tions under which the two were separated with ease in the presence of sulphuric acid.

Copper present	0.0745 gram.
Sodium tellurate present	0.2000 gram.
Concentrated sulphuric acid	1 cc.
Dilution	150 cc.
$N.D_{100}$	0.05 to 0.1 ampere.
Pressure	2 to 2.25 volts.
Temperature	65° C.
Time	6 hours.
Copper found	(*a*) 0.0747 gram. (*b*) 0.0748 gram.

The writer has had the assistance of Mr. S. H. Ross in working out the details of these experiments, for which he would here acknowledge his indebtedness.

UNIVERSITY OF PENNSYLVANIA.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, NO. 72.]

THE RAPID PRECIPITATION OF METALS IN THE ELECTROLYTIC WAY.[1]

BY FRANZ F. EXNER.

Received July 5, 1903.

SEVERAL years ago experiments were made in this laboratory looking to the electrolytic separation of molybdenum from tungsten. The anode was made to rotate by means of a small electric motor,[2] the idea being to use high currents with a high speed of rotation of the anode, thus hastening the deposition and, by agitation of the electrolyte, overcoming certain disturbances which had arisen. In the particular case referred to, the results were not entirely satisfactory. The idea, however, was shortly after tried upon other metals with success and led to the present investigation. Klobukow[3] called attention to the agitation of the electrolyte, but never published any results. Gooch and Medway[4] recently described successful experiments made with copper, nickel and silver solutions by *rotating the cathode*.

The purpose of the present investigation was to note the results

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

[2] Smith's "Electrochemical Analysis," p. 77, third edition, 1902.

[3] *J. prakt. Chem.*, **33**, 475 : **40**, 121.

[4] *Am. J. Sci.* [4], **15**, 320.

following the *rapid rotation of the anode* and to develop with care the conditions for successful work, in various electrolytes,[1] for a number of metals. The result has been that many of them can now be precipitated quantitatively by the current in such astonishingly short periods that the methods of electrolysis are made more available to the analyst than heretofore. The essentials are recorded in the paragraphs which follow and the reader is referred to the author's thesis for greater details.

An ordinary platinum dish was used as cathode. The anode was a spiral of heavy platinum wire. It was 2 inches in diameter. Its center was depressed to give it the form of a shallow bowl. It was usually made to perform from 500 to 600 revolutions per minute. At higher speeds, the electrolyte is thrown against the cover-glasses; this is not objectionable, unless the dish contains too much liquid. The solution may sweep around the edge of the dish under the cover-glasses throughout the entire operation without any loss. The volume of liquid never exceeded 125 cc. and was not less than 110 cc. In every case the electrolyte was heated almost to boiling before exposing it to the action of the current. No external heat was applied during the electrolysis. The high currents kept the liquid hot. When the decomposition was finished, the rotator was stopped and the current was reduced by the introduction of resistance, water being added to cover the exposed metal, thus preventing its oxidation. The liquid in the dish was siphoned off, the dish being kept full by the addition of water from a wash-bottle. The metallic deposit was washed and dried in the customary way.

EXPERIMENTAL PART.

Copper.—This metal was precipitated from these solutions: A, sulphuric acid; B, nitric acid; C, ammonium sulphate and ammonia; D, ammonium nitrate and ammonia; E, potassium cyanide. The results were as follows:

A

No. of exp.	$CuSO_4 = Cu$. Gram.	H_2SO_4 dil. 1 : 10. cc.	Current $N.D_{100} = A$.	V.	Time. Minutes.	Deposited Cu. Gram.	Error. mg.
1	0.4994	1	3	15	3[2]	0.4992	0.2
			4	15	2		
			5	14-9	5		
2	"	1	5	"	5	0.4994	"
3	"	1	5	"	5	0.4994	"

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[1] Consult Smith's "Electrochemical Analysis," 3rd edition, 1902.

[2] The brace indicates that all those current conditions were used in the experiment acording to the time given.

B.

No. of exp.	$CuSO_4$ = Cu. Gram.	Conc. HNO_3. cc.	Conc. NH_4OH sol. cc. After 10 min.	Current N.D_{100} = A.	V.	Time. Minutes.	Deposited Cu. Grams.	Error. mg.
1	0.2484	1.0	....	5	10	10	0.2385	−9.9
2	0.2484	1.0	....	5	10	15	0.2387	−9.7
3	0.2484	1.0	....	5	10	20	0.2393	−9.1
4	0.2484	0.5	 5	5 3	12 18	10 5	0.2482	—0.2
5	0.2484	1.0	 5	5 5	10 12	10 5	0.2483	—0.1
6	Identical with 5.							
7	0.4968	1.0	 5	5 5	8 10	10 5	0.4964	—0.4

In Experiments 4 to 7, the electrolysis was first conducted for ten minutes in a nitric acid solution and then 5 cc. of concentrated ammonia water were added. This caused a change in the resistance of the solution, especially when only 0.5 cc. of nitric acid was used. The change was less marked when 1 cc. of the nitric acid was added, and since it also gave better results, that quantity is to be preferred. The deposit from this electrolyte was very satisfactory. It was dense, bright, and metallic in appearance.

C.

No. of exp.	$CuSO_4$ = Cu. Gram.	$(NH_4)_2SO_4$. Grams.	Conc. NH_4OH. cc.	Current N.D_{100} = A.	V.	Time. Minutes.	Deposited Cu. Gram.	Error. mg.
1	0.2484	3	20	5	8	5	0.2488	+0.4
2	0.2484	1	20	5	12	5	0.2488	+0.4
3	0.4968	1.5	30	5	12	8	0.4966	—0.2

The conditions of 2 or 3 are preferable. The deposit is adherent. This electrolyte will be preferred, because in it the least time is required for complete precipitation.

D.

No. of exp.	$CuSO_4$ = Cu. Gram.	NH_4NO_3. Grams.	Conc. NH_4OH solution. cc.	Current N.D_{100} = A.	V.	Time. Minutes.	Deposited Cu. Gram.	Error. mg.
1	0.2484	3.0	20	5	8	10	0.2463	—2.1
2	0.2484	1.0	20	5	12	10	0.2483	—0.1
3	0.4968	1.5	30	5	12	15	0.4968	0.0
4	0.4968	1.5	30	5	12	14	0.4964	—0.4

The conditions of Experiments 2 and 3 are the most desirable. The deposits were excellent in character. A great advantage of this solution is, that in the ordinary course of analysis copper is usually obtained as nitrate. These conditions were made use of in the separation of copper from nickel.

E.

No. of exp.	$CuSO_4$ = Cu. Gram.	KCN.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Cu. Gram.	Error. mg.
1	0.2484	Least possible for sol.	5	12	10	0.2136	—34.8
2	0.2484	"	6	18	10	0.2465	— 1.9
3	0.2484	"	6	18	18	0.2479	— 0.5
4	0.2484	"	6	18	18	0.2478	— 0.6

The electrolyte yielded its last traces of copper very slowly, which was probably due to the solvent action of the solution upon the deposit. The deposits were of a golden red color and shone as if they had been polished.

NICKEL.

The solutions used in the following experiments were: A, ammonium sulphate and ammonia; B, acetic acid and ammonia; C, ammonium nitrate and ammonia; D, potassium cyanide.

A.

No. of exp.	$NiSO_4$ = Ni. Gram.	$(NH_4)_2SO_4$. Grams.	Conc. NH_4OH. cc.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Ni. Gram.	Error. mg.
1	0.2498	1.5	20	4	12	15	0.2496	—0.2
2	0.4996	1.5	30	5	10	15	0.4977	—1.9
3	0.4996	1.5	25	5	10	17	0.4998	+0.2

The conditions in 1 and 3 are the most desirable. The deposits were gray in color, dense, adherent and thoroughly satisfactory.

B.

No. of exp.	$NiSO_4$ = Ni. Gram.	30 % sol. $HC_2H_3O_2$ cc.	Conc. NH_4OH. cc.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Ni. Gram.	Error. mg.
1	0.2498	10	10	5	8.0	10	....	....
2	0.2498	10	25	5	6.5	10	0.2498	....

The conditions in 2 were very satisfactory. The deposits were much like those from electrolyte A.

An experiment was performed in which 3 grams of ammonium nitrate were substituted for the acetic acid in Experiment B, 2; but after ten minutes no deposit had been formed. This suggested a possible separation from copper, which was worked out later. The experiments made with potassium cyanide were abandoned.

ZINC.

A solution of the sulphate was used. The dishes in which the precipitations were made were coated with silver. The electrolytes were: A, sodium acetate and acetic acid; B, sodium hydroxide; C, sodium hydroxide and potassium cyanide.

A.

No. of exp.	$ZnSO_4$ = Zn Gram.	$NaC_2H_3O_2$ Grams.	$HC_2H_3O_2$ 30 per cent. solution. cc.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Zn. Gram.	Error. mg.
1	0.2502	1	0.2	3	18	10	0.2502	...
2	0.2502	3	0.2	4	15	10	0.2502	—0.2
3	0.5004	3	0.2	4	18	15	0.5001	—0.3
4	0.5004	3	0.2	4	10	15	0.5002	—0.2
5	0.5004	1	0.2	4	19	15	0.5000	—0.4

Experiments 1 and 5 show that 1 gram of sodium acetate requires too high a voltage to overcome the resistance, causing an undue amount of heat. The high voltage in Experiment 3 was due to the great speed of rotation. The latter was reduced to 600 revolutions per minute in Experiment 4. The conditions in Experiment 2 and 4 are to be preferred, and gave very satisfactory results. The deposits were light gray in color, and crystalline.

B.

No. of exp.	$ZnSO_4$ = Zn. Gram.	NaOH. Grams.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Zn. Gram.	Error. mg.
1	0.5004	7	5	5	15	0.5003	—0.1
2	0.5004	5	5	6	15	0.5003	—0.1
3	0.5004	8	5	5	15	0.5004	...
4	0.5004	12	5	5	15	0.5006	+0.2

Nothing is gained by the extra sodium hydroxide in Experiment 4. The conditions of Experiment 3 were most satisfactory. The deposit from this electrolyte was sufficiently adherent for washing and weighing. C. Two experiments were made in which the zinc sulphate solution was precipitated by sodium hydroxide, the precipitate redissolved in potassium cyanide, and electrolyzed in the usual way, but it was finally found necessary to abandon this electrolyte.

SILVER.

The only electrolyte tried was the cyanide. A speed of 700 revolutions per minute was found to be most satisfactory.

No. of exp.	$AgNO_3$ = Ag. Gram.	KCN. Grams.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Ag. Gram.	Error. mg.
1	0.2495	2	2	5	7	0.2492	+0.2
2	0.4990	2	2	5	10	0.4990	. .
3	0.4990	2	2	5	9	0.4992	+0.2

The silver came down as a dense white deposit, excepting the last traces, which were a little loose, but quite adherent.

BISMUTH.

This metal proved most troublesome. The speed of rotation employed varied from 700 to 900 revolutions per minute. Electrolyte: 0.5510 gram of bismuth dissolved in a small excess of nitric acid and electrolyzed with a current $N.D_{100} = 1$ A. $V = 2.5$. Time = 18 minutes. The deposit of bismuth weighed 0.5507 gram. It was white and crystalline at first, but later it became black and loose, yet sufficiently adherent for washing and weighing.

Ten different electrolytes were tested with current strengths varying from 4 to 5 amperes, but all gave more unsatisfactory results than when nitric acid alone was present. The only electrolyte that gave deposits sufficiently satisfactory for analysis was that containing a small excess of nitric acid.

The suggestion of Vortmann was then applied, and the bismuth deposited with mercury as an amalgam. Solutions of mercurous nitrate and bismuth nitrate, each dissolved in the smallest excess of nitric acid, were used.

No. of exp.	$Bi(NO_3)_3$ = Bi. Gram.	$HgNO_3$ = Hg. Gram.	Conc. HNO_3. cc.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Bi. Gram.	Error. mg.
1	0.2970	0.2920	..	4	12	15	0.2965	—0.5
2	0.2970	0.2920	..	4	12	12	0.2967	—0.3
3	0.2970	0.2920	1	5	8.5	8	0.2972	+0.2
4	0.2970	0.2920	1	5	8.5	8	0.2971	+0.1

The 1 cc. of nitric acid lowered the voltage and better results followed. The conditions of Experiments 3 and 4 are to be preferred. The deposit was iron-gray in color and very satisfactory.

MERCURY.

A solution of mercurous nitrate containing nitric acid was used. The speed of rotation was 700 revolutions per minute.

No. of exp.	$HgNO_3$ = Hg. Gram.	Conc. HNO. cc.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Hg. Gram.	Error. mg.
1	0.2920	1	4	11	15	0.2920	..
2	0.2920	1	7	12	5	0.2919	—0.1
3	0.5840	1	7	12	7	0.5837	—0.3

The deposits were perfect mirrors of pure mercury, and dissolved off the dish with great ease. The loss in weight of the dish in these three experiments was only 0.6 milligram.

COBALT.

A solution of cobalt sulphate was used. The speed of rotation was 700 revolutions per minute. The electrolytes were: A, ammonium sulphate and ammonia; B, sodium and ammonium acetates and ammonia.

A.

No. of exp.	$CoSO_4$ = Co. Gram.	$(NH_4)_2SO_4$. Grams.	Conc. NH_4OH sol. cc.	Current. N.D_{100} = A.	V.	Time. Minutes.	Deposited Co. Gram.	Error. mg.
1	0.2460	3	50	5	10	15	0.2433	—2.7
2	0.2460	3	50	5	10	17	0.2440	—2.0

The solution from Experiment 2 gave no test for cobalt with ammonium sulphocarbonate, but there was a small deposit upon the anode. Experiments to remedy the defects of this electrolyte were not carried out.

B.

No. of experiment.	$CoSO_4$ = Co. Gram.	30 per cent. solution $HC_2H_3O_2$ cc.	$NaC_2H_3O_2$. Grams.	Concentrated NH_4OH. cc.	Current N.D_{100} = A.	V.	Time. Minutes.	Deposited Co. Gram.	Error. mg.
1	0.2460	10	.	60	5	8	16	0.2404	5.6
2	0.2460	5	5	60	5	12 to 8	15	0.2462	+0.2
3	0.2460	.	2	60	5	15 to 12	15	0.2462	+0.2
4	0.2460	5	1	50	5	12 to 9	17	0.2459	—0.1
5	0.2460	5	1	50	5	10 to 7	15	0.2459	—0.1
6	0.4920	5	1	50	5	9 to 8	22	0.4916	—0.4
7	0.4920	5	1	50	5	9 to 8	22	0.4917	0—0.3

In Experiment 1 the deposit was excellent, but there was oxide on the anode.

The deposit in Experiment 2 was very good, but there was a slight discoloration of the anode.

The deposit of Experiment 3 was satisfactory. The anode was not coated.

The deposits in Experiments 4 to 7 were good. They were gray in color and quite metallic in appearance. The discoloration of the anode was so slight as to be negligible.

In the last three experiments, the speed was 500 revolutions per minute. These conditions are recommended. Sodium acetate hastens the deposit and prevents deposition upon the anode, while the ammonium acetate improves the deposit, but retards its formation.

CADMIUM.

The following electrolytes were used: A, sulphuric acid; B, sodium acetate and potassium sulphate; C, potassium cyanide. The speed of rotation was about 600 revolutions per minute.

A.

No. of exp.	$CdSO_4$ = Cd. Gram.	H_2SO_4 dil. 1 : 10. cc.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Cd Gram.	Error. mg.
1	0.2756	3	5	9	10	0.2756	....
2	0.2756	1	5	17–11	10	0.2755	—0.1
3	0.5512	1	5	8	15	0.5508	—0.4

Deposit 1 was crystalline at first, but the last portions of metal came down loose, but sufficiently adherent. Deposits 2 and 3 were beautifully crystalline and very satisfactory.

B, *Experiment 1.*—Electrolyte: Cd = 0,5512 gram. Sodium acetate = 2 grams. Potassium sulphate = 0.5 gram. Current $N.D_{100}$ = 5 A. V = 12. Time $\doteq$ 15 minutes. The deposited cadmium weighed 0.5514 gram. The potassium sulphate was added to increase the conductivity of the electrolyte.

Experiment 2.—Electrolyte: Cd = 0.5512 gram. It was precipitated with 0.5 gram of sodium hydroxide, the precipitate was dissolved with acetic acid, and 0.5 gram of potassium sulphate added. Current $N.D_{100}$ = 5 A. V = 11. Time = 15 minutes. Deposited Cd = 0.5514 gram.

C.

No. of exp.	$CdSO_4$ = Cd. Gram.	NaOH. Grams.	KCN. Grams.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Cd. Gram.	Error. mg.
1	0.5530	5	2	5	8	15	0.5531	+0.1
2	0.5530	5	2	5	8	10	0.5528	—0.2
3	0.5530	5	2	5	8	12	0.5528	—0.2
4	0.5530	5	2	5	8	10	0.5530	....

The deposits were white, compact and very satisfactory.

IRON.

A solution of ferric ammonium sulphate with ammonium oxalate was used as electrolyte. The speed of rotation was about 800 revolutions per minute.

No. of exp.	Ferric am. alum = Fe. Gram.	$(NH_4)_2C_2O_4$. Grams.	$H_2C_2O_4$ sat. sol. cc.	Current. $N.D_{100}$=A.	V.	Time. Minutes.	Deposited Fe. Gram.	Error. mg.
1	0.2461	7	1	7	7.4	25	0.2458	—0.3
2	0.2461	7.5	·	7	7.5	25	0.2463	+0.2
3	0.2461	7.5	·	7	7.5	25	0.2461	..
4	0.4922	7.5	·	7	7.5	35	0.4922	..

The deposits were so dense and light in color that they resembled the polished platinum dish.

LEAD.

The metal was deposited as dioxide upon a dish which had its inner surface roughened by a sand blast. Over a gram of deposited dioxide showed no tendency to scale off. The speed of rotation was about 600 revolutions per minute. The only electrolyte used was nitric acid.

No. of exp.	$Pb(NO_3)_2$ = Pb. Gram.	HNO_3 conc. cc.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited PbO_2. Grams.	Error. mg.
1	0.2807	20	10	4.5	10	0.2804	—0.3
2	0.2807	20	10	4.5	10	0.2804	—0.3
3	0.5614	20	10	4.5	13	0.5613	—0.1
4	0.5614	20	10	4.5	13	0.5614	....
5	1.1228	20	10	4.5	15	1.1228	....

The deposits of lead dioxide were of a uniform, velvety black color.

No success was had with manganese.

MOLYBDENUM.

Electrolyte: A solution of sodium molybdate containing 0.2250 gram of molybdenum trioxide was placed in the dish, and to this were added 2 cc. of sulphuric acid (dilute 1 : 10), and 1 gram of potassium sulphate. The current $N.D_{100} = 4$ A. $V = 15$. Time = 20 minutes. The molybdenum trioxide found weighed 0.2246 gram. The speed of the anode equaled 300 to 400 revolutions per minute.

TIN.

The electrolyte in this instance was a freshly prepared solution of stannous ammonium chloride, to which ammonium oxalate was added. The speed of the anode equaled 300 revolutions per minute.

No. of exp.	$SnCl_2.2NH_4Cl$ = Sn. Gram.	$(NH_4)_2C_2O_4$ hot sat. sol. cc.	Current. $N.D_{100}$=A.	V.	Time. Minutes.	Deposited Sn. Grams.	Error. mg.
1	0.5396	100	5	5	13	0.5392	—0.4
2	0.2193	100	5	5.5	15	0.2193	....
3	0.2193	100	5	5.5	15	0.2191	—0.2
4	0.2193	100	5	5.5	15	0.2191	—0.2
5	0.2193	100	5	5.5	15	0.2193	....
6	0.4355	100	5 8	5.5 6.5	15[1] 3	0.4353	—0.2
7	0.4355	100	5 8	5.5 6.5	15 5	0.4355	....
8	1.0800	100	5	4.5	20	1.0801	+0.1

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[1] In Experiments 6 and 7 the brace indicates two current conditions. The 5-A current running 15 minutes after which it was changed to 8-A.

GOLD.

The electrolyte was a solution of gold chloride to which potassium cyanide was added. The speed of rotation was about 500 revolutions per minute.

No. of exp.	$AuCl_3$ = Au. Gram.	KCN. Grams.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Au. Gram.	Error. mg.
1	0.0290	1.0	5	11	10	0.0289	0.1
2	0.0725	2.0	5	11	10	0.0725	...
3	0.1450	1.5	5	11	7	0.1447	—0.3

The deposits were excellent.

ANTIMONY.

Antimony chloride, with just enough hydrochloric acid to keep it in solution, was mixed with sodium hydroxide, sodium hydrosulphide and potassium cyanide, then electrolyzed as indicated below. The speed of rotation of the anode was 400 to 500 revolutions per minute.

No. of exp.	$SbCl_3$ + HCl = Sb Gram.	NaOH 10 % sol. cc.	NaHS. cc.	KCN. Grams.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Sb. Gram.	Error. mg.
1	0.3042	30	20	2	5	4.5	20	0.3042	...
2	0.3042	30	20	2	5	4.5	20	0.3041	—0.1
3	0.3042	30	20	2	5	4.5	18	0.3041	—0.1

The deposits under these conditions were excellent. They showed a dense, light, metallic appearance. This is a new electrolyte for antimony.

No success was had with the deposition of arsenic.

SEPARATIONS.

SEPARATION OF COPPER FROM NICKEL.

The sulphates of the metals were used. The speed of rotation was about 600 revolutions per minute.

No. of experiment.	$CuSO_4$ = Cu. Gram.	$NiSO_4$ = Ni. Gram.	Concentrated HNO_3. cc.	NH_4NO_3. Grams.	Current $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Cu. Gram.	Error. mg.
1	0.2457	0.25	0.25	3	4	5	15	0.2460	+0.3
2	0.2457	0.25	0.25	3	4	5	15	0.2458	+0.1

The deposits of copper were good and free from nickel. Several other experiments were performed in which more free nitric acid was used, and some in which less time was allowed, but in all of them the precipitation was incomplete. The separation is probably possible, also, in an ammonia solution in the presence of ammonium nitrate.

Analysis of Nickel Coin.

A nickel coin, weighing 4.8514 grams, was dissolved in nitric acid, then evaporated on the water-bath, and the crystallized residue dissolved in water. Twenty-five cc. of this solution were placed in a weighed platinum dish and 3 grams of ammonium sulphate, and 1 gram of ammonium nitrate added. This solution was diluted with water to 125 cc., heated nearly to boiling and electrolyzed with a current of $N.D_{100} = 5$ A. $V = 5$. Time = 20 minutes. The precipitated copper weighed 0.3629 gram, equal to 74.81 per cent. of the coin. The solution from this deposit was precipitated with sodium hydroxide and bromine water, and the precipitate filtered, washed and dissolved in dilute sulphuric acid, containing 2.5 cc. of concentrated acid. To this solution 30 cc. of concentrated ammonia water were added, the whole diluted to 125 cc., heated, and electrolyzed with a current of $N.D_{100} = 6$ A. $V = 5$. Time = 17 minutes. The deposited nickel weighed 0.1185 gram, corresponding to 24.42 per cent. of the coin.

The attempt was next made to see how quickly a complete analysis of a nickel coin could be made. This was done as follows:

Weight of coin = 4.925 grams.

It was dissolved in 20 cc. of concentrated nitric acid diluted with an equal volume of water. When the solution was complete, the liquid was exactly neutralized with ammonia, transferred to a 250 cc. measuring flask and this filled with water to the mark. 25 cc. of this liquid were then transferred to a weighed platinum dish and 3 grams of ammonium sulphate were added. This was diluted to 125 cc., heated and electrolyzed with a current of $N.D_{100} = 5$ A. $V = 5.5$ for 20 minutes. The precipitated copper weighed 0.3691 gram = 74.95 per cent. of the coin. The solution from the copper was precipitated with sodium hydroxide and bromine water, filtered and washed. The precipitate was then dissolved in 2 cc. of concentrated sulphuric acid diluted with water, 30 cc. of concentrated ammonia water added, diluted to 125 cc., heated, and the solution electrolyzed with a current of $N.D_{100} = 6$ amperes and 5 volts. Twenty minutes were required for the deposition. The precipitated nickel weighed 0.1217 gram, corresponding to 24.71 per cent. The solution from the nickel deposit was filtered, and the precipitate washed, ignited and weighed. The weight of ferric oxide was 0.0026 gram, which is equivalent to 0.35 per cent. of metallic iron. The total percentage equalled 100.01. The time required for the complete analysis was two and a half hours.

SEPARATION OF COPPER FROM ZINC.

The conditions which served for the separation of copper from nickel are applicable in its separation from zinc.

No. of exp.	$CuSO_4$ = Cu. Gram.	$ZnSO_4$ = Zn. Gram.	NH_4NO_3. Grams.	Conc. HNO_3. cc.	Current. $N.D_{100}$ = A.	V.	Time. Minutes.	Deposited Cu. Gram.	Error. mg.
1	0.2457	0.25	3	0.25	5	9	15	0.2458	+0.1
2	0.2457	0.25	3	0.25	5	9	15	0.2460	+0.3

This is sufficient to show the possibilities of this method of analysis. At the present time other separations are in progress.

UNIVERSITY OF PENNSYLVANIA.

[CONTRIBUTION FROM THE HAVEMEYER LABORATORIES, COLUMBIA UNIVERSITY, NO. 84.]

THE VOLUMETRIC DETERMINATION OF BISMUTH AS MOLYBDATE AND ITS SEPARATION FROM COPPER.[1]

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AT THE present time the quantitative determination of bismuth is conducted in the great majority of cases by precipitation as bismuth basic carbonate by ammonium carbonate, in spite of the fact that many different schemes have been published for its separation from other metals and determination, using other reagents.

It was with the intention of finding, if possible, a reliable volumetric method equal in accuracy to the carbonate method, that this work was undertaken.

As the work progressed and the molybdate method was to be applied to ores, to show its practical value, it developed that a shorter and simpler separation of bismuth from copper, especially, would make the method complete from the ore, and it was then that the precipitation of bismuth with hydrogen sulphide from a solution containing tartaric acid and made alkaline with

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[1] From author's thesis for the degree of Doctor of Philosophy. Read at the December meeting of New York Section of the American Chemical Society.