

completely dried without fusion, his experiments show a loss in weight on fusion of 0.002 per cent., while the average loss on fusion, as given on pages 1585 and 1586 is about 0.004 per cent. Moreover, Ladenburg's method of purifying silver iodide, by washing the precipitated salt with ammonia, could hardly be expected to remove last traces of silver chloride and silver cyanide "included" by the precipitate. These errors are all so small that they would not affect the second place of decimals. However, Ladenburg's, Scott's,¹ and Köthner and Aeuer's work all afford confirmation that the atomic weight of iodine is undoubtedly much higher than has been usually supposed.

The results of the research are, then, as follows:

(1) The atomic weight of iodine is found to be 126.975 ($O = 16.000$).

(2) Richards and Wells's value for the atomic weight of chlorine, 35.467, is confirmed.

(3) The existence of an element of the halogen family of higher atomic weight than iodine is shown to be improbable.

(4) The specific gravity of pure fused silver iodide is found to be 5.674 at 25° referred to water at 4°.

I am indebted to the Cyrus M. Warren Fund for Research, in Harvard University, for platinum vessels, quartz crucibles and balance, all indispensable in the research.

CHEMICAL LABORATORY OF HARVARD COLLEGE,
CAMBRIDGE, MASS., August 6, 1904.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY,
No. 89.]

THE USE OF THE ROTATING ANODE IN ELECTRO-ANALYSIS.

BY EDGAR F. SMITH.

Received September 21, 1904.

THE results obtained by Exner, in this laboratory, with a rotating anode in the electrolytic precipitation of metals led to further study along the same line. Thus, Ashbrook succeeded in effecting a number of separations reducing the time factor, in every instance, very considerably. Ingham exhaustively investigated the precipitation of zinc from various electrolytes, show-

¹ *Proc. Chem. Soc.*, 18, 112.

ing also how this metal could be quickly determined in its most frequently occurring ore, thereby making a very definite and valuable contribution to technical and mineral analysis.

To still further emphasize the advantages arising from the use of a rotating anode in electro-analysis, Ingham demonstrated the possibility of estimating nitric acid in the alkali nitrates with great accuracy in thirty minutes.

It is only fair to say that Exner in no sense exhausted the possibilities of metal precipitation, aided by the rotating anode. Other workers, in this laboratory, have followed him, giving more attention to details, thus gradually accumulating a vast amount of data relating to the various electrolytes and to the conditions most favorable for successful experimentation. That is the merit, in large measure, of Ingham's study of zinc. The facts presented in the present communication are of a similar character. It is our purpose to continue studies in this direction, and also to add to the separations already accomplished by Ashbrook.

The data here presented in regard to the estimation of nickel and cobalt electrolytically will be found decidedly helpful in many ways.

I. DETERMINATION OF NICKEL IN VARIOUS ELECTROLYTES.

BY GEORGE H. WEST.

It is not necessary to enumerate here the many recommendations made at various times by chemists for the electrolytic determination of this metal, but we will merely present the conditions under which the work was done, and give the results obtained by us. Neither is it necessary to occupy space with a description of the apparatus employed in arriving at these results, beyond stating that it was identical with that previously used by Exner, Ingham, and Ashbrook in this laboratory.

The dilution of solution in the various electrolytes ranged from 100 to 125 cc., which represented a cathode surface of 100 sq. cm. The speed of the anode varied from 500 to 650 revolutions per minute. The deposits of nickel were crystalline and gray in color from solutions in which there was an excess of ammonia, while from acid solutions the metal was brilliant and exceedingly metallic in appearance—closely resembling the platinum dish. Whenever peroxide appeared on the rotating anode it was made to disappear, in ammoniacal solutions, by the addition of more ammonia water

to the electrolyte, and when in acid solutions by lowering the current toward the end of the decomposition, and after a few minutes again increasing it, or by introducing into the acid liquid a few drops of a mixture consisting of 5 cc. of glycerol, 45 cc. of alcohol and 50 cc. of water. Pure nickel sulphate was the salt used. To indicate the rate of precipitation the results are plotted in each instance. In this form they become highly instructive.

(A) AMMONIUM ACETATE ELECTROLYTE.

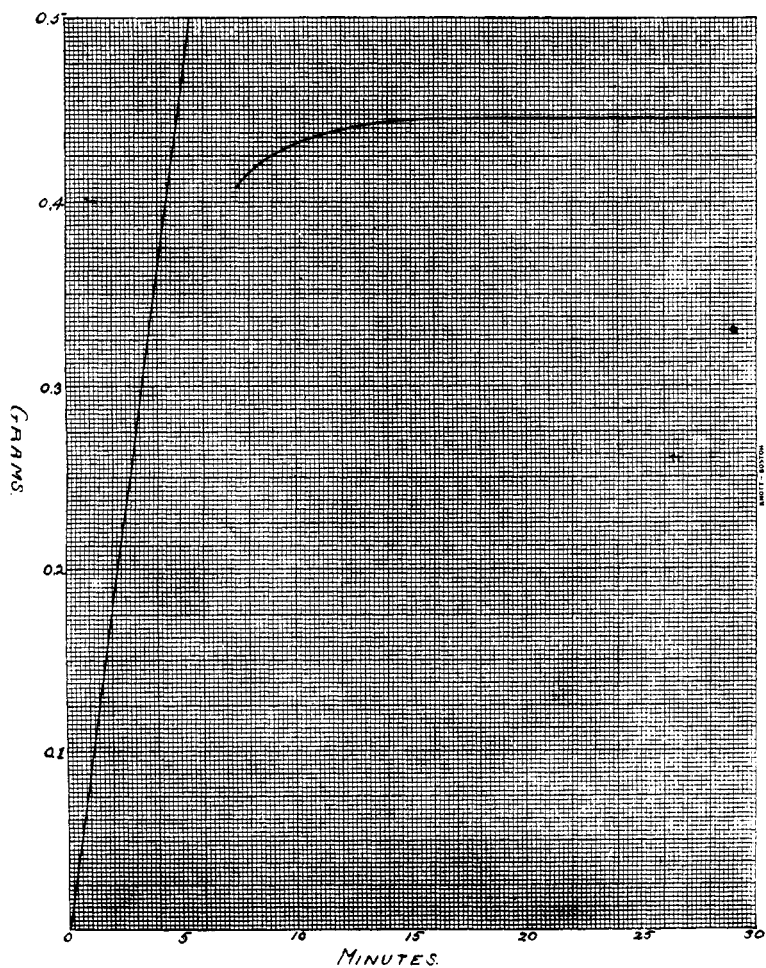
Expt. No.	Nickel present. Gram.	Acetic acid (26 per cent.)		Amperes.	Volts.	Time. Minutes.	Nickel found. Gram.
		Ammonia (sp. gr. 0.94). cc.	cc.				
1	0.4444	20	10	5	4.7	16	0.4438
2	0.4444	25	10	5	4.6	15	0.4404
3	0.4444	20	10	5	4.6	12½	0.4409
4	0.4444	25	10	5	4.6	15	0.4436
5	0.4444	25	10	5	4.6	20	0.4444
6	0.4444	25	10	5	4.6	7½	0.4082
7	0.4444	25	10	5	4.6	10	0.4326
8	0.4444	25	10	5	4.6	30	0.4454

Experiments 1, 4 and 5 record the most favorable conditions. Reference to Exner's experience with this same electrolyte shows that he precipitated about 0.25 gram of nickel completely in ten minutes. There was no anodic deposition in the above experiments, which are graphically shown in Fig. 1.

(B) SODIUM ACETATE ELECTROLYTE.

Nickel present. Gram.	Sodium acetate, 1 cc. = 0.1 gram salt.	Acetic acid (26 per cent.).	Amperes.	Volts.	Time. Minutes.	Nickel found. Gram.
	cc.	cc.				
0.4431	30	0.25	5	7.5	7½	0.4028
0.4431	30	0.25	5	7.5	10	0.4256
0.4431	30	0.25	5	7.0	15	0.4306
0.4431	30	0.40	5	7.0	20	0.4431
0.4431	30	0.25	5	7.5	20	0.4422
0.4431	30	0.25	5	7.0	20	0.4429
0.4431	30	0.25	5	7.5	25	0.4432
0.4431	30	0.25	5	7.5	30	0.4432
0.4431	30	0.25	5	7.5	30	0.4432

It is evident that the precipitation here was slower than in the preceding series of experiments. The solution, however, in this instance was acid, and under such conditions more time was invariably required for complete precipitation. In each of these



NICKEL FROM AMMONIUM ACETATE ELECTROLYTE

Fig. 1.

determinations there was also present 0.5 cc. of the glycerol—alcohol mixture. It effectually prevented any deposition upon the anode. The graphic representation of the results is given in Fig. 2.

(c) AMMONIA AND AMMONIUM SULPHATE ELECTROLYTE.

This is, of course, the time-honored solution from which nickel has been deposited by the current for analytical and other purposes. Exner gave it some attention in connection with the rotating anode. The appended results supplement and complete his very nicely. A study of the graphic representation in Fig. 3 will prove interesting and instructive.

No.	Nickel present. Gram.	$(\text{NH}_4)_2\text{SO}_4$. Grams.	NH_4OH (sp. gr. 0.94). cc.	Amperes.	Volts.	Time. Minutes.	Nickel found. Gram.
1	0.2522	1.2	20	4	5.5	1	0.0538
2	0.2522	1.2	20	4	5.5	2	0.0957
3	0.2522	1.2	20	4	5.5	3	0.1474
4	0.2522	1.2	20	4	5.5	4	0.1733
5	0.2522	1.2	20	4	5.5	5	0.2102
6	0.2522	1.2	20	4	5.5	7½	0.2444
7	0.2522	1.2	20	4	5.5	10	0.2493
8	0.2522	1.2	20	4	5.5	15	0.2522
9	0.2522	1.2	20	4	5.5	20	0.2531
10	0.2522	1.2	20	4	5.5	30	0.2525
11	0.5050	1.2	25	4	5.5	1	0.0571
12	0.5050	1.2	25	4	5.5	2	0.1164
13	0.5050	1.2	25	4	5.5	3	0.1549
14	0.5050	1.2	25	4	5.5	4	0.2000
15	0.5050	1.2	25	4	5.5	5	0.2510
16	0.5050	1.2	25	4	5.5	7½	0.3580
17	0.5050	1.2	25	4	5.5	10	0.4450
18	0.5050	1.2	25	4	5.5	15	0.5007
19	0.5050	1.2	25	4	5.5	20	0.5050
20	0.5050	1.2	25	4	5.5	30	0.5052
21	0.5050	1.2	25	5	7	7½	0.4518
22	0.5050	1.2	25	5	7	10	0.4876
23	0.5050	1.2	25	5	7	15	0.5042
24	0.5050	1.2	25	5	7	20	0.5049
25	0.5050	1.2	25	6	8.5	20	0.5046
26	1.0100	1.2	30	5.2	6.5	25	1.0093
27	1.0100	1.2	30	5.2	6.5	25	1.0093

(D) FORMATE ELECTROLYTE.

These results represent the first study of nickel in the presence

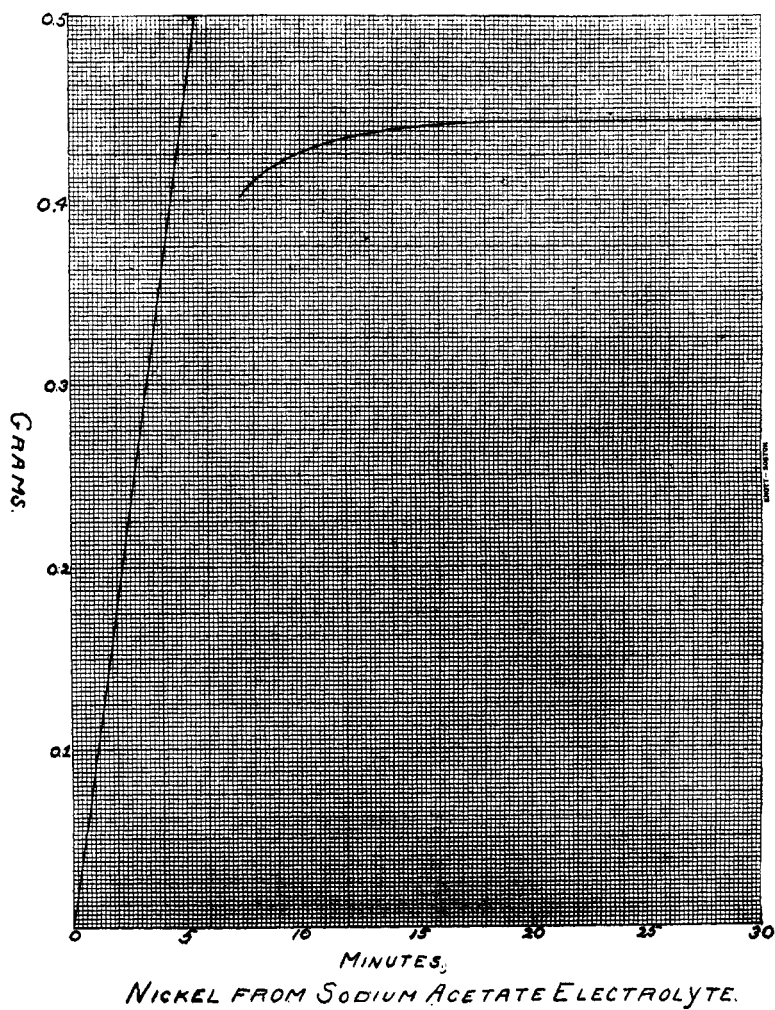


Fig. 2

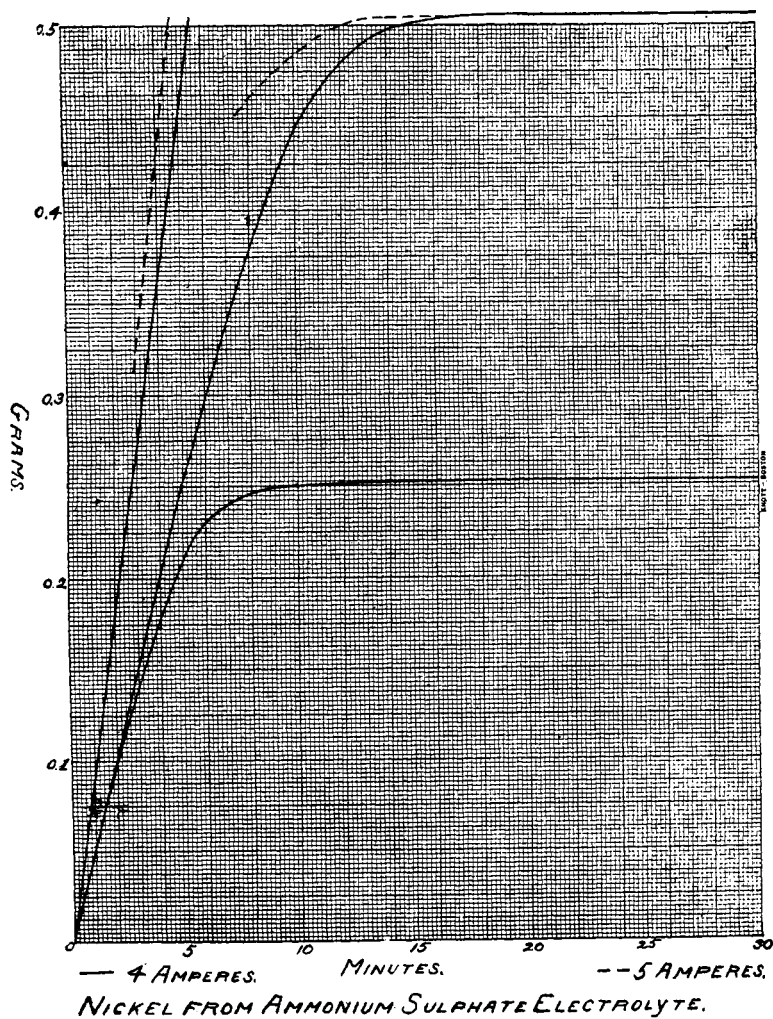


Fig. 3.

of formic acid, or formates, instituted because of the favorable outcome, under nearly similar conditions, with zinc, by Ingham. They are graphically represented in Fig. 4.

Nickel present. Gram.	NH ₄ OH. cc.	H.CO ₂ H (25 per cent.). cc.	Amperes.	Volts.	Time. Minutes.	Nickel found. Gram.
0.4444	25	10	5	6	7½	0.4214
0.4444	25	10	5	6	10	0.4393
0.4444	20	10	5	6	12½	0.4431
0.4444	20	10	5	6	15	0.4446
0.4444	20	15	5	4	15	0.4443
0.4444	20	10	5	4	15	0.4444
0.4444	20	12	5	4	20	0.4449

(E) SODIUM FORMATE ELECTROLYTE.

Pure sodium carbonate was added to the complete precipitation of the nickel from its sulphate, the precipitate dissolved in excess of formic acid, and the solution electrolyzed. The results were as follows and are graphically recorded in Fig. 5.

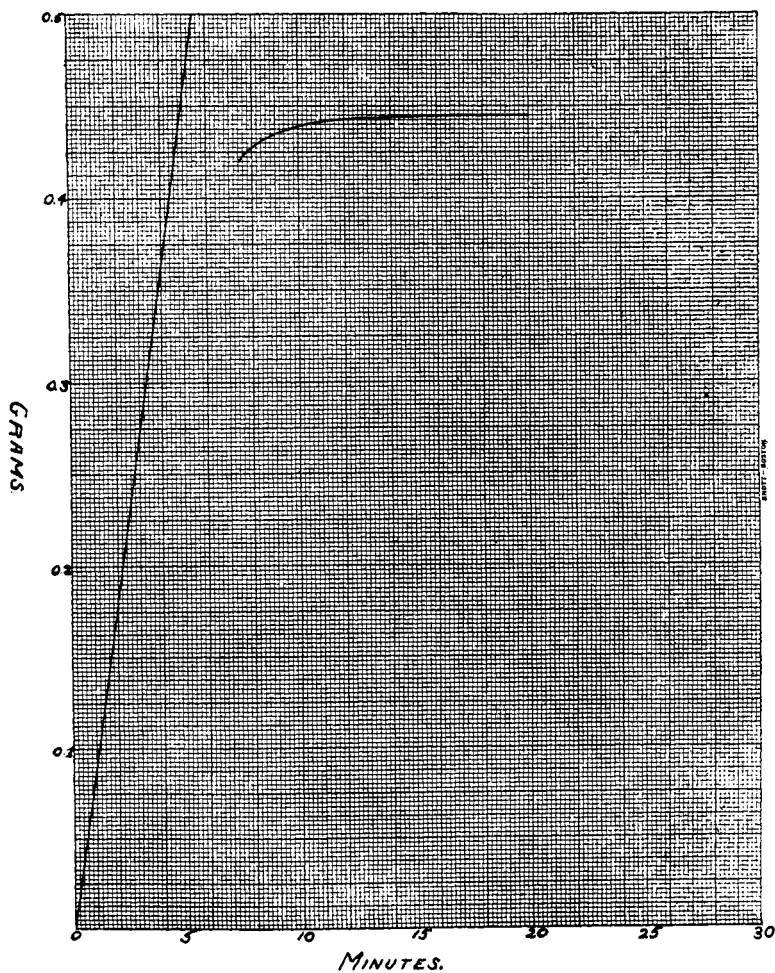
Nickel present. Gram.	Na ₂ CO ₃ . Grams.	H.CO ₂ H (25 per cent.). cc.	Amperes.	Volts.	Time. Minutes.	Nickel found. Gram.
0.4444	5	22	5	4	5	0.2474
0.4444	5	22	5	4	7½	0.3260
0.4444	5	22	5	4	10	0.3688
0.4444	5	22	5	4	15	0.4323
0.4444	5	22	5	4	20	0.4394
0.4444	5	22	5	4	30	0.4448

(F) AMMONIUM LACTATE ELECTROLYTE.

In estimating zinc Jordis¹ recommended its deposition from an ammonium lactate solution in the presence of lactic acid. As this electrolyte had never before been applied, to our knowledge, to nickel, a series of experiments were tried with the results indicated below and graphically represented in Fig. 6.

Nickel present. Gram.	NH ₄ OH (sp. gr. 0.94). cc.	Lactic acid. cc.	Amperes.	Volts.	Time. Minutes.	Nickel found. Gram.
0.4443	25	2.5	5	6.5-7.5	5	0.3151
0.4443	25	2.5	5	7.5	7½	0.4056
0.4443	25	2.5	5	7.5	10	0.4344
0.4443	25	2.5	5	7.5	15	0.4442
0.4443	25	2.5	5	7.5	15	0.4443
0.4443	25	2.5	5	7.5	20	0.4447
0.4443	25	2.5	5	7.5	20	0.4439
0.4443	25	2.5	5	7.5	22	0.4443
0.4443	25	2.5	5	7.5	25	0.4453
0.4443	25	2.5	5	7.5	30	0.4441

¹ *Zischr. Elektrochemie*, 2, 138, 563, 655.



NICKEL FROM AMMONIUM FORMATE ELECTROLYTE.

Fig. 4.

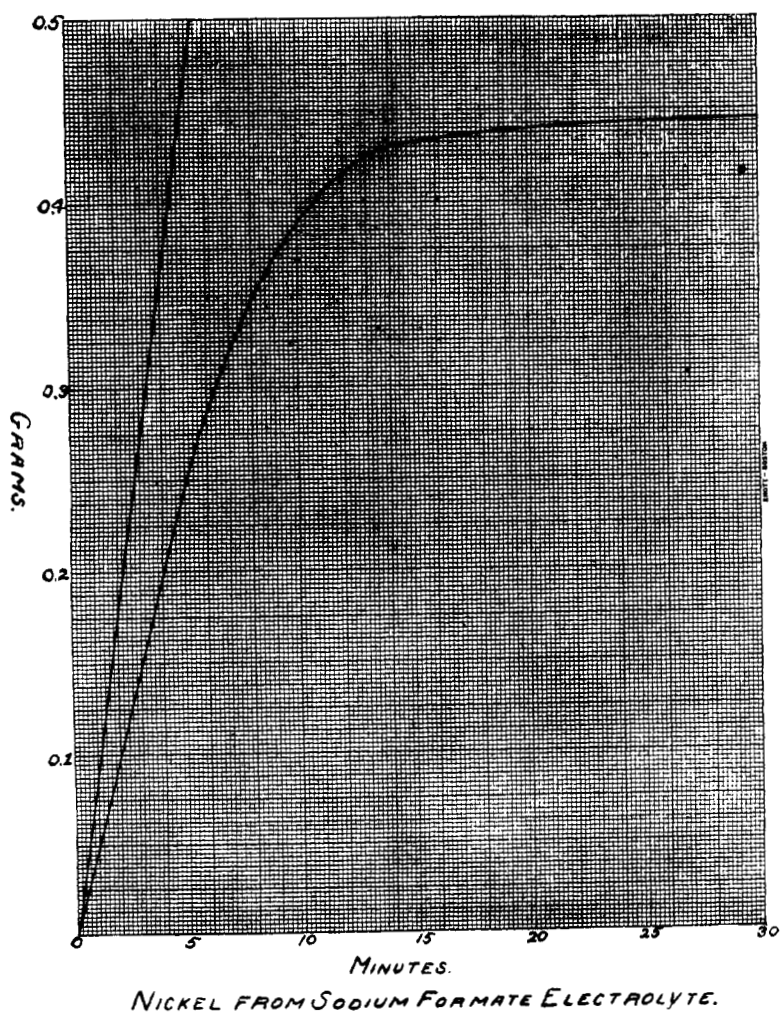


Fig. 5.

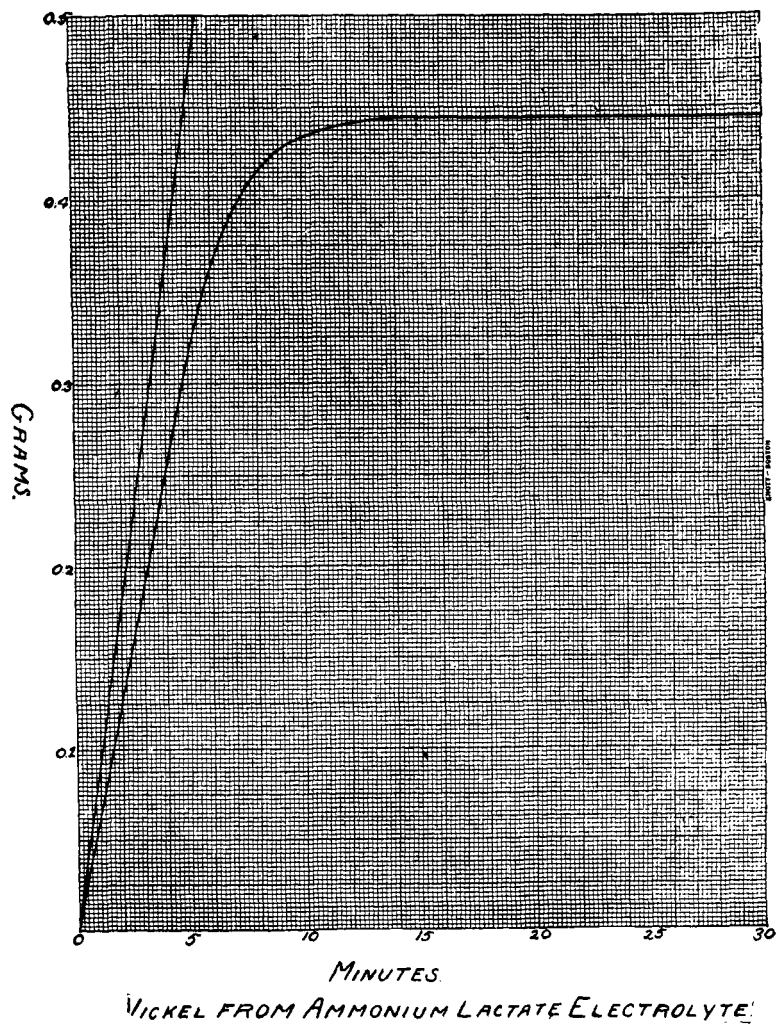


Fig. 6.

II. DETERMINATION OF COBALT IN VARIOUS ELECTROLYTES.

BY LILY G. KOLLOCK.

The speed of the rotating anode in these trials with cobalt, as well as other details, have been given in the introductory paragraphs, so that its deposition from the various electrolytes may be given without further comment.

(A) SODIUM FORMATE ELECTROLYTE.

To a solution of pure cobalt sulphate, of definite strength, were added 2.5 grams of sodium carbonate and 4 cc. of 94 per cent. formic acid. It was then heated to boiling and electrolyzed with a current of $N.D_{100} = 5$ amperes and 6 volts. After thirty minutes the solution was siphoned from the dish, water being substituted for it. The precipitation of metal was complete. It was so brilliant in appearance that it was hardly distinguishable from the platinum. It may not be superfluous to repeat that in this electrolyte there may occur a slight anodic deposition of peroxide. The glycerol mixture, previously referred to, causes it to disappear or prevents its formation. But it is preferable to lower the current to 1 ampere for a few minutes when the solution has almost lost its color and when the peroxide has disappeared from the anode to restore the current to its original strength. Re-deposition of oxide was never observed.

Much formic acid retards the precipitation of the cobalt. If the solution becomes alkaline, the metal is deposited very rapidly and is consequently very spongy. It was found advantageous to add the acid drop by drop from time to time. The quantity of acid indicated in the table was usually all that was necessary in order to insure good results.

Expt. No.	Cobalt taken. Gram.	Sodium carbonate. Grams.	Formic acid. cc.	Volts.	Amperes.	Time. Minutes.	Cobalt found. Gram.
1	0.3535	2.5	3.0	8	6	30	0.3530
2	0.3535	2.5	3.0	8	6	30	0.3535
3	0.3535	4.0	4.5	9	6	35	0.3540
4	0.3535	2.5	4.5	9	6	35	0.3540
5	0.3535	2.5	3.5	10	8	30	0.3530
6	0.3535	2.5	3.5	8	8	30	0.3531
7	0.3525	2.5	3.5	6	6.5	35	0.3535
8	0.3535	2.5	5.0	7	7	40	0.3535
9	0.2635	2.5	3.0	5	5	30	0.2635
10	0.2635	2.5	13.5	7.5	6	30	0.2643
11	0.2635	2.5	14.0	7.5	6	30	0.2630
12	0.2635	2.5	15.0	7.5	6	30	0.2635
13	0.2635	2.4	15.0	7	6	30	0.2630

The formic acid in Expts. 1 to 9 was 94 per cent., and 25 per cent. in those from 10 to 13.

The rate of deposition was studied. The curve was plotted from the following data:

In 5 minutes 0.1470 gram of metal was deposited.

"	7½	"	0.2096	"	"	"	"	"	"
"	10	"	0.2570	"	"	"	"	"	"
"	15	"	0.3066	"	"	"	"	"	"
"	20	"	0.3092	"	"	"	"	"	"
"	25	"	0.3142	"	"	"	"	"	"
"	30	"	0.3152	"	"	"	"	"	"

See Fig. 7.

(B) AMMONIUM FORMATE ELECTROLYTE.

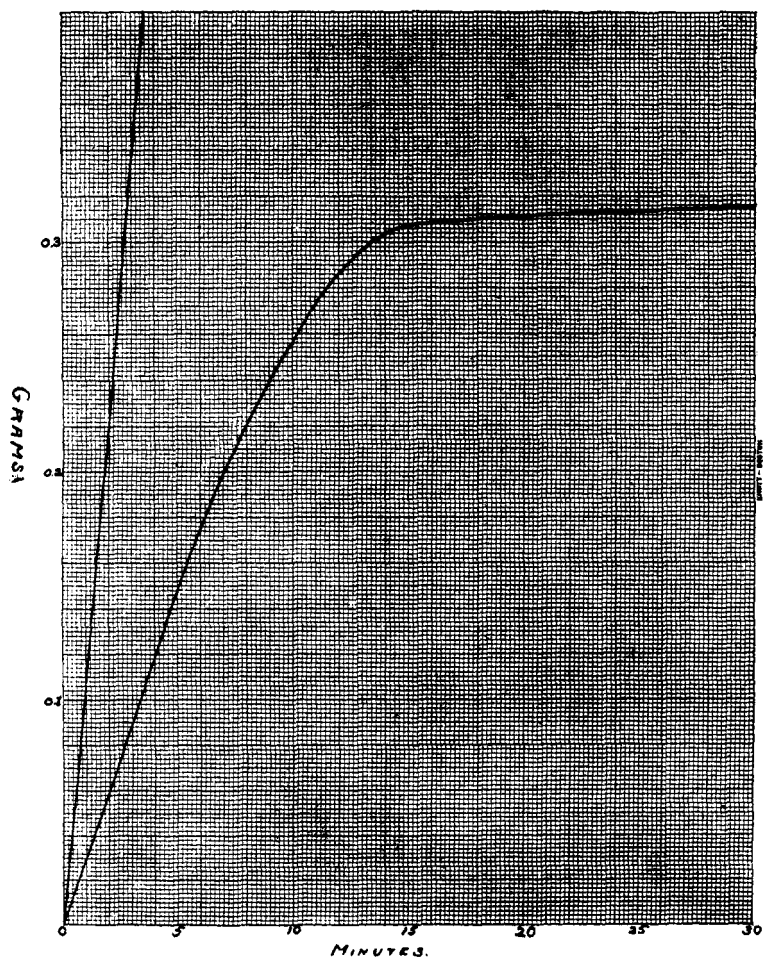
The solution may be alkaline or acid. From the former the metal separates in a compact form, but very dull in appearance, while from an acid solution it separates in a very brilliant form, but more slowly than from an ammoniacal solution. There was little tendency to anodic deposition in this electrolyte. The solution was colorless in fifteen minutes.

Expt. No.	Cobalt present. Gram.	Ammonium hydroxide. cc.	Formic acid (94 per cent.). cc.	Volts.	Amperes.	Time. Minutes.	Cobalt found. Gram.
1	0.1260	20	3	5.5	6	20	0.1265
2	0.3152	25	3	5	6	30	0.3160
2	0.3152	20	3.5	5.5	6	25	0.3152
4	0.3152	20	3.5	7	6.5	20	0.3152
5	0.3152	20	3.5	7	6.5	20	0.3150

(C) AMMONIUM ACETATE ELECTROLYTE.

The deposit of cobalt from this solution was very satisfactory. The metal was brilliant in appearance. The following conditions may be expected to yield excellent results. Peroxide on the anode was not observed in any of these determinations.

Expt. No.	Cobalt present. Gram.	Ammonium hydroxide. cc.	Acetic acid (26 per cent.). cc.	Volts.	Amperes.	Time. Minutes.	Cobalt found. Gram.
1	0.2980	25	10	6	5	20	0.2980
2	0.2980	25	10	6	5	20	0.2980
3	0.2980	25	10	6	5	20	0.2980
4	0.2980	25	10	6	5	20	0.2980
5	0.1324	25	10	5	5.5	25	0.1318
6	0.3310	25	10	6	5	25	0.3304
7	0.3310	25	10	6	5	25	0.3306
8	0.3310	25	10	5	5	25	0.3305
9	0.3310	25	10	5	5	30	0.3310



COBALT FROM SODIUM FORMATE ELECTROLYTE.

Fig. 7.

The conditions of Expt. 1 were observed in getting data for the time curve (Fig. 8).

In 5 minutes 0.2235 gram of cobalt was deposited.

"	10	"	0.2778	"	"	"	"	"
"	15	"	0.2950	"	"	"	"	"
"	20	"	0.2980	"	"	"	"	"
"	25	"	0.2980	"	"	"	"	"

(D) SODIUM LACTATE ELECTROLYTE.

Cobalt, like nickel, had never been previously studied in an electrolyte containing lactic acid or any of its salts. It proved a most satisfactory solution from which to deposit the metal; at least such was the case while working with the conditions given below. There was no peroxide deposition, and the deposits of metal were adherent and brilliant in appearance. A large excess of lactic acid exerted a retarding influence upon the precipitation.

Expt. No.	Cobalt present. Gram.	Sodium carbonate. Grams.	Lactic acid conc. cc.	Volts.	Amperes.	Time.	Cobalt found. Gram.
1	0.3152	2.2	5	8	5	25	0.3150
2	0.3152	2	5	11	6.5	30	0.3150
3	0.3152	2	4	9.5	5	25	0.3155

(E) AMMONIUM LACTATE ELECTROLYTE.

The results in this solution were, if anything, superior to those obtained in the preceding electrolyte. The liquid became colorless in twelve minutes, but the final traces of cobalt required a little more time for their complete removal. The large amount of ammonium hydroxide was used to increase the conductivity of the liquid.

Expt. No.	Cobalt present. Gram.	Ammonium hydroxide. cc.	Lactic acid. cc.	Volts.	Amperes.	Time. Minutes.	Cobalt found. Gram.
1	0.3152	10	2.5	10	6	30	0.3155
2	0.3310	20	4.5	7.5	6	25	0.3305
3	0.3310	20	4.5	7.5	6	25	0.3310
4	0.3310	30	7	5	6	25	0.3310

In getting data to construct a time curve (Fig. 9), the following conditions were observed: Ten cc. of ammonium hydroxide and 2.5 cc. of concentrated lactic acid were added to the cobalt sulphate solution, which was then electrolyzed with a current of $N.D_{100} = 6$ amperes and 7.5 volts.

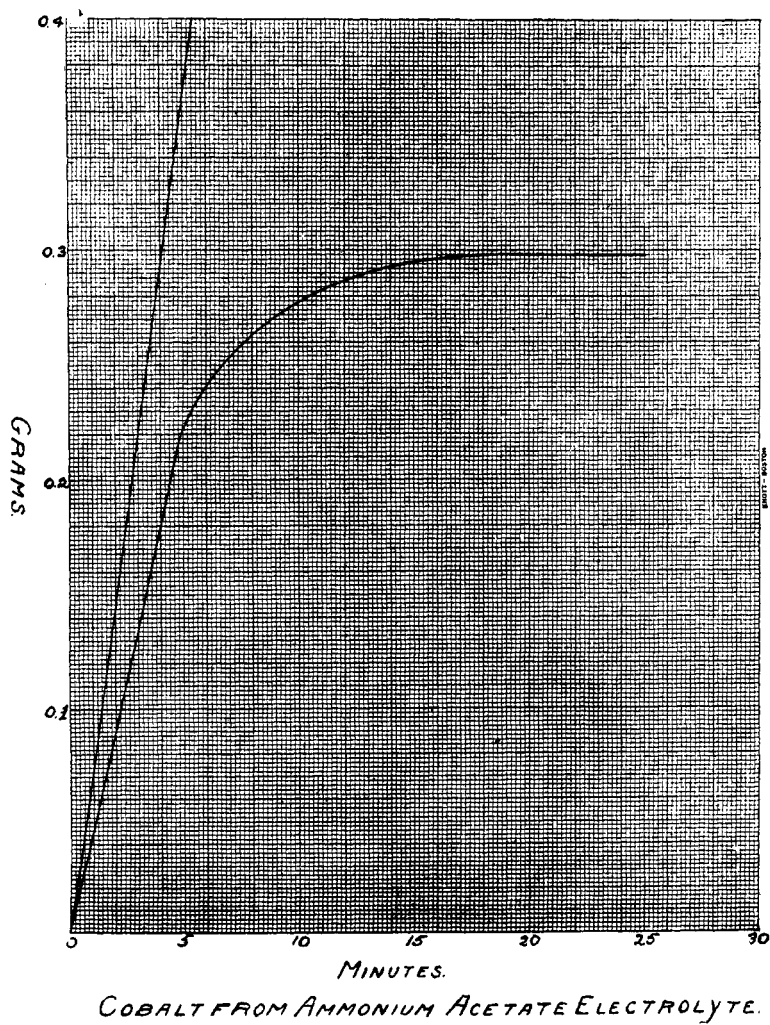
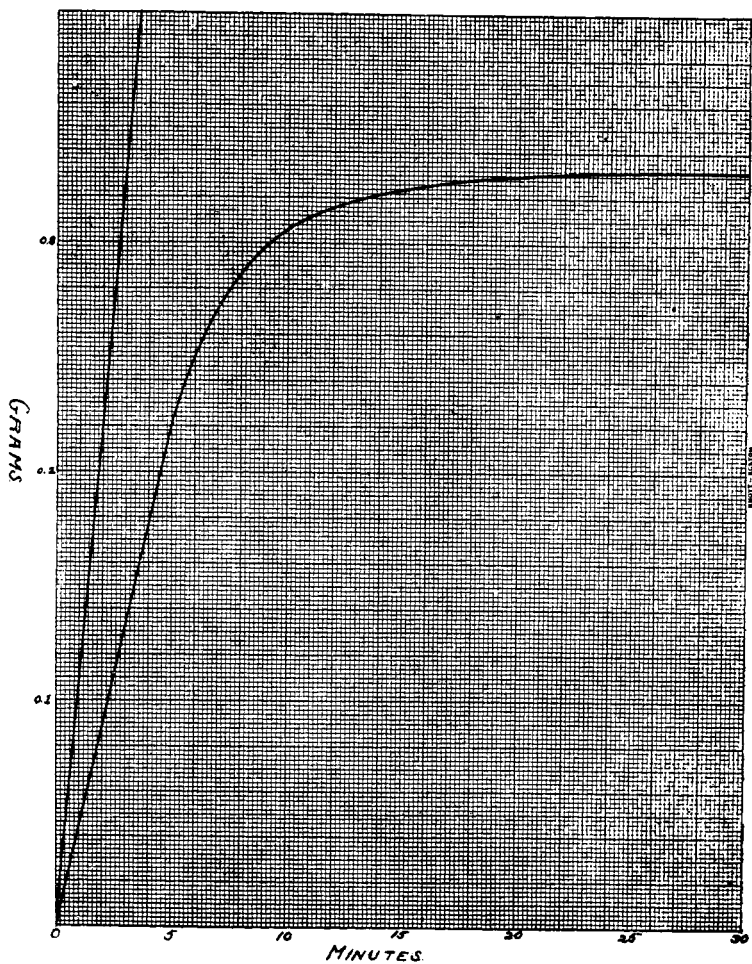


Fig. 8.



COBALT FROM AMMONIUM LACTATE ELECTROLYTE.

Fig. 9.

In 5 minutes 0.2215 gram of cobalt was deposited.

"	10	"	0.3060	"	"	"	"	"
"	15	"	0.3230	"	"	"	"	"
"	20	"	0.3290	"	"	"	"	"
"	25	"	0.3310	"	"	"	"	"
"	30	"	0.3310	"	"	"	"	"

(F) AMMONIUM SUCCINATE ELECTROLYTE.

In this solution there was a slight deposit on the anode, but it was removed in the manner indicated under the formate electrolytes. A little carbon was observed in the precipitated cobalt. Its quantity was inappreciable.

Expt. No.	Cobalt present. Gram.	Ammonium hydroxide. cc.	Succinic acid. Grams.	Volts.	Amperes.	Time. Minutes.	Cobalt found. Gram.
1	0.3310	25	4	5	5.6	30	0.3315
2	0.3310	25	3.5	6	5	25	0.3305
3	0.3310	25	3	6	5	30	0.3309
4	0.3310	25	3	8	6	25	0.3305

The liquid in Expts. 1 and 2 was acid in reaction, while that in Expts. 3 and 4 was alkaline.

In determining the time curve (Fig. 10) 3 grams of succinic acid and 25 cc. of ammonium hydroxide were added to the liquid which was electrolyzed with a current of $N.D_{100} = 5$ amperes and 8 volts. The results were:

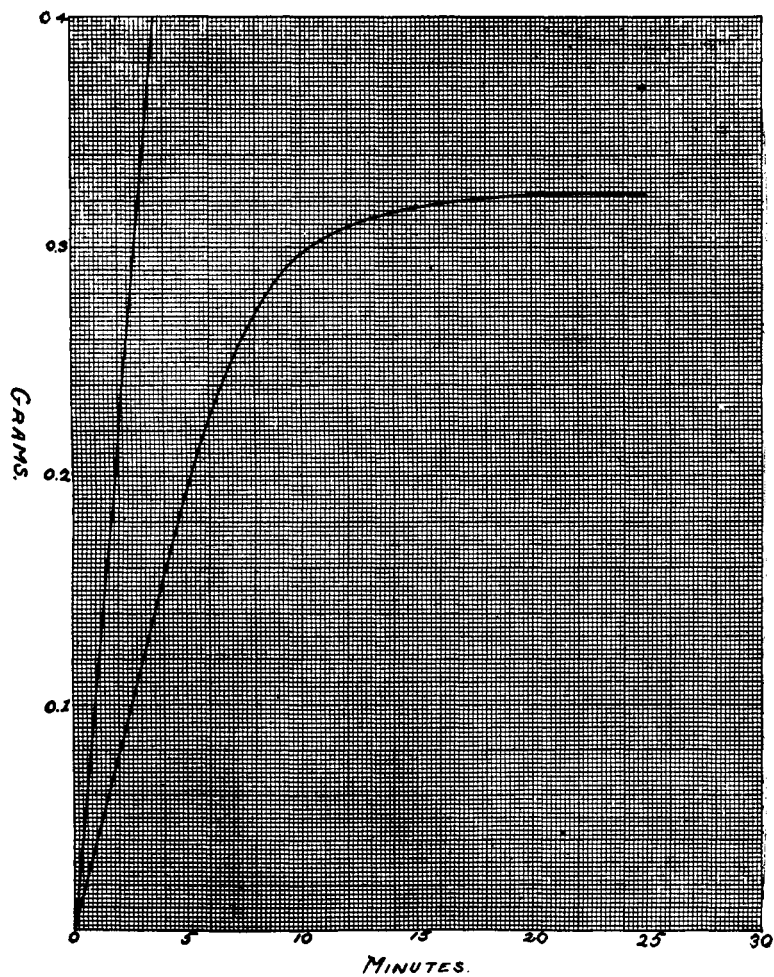
In 5 minutes 0.1935 gram of cobalt was deposited.

"	10	"	0.2975	"	"	"	"	"
"	15	"	0.3284	"	"	"	"	"
"	20	"	0.3310	"	"	"	"	"
"	25	"	0.3310	"	"	"	"	"

Our experience with sodium succinate was unsatisfactory. There was a great deal of peroxide deposited upon the anode, hence that electrolyte was abandoned.

SEPARATION OF COBALT FROM THE ALKALINE EARTH METALS.

In these separations the metals were present as formates. Care was taken not to have too great an excess of formic acid present, but to add it gradually in order that the solution might continue acid in its reaction throughout the decomposition.



COBALT FROM AMMONIUM SUCCINATE ELECTROLYTE.

Fig. 10.

1. COBALT FROM CALCIUM.

Cobalt present. Gram.	Calcium formate. Gram.	Ammonium hydroxide. cc.	Formic acid. cc.	Volts.	Amperes.	Time. Minutes.	Cobalt found. Gram.
0.2135	0.5	10	4	5	6	30	0.2135
0.2135	1	10	4	4.5	6	30	0.2134
0.2135	1.5	10	4.5	4.5	6	30	0.2133
0.2135	1	10	4.5	4.5	6	30	0.2132

2. COBALT FROM STRONTIUM.

Cobalt present. Gram.	Strontium formate. Gram.	Formic acid. cc.	Volts.	Amperes.	Time. Minutes.	Cobalt found. Gram.
0.2040	1.0	5	14	3	25	0.2045
0.2040	1.5	5	14	3	25	0.2135
0.2040	1.5	5	15	3	25	0.2045

In these separations the addition of ammonium formate was omitted. Its presence apparently operated adversely.

3. COBALT FROM BARIUM.

Cobalt present. Gram.	Barium formate. Gram.	Ammonium hydroxide. cc.	Formic acid after neutralization. cc.	Volts.	Amperes.	Time. Minutes.	Cobalt found. Gram.
0.2040	1	10	1	5	6	25	0.2045
0.2040	1.5	10	1	5	6	25	0.2042
0.2040	1	10	1	5	6	25	0.2043

4. COBALT FROM MAGNESIUM.

Cobalt present. Gram.	Magnesium formate. Grams.	Ammonium hydroxide. cc.	Formic acid in excess. cc.	Volts.	Amperes.	Time. Minutes.	Cobalt found. Gram.
0.2040	0.5	5	1	6	5	30	0.2045
0.2040	1	5	1	6	5	30	0.2033

It might seem on first thought that the separations outlined above would proceed without any trouble. Experience showed that it was necessary to observe certain points very carefully, hence the conditions under which we obtained success have been duly recorded.

In addition to the precipitation of metals with a rotating anode experiments have been conducted, in this laboratory, with a mercury cathode.¹ It seemed probable that a rotating anode might be used with advantage, together with the mercury cathode. Accordingly, the beaker with the layer of mercury as described by Smith

¹ This Journal, 25, 884; 26, 1124.

and by Myers, in the references cited above, was used. The anode was a spiral of heavy platinum wire, 1 cm. in diameter. It was made to rotate about 600 times per minute. A solution of copper sulphate, when acted upon under these conditions, with a current of 2 amperes and 4 volts, lost its color in fifteen minutes. This led to the following quantitative determinations of copper.

Copper present. Gram.	Volts.	Amperes.	Time. Minutes.	Volume of liquid. cc.	Copper found. Gram.	Separation of poles. cm.
0.2405	10 to 5	0.01 to 2.0	30	40	0.2401	2
0.2405	10 to 5	0.01 to 2.0	20	25	0.2401	1
0.4810	10 to 5	0.01 to 2.0	25	25	0.4805	1

The copper sulphate solution was neutral at the beginning of the decomposition, hence the low current in the early stages of the precipitation. The more concentrated the solution the more rapidly did the metal separate. It seems probable from other experiments that 0.5 gram of copper can be deposited in fifteen minutes. The several trials made with a nickel sulphate solution indicate that from 0.25 to 0.5 gram of that metal can also be precipitated *with a mercury cathode and rotating anode* in about the same period of time.

Mention is here made of these observations as experimentation in this particular direction will be undertaken immediately. Should success attend the same, as there is every reason to believe, one rather expensive factor in electro-analysis will be eliminated, *viz.*, the platinum dish, cone or cylinder.

UNIVERSITY OF PENNSYLVANIA.

COMPOSITION OF THE FOUR SULPHIDES OF MANGANESE.¹

BY J. C. OLSEN AND W. S. RAPALJE.

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LITERATURE.

THE composition of the two commonly known sulphides of manganese has been investigated by several workers. The results obtained have been so conflicting that it is impossible to decide from the literature whether either of these sulphides is anhydrous or not, much less to give the exact composition. The reason for this discrepancy, as we shall attempt to show, has been the failure

¹ Read before the New York Section of the American Chemical Society, April 8, 1904.