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 copper, disappears from the filter and finally four or five times

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

with hot water. Chlorides and sulphates are not permissible, excepting sulphates resulting from the decomposition of the persulphate. Manganese interferes, of course. A small amount of iron is harmless, but a large quantity would hinder complete oxidation.

Having obtained the lead peroxide, it occurred to the writer to endeavor to determine the lead in a manner somewhat analogous to Julian's[1] method for manganese, *viz.*, by dissolving in a measured excess of acidulated hydrogen peroxide and titrating the excess with potassium permanganate, according to the following equations:

(*a*) $PbO_2 + H_2O_2 + 2HNO_3 = Pb(NO_3)_2 + 2H_2O + O_2$.

(b) $5H_2O_2 + 2KMnO_4 + 6HNO_3 =$

$$2KNO_3 + 2Mn(NO_3)_2 + 8H_2O + 5O_2;$$

hence $2KMnO_4 = 5H_2O_2 = 5Pb$.

As the theoretical factor, $\frac{5Pb}{10Fe} = 1.851$, gives too low results, the empirical factor, 1.92, has been chosen, giving results agreeing closely with the standard gravimetric method, as follows:

High lead brass.

Gravimetric as sulphate. Per cent lead.	New volumetric. method.	
9.80	9.76	9.85
9.88	9.80	
....	9.87	
....	9.98	

To test further the accuracy of the method, a standard solution of lead nitrate was made up by dissolving 2 grams dried normal lead carbonate in nitric acid and diluting to a liter. The lead carbonate contained 76.93 per cent. lead, against 77.52 theoretically. This is the average of three determinations as sulphate, which happens to check exactly with the calculated percentage of lead from expelling the carbon dioxide by ignition (16.35 per cent.).

Standard lead nitrate taken. cc. Gram lead.	Found. Gram lead.
5 = 0.00769	0.00768
10 = 0.01539	0.01574
50 = 0.07693	0.07642
100 = 0.15386	0.15360
	0.25344
0.25387 = 99.83 per cent.	

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[1] This Journal, 15, 113.

The method is less suitable to high percentages of lead, owing o the tendency of the peroxide to go through the filter, thus oftenimes necessitating refiltration. It will also be advantageous to tandardize the potassium permanganate solution against standard ead nitrate rather than calculate the lead factor from the iron alue.

SOLUTIONS REQUIRED.

(1) *Hydrogen Peroxide.*—Suitable strength for low percentges of lead: 900 cc. distilled water; 50 cc. nitric acid, concenrated; 15 cc. hydrogen peroxide, U. S. P.

(2) *Standard Potassium Permanganate Solution.*—Dissolve .139 grams (theoretically 1.1324) to every liter of water and tandardize against pure iron wire or lead solution, as suggested bove. One cc. equals 0.00200 gram iron = 0.00384 lead. This s also a convenient strength for titrating small amounts of iron, nanganese and lime.

If the solution were to be employed for lead exclusively, a trength of 0.005 gram lead per cubic centimeter would be preerred.

Applying the method to brasses and bronzes the scheme is as ollows: Dissolve 1 gram drillings in 15 cc. nitric acid, sp. gr. .28 (2 parts acid of sp. gr. 1.42 to 1 of water), and boil slowly ill about 6 cc. remain, then add 100 cc. water, boil and allow to ettle. Filter on double 9 cm. Munktell's filter, No. 0, and wash vith 2 per cent. nitric acid solution, dry, ignite and weigh the tin lioxide as usual. If phosphorus is present, determine the amount nd make correction.

To the filtrate from tin add 25 cc. strong ammonia and gradally 3 to 4 grams of solid ammonium persulphate (Merck's) and oil for five minutes; allow to settle and filter on a double 9 cm. ilter with pulp and using suction. Wash first with dilute amnonia (1 part to 5 of water) to remove copper and zinc from the ilter, then with hot water four or five times. Transfer the filter vith precipitate to the beaker in which precipitation was made, nd add, with a pipette, 25 cc. hydrogen peroxide solution, stir vith a glass rod and bring the solution in contact with any lead eroxide adhering to the side of the beaker. When all or nearly ll dark particles disappear, add 20 cc. nitric acid (sp. gr. 1.28) nd 150 cc. water. Stir again, and when all is dissolved titrate with standard potassium permanganate solution until pink color appears. For instance:

	cc.	
If 25 cc. hydrogen peroxide blank require	18.5	standard pot. permang.
And sample requires	10.2	
Difference	8.3	
Then 8.3 × 0.002 × 1.92 × 100 = ...	3.19	per cent lead.

As mentioned above, iron is precipitated along with the lead but does not interfere. It can be determined by filtering off the suspended filter-paper after the lead titration, adding 10 cc. concentrated hydrochloric acid, precipitating with ammonia, redissolving in dilute hydrochloric acid and determined volumetrically or, if in exceedingly small amounts, colorimetrically, after v. Kéler and Lunge,[1] modified.

The ammoniacal filtrate from lead and iron is neutralized with dilute sulphuric acid with an excess of only a few cubic centimeters, heated to boiling and the copper precipitated with sodium thiosulphate; 30 to 40 cc. of a 20 per cent. solution generally suffices. Filter on an 11 cm. filter and wash with hot water. Redissolve the cuprous sulphide in dilute nitric acid (sp. gr. 1.20) filter and determine copper electrolytically; or dilute to 100 cc. pipette off 25 cc., neutralize nearly with ammonia and precipitate as cuprous sulphocyanide, according to Parr's method.[2] Use N/ potassium permanganate solution. This is the best volumetric method for copper that the writer has tried. Using Parr's factor however, the results were somewhat lower than the electrolytic but on applying the latest atomic weight for iron,[3] *viz.*,[4] 55.8 in calculating the copper factor, the results were very satisfactory Thus one obtains the copper factor by multiplying the iron value of the permanganate by 0.1626, against 0.1602, recommended by Parr.

To the filtrate from the cuprous sulphide is added 15 cc. concentrated hydrochloric acid, the solution is boiled until sulphur has separated out, filtered and the zinc is titrated, while the solution is warm, with standard potassium ferrocyanide, using uranium nitrate as indicator.

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[1] *Zeitschr. angew. Chem.*, 1894, 669.

[2] This Journal, **22**, 685, and **24**, 580.

[3] Küster's "Logarithmische Rechentafeln für Chemiker," 1902.

[4] This Journal, **26**, 239.

To apply the method to lead sulphide ores it will be necessary to convert into the carbonate,[1] dissolve in dilute nitric acid and peroxidize as directed above.

To our chief chemist, Mr. William Brady, the writer is indebted for valuable suggestions and for permission to publish this method.

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THE PHOTOMETRIC DETERMINATION OF SULPHUR IN COAL.

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Received July 20, 1904.

THE use of sodium peroxide as an oxidizing agent for the sulphur of coal has received considerable attention, but the violence of the reaction has brought disfavor upon the method. However, by means of a closed bomb, as in the Parr calorimeter, there has been fully demonstrated the practicability of using sodium peroxide for this purpose. Indeed, over two years ago Mr. Milton Hersey, of Montreal, Canada, in a communication to the author, reported the very satisfactory use of the residues from the calorimetric process for gravimetrically determining the sulphur. Later articles by Sundstrom[2] and by von Konek[3] have advocated the same method.

Coupling the sodium peroxide method of arriving at a combustion with the photometric method proposed by Mr. Hinds,[4] there seem to be possibilities well worth investigating.

The work with the photometer, however, either as outlined by Mr. Hinds, or as elaborated by Mr. Jackson,[5] was not found satisfactory. A careful study was made of the variable elements that entered into the method. The method prescribed a candle of standard power, maintained at a definite distance from the bottom of the graduated tube in which was read the depth of liquid through which the outline of the candle flame could be seen. It was soon found that the strength of the light had little to do with the matter. A stronger light would illuminate the

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[1] Furman's "Manual of Practical Assaying," page 139.

[2] This Journal, **25**, 184.

[3] *Ztschr. angew. Chem.*, 1903, p. 517.

[4] This Journal, **23**, 269.

[5] *Ibid.*, **23**, 799.