2.4 to 3 volts. In two hours there was deposited (1) 0.1272 gram, and (2) 0.1277 gram of gold.

To the auric chloride were added 50 cc. of water, then the sodium sulphide and the solution warmed carefully until it became clear, and finally diluted to 150 cc. and electrolyzed. The deposited gold was perfectly adherent, and of a shining yellow color, appearing like polished metal.

The separation of gold from arsenic, molybdenum and tungsten may be effected by observing the conditions given for the determination of gold from its sodium sulphide solution.

Separations of gold from mercury and tin were not effected.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, No. 86.]

THE USE OF A ROTATING ANODE IN THE ELECTROLYTIC ESTIMATION OF ZINC.[1]

BY LESLIE HOWARD INGHAM.

Received July 26, 1904.

THE purpose of the present investigation was to ascertain how rapidly the metal zinc could be electrolytically precipitated with the aid of a rotating anode.

The electrolytes employed were solutions of zinc in the presence of sodium hydroxide, sodium acetate. sodium formate and potassium cyanide.

The rate of precipitation of zinc from each of the electrolytes was also studied, after which experiments were instituted to discover how quickly the content of metal in zinc blende could be estimated. It is believed that these experiments indicate which electrolyte is preferable in each case, and demonstrate conclusively that the electrolytic procedure for the estimation of zinc is preferable to the ordinary gravimetric or volumetric methods.

The Apparatus.—The depositions were made upon platinum dishes of the usual form. The anode was a platinum wire 1 mm. in diameter, bent into a spiral 2 inches in diameter. The plane

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[1] From the author's thesis presented to the University of Pennsylvania for the Ph.D. degree.

of the spiral was nearly flat, but slightly depressed in the center in dish-form to conform approximately to the figure which the surface of the liquid assumes in the dish while agitated.

The anode was rotated by a small electric motor made by the Elektrochemische Werkstätte in Darmstadt, which received its current from the 110-volt lightning circuit by means of the usual drop-cord and plug. The motor rotated at about 1500 turns per minute and was provided with a three-step cone-pulley. A corresponding three-step pulley on the vertical shaft, which carried the anode, permitted the latter to be rotated at three different speeds approximating 230, 560 and 1750 revolutions per minute. The belt was usually run on the smallest pulley of the armature and the largest of the shaft, thus giving the anode the slowest speed. This speed is designated as I, the faster speeds being II and III. The latter was never used in the precipitation of zinc.

The amount of liquid in the dish was usually about 125 cc. As the agitation of the electrolyte caused the level of the liquid to rise at the walls of the dish, the amount of cathode surface exposed varied constantly. The figures given for current strength will, in every case, be those read directly from the ammeter, but since the cathode surface was kept as near 100 sq. cm. as was possible under the circumstances, these amperages may be considered as equivalent to those implied in the expression $N.D_{100} =$ ampere.

(A) SODIUM ACETATE ELECTROLYTE.

The conditions for the precipitation of zinc from an acetate electrolyte, as given by Exner,[1] were conformed to in the following experiments: The platinum dishes were first coated electrolytically with silver and this deposit washed with water and alcohol, ignited and weighed. The zinc solution was then pipetted into the dish, the desired reagents added and the solution heated nearly to boiling while being held over a free flame in nickel forceps. Heat was not applied during electrolysis, as the electrical energy expended was sufficient to keep the liquid near its boiling-point. The lack of agreement between the theoretical and required amount of zinc was not thought remarkable, since the specimen of zinc sulphate used was poorly crystallized and appeared to have been formed from a hot supersaturated solution.

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[1] This Journal, 25, 896.

The following results were obtained:

Zn. taken. Gram.	$NaC_2H_3O_2$. Grams.	$HC_2H_3O_2$ (30 per ct.). Drops.	Amperes.	Volts.	Time. Minutes.	Speed.	Zn. found. Gram.	Expt. No.
0.2503	3	4	4	12	10	I	0.2615	1
0.2503	3	4	4	14	10	II	0.2614	2
0.2503	3	4	4	14	10	II	0.2617	3
0.2503	3	4	4	14	10	II	0.2619	4
0.2503	3	6	4	17	15	II	0.2624	5
0.5005	3	6	4	12	15	I	0.5210	6
0.5005	3	4	4	13	15	I	0.5217	7
0.5005	3	6	4	12	15	I	0.5191	8
0.5005	3	6	4	12	15	I	0.5210	9
0.5005	3	6	4	17	15	I	0.5221	10
0.5005	3	6	4	17	15	II	0.5204	11
0.5005	3	6	4	17	15	II	0.5204	12
0.3504	3	6	4	11	15	I	0.3662	13
0.3504	3	6	4	11	15	I	0.3665	14
0.3504	3	6	4	11	15	I	0.3660	15
0.3504	3	6	4	11	15	I	0.3658	16

After the deposit was weighed the zinc and silver were removed together by heating gently with nitric acid (sp. gr. 1.2). The gray deposit dissolved very readily, leaving the platinum dish clean and bright. This was then resilvered and weighed as described above. This mode of cleaning the silver and zinc from the dish was subsequently found to be unnecessary, however, as a dilute solution of sulphuric acid will remove the zinc and leave the silver practically unattacked. Although this was not discovered until later, it will be convenient to describe here the manipulation which was finally adopted. After the dish with its deposit was weighed it was filled to the brim with sulphuric acid previously diluted with about fifty times its volume of water; the dish was then set aside until visible action ceased. The solution was then poured into a beaker and the dish rinsed with water and heated to a faint redness over a free flame while held in nickel forceps. It was then cooled under the faucet and filled a second time with the dilute acid, rinsed after a few moments, heated as before and given a third treatment with the same acid. After the weak acid had been poured away the dish was given a final rinsing in clean water, wiped dry externally and ignited, cooled in a desiccator and weighed. By this treatment the dish lost only about

0.5 mg. of silver each time it was used and one coat of silver sufficed for over a hundred determinations of zinc. The entire time spent in cleaning the dish need not exceed six minutes. Some annoyance was experienced at the outset in the form of high results and a brown anodic stain, which was found to be due to lead acetate in the sodium acetate, and entirely disappeared upon the removal of the lead. All the deposits from a sodium acetate electrolyte consisted of exceedingly minute flat crystals, gray in color and very adherent.

(B) SODIUM HYDROXIDE ELECTROLYTE.

The conditions given by Exner for the deposition of zinc from a solution of sodium zincate in the presence of a large excess of alkali were verified in the following experiments:

For each experiment 25 cc. of solution (= 0.2490 gram of zinc) were taken, together with the stated amount of sodium hydroxide in stick form. After all was dissolved the liquid was washed into a platinum dish which had been previously coated with silver and weighed, diluted to about 125 cc., then electrolyzed with the current and pressure given in the tabular exhibit below. At the expiration of the stated time the liquid in the dish was siphoned off and replaced by cold water and the circuit interrupted. The following are among the results obtained:

abt 8% err.

Zinc taken. Gram.	NaOH. Grams.	Amperes.	Volts.	Time. Minutes.	Zinc found. Gram.	No.	K_2SO_4. Gram.
0.2490	8	5	5	15	0.2481 -9	1	0.0
0.2490	8	5	5	15	0.2476 -14	2	0.0
0.2490	8	5	5	20	0.2492 +2	3	0.0
0.2490	8	5	6	15	0.2489 -1	4	0.0
0.2490	8	5	6	15	0.2487 -3	5	0.0
0.2490	8	5	6	15	0.2492 +2	6	0.0
0.2490	8	5	6	15	0.2492 +2	7	0.0
0.2490	8	5	6	15	0.2489 -1	8	equivalent
0.2490	8	5	6	15	0.2488 -2	9	"
0.2490	8	5	6	15	0.2494 +4	10	"
0.2490	8	5	6	15	0.2492 +2	11	"

In Experiments 8 to 11 sufficient potassium sulphate was added to form an equivalent with the zinc sulphate in the proportion $ZnSO_4.K_2SO_4$. This addition presents no advantage.

Blank experiments made with sodium hydroxide solution yielded 0.0000 and 0.0001 gram. The deposits of zinc obtained from the sodium hydroxide solution were very adherent, smooth, hard, gray in color, and so homogeneous that crystalline structure could not be detected by the unaided eye.

Experiments were conducted to ascertain the amount of sodium hydroxide needed for the best results. In each experiment 25 cc. of the zinc solution were used; to precipitate and redissolve the metal present required about 1 gram of sodium hydroxide, but more should be employed in order to increase the conductivity of the solution.

The results were as follows:

Zinc taken. Gram.	NaOH. Grams.	Amperes.	Volts.	Time. Minutes.	Zinc found. Gram.	No.
0.2490	2	5	10	20	0.2490	1
0.2490	3	5	8½	20	0.2489	2
0.2490	3	5	8½	20	0.2490	3
0.2490	4	5	6	20	0.2485	4
0.2490	4	5	6	20	0.2489	5
0.2490	5	5	5	20	0.2489	6
0.2490	5	5	5	20	0.2489	7
0.2490	6	5	5	20	0.2489	8
0.2490	6	5	5	20	0.2489	9
0.2490	7	5	5	20	0.2490	10
0.2490	7	5	5	20	0.2490	11
0.2490	10	5	5	20	0.2493	12
0.2490	12	5	4½	20	0.2490	13
0.2490	6	10	8	10	0.2472	14
0.2490	6	5	5	10	0.2480	15

It appears that the amount of sodium hydroxide used may vary within quite wide limits. The last two observations show that the time cannot well be reduced as low as ten minutes even by the use of a current as great as 10 amperes. Portions of the same zinc salt solution were also electrolyzed in the presence of sodium acetate, employing the conditions mentioned under that electrolyte in (A). The results were 0.2488 gram, 0.2491 gram, 0.2487 gram, 0.2486 gram, 0.2490 gram, agreeing excellently with those obtained in the sodium hydroxide electrolyte.

(C) SODIUM FORMATE ELECTROLYTE.

Twenty-five cc. of the zinc sulphate solution were pipetted into the silvered platinum dish and 0.5 gram of sodium carbonate, dissolved in a little water, was added. Formic acid was run in until all the carbonate was dissolved and a few drops of acid remained in excess. This solution was then diluted to 125 cc., as usual, warmed and electrolyzed. The first experiments showed that this electrolyte possessed a resistance so high that about 25 volts were required to produce a current of 3.5 amperes, and the heat evolved was excessive. The addition of increased amounts of sodium formate obviated this trouble. The preferable conditions are those of Experiments 5 to 8.

Zinc taken. Gram.	Na_2CO_3. Grams.	H.COOH (sp. gr. 1.22). cc.	Amperes.	Volts.	Time. Minutes.	Zinc found. Gram.	No.
0.2490	0.4	3.0	3½	23	10	0.2447	1
0.2490	0.6	3.5	4	20	10	0.2460	2
0.2490	2.5	3.5	5	10	20	0.2478	3
0.2490	2.5	3.5	5	10	20	0.2482	4
0.2490	5.0	4.6	5	8	20	0.2489	5
0.2490	5.0	4.6	5	8	20	0.2486	6
0.2490	5.0	4.6	5	8	20	0.2490	7
0.2490	5.0	4.6	5	8	20	0.2490	8

The zinc deposits were bluish gray in color and resembled those from the sodium hydroxide electrolyte, being fine-grained and very adherent.

(D) POTASSIUM CYANIDE ELECTROLYTE.

The results obtained with this electrolyte were so unsatisfactory that it was abandoned.

THE RATE OF PRECIPITATION OF ZINC FROM THE VARIOUS ELECTROLYTES.

These experiments were conducted precisely as those already described, save that at the end of the allotted time the liquid in the dish was very hastily siphoned off and the circuit interrupted as promptly as possible. Since the metal contained in the solution was not all deposited, it was impossible to allow much time for washing while the current was passing, nor, on the other hand, was it proper to interrupt the circuit at the instant of expiration of the desired time, thus leaving the acid liquor in contact with the deposits. A compromise was effected by the use of a large siphon which emptied the dish in a few seconds, the current being reduced to 1 ampere, and the liquid rapidly replaced by distilled water, after which the circuit was opened. The dish was then washed, dried and weighed as usual. The results for the first few minutes can be only approximate at best, as the metal was then being deposited very rapidly and the few seconds occupied in siphoning affected them considerably.

SODIUM FORMATE ELECTROLYTE.

Zinc taken. Gram.	Na_2CO_3. Grams.	H.CO_2H (sp. gr. 1.22). cc.	Amperes.	Volts.	Time. Minutes.	Zinc found. Gram.	No.	Speed.
0.2490	5	4.6	5	10	1	0.0839	1	I
0.2490	5	4.6	5	10	2	0.1418	2	I
0.2490	5	4.6	5	10	3	0.1723	3	I
0.2490	5	4.6	5	10	5	0.2095	4	I
0.2490	5	4.6	5	10	7	0.2244	5	I
0.2490	5	4.6	5	10	10	0.2464	6	I
0.2490	5	4.6	5	10	12	0.2483	7	I
0.2490	5	4.6	5	10	15	0.2490	8	I
0.2490	5	4.6	5	10	20	0.2490	9	I
0.4780	5	4.6	5	17	1	0.0990	10	II
0.4780	5	4.6	5	17	2	0.1721	11	II
0.4780	5	4.6	5	17	3	0.2738	12	II
0.4780	5	4.6	5	17	4	0.3398	13	II
0.4780	5	4.6	5	17	5	0.3916	14	II
0.4780	5	4.6	5	17	7	0.4430	15	II
0.4780	5	4.6	5	17	10	0.4595	16	II
0.4780	5	4.6	5	17	12	0.4638	17	II
0.4780	5	4.6	5	17	15	0.4603	18	II
0.4780	5	4.6	5	7	20	0.4522	19	II
0.4780	5	4.6	5	17-5	10 + 5	0.4688	20	II-o
0.4780	5	4.6	5	17-5	10 + 20	0.4753	21	II-o

With the intermediate speed, II, the pressure needed to produce 5 amperes was about 17 volts; this caused considerable heating and should be avoided if 0.25 gram of zinc or less be present, but when depositing larger quantities of metal the higher speed was preferable, as better deposits resulted. After about twelve minutes at this voltage the solution boiled freely from the heat evolved by the current and a visible re-solution of the metal sometimes took place. It was thought unnecessary to agitate the electrolyte during the entire time of deposition, as the greater part of the metal came down in the course of the first ten or twelve minutes, and the small amount of zinc remaining in solution would not be apt to come down in a spongy condition if the agitation were omitted; moreover, the lessening of the voltage required diminished the heat produced. Accordingly, in the above series of experiments in 20 and 21 the motor was stopped after ten minutes and the voltage lowered to such a value as would maintain the current at 5 amperes, that being the current employed during agitation. It was found that from 17 to 20 volts were needed while the anode was rotated, while 5 volts sufficed after the anode became stationary. The last four observations, noted above, indicate that this suggestion is useful. With a lower speed of rotation the precaution is probably superfluous.

In the last twelve experiments there was used a zinc solution of which 25 cc. contained 0.2390 gram of metal. It was employed also in the two series next following.

SODIUM HYDROXIDE ELECTROLYTE.

Experiments upon the rate of deposition from this electrolyte yielded the following figures:

Zinc taken. Gram.	NaOH. Grams.	Amperes.	Volts.	Time. Minutes.	Zinc found. Gram.	No.	Speed.
0.2490	6	5	8	1	0.0827	1	I
0.2490	6	5	8	2	0.1378	2	I
0.2490	6	5	8	3	0.1991	3	I
0.2490	6	5	8	4	0.2129	4	I
0.2490	6	5	8	5	0.2254	5	I
0.2490	6	5	8	7	0.2466	6	I
0.2490	6	5	8	10	0.2480	7	I
0.2490	6	5	8	15	0.2488	8	I
0.2490	6	5	8	20	0.2490	9	I
0.4780	6	5	6	1	0.1028	10	I
0.4780	6	5	6	2	0.1847	11	I
0.4780	6	5	6	3	0.2921	12	I
0.4780	6	5	6	4	0.3498	13	I
0.4780	6	5	6	5	0.4217	14	I
0.4780	6	5	6	7	0.4691	15	I
0.4780	6	5	6	10	0.4740	16	I
0.4780	6	5	6	12	0.4780	17	I
0.4780	6	5	6	15	0.4774	18	I
0.4780	6	5	6	10 + 20	0.4777	19	I
0.4780	6	5	6	20 + 10	0.4780	20	I

SODIUM ACETATE ELECTROLYTE.

Experiments upon the rate of deposition of zinc from this electrolyte gave the following figures:

Zinc taken. Gram.	$NaC_2H_3O_2$. Grams.	$HC_2H_3O_2$ (30 pct.) Drops.	Amperes.	Volts.	Time. Minutes.	Zinc found. Gram.	No.	Speed.
0.4780	3	6	4	12½	1	0.0933	1	I
0.4780	3	6	4	12½	2	0.1500	2	I
0.4780	3	6	4	12½	3	0.2326	3	I
0.4780	3	6	4	12½	4	0.2957	4	I
0.4780	3	6	4	12½	5	0.3773	5	I
0.4780	3	6	4	12½	7	0.4645	6	I
0.4780	3	6	4	12½	10	0.4736	7	I
0.4780	3	6	4	12½	12	0.4725	8	I
0.4780	3	6	4	12½	15	0.4766	9	I
0.4780	3	6	4	12½	20 + 40	0.4779	10	I

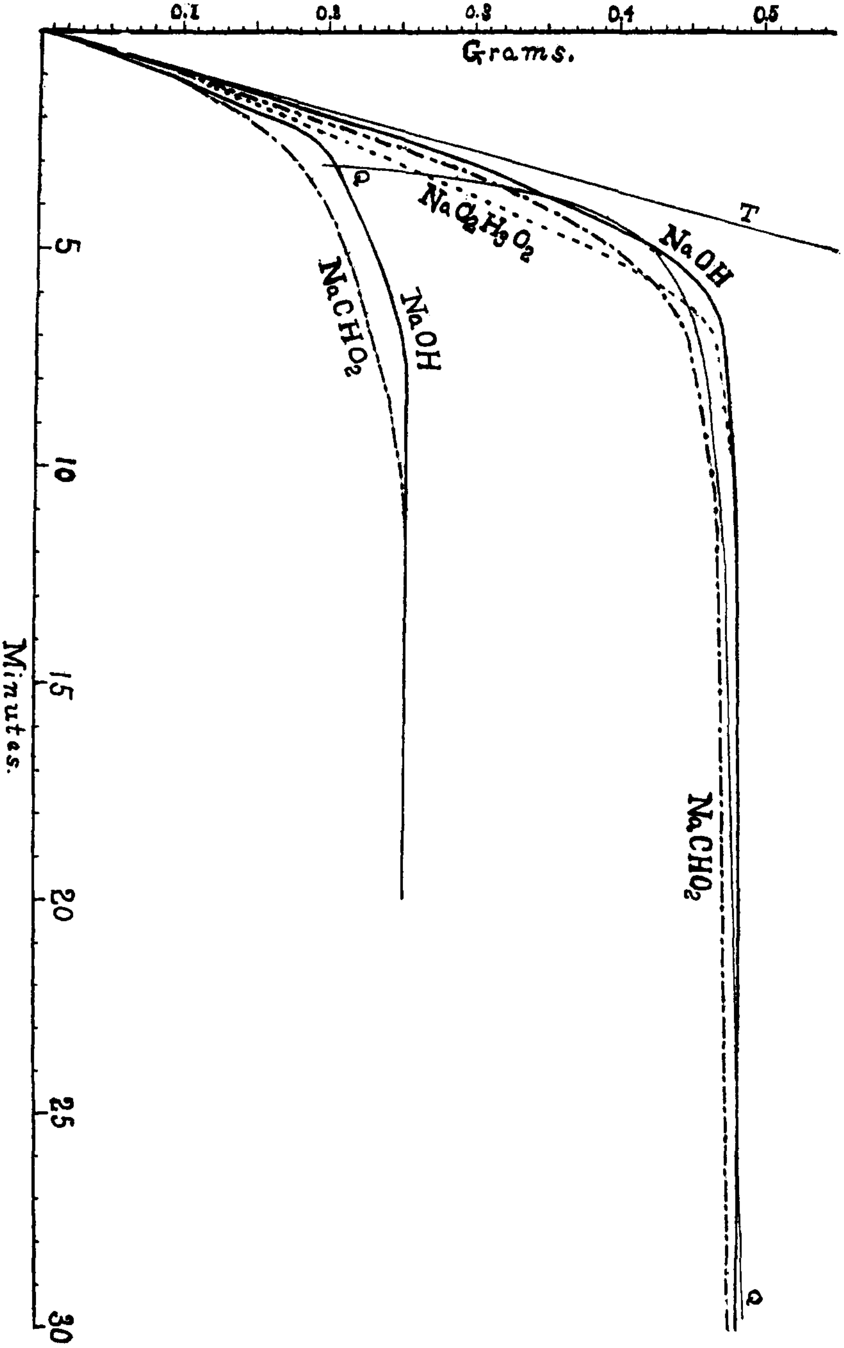
The above series of results may be expressed graphically, the number of minutes being used as the abscissa and the amount of metal deposited as the corresponding ordinate.

It will be noted that the graphs approximate the locus of the curve marked Q...Q, which is of the general form $[x\,y = c]$, an equilateral hyperbola, showing that after the first three minutes the amount of zinc deposited per minute decreases as the time increases. The straight line marked T represents the rate of deposition of zinc from a solution containing an unlimited amount of metal and is calculated from the electrochemical equivalent of zinc, in accordance with Faraday's law. This indicates that in the experiments in which nearly 0.5 gram of zinc was present at the outset, the current was depositing very nearly its maximum amount of metal during the first two minutes. The deposition curves thus appear to approach T at first, and later to approximate Q.

THE ELECTROLYTIC DETERMINATION OF THE ZINC CONTENT OF ZINC BLENDE.

The following experiments were conducted with a sample of very pure blende from Joplin, Missouri.

The mean of concordant gravimetric and volumetric determinations of its zinc content showed the presence of 65.70 per cent. of metal. For the determinations in the electrolytic way exactly 0.5000 gram of the powdered blende was weighed off, brushed into a No. 5 porcelain dish, moistened with water and covered with nitric acid (sp. gr. 1.41) and warmed gently on a stove plate. After about twenty minutes no further action appeared to take place and the glass cover was raised sufficiently to permit the escape of fumes and the liquid rapidly evaporated to dryness. The contents of the dish were then covered with pure hydrochloric acid (sp. gr. 1.21) and carried to dryness, and this treatment with hydrochloric acid repeated, care being taken to avoid overheating and consequent volatilization of zinc chloride. The dry salts were then moistened with strong hydrochloric acid and taken up in hot water. This operation occupied in all about one hour and ten minutes. Every sample of blende was treated in precisely the same manner.


0.1
0.2
0.3
0.4
0.5
Grams.
5
10
15
20
25
30
Minutes.
T
a
NaOH
$NaC_2H_3O_2$
NaOH
$NaCHO_2$
$NaCHO_2$
a

(A) SODIUM HYDROXIDE ELECTROLYTE.

The filtrate from the gangue was received in a small beaker and a solution of 6 grams of sodium hydroxide added, thereby precipitating the iron and redissolving the zinc as sodium zincate. This solution was filtered and the filtrate received in the silver-plated weighed platinum dish. The contents of the filter-paper were repeatedly washed with boiling water and the solution finally diluted to 125 cc. and electrolyzed, employing the conditions recommended in the earlier part of this paper, *viz.*, 5 amperes, 5 volts and speed I. The results were:

Blende taken. Gram.	Zinc found. Gram.	Zinc found. Per cent.
0.5000	0.3119	62.38
0.5000	0.3178	63.56
0.5000	0.3049	60.98
0.5000	0.3091	61.82
0.4000	0.2461	61.53
0.4000	0.2450	61.25

Suspecting the lack of concordance to be due to the presence of chlorides, the filtrate from the gangue was evaporated with sulphuric acid till fumes appeared, then taken up in water and treated as above. The results indicate that the chlorides present were not the disturbing factor.

Blende taken. Gram.	Zinc found. Gram.	Zinc found. Per cent.
0.5000	0.3131	62.62
0.5000	0.3088	61.76

An examination of the iron precipitates from the above samples of blende showed that they contained zinc in every case. It was found impossible to precipitate the ferric hydroxide free from zinc hydroxide by the use of sodium hydroxide as precipitant, and further study of this method of obtaining the zinc in a form suitable for electrolysis was abandoned.

An effort was made to omit the filtration of the iron precipitate and to electrolyze the solution of the sodium zincate in the presence of the suspended ferric hydroxide. An anode in the form of a platinum cylinder, suspended by a stout wire through its major axis and capable of rotation, was employed together with a semi-cylindrical cathode of platinum foil. The preliminary experiments showed the method to possess little of promise, and further study of it was postponed.

(B) AMMONIUM CHLORIDE ELECTROLYTE.

The thought arose that the solution of the blende containing the iron and zinc as chlorides might be treated with a solution of ammonium chloride and ammonium hydroxide, the ferric hydroxide being filtered off and the ammoniacal filtrate electrolyzed after the addition of an excess of sodium hydroxide, employing the conditions previously described under "sodium hydroxide electrolyte." Preliminary experiments with a solution of zinc sulphate of known strength gave excellent results, showing that ammonium chloride is not a disturbing factor.

Zinc taken. Gram.	HCl (sp. gr. 1.21). cc.	NH_4OH (sp. gr. 0.95). cc.	NaOH. Grams.	Amperes.	Volts.	Time. Minutes.	Zinc found. Gram.
0.2390	5	Sufficient	6	5	6	20	0.2396
0.2390	5	to make	6	5	6	20	0.2393
0.2390	5	ammoniacal	6	5	6	20	0.2388

Further study demonstrated that the addition of the sodium hydroxide was unnecessary, the filtrate from the iron precipitate being a good conductor, if plenty of ammonium chloride was present, and it yielded up its zinc readily and completely on electrolysis. Wrightson noted the separation of zinc in an ammoniacal solution long ago; the failure of Reinhardt and Ihle to obtain satisfactory deposits from a solution containing free ammonia was probably due to the absence of agitation.

Zinc taken. Gram.	HCl (sp. gr. 1.21). cc.	NH_4OH (sp. gr. 0.95). cc.	NH_4Cl. Gram.	Amperes.	Volts.	Time. Minutes.	Zinc found. Gram.
0.2387	5	25	1	5	5	20	0.2382
0.2387	5	25	1	5	5	20	0.2383
0.2387	5	25	1	5	5	20	0.2388
0.2387	5	25	1	5	5	20	0.2394

Fearing that the anode might be attacked by the chlorine liberated in contact with it, the platinum spiral was weighed before and after each of the above experiments, but it was found to have experienced no sensible change in weight.

Having thus found a new and most satisfactory elctrolyte, 0.5000 gram samples of the powdered blende were accordingly decomposed, as already described, and having removed the gangue and ferric hydroxide, the filtrate from the latter was received in the usual silvered and weighed platinum dish, the precipitate not being washed with water. The dish was now set aside and the porcelain dish in which the iron had been precipitated was placed under the funnel and the iron precipitate washed into it with dilute hydrochloric acid and the acid washed out of the paper with hot water. The iron was then reprecipitated in the same manner as at first and filtered on the same paper, the filtrate being received in the platinum dish. This precipitate was now thoroughly washed with boiling water and then the latter added to the filtrate to make it up to the usual 125 cc., if any further dilution were needed.

Careful study showed that more than two precipitations of the iron were not required in the case of a blende similar to that used in this investigation. The iron precipitate was invariably free from zinc and the solution free from iron. It was found convenient to use in the above operations a total of 10 cc. of hydrochloric acid (sp. gr. 1.21), and enough ammonium hydroxide (sp. gr. 0.95) to neutralize this and yield about 2 cc. in excess. To the platinum dish containing the zinc solution was now added 0.5 gram of ammonium chloride, preferably in the dry form, since the solution by this time nearly equaled the 125 cc. The hot water used in washing rendered further heating unnecessary and the liquid was immediately electrolyzed. The deposits were crystalline, slightly coarse, but not spongy. They were light gray in color and sufficiently adherent to present no difficulty in washing, although less adherent than those obtained earlier in this study. These deposits will not bear rubbing with the finger.

Blende taken. Gram.	HCl (sp. gr. 1.21). cc.	NH_4OH (sp. gr. 0.95). cc.	NH_4Cl. Gram.	Amperes.	Volts.	Time. Minutes.	Speed.	Zinc found. Per ct.
0.5000	10	Sufficient to make strongly alkaline	0.5	5	6	20	I	65.63
0.5000	10		0.5	5	6	20	I	65.75
0.5000	10		0.5	5	6	20	I	65.75
0.5000	10		0.5	5	6	20	I	65.65
0.5000	10		0.5	5	6	20	I	65.69
0.5000	10		0.5	5	6	20	I	65.68
0.5000	10		0.5	5	6	20	I	65.70
0.5000	10		0.5	5	6	20	I	65.69
0.5000	10		0.5	5	6	20	I	65.64

The time required to complete a determination of the zinc in blende by this method is about two hours and ten minutes from the time of weighing off the powdered ore to weighing its zinc content.

(C) SODIUM ACETATE ELECTROLYTE.

Attention was given to the precipitation of iron from blende as basic acetate and the electrolytic determination of the zinc in the filtrate, making use of the conditions given earlier for the use of an acetate electrolyte.

The blende having been decomposed, as described above, the solution containing the zinc and iron as chlorides was filtered off from the gangue and received in a No. 5 porcelain dish, nearly neutralized by sodium carbonate, 2.5 grams of sodium acetate added and the solution boiled for a few moments until the ferric basic acetate separated well. It was then filtered and the filtrate received in the usual silvered and weighed platinum dish. The precipitate was not washed, but was dissolved in dilute acetic acid and this solution allowed to run into the porcelain dish in which precipitation had been conducted before. The paper having been washed with hot water, this solution was nearly neutralized with sodium carbonate, 1 gram of sodium acetate added, and the precipitation made by boiling as before. This filtrate was received in the platinum dish and the iron redissolved and reprecipitated a third time. The combined filtrates were then made up to 125 cc. and electrolyzed.

Blende taken. Gram.	$NaC_2H_3O_2$. Grams.	$HC_2H_3O_2$ (30 pct). cc.	Amperes.	Volts.	Time. Minutes.	Zinc found.	
						Gram.	Per cent.
0.5000	3.5	1	5	13	25	0.3250	64.94
0.5000	3.5	2	5	13	25	0.3247	60.94
0.5000	4.5	0	5	13	25	0.3285	65.70
0.5000	4.5	0	5	13	30	0.3286	65.72
0.5000	4.5	0	5	13	30	0.3281	65.62
0.5000	4.5	0	5	13	30	0.3283	65.66
0.5000	4.5	0	5	13	30	0.3285	65.70

In the first two experiments free acetic acid was added before electrolyzing. The results show that this was not desirable, as the filtrate is sufficiently acid after the basic acetate separation of the iron.

The total time of an analysis of blende by this procedure is about two hours and thirty minutes.

(D) SODIUM FORMATE ELECTROLYTE.

The manipulation in this case was similar to that in the case of (C). The blende having been decomposed as usual and the gangue filtered off, the filtrate was received in a No. 5 porcelain dish and exactly 5 grams of dry sodium carbonate added. The dish was then covered to prevent loss by spattering, and 4 cc. of formic acid (sp. gr. 1.22) were run in. The solution was boiled and filtered hot into the weighed and silvered platinum dish. The unwashed precipitate was then rinsed back into the porcelain dish by 6 cc. of 1 : 10 formic acid and the paper washed; 5 grams of sodium carbonate were then added and the solution boiled, filtered into the platinum dish and the paper well washed. Two basic formate precipitations were found to be sufficient. To the contents of the dish were then added 2 cc. of the 1 : 10 formic acid and it was then heated nearly to boiling and electrolyzed. The deposits were excellent.

Blende taken. Gram.	Na_2CO_3. Gram.	HCOOH (sp. gr. 1.22). cc.	Amperes.	Volts.	Time. Minutes.	Zinc found. Gram.	Zinc found. Per cent.
0.5000	5.5	4.8	5	9	25	0.3284	65.68
0.5000	5.5	4.8	5	9	25	0.3281	65.62
0.5000	5.5	4.8	5	9	25	0.3298	65.96
0.5000	5.5	4.8	5	9	25	0.3289	65.78
0.5000	5.5	4.8	5	9	25	0.3295	65.90

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, No. 87.]

ELECTROLYTIC SEPARATIONS POSSIBLE WITH A ROTATING ANODE.[1]

BY DONALD S. ASHBROOK.

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A YEAR ago, Exner,[2] working in this laboratory, demonstrated that by using a high current density and great pressure with a rotating anode it was possible to determine fourteen metals in the electrolytic manner much more rapidly than had ever been done previously. The deposits were, in all instances, most satisfactory. These results constituted an important advance in electrochemical analysis. The objection once put forward, that too much time was necessary in such work, was completely removed. Indeed, Exner's investigation has wholly revolutionized electro-analysis. However, there remained the question as to how far this procedure

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

[2] This Journal, **25**, 896.