

pressure below that of the atmosphere. In the particular instruments which the author has had made for combustion gases, the stem of the burette is of smaller diameter than usual, so that the entire contents are not greater than 28 or 30 per cent. of the burette. This makes possible finer divisions being graduated thereon, which read to 0.01 per cent.

The cock located at the lower end of the burette has in all cases been made of metal, and has proven more satisfactory than those of glass; those at the top of the pipette formerly employed, the author was compelled to replace with pinch-cocks, owing to the difficulty of obtaining carefully made and durable glass cocks.

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[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, No. 95.]

## THE USE OF THE ROTATING ANODE AND MERCURY CATHODE IN ELECTRO-ANALYSIS.

By LILY G. KOLLOCK AND EDGAR F. SMITH.

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(FIRST PAPER.)

SEVERAL investigations made in this laboratory have shown that when in electro-analysis the anode is rotated, high currents can be used and metals be precipitated completely in very short periods of time; further, by the use of mercury cathodes most interesting determinations and separations of metals are possible.<sup>1</sup> In the latter case, however, the anode has been stationary, and the electrolyte consequently not agitated. Then, of course, the precipitation of the metal has been comparatively slow. Observing the splendid results obtained with the rotating anode, when platinum was the cathode, we determined to use a combination of rotating anode and mercury cathode. This was accordingly done, and in some preliminary trials made last August (1904), the results of which were briefly alluded to in a communication published in this Journal, 26, 1614, mention was made that 0.4810 gram of copper could be precipitated in twenty-five minutes, and that this success could be had with other metals. Since then we have made additional experiments which we desire to record here. Not only is the time factor reduced for the metals studied, but the plan of combining a mercury cathode with the rotating anode

<sup>1</sup> This Journal, 25, 884; 26, 1124.

gives an inexpensive form of apparatus which will eliminate the platinum dish, cone or cylinder from electro-analysis and thus remove an expensive factor.

#### APPARATUS.

The decomposition cell is a tube 3.5 cm. in diameter and 7.5 cm. in height, made from a test-tube. Soften the bottom of the tube in a blast-lamp flame, then push through it a platinum wire 2 cm. in length, so that its end projects 0.5 cm. into the tube. Flatten the bottom of the tube on an asbestos plate and anneal it in the ordinary way.

The anode, 7.5 cm. in length, is made from platinum wire 1 mm. in diameter, coiled into a flat spiral 1.5 cm. in diameter. It is inserted in a chuck carried by the rotator which is also provided with three pulleys varying from 2 to 5 cm. in diameter. These pulleys are connected by a belt to two pulleys on the motor. With this arrangement the rotation of the anode could be varied from 100 to 1800 revolutions per minute. During the decomposition an amperemeter, a voltmeter and a rheostat, allowing of resistance from 0.1 to 100 ohms, were kept in the circuit.

The precautions indicated by Myers in his paper with regard to the decomposition cell were observed. If care be taken to have the cell as clean as possible, there will be no trouble experienced with the amalgam subsequently adhering to its sides. The mercury, before using, should be washed with alcohol and ether, and after the odor of the latter has disappeared, be placed in the desiccator until it is weighed. It was generally allowed to remain for about five minutes on the balance pan before taking the final weight. In practice a beaker containing a large quantity of mercury, so prepared, should be kept in the desiccator ready for use. The mass of the mercury taken in a single experiment varied from 40 to 50 grams. This was frequently used for two or three determinations, except in the case of chromium, where it was found advisable to use it but once. The cathode surface in the first experiments upon zinc was 3.5 sq. cm., but throughout the rest of the work it was about 9 sq. cm. After weighing the decomposition cell and mercury the solution to be electrolyzed should be introduced. The volume of the electrolyte is always recorded in the accompanying tables. The cell should then be placed upon the copper plate and the anode lowered into the solution. The distance between the

cathode and anode depended upon the volume of the electrolyte. When the volume was 5 cc. the electrodes were 0.5 cm. apart and in other instances 1 cm. was their distance apart. The difference did not appear to materially affect the rate of deposition. The tube should be covered. The anode should next be rotated and the connection made with the required number of chloride accumulator cells. The speed of the anode was varied either by using less current for the motor or by changing the combination of pulleys. With the higher currents recorded, the solution was frequently heated to boiling. When this occurred the current invariably dropped sometimes as much as 1 ampere. But upon washing down the cover-glasses with cold water, it rose to its former strength. The dropping of the current is probably due to the accumulation of steam bubbles upon the electrodes. During the electrolysis some of the solution will, of course, be carried to the sides of the containing vessel and to the cover-glasses by the escaping gases or by the agitation of the liquid. After many trials it was found that it is unnecessary to wash down this portion when the higher currents are used. The condensed steam continually frees the sides from the solution. The cover-glasses may now and then be tilted against the sides of the tube in order to run off the water which collects in large drops.

It has been repeatedly observed in the present work that the greater the concentration of the electrolyte, the greater the rapidity of deposition, but the last traces of metal were always difficult to remove. For this reason, after a solution had become colorless, the electrolytic action was continued several minutes in order to precipitate the minute amount remaining unprecipitated. It is, therefore, also important to have the volume small toward the end of the decomposition.

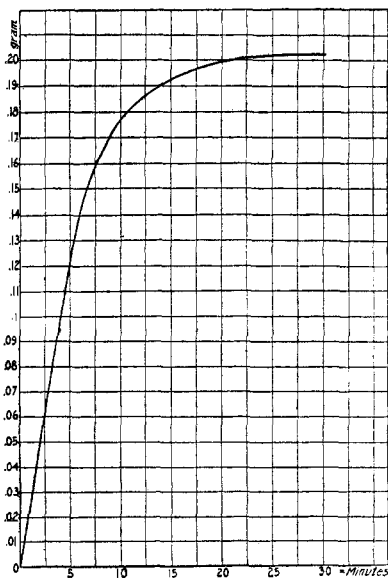
When the metal has been completely deposited, the anode should be stopped, the cover-glasses removed and the decomposition cell filled with distilled water. This should then be siphoned off to the level of the spiral and the liquid replaced by distilled water until the current drops to zero. This wash-water should always be put aside and tested in order to ascertain that the metal has been completely deposited. The current should next be interrupted and the tube removed and washed again with distilled water, inclining and twirling the cell in order to more completely wash the amalgam. As much of the water as possible should be

poured from the cell and the amalgam then be washed twice with absolute alcohol and twice with ether. It should be wiped dry on the outside and after the volatilization of the ether be placed in the desiccator and weighed as previously described.

### EXPERIMENTAL PART.

#### ZINC.

The first experiments made after those described in this Journal, 26, 1614, were upon zinc sulphate. They were conducted in order to ascertain the rate of deposition with varying concentration, current strength, electromotive force, speed of anode and how the quantity of metal in the mercury affected the subsequent rate of deposition. The solution for the first experiments contained 0.2025 gram of metallic zinc in 10 cc. This was determined by the electrolytic method, depositing it upon a plati-



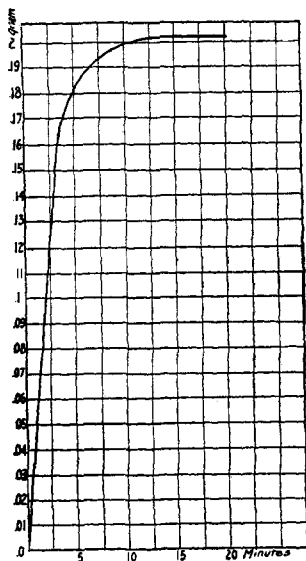
Curve 1. Zinc—1 ampere, 5 volts.

num dish from an ammonium acetate electrolyte. The speed of the anode was 400 revolutions per minute. The current strength was 1 ampere and the electromotive force was 5 volts. The volume of the zinc sulphate solution equaled 15 cc., the current acted thirty minutes. The solution siphoned from the tube

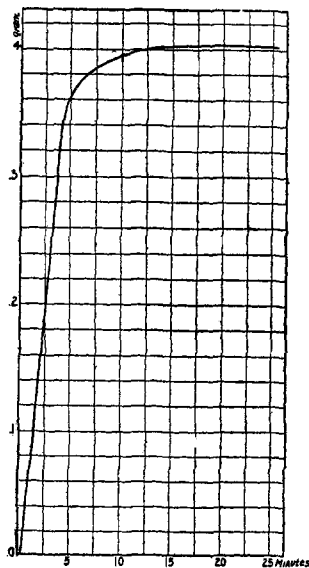
showed no trace of zinc. Consecutive experiments so conducted gave the following results in twenty-five minutes: 0.2027, 0.2030, 0.2025, 0.2025, 0.2021, 0.2027, 0.2025 gram. Two trials were made with the same conditions but using a volume of 10 cc. instead of 15 cc. It was found that the zinc was completely separated in twenty minutes.

Experiments were then made to determine the rate of deposition in successive periods of time and the curve constructed from the data thus obtained, using periods of time for abscissas and masses for ordinates. The conditions employed were those given above. The results were as follows: In five minutes, 0.1196 gram; in ten minutes, 0.1774 gram; in fifteen minutes, 0.1897 gram; in twenty minutes, 0.2002 gram; in twenty-five minutes, 0.2027 gram.

Upon employing a current of 2 amperes, adding sulphuric acid to increase the conductivity, the entire amount was deposited in



Curve 2. Zinc—2 amperes, 7 volts.



Curve 3. Zinc—2 amperes, 6 volts.

fifteen minutes. The following conditions were employed: Total volume, 15 cc.; sulphuric acid, 0.4 cc.; current strength, 2 amperes; pressure, 7 volts; speed of anode, 500 revolutions per minute.

In five minutes, 0.1860 gram of zinc was deposited; in ten minutes, 0.1998 gram; in fifteen minutes, 0.2020 gram.

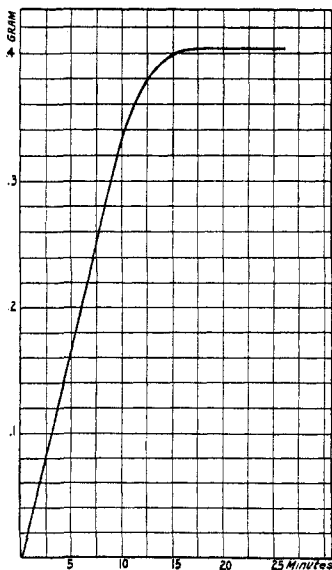
Double the quantity of zinc mentioned above was dissolved in 15 cc. To this was added 0.25 cc. of concentrated sulphuric acid, the anode was rotated at the rate of 800 revolutions per minute, and the solution electrolyzed. In thirty minutes the zinc was completely deposited, using a current of 1.5 amperes and 10 volts,

In ten minutes, 0.3701 gram was deposited; in fifteen minutes, 0.3997 gram; in twenty minutes, 0.4011 gram; in thirty minutes, 0.4058 gram.

Curve 3 was drawn from these results.

The same mass of zinc in 20 cc. was electrolyzed with a current of 2 amperes and 6 volts, other conditions being identical.

In ten minutes, 0.3352 gram was deposited; in fifteen minutes, 0.4010 gram; in twenty minutes, 0.4030 gram; in thirty minutes, 0.4050 gram.



Curve 4. Zinc—1.5 amperes, 10 volts.

Curve 4 was drawn from these results. A comparison of the third and fourth curves shows the effect of greater dilution upon the quantity of zinc deposited in the first ten minutes.

Two experiments were made to learn the effect of different

speeds of the anode upon the rate of precipitation. It was found that the amount of zinc deposited under a rotation of 440 revolutions per minute, and 1,000 revolutions per minute was only 0.0004, which is within experimental error, showing that between these limits there is no apparent effect. It was also discovered that when more than 1 gram of zinc was present in the mercury, the latter should not be further used, if it is desired to obtain results in the shortest period.

TABLE I. ZINC.

Experiment.	Zinc present. Gram.	Sulphuric acid present. cc.	Volume. cc.	Current. Amperes.	Volts.	Revolutions of anode per minute.	Time. Minutes.	Zinc found. Gram.	Error. Gram.
1	0.2025	0.0	15	1	7	750	30	0.2027	+0.0002
2	0.2025	0.0	15	1	7	750	25	0.2030	+0.0005
3	0.2025	0.0	15	1	7	750	25	0.2015	-0.001
4	0.2025	0.0	15	1	7	750	25	0.2020	-0.0005
5	0.2025	0.0	15	1	7	750	25	0.2025	...
6	0.2025	0.0	10	2	7	750	25	0.2024	-0.0001
7	0.2025	0.25	10	2	7	750	30	0.2027	+0.0002
8	0.4050	0.25	20	1.5	6	750	45	0.2054	+0.0004
9	0.2025	0.25	10	1	5	750	25	0.2025	...
10	0.2025	0.25	10	1	5	750	25	0.2029	+0.0004
11	0.2025	0.25	15	1	5	750	25	0.2025	...
12	0.2025	0.25	15	1	5	750	20	0.2027	+0.0002
13	0.2025	0.25	15	2	6	750	15	0.2030	+0.0005
14	0.2025	0.25	15	2	6	750	15	0.2020	-0.0005
15	0.2025	0.25	15	2	6	750	15	0.2021	-0.0004
16	0.4050	0.25	15	5	8	1400	6	0.4057	+0.0007
17	0.4050	0.25	15	5	8	480	6	0.4045	-0.0005
18	0.4050	0.25	15	5-6	7-5	480	8	0.4042	-0.0008
19	0.4050	0.25	10	5	7	640	5	0.4050	...

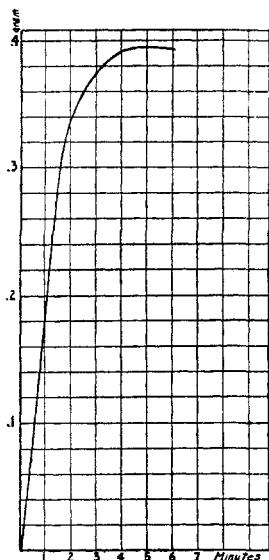
To 10 cc. of the zinc sulphate solution 0.4 cc. of concentrated sulphuric acid was added, after which it was electrolyzed by a current of 5 amperes and 7 volts, the speed of the anode being 640 revolutions per minute. Under these conditions 0.405 gram of zinc was precipitated in five minutes.

## COPPER.

Having found that 0.405 gram of zinc could be deposited in from five to eight minutes it was decided to try other conditions upon copper than those recorded in the previous paper, in order

to reduce the time factor. By using higher currents and greater concentration of the electrolyte this was accomplished.

A solution of copper sulphate containing 0.3945 gram of metallic copper in 5 cc. was used for these experiments. This quantity of metal was precipitated finally in five minutes. The solution became colorless in three minutes. Twice this quantity (0.789



Curve 5. Copper—5 amperes, 6 volts.

gram) was deposited in ten minutes, although the solution had become colorless at the expiration of seven minutes. The volume in this case being 10 cc. it appeared that the last traces of copper required more time for precipitation. A current of 5 amperes and 6 volts was used, sulphuric acid being introduced to increase the conductivity.

The current strength recorded in the following table was maintained during the greater part of the electrolysis. When it showed a tendency to rise, on the liberation of the acid, additional resistance was thrown into the circuit. The following rates of deposition of copper were determined under the preceding conditions. The anode made 640 revolutions per minute. In one minute, 0.1800 gram of copper was deposited; in two minutes, 0.3400 gram; in three minutes, 0.3664 gram; in four minutes, 0.3945 gram; in five minutes, 0.3945 gram.



TABLE II. COPPER.

Experiment.	Copper present. Gram.	Sulphuric acid present. cc.	Volume. cc.	Current. Amperes.	Volts.	Revolutions of anode per minute.	Time. Minutes.	Copper found. Gram.	Error. Gram.
1	0.7890	0.25	12	3.5	6	1200	10	0.7900	+0.001
2	0.3945	0.15	12	4	6	1080	5	0.3941	-0.0004
3	0.3945	0.25	12	3.5	6	1200	6	0.3942	-0.0003
4	0.3945	0.15	12	5	6.5	1200	5	0.3944	-0.0001
5	0.3945	0.0	10	2-4	9-7	1200	6	0.3946	+0.0001
6	0.3945	0.17	10	3.5	8.5	1200	4	0.3944	-0.0001
7	0.3945	0.17	10	4	6	1080	5	0.3946	+0.0001

## NICKEL.

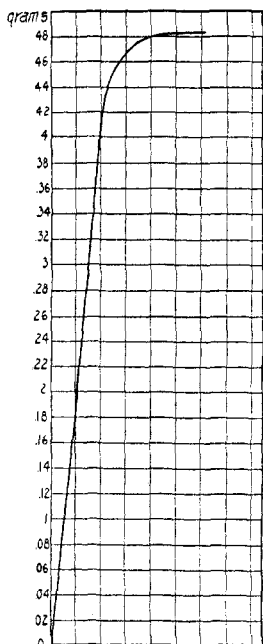
A nickel sulphate solution containing 0.4802 gram of metal in 10 cc. was used in the following experiments, and after finding that this quantity was completely deposited in the mercury in twenty minutes with a current of 2 amperes and 7 volts, the rate of deposition in succeeding periods of time was determined with a current of 2.5 amperes and 6 volts. In two and a half minutes, 0.2017 gram of nickel was deposited; in seven and a half minutes, 0.4095 gram; in ten minutes, 0.4651 gram; in twelve and a half minutes, 0.4774 gram; in fifteen minutes, 0.4802 gram.

TABLE III. NICKEL.

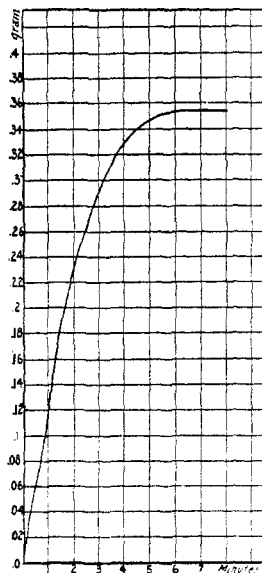
Experiment.	Nickel present. Gram.	Sulphuric acid. cc.	Volume. cc.	Current. Amperes.	Volts.	Revolutions of anode per minute.	Time. Minutes.	Nickel found. Gram.	Error. Gram.
1	0.4802	0.25	18	2	7	600	18	0.4802	...
2	0.4802	0.25	12	3.5	7	600	16	0.4799	-0.0003
3	0.4802	0.25	12	2-4	6.5	600	10	0.4806	+0.0004
4	0.4802	0.25	12	6	5	500	7	0.4804	+0.0002
5	0.4802	0.25	12	5	6.5	600	10	0.4796	-0.0006
6	0.9604	0.25	10-30	4	6	1100	10	0.9597	-0.0007
7	0.4802	0.25	12	3	7-5	1100	10	0.4806	+0.0004
8	0.4802	0.25	12	3	7	1100	10	0.4796	-0.0006
9	0.9604	0.25	12	3.5	7	1100	16	0.9604	...
10	0.4802	0.25	12	5	7	640	12	0.4809	+0.0007
11	0.4802	0.25	12	5	6	880	8	0.4806	+0.0004
12	0.4802	0.25	7	6	5	1200	9	0.4801	-0.0001
13	0.4802	0.25	7	6	6	1200	7	0.4801	-0.0001

On employing a current of 6 amperes and a pressure of 5 volts, the solution became colorless in four minutes. Not a trace of

nickel was found in the liquid after seven minutes. The amalgam was very bright and of the consistency of soft dough, when 1 gram of nickel was combined with the usual quantity of mercury (40 grams).



Curve 6. Nickel—2.5 amperes, 6 volts.



Curve 7. Cobalt—5 amperes, 5 volts.

#### COBALT.

This metal does not appear to enter the mercury with the same rapidity as nickel under similar conditions. The last minute traces are more difficult to remove. Various conditions were used. When no sulphuric acid was added the current was at first low, but it rapidly rose as the decomposition proceeded. The conditions, giving the total cobalt in the least time, were the following: 10 cc. of solution, containing 0.3535 gram of cobalt; 0.25 cc. of sulphuric acid and a current of 5 amperes with a pressure of 6 volts. The speed of the anode was 1,200 revolutions per minute. The solution became colorless in seven minutes, but ten minutes appeared to be necessary for the removal of the last traces of the metal. On using the same amount of cobalt in a volume of 5 cc., other conditions remaining unchanged, all of the metal sep-

arated in seven minutes, thus: In one minute, 0.1197 gram of cobalt was deposited; in three minutes, 0.2930 gram; in five minutes, 0.3300 gram; in six minutes, 0.3520 gram; in seven minutes, 0.3535 gram; in ten minutes, 0.3530 gram.

The curve (7) was constructed from these results.

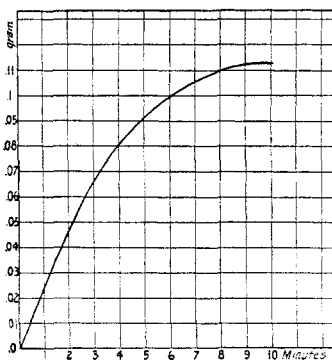
TABLE IV.—COBALT.

Experiment.	Cobalt present. Gram.	Sulphuric acid present. cc.	Volume. cc.	Current. Amperes.	Volts.	Revolutions of anode per minute.	Time, Minutes.	Cobalt found. Gram.	Error. Gram.
1	0.3525	0.35	15	5	7	1250	15	0.3522	—0.0003
2	0.3525	0.25	15	3	5	980	18	0.3524	—0.0001
3	0.3525	0.25	15	4	6	600	14	0.3523	—0.0002
4	0.3525	0.25	10	4	6	860	16	0.3530	+0.0005
5	0.3525	0.5	10	4	6	1000	15	0.3530	+0.0005
6	0.3525	0	10	4	6	1240	16	0.3528	+0.0003
7	0.3525	0.25	10	3	6	1200	10	0.3521	—0.0004
8	0.3525	0.5	10	6	6	1200	10	0.3530	+0.0005
9	0.3525	0.25	10	5	8	800	10	0.3522	—0.0003
10	0.3525	0.25	10	3	8	1400	12	0.3523	—0.0002
11	0.3525	0.5	10	6	5	800	11	0.3530	+0.0005
12	0.7050	0.5	15	6	7	1200	30	0.7052	+0.0002
13	0.1762	0.35	10	4	8	560	7	0.1762	....

## CHROMIUM.

A solution of chromium sulphate was electrolyzed with currents varying from 1 to 4 amperes and 7 to 12 volts, and with a varying quantity of sulphuric acid. It was found by Myers that the addition of the acid was necessary, otherwise, there was a separation of the oxide of chromium throughout the liquid; but too much acid retards or entirely prevents the decomposition. When 10 drops (40 drops = 1 cc.) were added and a current of 2.5 amperes and 6 volts applied, one half hour was necessary to deposit 0.23 gram of chromium. With 0.5 cc. of acid and a current of 5 amperes and 4 volts, the solution at the end of sixty minutes did not appear to have lost its color. Experiments were then made to learn how much chromium, if any, was deposited when the acid was present in large quantity. Thus, with a current of 4 amperes and 7 volts, the solution containing 1 cc. of acid, 0.05 gram of metal was precipitated in forty-five minutes, while with 2 cc. of acid and a current of 1 ampere and 4 volts the mercury showed no increase in weight after thirty minutes. The following results, ob-

tained with the use of smaller amounts of acid, confirm this. By adding 10 drops of acid ( $=0.25$  cc.) and employing a current of 4 amperes and 7 volts, the liquid became colorless in thirty minutes, but forty minutes were necessary for the complete removal of the metal. With the same quantity of acid, and a current of 5 amperes and 8 volts, the chromium was completely precipitated in thirty minutes. With five drops of acid and a current of 3 to 4.5 amperes and 8 volts, the solution became colorless in eleven minutes. It, therefore, seems that more than three drops of acid are sufficient to materially affect the rate of precipitation. More than two drops of acid must be present to prevent the separation of chromic oxide



Curve 8. Chromium—3.5 amperes, 11-10 volts.

which always took place with less than that amount of acid. The following conditions gave the most rapid determination: A volume of the solution, containing 0.1190 gram of chromium and three drops of sulphuric acid (40 drops = 1 cc.), was electrolyzed with a current of 4 to 5 amperes and 6 volts, the speed of the anode being 400 revolutions per minute. In four minutes the solution was colorless and in six minutes the chromium was found to be completely deposited. The solution was siphoned off in the manner previously described, but after the cell was removed anhydrous alcohol was poured in as quickly as possible and the operation repeated twice and followed by two washings with ether in order to prevent, if possible, oxidation of the chromium. Oxidation, if it occurred, was but slight, for the error never exceeded 0.0007 gram.

Curve 8 was constructed from the results given below: In two minutes, 0.048 gram of chromium was deposited; in four minutes,

0.085 gram; in six minutes, 0.1000 gram; in eight minutes, 0.1105 gram; in nine minutes, 0.1185 gram; in ten minutes, 0.1185 gram.

TABLE V.—CHROMIUM.

Experiment.	Chromium present. Gram.	Sulphuric acid (40 drops = 1 cc.).	Volume. cc.	Current. Amperes.	Volts.	Revolutions of anode per minute.	Time. Minutes.	Chromium found. Gram.	Error. Gram.
1	0.1180	5	10-15	3-4	7	280	15	0.1186	+0.0006
2	0.1180	3	10-15	2-4	11-9	280	15	0.1187	+0.0007
3	0.1180	3	10-15	1-3	9	640	20	0.1185	+0.0005
4	0.1180	3	8-15	1.5-3	10-8	220	15	0.1186	+0.0006
5	0.1180	3	10-15	1-3	11-9	520	20	0.1186	+0.0006
6	0.1180	3	5-15	1-2	11-9	640	17	0.1175	-0.0005
7	0.1180	3	5-15	2-4	9-8	480	15	0.1180	....
8	0.2360	3	5-15	2.5	10	520	50	0.2355	-0.0005
9	0.1180	5	5-15	3	7.5	400	15	0.1179	-0.0001
10	0.1180	3	7-15	4-5	8	640	6	0.1175	-0.0005
11	0.1180	3	7-15	3-4	10-9	640	10	0.1180	....
12	0.1180	7	7-15	3-4	10-8	200	13	0.1187	+0.0007
13	0.1180	3	5-15	3.5	8	640	11	0.1177	-0.0003
14	0.2360	4	5-15	3	12	640	35	0.2359	-0.0001
15	0.1180	3	5-15	3-4	10-8	320	11	0.1179	-0.0001
16	0.1180	3	5-15	3-4	10	540	11	0.1182	+0.0002

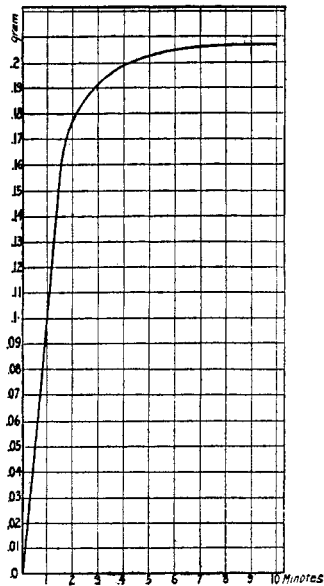
## IRON.

In experimenting with salts of this metal it was soon discovered that sulphuric acid in large amount retarded its precipitation. It was also noticed when higher currents were used that the solution became very hot and assumed a decidedly pink color,<sup>1</sup> which disappeared on the addition of cold water or when the cover-glasses were removed, allowing the steam to escape rapidly and thus decreasing the pressure and consequently the temperature of the boiling solution. The color reappeared a few seconds after the cover-glasses were replaced.

The conditions which gave the most satisfactory results were as follows: Volume of solution 5 cc. containing 0.275 gram of metallic iron, 3 drops of concentrated sulphuric acid and a current of 3 to 4 amperes and 7 volts. The rotation of the anode varied from 520-920 revolutions per minute. The iron was completely deposited in seven minutes. The following observations on rate of deposition were made under the conditions just given: In two minutes, 0.1760 gram of iron was deposited; in four minutes,

<sup>1</sup> Due to traces of manganese.

0.2000 gram; in six minutes, 0.2050 gram; in eight minutes, 0.2075 gram.



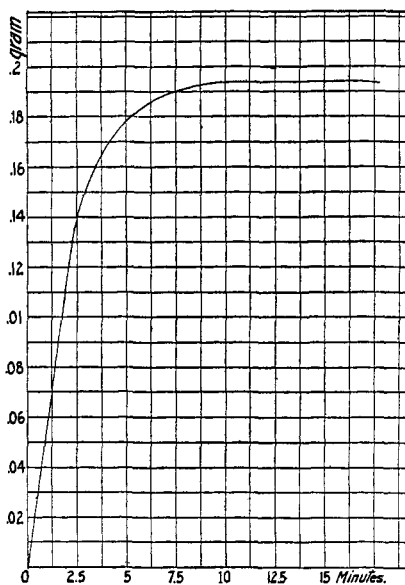
Curve 9. Iron—3.5 amperes, 7 volts.

In addition to the results just described a solution of ferrous sulphate containing 0.1945 gram of iron in 10 cc. was used to get further working conditions. By using a current of 3.5 amperes and 10–9 volts, with about 900 revolutions per minute of the anode, the total iron content was deposited in fifteen minutes. The

TABLE VI.—IRON.

Experiment.	Iron present. Gram.	Sulphuric acid in drops (40 drops=1 cc.).	Volume. cc.	Current. Amperes.	Volts.	Revolutions of anode per minute.	Time. Minutes.	Iron found. Gram.	Error. Gram.
1	0.2075	7	5	4.5	8-7	520	14	0.2072	-0.0003
2	0.2075	4	5-15	5.4	6.5-5	680	14	0.2078	+0.0003
3	0.2075	5	5-10	3.2-4	6.5	680	15	0.2077	-0.0003
4	0.2075	3	5	2-2.5	7-6	680	15	0.2073	-0.0002
5	0.2075	3	5	4	6-5	680	10	0.2080	+0.0005
6	0.2075	3	5	3-4.5	7-6	920	7	0.2078	+0.0003
7	0.2075	3	5	2-3	6	740	9	0.2076	+0.0001
8	0.2075	3	5	2-4	6.5-5.5	700	9	0.2076	+0.0001

residue from the decomposition cell, when oxidized by nitric acid and tested with potassium sulphocyanide, gave no color. The experiments made to determine the rate of deposition with a lower current (1-2.5 amperes and 10-9 volts), while the other conditions



Curve 10. Iron—1 to 2.5 amperes, 10-9 volts.

remained as above, gave the following results which appear in Curve 10.

In two and a half minutes, 0.1141 gram of iron was deposited; in five minutes, 0.1787 gram; in seven and a half minutes, 0.1945 gram; in ten minutes, 0.1950 gram.

When the residue was tested with potassium sulphocyanide no iron was detected. By the addition of 3 drops (40 drops = 1 cc.) of sulphuric acid and using a higher current (3.5 amperes) in ten minutes a faint reaction for iron was observed, indicating that the acid has some retarding influence. In fifteen minutes under these conditions the iron had completely separated. By using a higher current, 3 amperes and 9 volts, under the same condition, all the iron was deposited in ten minutes.