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REDETERMINATION OF THE ATOMIC WEIGHT OF ALUMINUM

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ABSTRACT

In this redetermination of the atomic weight of aluminum, weighed portions of the metal were converted to the hydroxide and sulphate, respectively. These were then heated until they were changed to the oxide, Al_2O_3 , which was finally ignited at 1,200 to 1,300° C. By this procedure, the atomic weight is related directly to that of oxygen.

Careful analyses showed that the metal contained only very small quantities of impurities and disclosed no gases in the highly ignited oxide. Proper corrections were made for the small amount of impurities in the metal, and special precautions were taken to prevent absorption of moisture by the ignited oxide during weighing.

The value for the atomic weight based on this work is 26.974 ± 0.002 .

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I. INTRODUCTION

All recent determinations of the atomic weight of aluminum have been made by the analysis of aluminum halides,^{1 2 3} and consequently the values depend on the purity of the halide and on the atomic

¹ T. W. Richards and H. Krepelka, J. Am. Chem. Soc. **42**, 2221 (1920).

² H. Krepelka, J. Am. Chem. Soc. **46**, 1343 (1924).

³ H. Krepelka and N. Nikolic, Chem. Listy **19**, 158 (1925), and Chem. Abs. **19**, 3179 (1925).

weights of the silver and of the halogen involved. The successful determination of the atomic weight of gallium by converting the metal to the oxide ⁴ suggested that the atomic weight of aluminum could be similarly determined, thus directly relating it to that of oxygen and avoiding errors that might exist in determinations by less direct methods.

Previous investigators have determined the atomic weight of aluminum by converting the metal to the oxide, but the results are of doubtful value because the metal and oxide were probably impure. In 1858 Tissier ⁵ obtained 27.185 for the atomic weight of aluminum by converting the metal, first to the chloride, then to the nitrate, and finally to the oxide. By essentially the same method Isnard ⁶ obtained a value of 27. The last attempt to establish the direct ratio between aluminum and oxygen by converting the metal to the oxide was made by Kohn-Abrest ⁷ in 1905 when he obtained 27.23 for the atomic weight. As the above-mentioned values are in disagreement with the present accepted value,⁸ and since aluminum of very high purity is now available, the present redetermination was undertaken.

II. SUITABILITY OF ALUMINUM OXIDE FOR ATOMIC-WEIGHT WORK

At the outset, it was necessary to make sure that both the metal and the oxide, Al_2O_3 , are suitable for this type of work. Careful analyses, which will be discussed later, showed that the aluminum metal available was satisfactory, and it remained only to determine whether the oxide, Al_2O_3 , is a suitable compound for establishing the direct ratio between aluminum and oxygen. For these tests, two portions of oxide were prepared, one by dissolving aluminum of high purity in diluted sulphuric acid, evaporating the solution to dryness and igniting the residue to the oxide, the other by dissolving the metal in diluted hydrochloric acid, precipitating with ammonium hydroxide, filtering, and igniting the precipitate to the oxide. Both portions of oxide were ignited between 1,250 and 1,300° C for 20 hours, after which they were placed in a desiccator, to be used in the tests that follow.

T. W. Richards and E. F. Rogers ⁹ found that many oxides contained gases (chiefly oxygen and nitrogen) which they did not succeed in driving out by heating in a vacuum or by heating with a blast lamp. Our work on gallic oxide ¹⁰ indicated that prolonged ignition at 1,200 to 1,300° C tends to free gallic oxide from tightly held gases. Aluminum oxide, formed in the same way, could reasonably be expected to be free from gases. To test this, samples of both portions of the prepared oxides were analyzed for their gas content by a procedure similar to that described in the paper just cited. For this purpose, a modification of the ordinary tubulated Gooch crucible was designed so that the aluminum oxide could be fused with borax at 1,000 to 1,100° C without any danger of softening the cement that was used to seal the cover on the crucible. This water-jacketed Gooch crucible is shown in figure 1.

⁴ G. E. F. Lundell and James I. Hoffman, *J. Research NBS* **15**, 409 (1935) RP838.

⁵ Ch. Tissier, *Compt. rend.* **46**, 1105 (1858).

⁶ Note in *Compt. rend.* **66**, 508 (1868).

⁷ Emile Kohn-Abrest, *Bul. soc. chim.* [3] **33**, 121 (1905).

⁸ *J. Am. Chem. Soc.* **43**, 541 (1926), and **58**, 541 (1936).

⁹ *Proc. Am. Acad. Arts Sci.* **28**, 200 (1893).

¹⁰ *J. Research NBS* **15**, 409 (1935) RP838.

A 6-g portion of borax, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, was heated in the open crucible until water and visible bubbles of gas were expelled. By means of Cementyte a small glass cap was sealed over the end of tube B; then the cover was sealed to the crucible, and tube A to a manometer. The fused borax was then heated for 3 hours at about $1,000^\circ\text{C}$ in a vacuum. After cooling, the cover was removed, 1 g of the highly ignited aluminum oxide was quickly placed on top of the borax, and the cover was again sealed in place. After evacuating the system (having a total volume of about 45 ml), the borax and aluminum oxide were heated at $1,000$ to $1,100^\circ\text{C}$ for 40 to 60 minutes. In this time practically all the aluminum oxide was dissolved in the borax. When the system had cooled to the original temperature, the change in pressure was noted and compared with the change obtained in a blank determination involving only the borax. In

a series of 10 determinations, including samples from both portions of oxide, the average difference between the blanks and the determinations involving 1 g of oxide was equivalent to less than 0.1 ml of gas, as calculated from the volume of the system and the difference in pressure before and after the fusions (1 mm of Hg being equivalent to approximately 0.06 ml). In checking this procedure, 0.43 and 0.60 ml of gas were obtained, respectively, from 0.5-g portions of zinc oxide which was shown to contain 0.96 ml of gas per gram. The amount of gas in the zinc oxide was determined by dissolving the oxide in diluted hydrochloric acid ¹¹ (1+4) and collecting the gas in a graduated tube. Some of the cooled melts resulting from the fusion of the aluminum oxide in borax were also dissolved in diluted hydrochloric acid (1+4), but no gas was obtained. As a whole, the blanks were high and the results obtained in the determination of gases did not check as well as might be desired, but it can be stated definitely that no significant quantity of gas was contained in the aluminum oxide which had been ignited for 20 hours or more at $1,200$ to $1,300^\circ\text{C}$.

As it is known to be difficult to obtain constant weight by igniting aluminum hydroxide to the oxide, tests for small quantities of water

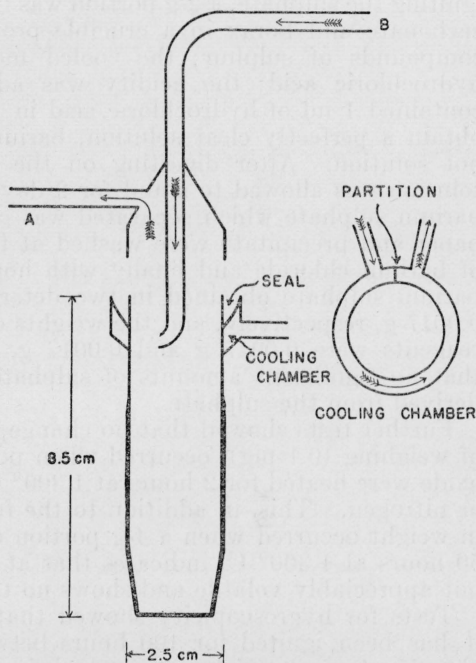


FIGURE 1.—Water-jacketed tubulated Gooch crucible.

The crucible is made of platinum containing 3.5 percent of rhodium and is of extra thickness in the lower part to prevent distortion during fusions.

¹¹ Diluted hydrochloric acid means the concentrated reagent to which an indefinite volume of water has been added. Diluted hydrochloric acid (1+4) denotes 1 volume of concentrated hydrochloric acid (sp gr 1.18) diluted with 4 volumes of water. If no dilution is specified, the concentrated reagent is meant. This system of specifying diluted acids is used throughout this paper.

in the final ignited product seemed desirable. The oxide which was derived from the hydroxide did not yield any gas during fusion with borax in a vacuum. This alone was probably sufficient evidence to show that no appreciable amount of water was retained, but it was deemed advisable to add some substance containing a known amount of moisture and then fuse the mixture with borax in a vacuum. One milligram of bauxite containing 0.27 mg of water was accordingly added to 1 g of the highly ignited aluminum oxide, and the mixture was fused. After cooling to the original temperature, the manometer reading definitely showed an increase in pressure and indicated 0.25 mg of water. This shows that a small amount of water would have been revealed by the fusion in a vacuum if it had been present.

To test for residual sulphate in the oxide that was formed by igniting the sulphate, a 2-g portion was fused with a mixture of sodium carbonate and borax in a crucible protected from gases containing compounds of sulphur; the cooled melt was dissolved in diluted hydrochloric acid; the acidity was adjusted so that the solution contained 1 ml of hydrochloric acid in 100 ml, and, after filtering to obtain a perfectly clear solution, barium chloride was added to the hot solution. After digesting on the steam bath for 1 hour, the solution was allowed to stand for 2 days at room temperature. The barium sulphate which separated was collected on a small filter, the paper and precipitate were washed at first with a 1-percent solution of barium chloride and finally with hot water. The weights of the barium sulphate obtained in two determinations were 0.0016 g and 0.0017 g, respectively, and the weights obtained in two blanks on the reagents were 0.0017 g and 0.0017 g, respectively, which indicates that no significant amounts of sulphate were retained by the oxide derived from the sulphate.

Further tests showed that no change, within the limits of accuracy of weighing (0.1 mg), occurred when portions of the strongly ignited oxide were heated for 2 hours at $1,300^{\circ}\text{C}$ in an atmosphere of oxygen or nitrogen. This, in addition to the fact that no detectable change in weight occurred when a 4-g portion of oxide was heated in air for 50 hours at $1,300^{\circ}\text{C}$, indicates that at this temperature the oxide is not appreciably volatile and shows no tendency to dissociate.

Tests for hygroscopicity showed that aluminum oxide, even after it has been ignited for 100 hours between $1,200$ and $1,300^{\circ}\text{C}$, will absorb sufficient moisture to make it impossible to weigh it accurately in a container that is open to the atmosphere. For example, an 8-g portion of the strongly ignited oxide absorbed 0.0113 g of moisture when it was exposed in an open platinum crucible for 2 hours in an atmosphere having a relative humidity of 80 percent. Three additional days of exposure, however, caused practically no further change in weight. The troubles caused by hygroscopicity were overcome by transferring the crucibles directly from the furnace to a Pyrex glass weighing bottle shown in figure 2, and described in a later section.

By taking into consideration (1) that aluminum oxide that has been derived from the hydroxide or sulphate and has been ignited for 20 hours or more at $1,200$ to $1,300^{\circ}\text{C}$ contains no appreciable amount of gas, (2) that the oxide shows great stability under varying conditions, (3) that the oxide derived from the hydroxide retains no significant quantity of water, (4) that the oxide derived from the sulphate

contains no significant quantity of residual sulphate, and (5) that the difficulties caused by hygroscopicity can be overcome by a special type of weighing bottle, it was concluded that this oxide, Al_2O_3 , ignited at $1,300^\circ\text{C}$, is a suitable compound to use in determining the atomic weight of aluminum.

III. ANALYSIS OF THE ALUMINUM METAL

Two lots of aluminum were used in this work. The purer lot was furnished in the form of notch-bars through the courtesy of H. V. Churchill and R. W. Bridges, Chief Chemist and Assistant Chief Chemist, respectively, of Aluminum Research Laboratories, Aluminum Co. of America, New Kensington, Pa. In the discussion which follows, this lot will be referred to as aluminum A. The other lot is the standard sample 44c, furnished by the National Bureau of Standards as a melting-point standard.

1. SPECTROCHEMICAL ANALYSIS

Samples from both lots were tested spectrochemically by B. F. Scribner of the Bureau's Spectroscopy Section, and the spectra were examined for the sensitive lines of Ag, As, Au, B, Be, Bi, C, Ca, Cd, Ce, Co, Cr, Cu, Fe, Ga, Ge, Hf, Hg, In, Ir, K, Li, Mg, Mn, Mo, Na, Ni, Os, Pb, Pd, Pt, Rh, Ru, Sb, Sc, Si, Sn, Sr, Ta, Th, Ti, Tl, U, V, W, Y, Zn, and Zr. The elements, other than aluminum, that were detected are listed in table 1. The scale used by the Spectroscopy Section in qualitatively designating increasing amounts of impurities is: *trace*, *very weak*, *weak*, *moderate*, and *strong*. The designation *faint trace* is used only when the most sensitive lines of the impurity appear near the limit of visibility.

TABLE 1.—Results of chemical and spectrochemical analyses of aluminum metal

Elements found or for which special tests were made	Aluminum A		Aluminum sample 44c	
	Chemical determination (figures are expressed in percent)	Qualitative spectrochemical designation ¹	Chemical determination (figures are expressed in percent)	Qualitative spectrochemical designation
B	Not detected ²	Not detected	Not detected	Not determined.
Be	do	do	do	Not detected.
C	do	do	do	Do.
Ca	Not determined ³	do	do	Faint trace.
Cr	Not detected	do	do	Do.
Cu	0.002	Trace	0.006	Trace.
Fe	0.003	do	0.007	Do.
Ga	(4)	Faint trace	0.0004	Faint trace.
Mg	Not detected	Trace	Not detected	Trace.
Mn	<0.0002	Faint trace	do	Faint trace.
Mo	(4)	Not detected	0.00002	Not detected.
Na	Not determined	Faint trace	Not determined	Faint trace.
Pb	Not detected	Not detected	Not detected	Do.
P	<0.001	Not determined	do	Not determined.
S	Not detected	do	0.0001	Do.
Si	0.006	Trace	0.011	Trace.
Ti	(4)	Faint trace	0.0001	Not detected.
V	(4)	Not detected	Not detected	Do.
Zr	(4)	do	0.0003	Do.

¹ The same qualitative spectrochemical designation is given for iron and for gallium in both lots of metal, but the lines indicated that standard sample 44c contains more iron and gallium than aluminum A.

² "Not detected" means that the material was examined, but the element was not found.

³ "Not determined" means that the material was not examined for the element.

⁴ See (b) and (c) under Chemical Analysis in text, page 5, 6 and 7.

2. CHEMICAL ANALYSIS

For most of the determinations, the conventional methods of analysis were used. The careful analysis of sample 44c was made by J. A. Scherrer, who determined gallium and most of the other impurities by the methods described or suggested in *Determination of Gallium in Aluminum*.¹² Aluminum *A* was analyzed by one of the authors. Details will be given only in those cases in which the determinations seem to be of especial interest, or in which unexpected difficulties were encountered. Careful blank determinations in duplicate were made on the reagents in all the following tests.

(a) SILICON

A 10-g sample of aluminum *A* was transferred to a 600-ml Pyrex beaker and dissolved in 500 ml of a mixture of acids prepared by mixing 485 ml of water, 115 ml of sulphuric acid, 200 ml of hydrochloric acid, and 200 ml of nitric acid. The beaker was immersed in ice water to retard the reaction. The solution was evaporated until fumes of sulphuric acid appeared. Warm water was then added to the partially cooled residue, and the beaker was placed on the steam bath until the aluminum sulphate was dissolved. The solution was poured through a small filter, the paper was washed with hot water, and the filtrate and washings were again evaporated until fumes of sulphuric acid appeared. After the aluminum sulphate was dissolved, the solution was poured through a small filter, the paper was washed with hot water, and the silicon in the two papers was determined in the usual manner by igniting the papers, weighing the residue, then treating it with hydrofluoric and sulphuric acids, and again igniting and weighing. The small nonvolatile residue that remained after the silica was volatilized was fused with a small quantity of sodium carbonate, the cooled melt was dissolved in diluted hydrochloric acid, and the resulting solution was evaporated to dryness. The dried residue was treated with 20 ml of diluted hydrochloric acid (1+20), and the solution was examined for silica. None was found, which indicates that all the silicon was oxidized by the mixture of acids, and that no silicon remained in the nonvolatile residue.

(b) ELEMENTS THAT ARE PRECIPITATED BY HYDROGEN SULPHIDE IN ACID SOLUTION (COPPER)

A 100-g sample of aluminum *A* was dissolved in a slight excess of diluted hydrochloric acid (1+2). The resulting solution was diluted to 2 liters, and the acidity was adjusted so that about 2 percent by volume of hydrochloric acid was present. The solution was heated nearly to boiling, and a rapid stream of hydrogen sulphide was passed into it for 3 hours while it cooled. After standing overnight, the small precipitate which formed was collected on a filter. The paper and precipitate were decomposed in a mixture of nitric and sulphuric acids, and nitric acid was expelled by evaporating until fumes of sulphuric acid appeared. The cooled solution was diluted with water and filtered to remove any silica that might have separated. The acidity of the filtrate was adjusted to 2 percent by volume, and the

¹² J. A. Scherrer, *J. Research NBS* **15**, 585 (1935) RP853.

precipitation with hydrogen sulphide was repeated. The resulting precipitate was collected on a filter, ignited at 500° C in a porcelain crucible, cooled, and weighed. The residue weighed 0.0027 g. This was dissolved in nitric acid, and the copper in it was determined colorimetrically.¹³ The determination indicated 0.00175 g of copper, equivalent to 0.0022 g of CuO. This leaves only the difference between 0.0027 g and 0.0022 g or 0.0005 g of oxides to be distributed among all the other elements that are precipitated by hydrogen sulphide in acid solution. Since 0.0005 g in a 100-g sample is without significance in this work, no attempt was made to separate this small weight of oxides into its constituents.

(c) ELEMENTS THAT ARE PRECIPITATED BY CUPFERRON (IRON)

Iron, vanadium, titanium, zirconium, tin, and gallium are completely precipitated by cupferron in a solution containing 5 ml of sulphuric acid in 100 ml. It was therefore decided to precipitate all these elements and to determine their composite weight in the form of oxides. Filtrates from the silicon determinations were combined so that the solution contained the equivalent of 21.0 g of aluminum in a volume of 1 liter. The acidity was adjusted to 5 ml of sulphuric acid in 100 ml, the solution was cooled, and an excess of cupferron was added. After standing in ice water for 3 hours, the precipitate was collected on a filter and washed with cold diluted sulphuric acid (1+100) containing a little cupferron. The paper and precipitate were decomposed by digestion with nitric and sulphuric acids, and the nitric acid was expelled by evaporating until fumes of sulphuric acid appeared. After cooling, 100 ml of water was added, and the acidity was adjusted so that the solution contained 5 ml of sulphuric acid. The precipitation with cupferron was repeated, and the precipitate was collected on a filter, ignited in platinum, treated with hydrofluoric and sulphuric acids, again ignited, and weighed. The weight of the residue was 0.0011 g. Iron oxide in this residue was determined by leaching the oxides with hydrochloric acid and applying the colorimetric method of Stokes and Cain¹⁴ to the solution. Iron, equivalent to 0.0009 g of Fe₂O₃ was found. Since this left only 0.0002 g to be distributed among all the other oxides of the elements that are precipitated by cupferron, and since such a small weight, compared with the weight of sample taken, is without significance in this work, no further efforts were made to determine the constituents of the residue. Qualitative tests showed it to be free from copper.

The percentage of iron, as calculated from the quantity found by the above procedure, is 0.003. By cutting out little pieces, such as were used in the atomic weight determinations, dissolving them in diluted hydrochloric acid, and applying the colorimetric method of Stokes and Cain, 0.0027 percent of iron was found. This shows that no significant amount of iron was introduced by the tools used in preparing the samples for the atomic weight determination.

(d) MAGNESIUM

A 14-g sample of aluminum was placed in a Pyrex beaker containing 500 ml of cold water, and 25-ml portions of a 30-percent solution of sodium hydroxide were added at intervals until the alumi-

¹³ See G. E. F. Lundell, J. I. Hoffman, and H. A. Bright, *Chemical Analysis of Iron and Steel*, p. 274 (J. Wiley and Sons, New York, N. Y., 1931).

¹⁴ H. N. Stokes and J. R. Cain, *Bul. BS* 3, 115 (1907) S53.

num was dissolved. The solution was then heated on the steambath for 1 hour, cooled to room temperature, and filtered. After washing the precipitate with a 1-percent solution of sodium hydroxide, it was dissolved in diluted hydrochloric acid, 1 g of citric acid was added to the solution, and the magnesium was determined by the usual procedure of twice precipitating with diammonium phosphate and igniting the precipitate to the pyrophosphate, $\text{Mg}_2\text{P}_2\text{O}_7$. The weights of $\text{Mg}_2\text{P}_2\text{O}_7$ obtained in two determinations were the same as that obtained in a blank determination involving only the reagents. As a check on the method, 0.0014 g of magnesium powder was added to one blank at the start. After allowing for the blank on the reagents, the weight of $\text{Mg}_2\text{P}_2\text{O}_7$ obtained (0.0064 g) corresponded exactly to the weight of magnesium added. These tests indicate that if magnesium is present, the amount is entirely negligible.

(c) MANGANESE

A 10-g sample was dissolved in diluted hydrochloric acid (1+1), 50 ml of sulphuric acid was added, and the hydrochloric acid was expelled by heating until fumes of sulphuric acid appeared. The cooled residue was dissolved in water, then nitric and phosphoric acids were added, and the solution was boiled with potassium periodate.¹⁵ A barely visible pink color appeared which was estimated colorimetrically to amount to less than 0.0002 percent of manganese.

(f) BERYLLIUM

A 20-g sample of aluminum *A* was dissolved in diluted hydrochloric acid (1+1), and after the volume was adjusted to 600 ml, the solution was cooled to about 5° C and saturated with hydrochloric acid gas. The bulk of the aluminum was precipitated as the hydrated chloride, $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$, and removed by filtration.¹⁶ The precipitate was washed with cold hydrochloric acid saturated with hydrochloric acid gas. The filtrate and washings were concentrated to a volume of 20 ml, the solution was cooled, an equal volume of ether was added, and then the solution was saturated with hydrochloric acid gas. The small precipitate of hydrated aluminum chloride was removed by filtration and washed with a cold solution prepared by mixing equal volumes of hydrochloric acid and ether and saturating it with hydrochloric acid gas.

The filtrate and washings were evaporated to dryness, the residue was dissolved in diluted hydrochloric acid, and the iron and the small quantity of aluminum that might have remained in the solution¹⁷ were precipitated by 8-hydroxyquinoline from an acetic acid solution, buffered with ammonium acetate (pH of approximately 6.8). The procedure described by Knowles for the separation of beryllium from aluminum was followed.¹⁸ The filtrate was evaporated with nitric, sulphuric, and perchloric acids to destroy the excess of 8-hydroxyquinoline. When the reagent was destroyed, 50 ml of water was added and the solution was rendered slightly ammoniacal. After standing for 4 hours, a precipitate appeared which, after ignition,

¹⁵ See G. E. F. Lundell, J. I. Hoffman, and H. A. Bright, *Chemical Analysis of Iron and Steel*, p. 199 (J. Wiley and Sons, New York, N. Y., 1931).

¹⁶ See F. A. Gooch and F. S. Havens, *Am. J. Sci.* [4] **2**, 416 (1896).

¹⁷ The precipitate was very small and appeared to be mostly iron, which indicates that the aluminum was practically quantitatively precipitated by the treatment with hydrochloric acid and ether.

¹⁸ H. B. Knowles, *J. Research NBS* **15**, 87 (1935) RPS13.

weighed 0.2 mg more than that obtained in a blank carried through all the steps of the method. Although this weight is insignificant, the ignited residue was subjected to spectrochemical analyses by B. F. Scribner. No beryllium was detected, but faint traces of vanadium, bismuth, and titanium were indicated, which may have accounted for the 0.2-mg excess in the weight of the residue.

(g) NITROGEN

A 10-g sample of the metal was dissolved in diluted hydrochloric acid (1+1) in a flask fitted with a trap to prevent access of ammonia from the air. After solution was complete, the trap was removed, a funnel was placed in the neck of the flask, and a 30-percent solution of sodium hydroxide was poured in until the solution was strongly alkaline. A moist piece of red litmus paper was then pinched between the funnel and the inner surface of the mouth of the flask. The red litmus did not turn blue during 3 hours of heating on the steam bath. In a similar determination in which 0.4 mg of ammonium chloride, equivalent to 0.1 mg of nitrogen, had been added to the solution, the red litmus had turned blue at the end of 15 minutes. This indicates that if nitrogen is present as the nitride, its amount is less than 0.02 mg in a 2-g sample, which is entirely negligible.

(h) OCCLUDED GASES

A small piece of aluminum, weighing 5.0 g, was fused in a vacuum in the apparatus used for determining gases in the oxide. A small porcelain crucible was used as the container for the metal inside of the tubulated Gooch crucible to prevent the aluminum from coming in contact with the platinum. The manometer showed a slight increase in pressure, but this did not differ by more than 1 mm of Hg from that obtained when the porcelain crucible alone was heated in the apparatus in a blank determination. This indicates that 5.0 g of the metal contained less than 0.1 mg of occluded gases.

(i) CARBON

Aluminum shavings, such as are obtained in the usual sampling operations, do not burn rapidly in air or oxygen even at a temperature of 1,300° C. The coating of oxide that forms on the surface prevents burning of the metal within, and heating at 1,300° C. in oxygen for as much as 4 or 5 hours usually does not effect complete oxidation of the metal. It is obvious that an accelerator or flux must be used. Various substances were tried, but only one, copper oxide, was found to be satisfactory.

In the procedure finally used, the aluminum shavings were burned in the type of furnace used in the determination of carbon in steel.¹⁹ Because of the high temperature required, the type that is heated by silicon carbide elements was used. A nickel boat, previously ignited in oxygen, was filled with 90-mesh alundum, and then 1 g of aluminum shavings was placed in a groove in the alundum. After distributing the aluminum uniformly throughout the length (10 cm) of the boat, 0.5 g of copper oxide, CuO, was evenly distributed on top of the metal. A nickel cover was placed over the boat, and the charge was put in the combustion tube which had previously been heated to 1,000° C. As

¹⁹ G. E. F. Lundell, J. I. Hoffman, and H. A. Bright, *Chemical Analysis of Iron and Steel*, p. 154-18 (J. Wiley and Sons, New York, N. Y., 1931).

soon as the tube was closed, a rapid stream of oxygen was admitted, and the temperature of the furnace was raised from 1,000 to $1,325 \pm 25^\circ \text{C}$, within 20 minutes. The latter temperature was maintained for one-half hour. The gases issuing from the furnace were passed through a tube containing platinized silica gel designed to remove compounds of sulphur, and then through a small tube containing Ascarite (sodium hydroxide on asbestos). The exit-end of the absorption tube contained anhydrous magnesium perchlorate, and a guard tube containing Ascarite and anhydrous magnesium perchlorate was attached to prevent moisture or carbon dioxide from the air from entering the absorption tube in case of back pressure which might result from rapid combustion of the aluminum.

By this procedure the aluminum was fairly completely burned, but occasionally a little of the metal remained in places where the slag ran together into rather thick nodules. Judging by the amount of hydrogen that was evolved when the pulverized slag was treated with hydrochloric acid, all but 1 or 2 percent of the metal was burned in every case. In the case of aluminum A, the average increase in weight of the absorption tube in four blank determinations was the same as in four determinations when aluminum was being burned. This indicates that there is no carbon in this material. In the case of aluminum sample 44c, there was a slight indication of the presence of carbon, but calculations showed that the amount could not have been greater than 0.003 percent. Tests of the method were made by adding chips of steel, containing carbon equivalent to 0.4 mg of carbon dioxide, to the chips of aluminum. The amount of carbon added was recovered in every case. Spectrochemical tests failed to show carbon in either of the two materials, but the sensitivity of the spectrochemical test is not known in the case of carbon.

(I) ALUMINUM OXIDE (Al_2O_3)

Metallic aluminum is said to contain small quantities of oxide²⁰ which may have been formed in the process of melting and pouring, or as a thin surface film by exposure to the atmosphere. In this work the total amount of aluminum oxide was determined by analyzing the residue left when the metal was subjected to a stream of dry hydrochloric acid gas and hydrogen at temperatures between 400 and 600°C . In general, the procedure described by Withey and Millar was used. Under these conditions metallic aluminum reacts to form the anhydrous chloride which is volatilized, while any aluminum oxide remains as a nonvolatile residue. In order to prevent oxidation of the metal, it was found that all air and moisture had to be displaced from the apparatus by passing a rapid stream of dry hydrogen through it for 4 to 6 hours before the metal was heated. In all determinations the hydrogen was purified by passing it over hot copper gauze and then through Ascarite (sodium hydroxide on asbestos) followed by anhydrous magnesium perchlorate. The hydrochloric acid gas was passed through a tower of anhydrous calcium chloride and then through anhydrous magnesium perchlorate.

Pieces of aluminum, weighing from 2 to 3 g and having a surface area of 6 to 8 cm^2 were cut out of an ingot with a sharp chisel. The

²⁰ Hirsch Löwenstein, *Z. anorg. allgem. Chem.* **199**, 48 (1931), reported 0.008 percent as the normal content of the Al_2O_3 in new aluminum metal. W. H. Withey and H. E. Millar, *J. Soc. Chem. Ind., Trans.* (London) **45**, 170 (1926), found as much as 0.066 percent in thin sheets.

surfaces of the pieces were then shaved smooth and bright with a sharp penknife. By subjecting these pieces to the procedure just described, 0.013 percent of white residue was left from aluminum *A* and 0.014 percent from aluminum sample 44c. The residues from four of these determinations, representing 15.3 g of metal, were ignited and weighed. After treating with hydrofluoric and sulphuric acids, they were again ignited and weighed. The loss in weight was only 0.2 mg, which indicates that but little if any of the silicon in the samples was present as silica. After the treatment with hydrofluoric acid, the residue was fused with sodium carbonate, the cooled melt was dissolved in diluted hydrochloric acid, ammonium hydroxide was added, and the resulting precipitate, which contained no significant amount of titanium and zirconium, was identified as aluminum hydroxide. This precipitate was ignited and weighed, then it was treated with hydrofluoric and sulphuric acids, and again ignited and weighed. The fact that a loss of only 0.1 mg resulted from the treatment with hydrofluoric and sulphuric acids indicates that practically all the very small amount of silicon in this material was volatilized during the treatment with hydrochloric acid gas and hydrogen.

Any oxide that was formed during the melting and pouring of the aluminum could reasonably be expected to remain in the nonvolatile residue because it must have been heated to at least 660° C (the melting point of aluminum). However, it was not at all certain that the film of oxide which forms on the surface of the metal at room temperature would remain in the nonvolatile residue. Accordingly, three portions of a standard solution of aluminum chloride, each equivalent to 0.0095 g of Al_2O_3 , were converted to the hydroxide. The first portion was ignited for 1 hour at 600° C, the second was dried for 20 hours at 95° C, and the third was dried at room temperature over concentrated sulphuric acid for 20 hours. These were then separately subjected to the procedure of volatilization just described. No volatilization occurred in any case.

It was realized, however, that the evidence for the nonvolatility of the oxide in hydrochloric acid gas and hydrogen would be more convincing if the determination could be made on a piece of aluminum with a film of oxide of known weight. In an attempt to produce such a film, two bright pieces of aluminum, having a surface area of 10.5 cm^2 , were placed in a tube through which a stream of oxygen was passed. No weighable film was formed in 3 days at room temperature. Intermittent heating in oxygen between 50 and 200° C for 24 hours also failed to produce a weighable film. A film of hydrated oxide was finally obtained by placing the pieces of aluminum in a platinum crucible and keeping them partially covered with water at temperatures between 25 and 95° C for 20 days. These pieces of metal were then dried in an oven at 95° C. The increase in weight was 0.0014 g. The weight of the oxide, Al_2O_3 , after volatilizing the metal as chloride, was 0.0018 g.

By spreading aluminum hydroxide in a very thin film on the inside surface of a platinum crucible and drying at 95° C for 20 hours, a hydrated oxide corresponding approximately to $\text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$ was obtained. If it is assumed that the increase in weight of 0.0014 g was caused by the formation of $\text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$ on the pieces of aluminum, then 0.0008 g of oxygen was combined with aluminum to form the

oxide, Al_2O_3 . This corresponds to 0.0017 g of Al_2O_3 ²¹. A determination of oxide on freshly cut pieces of aluminum, similar to those on which the film of oxide was formed, yielded a residue of 0.0003 g. The weight of oxide that should have been found is, therefore, 0.0017 g + 0.0003 g = 0.0020 g, as compared with 0.0018 g, the weight actually found. If the uncertainty of the composition of the film is taken into account, this must be considered as fairly good evidence that no volatilization of aluminum oxide took place.

Observations indicated that if aluminum is converted to the *anhydrous chloride*, the latter is volatile in a stream of dry hydrochloric acid gas and hydrogen, but that if the chloride is allowed to *hydrate*, it remains in the nonvolatile residue. To confirm this, 0.1000 g of the hydrated chloride, $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$, was put in a platinum boat and subjected to a stream of hydrochloric acid gas and hydrogen exactly as was done in the determination of oxide in the metal. All the aluminum chloride remained in the platinum boat. This behavior accounts for the fact that large nonvolatile residues were obtained in the preliminary experimental work when the hydrochloric acid gas and hydrogen were inadequately dried before they came in contact with the metal.

(k) PHOSPHORUS

A 5.0-g portion of metal was dissolved in diluted nitric acid (1+1) to which small portions of hydrochloric acid were added from time to time. The solution was evaporated nearly to dryness on the steam-bath, 50 ml of nitric acid was added, and the solution was again evaporated nearly to dryness. The sirupy residue was dissolved in 100 ml of diluted nitric acid (1+2), the solution was nearly neutralized with ammonium hydroxide, and molybdate reagent was added. The solution was shaken for 10 minutes and allowed to stand for one-half hour. No precipitate appeared. In a solution similarly prepared but to which 0.05 mg of phosphorus was added in the form of a standard solution of phosphoric acid, a definite yellow precipitate appeared at the end of one-half hour. This indicates that if phosphorus is present, the amount is less than 0.001 percent.

IV. PROCEDURES AND RESULTS

1. CHEMICAL REAGENTS AND PLATINUM CRUCIBLES

Hydrochloric acid, sulphuric acid, and ammonium hydroxide were the only reagents used in this part of the work. The acids, bought on the specifications for analytical reagent chemicals recommended by the Committee on Analytical Reagents of the American Chemical Society, were of such good quality that it was not necessary to purify them. None of them yielded a nonvolatile residue exceeding 0.2 mg when 100-ml portions were evaporated and the residue ignited. As less than 30 ml of the combined acids was involved in any determination, and especially since exactly the same quantities were used in blank determinations, these small amounts of impurities were entirely negligible. The ammonium hydroxide was prepared by passing gaseous ammonia into distilled water contained in a Pyrex flask whose inside surface was covered with paraffin. A 100-ml portion of this

²¹ When $\text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$ is formed on the surface of the metal, the addition of oxygen and water causes the increase in weight. Therefore, $3 \text{ O} + 2\text{H}_2\text{O} = 0.0014 \text{ g}$. The oxygen combined with Al to form $\text{Al}_2\text{O}_3 = 4/7 \times 0.0014 = 0.0008 \text{ g}$. And $0.0008 \text{ g O} \div 0.0017 \text{ g Al}_2\text{O}_3$.

ammonium hydroxide yielded a residue weighing less than 0.1 mg, even after the solution had stood in the flask for over 9 months.

In work involving prolonged ignitions at high temperatures it is essential that the crucibles do not change weight or that two or more crucibles are available that change weight at a uniform rate. In the determinations reported in this paper one platinum crucible, from a series of four having the same composition, was used as a tare. The crucibles were made of platinum containing 3.5 percent of rhodium and had a capacity of 40 ml. The whole set was heated in a platinum-wound muffle furnace for 5 hours at 1,200 to 1,300° C, cooled, and weighed. Then the crucibles were again heated for 15 to 25 hours at 1,200 to 1,300° C, cooled, and weighed. One crucible, compared with the tare, showed an apparent change in weight of more than 0.1 mg during this latter period of heating and was therefore rejected.

2. CONVERSION OF METALLIC ALUMINUM TO ALUMINUM OXIDE, Al_2O_3 , THROUGH THE HYDROXIDE

For the conversion of the metal to the oxide through the hydroxide, a piece of aluminum weighing approximately 2 g was cut out and prepared by the procedure described under III, j, p. 10. It was then carefully weighed and transferred to a 125-ml Pyrex Erlenmeyer flask whose neck was drawn to about one-half its original diameter and three times its original length. After adding 90 ml of diluted hydrochloric acid (1+2), the flask was covered with a Pyrex watch glass and placed on the steambath. These precautions prevented loss by spraying during the very slow solution of the metal, which required 3 or 4 days. For the determination of a "blank", about 4 mg of the same aluminum was carefully weighed and transferred to a similar flask to which the same quantity of acid had been added. From this point the contents of the two flasks were subjected to the same treatments, and the proper correction for the aluminum added to the blank was applied at the end of the determination. The small amount of aluminum was added to the blank to serve as a gatherer of traces of impurities that might otherwise have escaped precipitation.

After the aluminum was dissolved, the solution was transferred to a 250-ml Pyrex beaker and diluted to 150 ml. After adding a few drops of methyl red indicator, ammonium hydroxide was added until the color of the indicator just turned yellow. The beaker was allowed to stand on the steambath for 5 minutes. Then the precipitate was transferred to large ashless filters and washed thoroughly with hot water to remove ammonium chloride which, if not removed, has a tendency to crystallize at the edge of the paper and cause mechanical losses during the drying of the precipitate. The filtrate and washings were slightly acidified with hydrochloric acid; the solution was evaporated to a volume of 40 to 50 ml; and ammonium hydroxide was again added until the color of the indicator just turned yellow. After digesting on the steambath for 15 minutes, this solution was allowed to stand overnight at room temperature, and the small precipitate which separated was removed by filtration and combined with the first precipitate. A dilute solution of ammonium chloride, whose pH was adjusted to about 6.2, was used for washing this last precipitate.

The filtrate was transferred to a clean platinum dish and heated until nearly all ammonium salts were expelled. A few drops of hydrochloric acid and 5 ml of water were added to the residue. The dish and contents were heated on the steambath for a few minutes, and the solution was poured through a small filter. The filtrate, having a volume of about 15 ml, was neutralized with ammonium hydroxide as before and allowed to stand for 24 hours. Although no precipitate was visible, the solution was poured through a small filter which was then added to the precipitates previously obtained.

The filters containing the precipitates were transferred to platinum crucibles which had been tested for constancy of weight, as previously described. In each case the filter paper containing the small amount of precipitate obtained from the aluminum that was added to the blank was placed in the crucible used as a counterpoise. Special care was taken to use in the blank the same number of filter papers of the same size and from the same lot as were used in the regular determination.

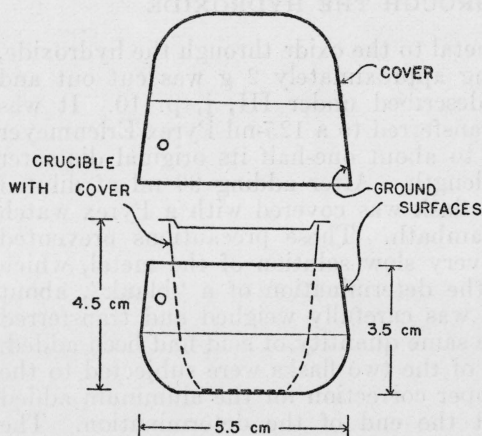


FIGURE 2.—Glass weighing bottle.

Tests for uniformity of the ash content of the filter papers were also made. The weights of the ash from three sets of papers, similar to those used to collect the aluminum hydroxide, were found to be identical within the limits of accuracy of weighing. After the precipitates were dried in an oven, the crucibles were partially covered and placed in a cold muffle furnace which was gradually heated to $1,200^{\circ}\text{C}$ during the course of about 10 hours. The residues were then heated in the covered cruci-

bles at $1,200$ to $1,300^{\circ}\text{C}$ until constant weight was obtained. All final weights remained constant within 0.1 mg during heating for at least 20 hours between $1,200$ and $1,300^{\circ}\text{C}$, and two of the ignited precipitates were heated for over 50 hours between these two temperatures without showing any change in weight.

Since it was found that aluminum oxide is still slightly hygroscopic after these prolonged ignitions, the hot crucibles were taken from the furnace and immediately placed in Pyrex weighing bottles, one of which is illustrated in figure 2. The lower portion of the bottle is only about 3.5 cm high so that the crucible can be set in it with Blair crucible tongs without disturbing the lid of the crucible. The covers were then placed on the bottles so that there was only the slightest meshing between the small holes of the cover and bottle to permit equalization of pressure during cooling. After cooling for 1 hour in a desiccator over concentrated sulphuric acid, the bottles containing the crucibles and aluminum oxide were weighed. In all weighings a similar bottle, containing the platinum crucible with the small quantity of oxide derived from the aluminum added to the blank, was

used as a counterpoise. In calculating the atomic weight, the weight of the sample of aluminum (about 2 g) minus the small amount of metal (about 4 mg) added to the blank was taken as the weight of aluminum metal that was converted to the oxide. Then the difference between the two weights of oxide thus obtained represented the weight of oxide that corresponded to this weight of aluminum, since the blanks on the reagents and glassware were equally operative in both cases and cancelled each other. The determinations were usually made in pairs, and in a few cases as much as 0.1 g of pure aluminum was added to the blank.

All weighings were made with a Ruprecht balance which had a sensitivity of seven scale divisions for 1 mg at a 2-g load and five scale divisions for 1 mg at a 135-g load, the former being the load during the weighing of the metal, and the latter the load during the weighing of the aluminum oxide, including the platinum crucibles and glass weighing bottles. The weight of the metal could easily be reproduced within 0.03 mg on repeated weighings, but reproducibility within 0.1 mg is the best that could be attained in weighing the oxide. It was more convenient to make direct weighings than to weigh by substitution. Because the ratio of the length of the balance arms changed slightly with load, corrections for this effect were made, although these were practically negligible. The weights were carefully calibrated by the Mass Section of the National Bureau of Standards before this work was started and after it was finished, and the proper corrections were made. By weighing with a counterpoise, the use of brass weights in excess of 10 g was avoided. Errors caused by the effect of changes in humidity on the brass weights were therefore negligible. All weights were corrected to the vacuum standard, 2.702 being used as the density of the metal and 4.00 as the density of aluminum oxide,²² Al_2O_3 . Corrections for the very small amounts of impurities in the metal were made by subtracting the weight of the impurities as the elements from the weight of the metal and then subtracting their calculated weight as the oxides from the weight of the ignited oxide.²³ The correction for the small amount of oxide contained in the aluminum (see III, 2, j, p. 10) was made by subtracting its weight both from that of the metal and from that of the ignited oxide.

In three cases it was possible to remove the alumina completely from the crucibles by rubbing gently with the fingers and moist filter paper after the ignitions and weighings were finished. The weights of the crucibles, as compared with the counterpoise, had not changed more than 0.1 mg, and a careful spectrochemical test, capable of easily detecting 0.003 percent of platinum, revealed no platinum in this oxide. The results that were checked in this manner are marked with an asterisk (*) in table 2. It is obvious that such a check was not possible in the series in which the oxide was formed by ignition of the sulphate.

The results obtained by converting the metal to the oxide through the hydroxide are shown in table 2.

²² Int. Critical Tables 1, 103, 136 (McGraw-Hill Book Co., New York, N. Y., 1926).

²³ For example, aluminum A contains 0.006 percent of silicon which in a 2-g sample is equivalent to 0.12 mg of Si and 0.26 mg of SiO_2 . Thus, in the case of silicon, 0.12 mg was subtracted from the weight of the metal and 0.26 mg from the weight of the oxide.

TABLE 2.—Results obtained for the atomic weight of aluminum by converting the metal to the oxide through the hydroxide

Number	Weight of aluminum	Weight of Al_2O_3	Calculated atomic weight
	g	g	
1 ^a	2.00100	3.78105	26.979
2 ^b	1.89511	3.58079	26.982
3 ^b	1.83837	3.47351	26.983
4 ^a	1.88787	3.56752	26.975*
5 ^a	1.90155	3.59348	26.974*
6 ^b	2.33772	4.41805	26.970
7 ^b	1.99419	3.76859	26.973*
Average.....			26.977 ± 0.004

^a Aluminum sample no. 44c.^b Aluminum A.

* The values marked with an asterisk (*) were checked by weighing the platinum crucibles after the prolonged ignitions.

3. CONVERSION OF METALLIC ALUMINUM TO ALUMINUM OXIDE, Al_2O_3 , THROUGH THE SULPHATE

The metal (about 2 g) was dissolved as described in the previous procedure, and the hydrochloric acid was removed by adding 7 to 10 ml of sulphuric acid and evaporating until fumes of the latter appeared. The residue was cooled, 15 to 20 ml of water was added, and the resulting solution was transferred to a platinum crucible that had been tested for constancy of weight as compared with a similar crucible as a counterpoise in the manner previously described. Exactly the same quantities of acids were carried along as a blank, and the residue resulting from the blank was ignited in the platinum crucible used as a counterpoise. The small flasks in which the metal was dissolved were repeatedly washed with water and the rinsings added to the contents of the crucibles. These additions were made possible by evaporating part of the water from the crucibles between rinsings.

TABLE 3.—Results obtained for the atomic weight of aluminum by converting the metal to the oxide through the sulphate

Number	Weight of aluminum	Weight of Al_2O_3	Calculated atomic weight
	g	g	
1 ^a	1.88650	3.56504	26.973
2 ^b	2.00812	3.79482	26.974
3 ^b	1.63804	3.09555	26.973
4 ^b	2.65087	5.00956	26.973
5 ^b	2.64428	4.99696	26.975
6 ^b	2.04031	3.85588	26.971
7 ^b	1.72393	3.25736	26.982
Average.....			26.973 ± 0.001

^a Aluminum sample 44c.^b Aluminum A.

* The value in parentheses was omitted in averaging the results because its deviation from the mean of the other results is greater than 4 times the average deviation.

When it was certain that all the aluminum sulphate had been transferred, as much as possible of the water was evaporated on the steam-bath. Then the crucibles were partially covered and transferred to radiators,²⁴ and heat was *very* gradually applied until the excess

²⁴ See W. F. Hillebrand and G. E. F. Lundell, *Applied Inorganic Analysis*, p. 22 (J. Wiley & Sons, N. Y. 1929 ed.).

sulphuric acid was volatilized. The crucibles and contents were next placed in a muffle furnace, which was gradually heated until sulphur trioxide began to be evolved. This occurred at about 800°C , and during this evolution the temperature was regulated to remain practically constant in order to avoid too rapid a decomposition of the sulphate, which might result in mechanical losses. After a noticeable evolution of sulphur trioxide had ceased, the crucibles were covered, transferred to a platinum-wound furnace, and the temperature was raised to $1,200$ to $1,300^{\circ}\text{C}$. The ignition was then continued until constant weight was obtained. The weighings were made as in the previous procedure. The results obtained are shown in table 3.

V. DISCUSSION OF RESULTS

Tests indicate that the recorded weights of the metal are accurate within 0.03 mg and the weights of the oxide within 0.1 mg . If errors of this magnitude were made in weighing, and if they were in the same direction in weighing the metal as in weighing the oxide, they were practically without effect; but even if they were in opposite directions, they would cause an error of only 0.0024 in the atomic weight, assuming that 2 g of metal was converted to the oxide.

The result of the hydroxide series is 26.977 ± 0.004 ; that of the sulphate series is 26.973 ± 0.001 ; and their average, weighted inversely²⁵ as the indicated uncertainties, is 26.974 ± 0.002 . The indicated uncertainties are in all cases three times the probable error.

Krepelka,²⁶ from the ratio, $\text{AlCl}_3:3\text{Ag}$, obtained 26.972 ± 0.001 by using 107.880 and 35.458 for the atomic weights, respectively, of silver and chlorine. If the present atomic weight of chlorine (35.457) is used, this value becomes 26.975 , and if the uncertainty is expressed as three times the probable error, Krepelka's value becomes 26.975 ± 0.003 . Later, Krepelka and Nikolic²⁷ from the ratio, $\text{AlCl}_3:3\text{Ag}$, obtained 26.974 ± 0.001 by using 107.880 and 35.457 for the atomic weights, respectively of silver and chlorine. From the ratio $\text{AlCl}_3:3\text{AgCl}$, they obtained 26.972 . If the average of these two values is taken and an uncertainty of ± 0.003 is assigned to it, their value becomes 26.973 ± 0.003 .

According to Krepelka's work, the atomic weight of aluminum lies between 26.972 and 26.978 , according to Krepelka and Nikolic's work it lies between 26.970 and 26.976 , and according to the present work it lies between 26.972 and 26.976 . Since the values between 26.972 and 26.976 are common to the three, and since three different ratios were employed in obtaining them, the atomic weight of aluminum might properly be given in the International Atomic Weights to three decimal places somewhere between the limits 26.972 and 26.976 .

Aston's work²⁸ with the mass-spectrograph indicates that aluminum has no isotopes and that the mass of Al^{27} is 26.9909 ²⁹ ($\text{O}^{16}=16.0000$). This provisional value, when corrected to the chemist's scale by using the conversion factor 1.00020 to take into account the heavier isotopes of oxygen, becomes 26.9855 . This leaves a discrepancy of approximately 0.01 between the chemically determined atomic weight and

²⁵ Usually such values are weighted inversely as the square of their probable errors, but since three of the results given in the hydroxide series were checked by weighing the crucibles after the ignitions, it seems unreasonable to give the average of the hydroxide series only $1/2$ the weight given to the sulphate series.

²⁶ H. Krepelka, *J. Am. Chem. Soc.* **46**, 1343 (1924).

²⁷ H. Krepelka and N. Nikolic, *Chem. Listy* **19**, 158 (1925) and *Chem. Abstracts* **19**, 3179 (1925).

²⁸ F. W. Aston, *Mass-Spectra and Isotopes*, p. 125 (Edward Arnold and Co., London, 1933).

²⁹ *Nature* **137**, 613 (1936). The value given is stated to be only provisional.

that obtained by the use of the mass-spectrograph. If it is assumed that the latter is correct, an error would have to exist in the present chemical determination which could be explained only by assuming (1) that the aluminum metal contained impurities other than those detected chemically or spectrochemically, or (2) that an average positive error of 0.7 mg was made in determining the amount of aluminum oxide. Impurities that could possibly cause the weights of oxide to be high, and the atomic weight to be low, are boron, silicon, beryllium, lithium, sodium, potassium, magnesium, calcium, and strontium. The first three have higher factors than aluminum for conversion of the element to the oxide, and the others might be retained as sulphates when the aluminum metal is converted to the oxide through the sulphate. The last six can be excluded because (1) chemical and spectrochemical tests indicated the absence of significant amounts of these, (2) no sulphate could be detected in the oxide that was formed by converting the metal to the oxide through the sulphate, and (3) the values obtained by igniting the sulphate are in such excellent agreement with the best values (marked * in table 2) obtained by igniting the hydroxide after the aluminum was precipitated by ammonium hydroxide. In this latter procedure most of all six elements last mentioned would have been eliminated.

Boron in the original metal would have caused low weights of oxide because it is readily lost as boron hydride or by volatilization with steam as boric acid during solution of the metal in acid.³⁰ Furthermore, a special spectrochemical test whose sensitivity is not definitely known but which was capable of detecting 0.001 percent of boron in silver metal failed to reveal any boron in the aluminum. The care with which silicon was determined and the special test that was made for beryllium show clearly that no significant error could have been introduced by silicon and beryllium. This excludes all obvious possibilities of accounting for a discrepancy of 0.01 in the atomic weight by assuming the presence of impurities in the metal. Spectrochemical tests showed that the alumina obtained in both procedures contained less than 0.001 percent of boric oxide. This, together with the tests on the aluminum oxide previously described, indicates that an average positive error of 0.7 mg in determining the amount of oxide is altogether unlikely.

If our value for the atomic weight, 26.974, is converted to the physicist's scale ($O^{16}=16.0000$), 26.9794 is obtained, and the packing fraction calculated from this is -7.6 as compared with Aston's value of -3.4 . If these values for packing fraction are plotted as ordinates against mass numbers as abscissas, our value falls considerably below the curve obtained for the lighter elements, whereas Aston's value falls more nearly on the curve.³¹ Of course, any errors that may exist in the values appear magnified by plotting a curve in this manner, and no definite conclusions can be drawn until more work is done with the mass-spectrograph and until a reasonable uncertainty is assigned to the value thus obtained.

The value for the atomic weight of aluminum based on the conversion of the metal to the oxide is 26.974 ± 0.002 .

WASHINGTON, October 13, 1936.

³⁰ H. V. Churchill and R. W. Bridges, *Chemical Analysis of Aluminum*, Aluminum Research Laboratories, New Kensington, Pa. (1935).

³¹ See *Mass-spectra and Isotopes*, p. 168 (1933).