeared. Reversing the order of the ignition failed to change the nature or magnitude of the difference. In one of the later analyses a comparatively new boat, which had never been contaminated with iron, was tested in a similar way with an exactly similar result. While the cause of the difficulty was not further investigated, a probable explanation of the difference in weight is the presence of a trace of iron or a similar metal in the material of the boat or lining. In any case, error could obviously be avoided by determining the difference in the weight of the boat when ignited in air and in hydrogen, and allowing for this difference in computing the weights of the ferric oxide and the metallic iron. This method of correction was actually followed.

After the boat with its lining had been treated as above and weighed, it was filled with the mixture of oxide and basic nitrate which had been powdered in an agate mortar and it was heated in the quartz tube in a current of pure dry air, gently at first until decomposition of the nitrate was essentially complete, finally at about 1000° for several hours. After the boat had cooled it was transferred to the weighing bottle by means of the bottling apparatus and weighed. The heating in air was then continued until the weight of the boat and contents became constant within a few hundredths of a milligram. Usually ten to fifteen hours was a sufficiently long period to produce this result, although for the sake of convenience the heating was frequently prolonged over night.

Although the very fact that the weight of the oxide becomes constant when it is treated in this way is evidence that the ferric oxide does not appreciably dissociate into a lower oxide and oxygen even at the highest temperatures employed, experiments were undertaken to test this point early in the investigation. One specimen of oxide was several times ignited in a current of oxygen, and, after it had cooled, it was bottled in dry air. As soon as the weight was constant, the material was ignited and cooled in air in the usual way. A loss in weight of 0.2 milligram in 5 grams of oxide was found. Re-ignition in oxygen yielded the original weight, and upon a second ignition in air the weight became practically the same as after the first ignition in air.

The higher weight of the oxide after ignition in oxygen was confirmed in a second similar series of experiments. Furthermore, it was found that the boat and lining when treated similarly showed no perceptible difference in weight under the different conditions.

It seemed hardly probable that the difference was due to dissociation of the ferric oxide, however, but rather to occlusion of either oxygen or a higher oxide by the ferric oxide. This view was substantiated by experiments in which ferric oxide was heated in nitrogen and in a vacuum. Nitrogen, free from both oxygen and hydrogen, was prepared in an all glass apparatus kindly loaned by Dr. C. J. Moore.[1] Ferric oxide, which had been ignited to constant weight in air, was heated for four hours in nitrogen and cooled in this gas before being bottled in air. As a result of this treatment, 7.5 grams of oxide lost 0.6 milligram. In order to prove conclusively that this loss was due to dissociation of the ferric oxide into a lower oxide and oxygen owing to low oxygen pressure, another sample of oxide was heated in the bottling apparatus which was kept as completely exhausted as possible by means of an efficient Töpler pump. During this treatment a small amount of gas was evolved continually and a loss in weight of 1 milligram was observed. Both of the latter experiments lead to the conclusion that the dissociation pressure of the oxygen from ferric oxide is at any rate very low at the highest temperatures employed in this work and therefore that no loss of oxygen could have taken place from this cause when the oxide was heated in air.

The results of the investigations of others support this view. Walden[2] found by heating ferric oxide in a vacuum no measurable pressure of oxygen below 1050° and at 1100° the observed pressure was only 5 mm. Hilpert[3] found by chemical tests no lower oxides in ferric oxide which had been heated to 1200° in air, and states that even at higher temperatures the velocity of dissociation is very small.

The safest course to pursue seems to be to assume the weight of the oxide after ignition to constant weight in air to be correct, although it is worth pointing out that if the weight after ignition in oxygen is used, the results of this research will be lowered by about seven one-thousandths of a unit.

Attention has been called by Richards[4] to the fact that most oxides made by the ignition of nitrates retain small amounts of nitrogen and oxygen, but Richards and Baxter,[5] in the earlier research upon ferric

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[1] Baxter, Moore and Boylston, *Proc. Am. Acad.*, **47**, 588 (1912); THIS JOURNAL, **34**, 261; *Z. anorg. Chem.*, **74**, 365.

[2] THIS JOURNAL, **30**, 1350 (1908).

[3] *Ber.*, **42**, 4893 (1909).

[4] *Proc. Am. Acad.*, **26**, 281 (1891); **28**, 200 (1893).

[5] *Ibid.*, **35**, 257 (1900); *Z. anorg. Chem.*, **23**, 250.

oxide, showed that ferric oxide is an exception to this rule, in that the quantity of occluded gases is negligible for the purpose in hand.

The Reduction of Ferric Oxide.

In order to reduce the ferric oxide, the boat and bottle were placed in their proper places in the bottling apparatus and, when the air had been nearly displaced by pure hydrogen, the boat was gradually heated until the reduction progressed rapidly. Owing to the fact that the reaction is an exothermic one, it was usually necessary to retard the reduction by removing the heating coil after the reduction had commenced. As soon as this portion of the process was past, heating was resumed and continued at dull redness as long as perceptible amounts of water were evolved. Since the reduced metal sinters to a considerable extent even at a relatively low temperature, it seemed better to avoid this effect as far as possible until reduction was essentially complete. The temperature was then very gradually increased to a bright red heat, and finally was maintained at 1050–1100° for several hours. The boat was next cooled in hydrogen, the hydrogen was displaced by air and the boat was bottled and weighed as in the case of the oxide. The metal was then heated for several hours at bright redness and again cooled and weighed, and the process was repeated until the weight became constant within a few hundredths of a milligram. In the earlier analyses four or five ignitions of from five to twenty-five hours were found necessary to secure constant weight. Eventually, time and labor were saved by prolonging the first heating in hydrogen to 60 hours or more. When this was done, no change in weight of more than 0.1 milligram was ever observed after the first heating, even when the second heating was as long as 50 hours. This constancy in the weight of the metal indicates not only that reduction had proceeded as far as it would go under the conditions of the experiment, but also that neither the platinum nor the iron volatilized appreciably even at the highest temperature employed.

While it is a well known fact that very finely divided iron is pyrophoric, we were able to secure evidence of only very slow oxidation of the highly sintered and compact metal which resulted from the reduction. The appearance of the metal did not alter on long standing even in an atmosphere of average humidity. Furthermore on, standing eighteen hours in the weighing bottle in a desiccator in five experiments, the observed gain in weight was only 0.12, 0.03, 0.01, 0.04 and 0.05 milligram, respectively. In the light of these facts it seems reasonably certain that in dry air iron oxidizes so slowly that, at any rate, no error was introduced from this source before the metal was weighed.

After five analyses had been completed, it was noticed in the next experiment while the hydrogen was being displaced by air, that a slight mist appeared on the tube beyond the boat and that the portion of the tube containing the boat became perceptibly warmer than the adjacent portions. In a short time the water evaporated into the current of dry air, leaving the iron unchanged in appearance. This phenomenon was not observed in the earlier experiments, because in these the boat was concealed by the heating coil which had been allowed to cool *in situ*. In a later experiment when the empty boat was treated in the same way a similar occurrence took place, so that the first conclusion that the union of the hydrogen and oxygen was catalyzed by the finely divided iron is by no means certain. Although no attempt was made to settle the latter point, experiments were immediately undertaken to determin whether perceptible oxidation of the iron was brought about during the formation of the moisture or by its presence. This was done by first weighing the reduced iron after displacing the hydrogen with air. Then the metal was again ignited and cooled in hydrogen, but before air was admitted to the bottling apparatus, this was exhausted as completely as possible by means of a Töpler pump. The iron did not lose perceptibly in weight during this treatment and a repetition of the experiment yielded the same result. While it is thus evident that the earlier experiments had fortunately not been vitiated by this newly discovered occurrence, in all subsequent analyses the precaution was taken to exhaust the bottling apparatus, after the iron had been allowed to cool in hydrogen, before admitting the air. The result of these experiments is entirely in accord with the experience of Friend, Hull and Brown[1] and of Friend;[2] for the former have shown that steam affects iron only slightly at 500° while the latter proved that steam has no effect at 200–300°.

Although Baxter[3] has already investigated the occlusion of hydrogen by finely divided iron which has been heated and cooled in an atmosphere of the gas, and found no positive evidence of occlusion, the subject was further investigated in several of the analyses of this research. The boatload of iron which had been brought to constant weight when cooled in an atmosphere of hydrogen was again ignited in hydrogen and while it was still red hot, the bottling apparatus was rapidly exhausted so that the metal cooled in a vacuum. Then air was admitted and the boat was bottled and weighed. No change in weight beyond the error of weighing could be detected and a repetition of the experiment yielded the same result. Even though it was thus proved to be safe to allow the metal to cool in hydrogen, in many of the later experiments the final weight of the metal was obtained after cooling in a vacuum.

Sieverts[4] found that iron, at 800°, dissolves per 100 grams only 0.2 mg.

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[1] *J. Chem. Soc.*, 99, 969 (1911).

[2] *J. Iron and Steel Inst.*, 2, 172.

[3] *Am. Chem. J.*, 22, 363 (1899).

[4] *Electrochem. Z.*, 16, 707 (1910).

hydrogen and that the amount dissolved diminishes with decreasing temperature.

From the above experiments it is obvious also that the platinum of the boat and lining could have occluded no appreciable amount of hydrogen. Sieverts and Jurisch found that platinum heated and cooled in hydrogen retained no measurable amount of the gas.[1]

In the earlier paper upon the analysis of ferric oxide, the effect of the earth's magnetism upon the weight of the iron was shown both theoretically and experimentally to be insignificant.

The following tables include the results of all the analyses which were undertaken, with the exception of one preliminary analysis in which impure hydrogen made from hydrone was employed.

The weighings were made entirely upon a No. 10 Troemner balance sensitive to 0.02 milligram, which is used only for the most exact work. The weighing bottle was always compared by substitution with a counterpoise of the same weight and shape containing a mass of platinum equal to that of the boat. The weights were standardized to hundredths of a milligram by the method proposed by Richards.[2] Ample time was always allowed the boat and counterpoise to come to equilibrium with the atmosphere of the balance case.

A vacuum correction of +0.000086 gram was added for every apparent gram of ferric oxide and of +0.000008 for every apparent gram of metal, the densities of these substances and the weights being assumed to be 5.2, 7.9 and 8.3, respectively.

In the first two analyses the weight of the empty boat after ignition in hydrogen was not determined. Since, however, a new boat with its lining was found to lose in weight by 0.1 milligram when ignited in hydrogen after ignition in air, a correction of this amount is applied in Analyses 1 and 2. In the first five analyses after the reduction of the oxide to metal the hydrogen was displaced by a current of air, but in all others the bottling apparatus was freed from hydrogen by exhaustion before air was admitted.

An examination of these results shows very satisfactory agreement, not only between the results in each series, but also between the averages of the two series. The highest value found for the atomic weight of iron in either series is 55.853 and the lowest 55.843. The difference, 0.010, corresponds to a variation in the weight of iron obtained from 5 grams of oxide of only about 0.0002 gram.

It is obvious that terrestrial and meteoric iron are identical so far as this examination is capable of testing the point. No other outcome was

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[1] *Ber.*, **45**, 221 (1912).

[2] THIS JOURNAL, **22**, 144 (1900).

to be expected, however, especially in the light of the previous comparison of the two sorts of material by Baxter and Thorvaldson.[1]

SERIES I.—O = 16.000.

Number of analysis.	Sample of Fe_2O_3.	Weight of Fe_2O_3 in vacuum. Grams.	Weight of Fe in vacuum. Grams.	Ratio $Fe_2 : Fe_2O_3$.	Atomic weight of iron.
1	A_1	4.86111	3.39995	0.699418	55.845
2	A_1	7.59712	5.31364	0.699428	55.847
3	A_1	6.50945	4.55298	0.699442	55.841
4	A_1	8.00040	5.59576	0.699435	55.849
5	A_1	7.27260	5.08661	0.699421	55.846
6	A_2	7.69441	5.38155	0.699410	55.843
7	A_2	7.33754	5.13213	0.699435	55.849
			Average,	0.699427	55.847
SERIES II.—O = 16.000.					
8	M	4.46431	3.12240	0.699414	55.844
9	M	4.85179	3.39351	0.699435	55.849
10	M	5.21397	3.64674	0.699417	55.845
11	M	5.99087	4.19030	0.699448	55.853
12	M	6.70197	4.68752	0.699425	55.847
			Average,	0.699428	55.848
		Average of all 12 analyses,		0.699427	55.847

The average of all twelve analyses, 55.847, is lower than that previously obtained by the reduction of ferric oxide, 55.883, by 0.036 unit. This difference is undoubtedly due to more complete reduction, owing to the higher temperature secured, although the longer periods of continuous ignition in hydrogen must have produced beneficial effects in the same direction.

On the other hand, the average result is almost exactly 0.01 unit higher than that found by Baxter, Thorvaldson and Cobb[1] and by Baxter and Thorvaldson[1] through the analysis of ferrous bromide, 55.838 (Ag = 107.880). It is hardly probable that the difference between the results of the analyses of ferrous bromide and ferric oxide is due to an incorrect assumption concerning the atomic weight of silver in the calculation of the bromide analyses, as is shown by the following table containing the results of the latter analyses calculated on the basis of various values for silver.

If Ag =	107.910	Fe =	55.854
	107.900		55.848
	107.890		55.843
	107.880		55.838
	107.870		55.833

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[1] *Loc. cit.*

In order to produce agreement between the bromide and oxide series of analyses, it would be necessary to assume the unreasonably high value for the atomic weight of silver of 107.90.

If the bromide analysis is assumed to be correct and to yield the value 55.838 (Ag = 107.880), this would indicate incomplete reduction of the ferric oxide, to the extent of 0.0002 gram residual oxygen in the metal resulting from 5 grams of oxide. Such a result would not be at all surprising, however, when the great difficulty in completely converting one solid into another without intermediate fusion or solution is considered.

Whether the atomic weight of silver is taken as 107.880 or 107.870, the averages of the bromide and oxide results, 55.843 and 55.840, when expressed to two decimal places are identical.

The outcome of this research may be briefly expressed as follows:

1. Pure ferric oxide on reduction in hydrogen is found to contain 69.9427% of iron, whence the atomic weight of iron is 55.847.
2. Terrestrial and meteoric material are found to give identical results.
3. When the result of this investigation is combined with that of the analysis of ferrous bromide by Baxter, Thorvaldson and Cobb, the atomic weight of iron is found to be 55.84.

We are greatly indebted to the Carnegie Institution of Washington for generous pecuniary assistance in carrying out this investigation.

CAMBRIDGE, MASS.

THE ALLEGED COMPLEXITY OF TELLURIUM.[1]

BY WILLIAM CONGER MORGAN.

Received September 28. 1912.

The accepted atomic weight of tellurium, 127.5, is higher than that of iodine, 126.9, and the so called anomalous position of tellurium in the periodic system resulting therefrom has been the incentive to many a research inquiring as to the homogeneity of this element. Brauner in 1889[2] was led to believe that "tellurium is not a simple substance" and offered this as an explanation of the lack of uniformity in the results obtained by his many different methods of determining its atomic weight. The same year in the Faraday Memorial Lecture, Mendeleeff stated his belief that with true tellurium, the atomic weight of which is about 125, there is associated another element of higher atomic weight belonging to the sulfur group. For this he suggested the name dvitellurium.

Though much careful work has been done upon this problem, every subsequent investigator has acknowledged his failure to obtain any direct evidence of the complexity of tellurium until the publication of

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[1] Results presented before the Eighth International Congress of Applied Chemistry, September 7, 1912.

[2] *J. Chem. Soc.*, **55**, 382.