to several commercial brands of copper containing much arsenic, but not over 0.005 or, at most, 0.01 per cent. of antimony. In the latter case, to avoid contamination of the large cathode, the last traces of copper had better be taken out with a narrow 1 inch cathode, if antimony commences to show on the copper. If much selenium or tellurium are present with the excessive arsenic, remove them first by Method II. Dissolve 5 grams of copper in nitric acid, dilute to 50 cc., and add only 3 cc. of concentrated sulphuric acid. Add ammonia until a slight permanent precipitate forms and redissolve this by exactly 1 cc. of sulphuric acid (sp. gr. 1.84) to prevent deposition of nickel or zinc. Now stir in dry, powdered, chemically pure ammonium nitrate (free from traces of chlorine) until the solution is thoroughly saturated and a little remains undissolved. Electrolyze with a current $N.D._{100} = 0.7$ ampere, and 2.7 to 2.8 volts for twelve hours, then reduce to 0.5 ampere. The total time for a 5-gram sample is about thirty hours. Test as usual with hydrogen sulphide water. Any trace of copper will show at once; arsenic a little later. Results: Sample (A), 99.64 per cent. In presence of 0.1 per cent. antimony, black plate, 99.88 per cent.

After removing the large cathode, test the solution with a small cathode, if the end-point is doubtful. If the solution is too acid, the copper will not deposit properly, but a trace of free acid is necessary.

Less time and chemicals should be allowed for in any of these methods, if only 1 gram of copper is taken for the assay.

One or the other of the plans devised may be selected so as to permit accurate control work with any brand of metallic copper.

HUBBELL, MICHIGAN.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, No. 81].

RESULTS OBTAINED IN ELECTROCHEMICAL ANALYSIS BY THE USE OF A MERCURY CATHODE.[1]

BY RALPH E. MYERS.

Received July 26, 1904.

IT HAS been clearly demonstrated in recent years[2] that valuable

---

[1] From the author's thesis for the Ph.D. degree.

[2] Gibbs: *Chem. News*, **42**, 291; Gibbs: *Am. Chem. J.*, **13**, 571; Luckow: *Ztschr. anal. Chem.*, **25**, 113; Vortmann: *Ber. d. chem. Ges.*, **24**, 2749; Drown and McKenna: *J. Anal. Chem.*, **5**, 627; Smith: This Journal, **25**, 883; Coehn and Kettembeil: *Ztschr. anorg. Chem.*, **38**, 198; Kettembeil: *Ztschr. anorg. Chem.*, **38**, 213.

electrolytic determinations and separations of metals are possible with the aid of a mercury cathode.

The purpose of the present investigation was to extend this method to elements which had never been studied under such conditions. The results have been most satisfactory and will doubtless be helpful to the analyst.

The decomposition cell used by Smith consisted of a small beaker (50 cc. capacity), on the side of which, near the bottom, was introduced a thin platinum wire.[1] This wire was in contact internally with the mercury, contained in the beaker, and externally with a copper plate, upon which the beaker rested during electrolysis. The copper plate being connected with the negative electrode of a battery, the mercury became the cathode.

Six cells of the above form were used in this investigation. They were of three sizes, designated as Nos. 1, 2 and 3. The largest, No. 1, was 8.5 cm. high and about 3.5 cm. in diameter. It held 70 cc. when filled to within 1.5 cm. of the top, and the upper suface of its mercury content, approximated closely to 10 sq. cm. in area. Cell No. 2 was 8 cm. by 3 cm.; it held about 45 cc. and had a mercury surface of 7 sq. cm. Cell No. 3 also had a mercury surface of 7 sq. cm., but held only 35 cc., as it was only 6.5 cm. high. It is to be understood that these cells were used interchangeably in the work which follows, unless special mention is made to the contrary, as under chromium and molybdenum. Therefore, the dilution, except in special cases, was not an important factor, and varied within wide limits. The beakers, Nos. 1, 2 and 3 weighed 22, 26 and 20 grams; hence when filled so that the gross weight was between 95 to 100 grams, they contained anywhere from 70 to 80 grams of mercury.

In order to insure similar conditions in the weighing of the mercury, before and after the deposition, it was first washed with water, alcohol and ether, as described below, before the original tare was taken. Watch crystals should be used to cover the cells when carrying them to and from the balance room, as minute globules of mercury are readily detached, even by the motion produced in walking. Too much stress cannot be laid upon this point, and if due precaution is observed, the experimenter will not be troubled by losses, which seem unaccountable.

The time necessary for a deposition varied from five to twenty-

---

[1] This Journal, 25, 887.

four hours, depending on the amount of metal to be deposited.[1] When the necessary time had elapsed for the deposition, the acid liquid was siphoned off, distilled water being added as the level dropped to the spiral of the anode. When the ammeter needle returned nearly to zero, the washing was interrupted.

The washing and drying were accomplished as follows: The cell was filled one-third full with water and was rotated around its longitudinal axis after the latter had been tilted 45° to the vertical. By this means the whole surface of the mercury, as well as the whole inner surface of the cell, was completely freed from the last traces of the electrolyte. This operation was repeated with two or three changes of water. Alcohol was then substituted for water, the first washings being discarded. The mercury was then treated three times with ether. Absolute alcohol and ether, though preferable, are not absolutely essential. After five to ten minutes the odor of ether disappeared from the cell. It was then cleaned externally by means of a cloth and the moisture from the breath. The final traces of ether and moisture were removed by allowing the cell to remain in a desiccator over sulphuric acid for a period of fifteen minutes. In order that the cell might come to the temperature of the balance case, it was allowed to remain for five minutes on the balance pan, before weighing.

DETERMINATION OF CHROMIUM.

Text-books on electrochemical analysis make no mention of the estimation of the element chromium. It has never been determined electrolytically. When it was found that a solution of chromium sulphate, slightly acidulated with sulphuric acid, yielded its chromium to a mercury cathode, it was hoped that a method for the quantitative estimation of this metal might be secured. Further investigation proved that this could be accomplished. A solution of chromium sulphate (Merck), containing 0.1080 gram of chromium per each 10 cc., was used as the stock solution. The conditions and results are given in Table I. The wash-waters gave no evidence of the presence of chromium, even after concentration.

---

[1] When fourteen hours is given as the time used for a deposition, the precipitation was made during the night, and really consumed only about four hours of the operator's time.

TABLE I.—DETERMINATION OF CHROMIUM.

	Chromium present in grams.	Chromium found in grams.	No. of cell used.	Sulphuric acid (sp. gr. 1.832) present in drops.	Time. Hours.	Conditions.			
						Amperes.	Volts.	Amperes.	Volts.
1	0.1080	0.1079	2	2	3	0.3	7	0.55	5.5
2	0.1080	0.1080	1	3	14	0.3	7	0.55	5.5
3	0.2160	0.2157	1	4	14	0.4	7.5	0.7	6
4	0.2160	0.2160	1	4	14	0.4	7.5	0.7	6
5	0.3240	0.3235	1	8	30	0.7	7	2	6.5
6	0.3240	0.3222[1]	1	6	30	0.65	7	2.5	8

The initial voltage and amperage are given to the left in the table. The acid liberated during the course of the electrolysis caused the potential to fall and the current to rise to the final voltage and amperage, shown to the right. Chromium amalgam decomposes quite readily, especially in contact with water, metallic chromium appearing as a fine black powder on the surface of the mercury. Hence, it was found advisable to wash the amalgam as rapidly as possible. The same portion of mercury should not be used for more than one decomposition. It is the hope of the author that this ready decomposability of chromium amalgam can be used in the preparation of chromium of a high degree of purity, as all other metals liable to be present would be soluble in nitric acid. This point will receive further investigation

Experments 5 and 6 were added to the table to show that under favorable conditions 0.3 gram of chromium could be taken up by the 75 grams of mercury used in a cell of this size. Trouble was experienced, however, in washing an amalgam of this strength, the chromium appearing on the surface of the mercury before the necessary washing had taken place. The low result in Experiment 6 was due to loss of chromium in the wash-water. The appearance of oxide of chromium in the electrolyte indicates the presence of an insufficient amount of acid for the current used. In such a case a new decomposition should be started, more acid or a lower current being used. A fairly anhydrous alcohol was found to be preferable in washing chromium.

SEPARATION OF CHROMIUM FROM ALUMINUM.

The separation of chromium from aluminum in a gravimetric

---

[1] Some chromium floated off in wash-water (see discussion).

way is not all that could be desired, and no electrolytic method whatever has been offered. Upon electrolyzing a solution containing a mixture of chromium and aluminum sulphates, the metallic chromium will be deposited as such in the mercury, the aluminum remaining in solution. It may then be determined in the usual way. Thus both the chromium and aluminum can be weighed directly. A stock solution of aluminum sulphate was prepared, containing 0.1421 gram of alumina per each 10 cc.

The aluminum in the wash-water from depositions Nos. 1 and 2 was precipitated with ammonia and weighed as oxide, with the usual precautions.

The initial voltage and amperage are given to the left of the last column of Table II. The current strength was gradually increased as the decomposition progressed and was raised to the values, given to the right in the table, during the last hour or two. The precautions given under the Determination of Chromium were observed here. That is, the amalgam was washed rapidly with fairly anhydrous alcohol and ether, and no more than 0.2 gram of chromium was deposited in a cell containing 75 grams of mercury. Reference to Table II shows that chromium and aluminum can not only be separated when present in nearly equal quantities, but also when a preponderance of either one is present.

TABLE II.—SEPARATION OF CHROMIUM FROM ALUMINUM.

	Chromium present in grams.	Chromium found in grams.	Alumina present in grams.	Alumina found in grams.	No. of cell.	Sulphuric acid (sp. gr. 1.832) present in drops.	Conditions.				
							Time. Hours.	Amperes.	Volts.	Amperes.	Volts.
1	0.1080	0.1080	0.1421	0.1423	1	6	14	0.35	6	0.8	6.5
2	0.1080	0.1081	0.1421	0.1426	2	4	14	0.3	6	0.8	6.5
3	0.0108	0.0107	0.2842	.....	1	6	2	0.3	5.5	0.7	7
4	0.0108	0.0107	0.2842	.....	3	5	1½	0.3	5.5	0.85	7.5
5	0.2160	0.2162	0.0142	.....	1	6	24	0.6	6	1.8	7.5
6	0.2160	0.2158	0.0142	.....	1	5	14	0.4	8	1	7.5

SEPARATION OF CHROMIUM FROM BERYLLIUM.

Chromium was also separated from beryllium in an analogous manner, the sulphate of beryllium yielding nothing to the cathode of mercury. The stock solution of berryllium sulphate contained

0.0818 gram of oxide per each 10 cc., and was free from iron and other elements which would enter the mercury under the conditions existing during the electrolysis. As indicated in Table III, a wide variation in the time necessary for this separation is permissible without injury to the deposit. No deleterious effects were produced by the prolonged action of the current, even for a period of ten hours after the metal was completely deposited. This statement is not restricted to this separation alone, but applies in general to all depositions made by the author, with the mercury cathode.

TABLE III.—SEPARATION OF CHROMIUM FROM BERYLLIUM.

	Chromium present in grams.	Chromium found in grams.	Beryllium oxide present.	No of cell used.	Sulphuric acid (sp. gr. 1.832) present in drops.	Conditions.				
						Time. Hours.	Amperes.	Volts.	Amperes.	Volts.
1	0.1080	0.1079	0.0818	1	4	14	0.3	6	3.5	5
2	0.1080	0.1078	0.0818	1	4	4.5	0.3	6	3.5	5

DETERMINATION OF MOLYBDENUM.

The element molybdenum is ordinarily precipitated by the current on the cathode as the black hydrated sesquioxide from solutions of sodium molybdate acidulated with sulphuric acid. This hydrated sesquioxide must be dissolved from the platinum dish in nitric acid and evaporated carefully to dryness and weighed as molybdenum trioxide. It was found that an aqueous solution of molybdenum trioxide, acidulated with sulphuric acid, when electrolyzed with a cathode of mercury, gave up its molybdenum completely, a brilliant white amalgam being formed. It thus became possible to weigh molybdenum directly as a product of the electrolysis. Experimentation proved that a solution of sodium molybdate, acidulated with sulphuric acid, could be substituted for the aqueous solution of the trioxide, with like results. Accordingly, a stock solution of sodium molybdate was prepared containing 0.0950 gram of molybdenum per each 10 cc. Portions of this were electrolyzed with the results and the conditions given in Table IV.

All depositions which could not be completed in seven or eight hours were run during the night with a current of about 1.5 amperes. It is not advisable to use a current of more than 2 amperes in the presence of a large amount of sulphuric acid, as the latter, under the influence of the heat which is developed, seemed to exert a slight solvent action on the mercury cathode. The drops of sulphuric acid given in the table were of such size that five measured exactly 0.2 of a cubic centimeter in volume. The use of too high a current for the amount of acid present was also attended by the formation of a lower oxide, which precipitated out, and spoiled the decomposition, as it was insoluble in dilute acid.

TABLE IV.—DETERMINATION OF MOLYBDENUM.

	Molybdenum present in grams.	Molybdenum found in grams.	No. of cell used.	Sulphuric acid (sp. gr. 1.832) present in drops.	Time. Hours.	Conditions.				
						Amperes.	Volts.	Amperes.	Volts.	
1	0.0950	0.0950	3	13	14	1.2	6	1.6	6.5	(2 hrs.)
2	0.0950	0.0946	3	13	22	1.2	6	1.6	6	(2 hrs.)
3	0.1900	0.1906	2	30	18	1.6	5.5	1.4[1]	7	(4 hrs.)
4	0.1900	0.1903	2	25	20	1.6	5.5	1.4[1]	7	(4 hrs.)

SEPARATION OF MOLYBDENUM FROM VANADIUM.

A solution of sodium vanadate, acidulated with sulphuric acid, was electrolyzed; the vanadium was reduced to a brilliant blue, but no increase was observed in the weight of the mercury. A stock solution of sodium vanadate was then prepared, containing 0.1002 gram of vanadium to each 10 cc. Portions of this solution were removed by means of a pipette, as usual. The same sodium molybdate solution was used as under the Determination of Molybdenum. The conditions favorable for the separation of molybdenum from vanadium are given in Table V. As indicated in Experiments Nos. 3 and 4 in Table IV, also in Experiments Nos 3 and 4 in Table V, it was found best to neutralize a portion of the sulphuric acid present with potassium hydroxide, after all but the last traces of molybdenum had been deposited. Large amounts of the acid seemed to exert a retarding influence upon the precipitation of the last traces of molybdenum. On the other hand, the neutralization must not be carried too far, as an oxide of vanadium appeared at the anode, when insufficient acid was present. When the molybdenum was completely deposited, the solution was blue in color and not green; this served as a signal

---

[1] Some chromium floated off in wash-water (see discussion).

for the interruption of the current. The values given in Table V are for molybdenum and vanadium, and not for their oxides. An attempt was made to show that the method was applicable when 0.0095 gram of molybdenum was present with 0.2 gram of vanadium, but after many trials the idea was abandoned, as a vanadium solution of this concentration seemed to prevent the complete deposition of the molybdenum. The method would, doubtless, work with these proportions, provided that a decomposition cell of double the capacity were used.

TABLE V.--SEPARATION OF MOLYBDENUM FROM VANADIUM.

	Molybdenum present in grams.	Molybdenum found in grams.	Vanadium present in grams.	No. of cell used.	Sulphuric acid (sp. gr. 1.832) present in drops.	Time. Hours.	Conditions.				
							Amperes.	Volts.	Amperes.	Volts.	
1	0.0950	0.0950	0.1002	2	20	20	1.6	6.5	1.5	5.5	(3 hrs.)
2	0.0950	0.0940	0.1002	3	20	18	2	5	1	5	(3 hrs.)
3	0.1900	0.1895	0.0100	2	30	18	1.6	4.5	1.5[1]	6	(3 hrs.)
4	0.1900	0.1887	0.0100	2	30	20	1.4	4.5	1.2[2]	5.5	(3 hrs.)

SEPARATION OF IRON FROM THE RARE EARTHS.

Wolcott Gibbs[3] in the year 1883 first deposited iron, using a mercury cathode. The decomposition of the iron salt was complete, as the presence of that metal could not be detected in the wash-water.

Drown and McKenna[4] separated iron from aluminum and phosphoric acid. Smith[5] in his paper "On the Uses of a Mercury Cathode in Electrochemical Analysis" described methods for the separation of iron from uranium, titanium, zirconium and thorium. Following a suggestion given in the latter paper, experiments were undertaken to determine the possibility of a separation of iron from rare earths. Preliminary trials disclosed the fact that the sulphates of the earths gave nothing to the mercury, when electrolyzed under conditions suitable for the deposition of iron. Accordingly, a stock solution of ferrous sulphate was prepared,

---

[1] Neutralized with caustic potash to 15 drops of sulphuric acid and then run under final conditions for time given.

[2] Neutralized with caustic potash to 20 drops of sulphuric acid and then run under final conditions for time given.

[3] *Am. Chem. J.*, **13**, 571

[4] This Journal, **25**, 888.

[5] *J. Anal Chem.*, **5**, 627.

one drop of sulphuric acid (sp. gr. 1.832) being added for every 10 cc. of the solution. The average of five volumetric determinations of the iron content of 10 cc. of this solution gave 0.1056 gram of metallic iron. The following results were obtained by electrolysis, using the mercury cathode: 0.1057 gram, 0.1055 gram, 0.1058 gram, 0.1057 gram, 0.1055 gram, 0.1056 gram and 0.1055 gram of iron. The wash-waters gave no tests for iron with sodium sulphide. The deposition of iron with a mercury cathode presented no difficulty whatever. The various cells, which have been previously described, were used interchangeably in the following separations. The dilution, therefore, was not an important factor. Wide variations in the current density produced no marked effect, except in the time necessary for the deposition. The iron amalgam washed easily and showed no such tendency to throw off metal in a fine state of division, as was observed with the chromium amalgam. After weighing a deposit of iron it was unnecessary to replace the amalgam in the cell with pure mercury before starting a new deposition. as the same mercury can be used repeatedly for successive depositions.

The earths available for this work were cerium, lanthanum, praseodymium, neodymium and yttrium, most of which were prepared by Shapleigh of the Welsbach Company. Stock solutions of the sulphates were prepared, and sulphuric acid added where necessary to prevent the formation of basic salts.

A glance at the following tables will show that satisfactory separations of iron from each of the earths were obtained. As no difficulties were encountered, and as the tables express all that is necessary for the successful application of the method, no further discussion is necessary.

TABLE VI.—SEPARATION OF IRON FROM CERIUM.

	Iron present in grams.	Iron found in grams.	Cerium dioxide present in grams.	Sulphuric acid (sp. gr. 1.832) present in drops.	Time. Hours.	Conditions.			
						Amperes.	Volts.	Amperes.	Volts.
1	0.1056	0.1055	0.0635	8	14	0.8	6	1	5.5
2	0.1056	0.1058	0.0635	8	14	0.8	6	1	5.5
3	0.0105	0.0103	0.1905	20	6	0.6	4.5	1	5
4	0.0105	0.0106	0.1905	20	6	0.6	4.5	1	5
5	0.2112	0.2112	0.0064	3	14	0.4	8	0.8	6
6	0.2112	0.2113	0.0064	3	14	0.5	8	1	7

TABLE VII.—SEPARATION OF IRON FROM LANTHANUM.

	Iron present in grams.	Iron found in grams.	Sesquioxide of lanthanum present in grams.	Sulphuric acid (sp. gr. 1.832) present in grams.	Time. Hours.	Conditions.			
						Amperes.	Volts.	Amperes.	Volts.
1	0.1056	0.1057	0.0432	5	6	0.6	6.5	0.8	7
2	0.1056	0.1053	0.0864	2	14	0.4	8	0.8	8
3	0.0105	0.0103	0.1728	6	14	0.4	7.5	0.6	9
4	0.0105	0.0101	0.1728	7	14	0.4	7.5	0.6	9
5	0.2112	0.2116	0.0129	3	14	0.4	6	1.2	8
6	0.2112	0.2112	0.0129	3	14	0.4	6	1.4	8

TABLE VIII.—SEPARATION OF IRON FROM PRASEODYMIUM.

	Iron present in grams.	Iron found in grams.	Oxide of praseodymium present in grams.	Sulphuric acid (sp. gr. 1.832) present in drops.	Time. Hours.	Conditions.			
						Amperes.	Volts.	Amperes.	Volts.
1	0.1056	0.1055	0.1200	5	5½	0.5	6.5	1	7
2	0.1056	0.1056	0.1200	5	14	0.5	6	1.2	9.5
3	0.0105	0.0104	0.2400	7	5	0.6	6	0.8	7
4	0.0105	0.0105	0.2400	6	5	0.6	7	0.8	7.5
5	0.2112	0.2113	0.0160	4	14	0.4	6	1.2	8
6	0.2112	0.2104	0.0160	4	14	0.4	6	1.2	8

TABLE IX.—SEPARATION OF IRON FROM NEODYMIUM.

	Iron present in grams.	Iron found in grams.	Oxide of neodymium present in grams.	Sulphuric acid (sp. gr. 1.832) present in drops.	Time. Hours.	Conditions.			
						Amperes.	Volts.	Amperes.	Volts.
1	0.1056	0.1056	0.1089	2	14	0.5	6	1	8
2	0.1056	0.1056	0.1089	2	14	0.5	6	1	8
3	0.0105	0.0102	0.2178	4	4½	0.6	9	0.8	9
4	0.0105	0.0102	0.2178	4	14	0.5	7	0.9	8
5	0.2112	0.2108	0.0145	3	14	0.4	6.5	1.2	7
6	0.2112	0.2112	0.0145	3	14	0.4	6.5	1.2	7

TABLE X.—SEPARATION OF IRON FROM YTTRIUM.

	Iron present in grams.	Iron found in grams.	Yttria present in grams.	Sulphuric acid (sp. gr. 1.832) present in drops.	Time. Hours.	Conditions.			
						Amperes.	Volts.	Amperes.	Volts.
1	0.1056	0.1054	0.0320	6	14	0.5	6	0.8	7
2	0.1056	0.1056	0.0320	6	6	0.6	6.5	0.8	7
3	0.0105	0.0105	0.1280	10	6	0.6	6	0.8	6
4	0.0105	0.0104	0.2080	12	4½	0.8	6	1.2	7
5	0.2112	0.2112	0.0032	3	14	0.4	7	1	6
6	0.2112	0.2111	0.0032	3	14	0.4	7	1	6

SEPARATION OF IRON FROM VANADIUM.

It was quite probable that a separation of iron from vanadium could be obtained.

The results given in Table XI show this to be the case. The same difficulty was encountered here as under the separation of molybdenum from vanadium, when 0.2 gram of the latter was present. Just as 0.2 gram of vanadium prevented the complete deposition of a small amount of molybdenum, so here it prevented the complete deposition of 0.0105 gram of iron. As suggested under molybdenum, the use of a larger cell or the division of the solution will overcome the difficulty.

TABLE XI.—SEPARATION OF IRON FROM VANADIUM.

	Iron present in grams.	Iron found in grams.	Vanadium present in grams.	Sulphuric acid (sp. gr. 1.832) present in drops.	Time. Hours.	Conditions.			
						Amperes.	Volts.	Amperes.	Volts.
1	0.1056	0.1054	0.1002	12	17	0.4	7	1	8.5
2	0.1056	0.1051	0.1002	13	14	0.6	7	1	9
3	0.2112	0.2113	0.0200	5	14	0.3	7	1	7.5
4	0.2112	0.2112	0.0200	5	14	0.4	7	1	7

SEPARATION OF IRON FROM BERYLLIUM.

From the fact that chromium can be separated from beryllium there was every reason to suppose that iron could be separated from beryllium also. As shown in Table XII, the results were highly satisfactory. For the purpose of further demonstrating that the element remaining in the wash-water was in a suitable condition to be estimated, ammonia was added in each case and the beryllium hydroxide ignited to oxide and weighed. The results given are not selected results, but are consecutive determinations.

TABLE XII.—SEPARATION OF IRON FROM BERYLLIUM.

Iron present in grams.	Iron found in grams.	Beryllium oxide present in grams.	Beryllium oxide found in grams.	Sulphuric acid (sp. gr. 1.832) present in drops.	Time. Hours.	Conditions.			
						Amperes.	Volts.	Amperes.	Volts.
0.1056	0.1057	0.0818	0.0821	2	7	0.5	7	0.8	6.5
0.1056	0.1059	0.0818	0.0820	2	14	0.5	7	0.8	6.5
0.0105	0.0105	0.1636	0.1633	2	4½	0.6	8	0.8	8
0.0210	0.0208	0.1636	0.1630	2	14	0.6	8	0.8	8
0.2112	0.2113	0.0082	0.0082	2	14	0.4	6.5	1.2	7
0.2112	0.2112	0.0082	0.0083	2	14	0.4	6.5	1.2	7

SEPARATION OF IRON FROM ALUMINUM.

Drown and McKenna's separation of iron from aluminum was confirmed by a number of experiments. In the opinion of the writer it is the best separation of these metals extant.

NEW VOLUMETRIC METHOD FOR THE DETERMINATION OF LEAD.

BY ERIC JOHN ERICSON.

Received July 7, 1904.

LAST year Messrs. Walters and Affelder described their scheme[1] for the analysis of bronzes and bearing metals, with particular attention to a new volumetric method for lead. While trying that method the writer encountered the same difficulty as with Low's iodometric method for copper, *viz.*, uncertainty of end-reaction and, besides, a tendency to too low results.

However, the idea to oxidize lead to peroxide by means of ammonium persulphate in an alkaline solution is excellent and is dhered to in this method up to the point of filtering, which takes lace without acidifying, thus removing iron along with the lead, washing with dilute ammonia (1 : 5) until the blue color, due to opper, disappears from the filter and finally four or five times

---

[1] This Journal, **25**, 632.