The deposits of mercury were always adherent and very satisfactory, so that we feel that this course may be recommended without hesitation to analysts.

UNIVERSITY OF PENNSYLVANIA.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEM-ISTRY, NO. 92.]

THE ELECTROLYTIC DETERMINATION OF CADMIUM WITH THE USE OF A ROTATING ANODE.¹

BY ALICE L. DAVISON. Received August 28, 1905.

INTRODUCTION.

IN VIEW of the work recently done in the rapid determination of metals electrolytically by the use of a rotating anode² it has seemed desirable to investigate each metal separately, studying the conditions under which it may be precipitated from different electrolytes. In this way, zinc, nickel and cobalt have been studied,³ and data accumulated relating to the rapid precipitation of these metals. The purpose of the present work was to make a similar study of cadmium.

This metal may be determined easily and accurately by the electric current from a number of electrolytes, and its deposition by the aid of a rotating anode was shown by Exner³ to be accomplished completely in a very short time. The apparatus used in the present investigation was the same as that employed by Exner, Ingham and Ashbrook³ working in this laboratory, and the same general method of procedure was followed. The dilution of the electrolyte was from 90-125 cc., giving an average cathode surface of 100 sq. cm., and the average distance between the electrodes was 1 cm. With the motor used, six speeds of rotation were practicable for the anode, *viz.*, 430, 580, 630, 700, 810 and 840 revolutions, respectively, per minute. These are designated in the tabulated conditions by the numerals I to VI.

In each electrolyte special attention was given to the determination of the rate of precipitation, and the results were plotted graphically. In every case the solution, before electrolyzing, was heated until bubbles appeared on the bottom of the dish.

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

² This Journal, 25, 896; 26, 1251, 1269, 1283, 1595.

³ Loc. cit.

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EXPERIMENTAL PART.

(A) SULPHURIC ACID ELECTROLYTE.

Wrightson¹ maintained that results obtained in the use of cadmium sulphate solutions were far from satisfactory, but it was later demonstrated² that he was in error. The conditions used by Exner in depositing cadmium from this electrolyte were useful as a guide in the further study of it. It was found that a large quantity of acid retarded the complete deposition of metal, and that the small amounts used in the experiments here recorded seemed favorable to the best results. The rate of deposition from this electrolyte is shown, together with that from the other inorganic electrolytes, in Fig. 1. The deposits were gray, crystalline, and quite adherent.

TABLE I

CdSO₄. =⊖= Cd.	H ₂ SO ₄ .	N. D. 100			Time.	
Gram.	1 : 10 cc.	amperes.	Volts.	Speed.	Minutes.	Cd found.
0,2867	1.0	3-5	13	III	15	0.2866
0.2867	1.0	5	13	III	14	0.2869
0.2867	0.5	3	13	III	I	0,1020
0.2867	0.5	3	13	III	2	0.1669
0.2867	0.5	3	13	III	5	0.2857
0.2867	0.5	3	12	III	$7\frac{1}{2}$	0.2860
0.2867	0.5	3	12	III	8	0.2861
0.2867	0.5	3	12	III	9	0.2867
0.2867	0.5	3	12	III	15	0.2867
0.5760	0.5	4	14	III	I	0.1190
0.5760	0.5	4	14	III	2	0.2245
0.5760	0.5	4	14	III	3	0.3417
0.5760	0.5	4	14	III	5	0.5217
0.5760	0.5	4	14	III	7 1	0.5760
0.5760	0.5	4	14	III	8	0.5762

It was thought by adding a few cubic centimeters of ammonium hydroxide when most of the metal had been precipitated, that the last traces of cadmium would be brought down more quickly. This was shown to be true by the following results:

			Таві	LE II.			
CdSO ₄ =C=Cd. Gram.	$H_2SO_4.$ 1 : 10 cc.	NH4OH. cc.	N.D. ₁₀₀ amperes,	Volts.	Speed.	Time. Minutes.	Cd found.
0.2867	0.5	2	3	13	III	5 <u>]</u> 2	0.2868
0.2867	O.5	2	3	13	III	$\begin{bmatrix} 5\\1 \end{bmatrix}$	0.2870
0.2867	0.5	2	3	13	III	5	0.2870
1 Z. ana	l. Chem., 15,	3 0 3.				-	

² Am. Chem. J., 2, 42.



Fig. 1.

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(B) AMMONIUM HYDROXIDE AND AMMONIUM SULPHATE ELECTRO-LYTE.

Clarke¹ attempted the precipitation of this metal from an ammoniacal solution. It was deposited in such a spongy nonadherent form that analysts since his day have wholly discarded solutions containing such salts. Since, however, Ingham² proved that zinc was deposited quantitatively and satisfactorily from an ammoniacal solution by the aid of a rotating anode it was thought that cadmium also might be determined in this way. Cadmium hydroxide was precipitated and redissolved by ammonium hydroxide, and then sulphuric acid neutralized by ammonium hydroxide was added to increase the conductivity. With a current of 5 amperes gray deposits were obtained, which, though somewhat spongy, were quite adherent and satisfactory. The liquid was always alkaline at the end of precipitation.

=C= Cd. Gram.	NH4OH. cc.	H ₂ SO ₄ . 1 : 10 cc.	N.D. ₁₀₀ amperes,	Volts,	Speed.	Time. Minutes.	Cđ found.
0.2887	5+6	10	5	$6\frac{1}{2}$	II	10	0.2887
0.2887	5+6	10	5	6	II	10	0.2886
0.2887	5 + 6	10	5	6 1	II	10	0.2885
0.2887	5 + 6	10	5	6	II	10	0,2888
0.2887	5 + 6	10	5	6	II	I	0.1312
0.2887	5 + 6	10	5	6	II	2	0.2708
0.2887	5 + 6	10	5	6	II	3	0.2868
0.2887	5+6	10	5	6	II	5	0.2889
0.2887	5 + 6	10	5	6	II	6	0.2887
0.2887	5+6	10	$\begin{cases} 4\\6\\7 \end{cases}$	5 6 1 6 1	II	$\begin{pmatrix} 3 \\ 1 \\ 1 \end{pmatrix}$	0.2879
0.2887	5+6	10	$\begin{cases} 4\\6\\8 \end{cases}$	5 6 1 7 1	II	$ \begin{array}{c} 2\\ 2\\ 1 \end{array} $	
0.2887	7+6	10	{ 5 { 6	5 6 1	11	4 }	0.2879
0.2887	5+12	10	$\left\{\begin{array}{c}5\\6\end{array}\right.$	6 1 6 1	11	$\left\{\begin{array}{c}4\\1\end{array}\right\}$	

TABLE	III.
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An attempt to shorten the time of deposition by increasing the current and the amount of ammonium hydroxide is shown by

¹ Z. anal. Chem., 18, 104.

2 Loc. cit.

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the last four experiments to have been unsuccessful, the only result being to increase the sponginess of the deposit, in two instances to such a degree that weighing was omitted. The rate of precipitation from this electrolyte is represented graphically in Fig. 1.

(C) SODIUM FORMATE ELECTROLYTE.

Formic acid as an electrolyte for the determination of cadmium was first suggested by Warwick¹ and later used by Avery and Dales.² Ingham also demonstrated that with a rotating anode formic acid was an excellent electrolyte for the deposition of zinc, and the conditions recommended by him were used as a guide in the following experiments with cadmium. From this electrolyte the metal was completely precipitated in four minutes as a dark gray, spongy, though adherent deposit, the liquid siphoned off giving no test for cadmium. The following results prove that the quantity of sodium formate may vary widely.

TABLE	IV.
	-

CdSO ₄ =C= Cd. Gram.	Na ₂ CO ₃ . (sp Grams.	HCOOH , gr. 1.06 cc,	i). N.D. ₁₀₀ amperes.	Volts.	Speed.	Time. Minutes.	Cd found.
0.2898	5	16	5	5	II	15	0.2900
0.2898	4	15	5	5 1	II	15	0.2903
0.2898	3	10	5	6]	II	15	0.2902
0.2898	2	8	5	7	II	10	0.2898
0.2898	I	4	5	111	II	10	0.2894
0.2898	1.2	5	5	8	II	I	0.1645
0.2898	1.2	5	5	8	II	2	0.2816
0.2898	1.2	5	5	9	II	3	0.2891
0.2898	1.2	5	5	8 1	II	4	0.2896

(D) AMMONIUM FORMATE ELECTROLYTE.

After the satisfactory results obtained from the use of sodium formate as an electrolyte, ammonium formate was suggested. Ammonium hydroxide was added in excess, and then varying amounts of formic acid. The results indicate that the liquid may be either alkaline or acid without affecting the deposit or the time.

¹ Z. anorg. Chem., 1, 258.

² This Journal, 19, 380.

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			TABLE 7	v.			
CdSO4 =C= Cd. Gram.	NH4OH. cc.	HCOOH (sp. gr. 1.06) cc.	N.D. ₁₀₀ amp ere s.	Volts.	Speed.	Time. Minute s.	Cd found.
0.2898	5	5	5	6	II	5	0.2900
0.2898	5	10	5	6	II	5	0.2910
0.2898	5	10	5	5	II	5	0.2898
0.2898	5	6	5	6	II	5	0.2899
0.2898	5	6	5	$5\frac{1}{2}$	II	I	0.1612
0.2898	5	6	5	6	II	2	0.2850
0.2898	5	6	5	6	II	3	0.2904
0.2898	5	10	5	5	II	I	0.1766
0.2898	5	10	5	6	II	2	0.2870
0.2898	5	10	5	6	II	3	0.2904

The deposits from this electrolyte were very similar to those from a sodium formate solution, and the time curves of the two electrolytes nearly coincide, as is seen in Fig. 2.



(E) SODIUM ACETATE ELECTROLYTE.

This electrolyte was one of the first from which cadmium was successfully precipitated,¹ and has usually been conceded to be one of the most satisfactory means of obtaining the metal electrolytically.² With the rotating anode Exner got excellent results from this electrolyte, but his conditions were not used, more sodium acetate being added, and a small amount of acetic acid being substituted for the potassium sulphate which was added to increase the conductivity. The electrolyte proved to be very like the formate solutions in its resulting deposits. The rate of precipitation from both sodium acetate and ammonium acetate will be seen to be very rapid (Fig. 2).

CdSO.							
$= \bigcirc = \mathring{C}d.$ Gram.	NaC ₂ H ₃ O ₂ . Grams.	$HC_2H_8O_2.$	N.D. ₁₀₀ amperes.	Volts,	Speed.	Time. Minutes.	Cd found.
0.1992	3	0.25	5	$8\frac{1}{2}$	II	10	0.1992
0.1992	3	0.25	5	8 1	II	10	0. 1992
0.3984	3	0.25	5	$8\frac{1}{2}$	II	I	0.1601
0.3984	3	0.25	5	8 1	II	2	0,2863
0.3984	3	0.25	5	$8\frac{1}{2}$	II	3	0.3963
0.3984	3	0.25	5	9	II	4	0.3987
	1.5						

(F) AMMONIUM ACETATE ELECTROLYTE.

That an alkaline acetate electrolyte is inferior to one that is acid was shown by the following experiments. At the beginning a slightly acid solution of ammonium acetate was used. The results of sixteen experiments were as follows:

CdSO.			TABLE V	/II.		
$= \bigcirc = Cd.$ Gram.	NH ₄ C ₂ H ₃ O ₂ . N Grams. am	I.D. ₁₀₀	Volts.	Speed.	Time. Minutes.	Cd found
0.4000	3	5	7	II	10	0.4010
0.4000	2	5	9 1	II	10	0.3997
0.4000	2	5	8 1	II	10	0.4002
0,4000	2	5	8 1	II	10	0.3993
0.4000	2	5	8 1	II	10	0.3989
0.4000	2	5	8 1	II	I	0.1617
0.4000	2	5	9	II	2	0.3255
0.4000	2	5	8 1	II	3	0.3928
0.4000	2	5	9	II	4	0.3981
0.4000	2	5	9	II	5	0.3990
0.4000	2	5	9	II	6	0.3995
0.4000	2	5	9 1	II	7	0.3995
0.4000	2	5	9 1	II	8	0.3994
0.4000	2	5	9 1	II	9	0.4002
0.3588	2	5	$8\frac{1}{2}$	II	10	o.3588
0.3588	2	5	9 1	II	10	o.3588

¹ Am. Phil. Soc. Proc., 1878; Z. anal. Chem., 19, 16.

² This Journal, 19, 870 : C. R., 131, 384.

It was evident from these experiments that the last traces of metal were very hard to precipitate, so that a series of four trials was made in which a few cubic centimeters of aminonium hydroxide were added after the electrolysis had proceeded three minutes. The result was to hinder deposition. Then the addition of 3 cc. of acetic acid after two minutes was tried, with the following satisfactory results. In the last five experiments the excess of acid was present from the beginning. The deposits were excellent.

CdSO₄ =⊖= Cd. Gram.	NH4C2H3O2. Grams.	$\mathbf{HC_{2}H_{3}O_{2}}$. cc.	N.D. ₁₀₀ amperes.	Volts.	Speed.	Tim e . Minutes.	Cd found.
0.3575	2	3	5	$8\frac{1}{2}$	II	5	0.3575
0.3575	2	3	5	8	II	5	0.3574
0.3575	3	3	5	8 1	II	5	0.3575
0.3575	3	3	5	8	II	5	0.3574
0.3575	3	3	5	$8\frac{1}{2}$	II	5	0.3577
0.3575	3	3	5	9	11	I	0.1647
0.3575	3	3	5	$8\frac{1}{2}$	II	2	0.3143
0.3575	3	3	5	7	II	3	0.3575
0.3575	3	3	5	7	II	4	0.3573

TABLE VIII.

(G) POTASSIUM CYANIDE ELECTROLYTE.

Beilstein and Jawein¹ made most satisfactory determinations of cadmium from this electrolyte, as has been done by others.² It was studied to some extent by Exner, and the conditions used by him, modified somewhat, were those employed in the following experiments. The addition of sodium hydroxide was necessary to decrease creeping, which was so considerable that the dilution could not exceed 100 cc. It was found that when comparatively large amounts of sodium hydroxide were used the deposits had a cloudy gray appearance, but with the minimum amount (about 1 gram), the deposit of cadmium was lustrous and of a beautiful silver-white color. In preparing the solution, potassium cyanide in excess was added first and then the sodium hydroxide. Fig. 1 shows that the rate of precipitation in this electrolyte was considerably slower than in any of the others.

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¹ Ber., 12, 759.

² Z. angew. Chem., 1892; this Journal, 19, 870; Z. anorg. Chem., 28, 233; this Journal, 21, 911.

CdSO4 =C= Cd. Gram.	NaOH. Grams.	KCN. Grams.	N.D. ₁₀₀ amperes.	Volts.	Speed.	Time. Minutes.	Cd found.
0.3565	5	4	5	4 1	II	10	0.3550
0.3565	2	4	5	$4\frac{1}{2}$	II	15	0.3558
0.3565	3	4	5	$4\frac{1}{2}$	II	15	0.3568
0.3565	2	4	5	5	II	15	0.3561
0.3565	2	4	5	5	II	15	0.3566
0.3565	2	4	5	4	II	15	0.3564
0.3565	1	3	5	6	II	15	0.3563
0.3565	I	3	5	5 1	II	I	0.1385
0.3565	I	3	5	5 1	II	2	0,2276
0.3565	1	3	5	5 1	II	3	0.2670
0.4568	I	3	5	5	II	I	0.1808
0.4568	1	3	5	5	II	2	0.2585
0.4568	1	3	5	5	II	3	0.3291
0.4568	I	3	5	5 1	II	5	0.3778
0.4568	I	3	5	5	II	7 1	0.4348
0.4568	τ	3	5	5	II	10	0.4534
0.4568	I	3	5	5	II	15	0.4568
0.4568	I	3	5	4 1	II	15	0.4578
0.4568	I	3	5	$5\frac{1}{2}$	II	20	0.4561
0.4568	I	3	5	51	II	20	0.4569

TABLE IX.

The anode spiral and the cathode dish were found during these determinations to have each lost about 4 mg. in weight.

(H) AMMONIUM LACTATE ELECTROLYTE.

The results in this electrolyte were not very satisfactory, a trace of cadmium being found in the liquid siphoned off in nearly every experiment. The deposits were similar to those from other organic electrolytes.

			Тав	le X.			
$\begin{array}{c} CdSO_3\\ =\bigcirc = Cd.\\ Gram. \end{array}$	NH4OH. cc.	Lactic acid. cc.	N.D. ₁₀₀ amperes.	Volts.	Speed.	Time. Minutes.	Cd found.
0.4568	4	2	5	$7\frac{1}{2}$	II	5	
0.4568	12	3	5	7 1	II	10	0.4647
0.4568	2	I	5	7 1	II	10	0.4046
0.4568	10	5	5	7 1	II	10	0.4344

(I) SODIUM LACTATE ELECTROLYTE.

Equally unsatisfactory results were obtained from this electrolyte, the last traces of metal seeming to be precipitated with great difficulty.

$= \bigcirc = Cd.$	NaOH.	acid.	$\mathbf{N},\mathbf{D}_{\cdot 100}$			Time.	
Gram.	Gram,	cc.	amperes.	Volts,	Speed.	Minutes.	Cd found.
0.4568	I	$2\frac{1}{2}$	5	10	II	10	0.4340
0.4045	I	$3\frac{1}{2}$	5	9 1	II	15	0.4021
0.4045	I	$3\frac{1}{2}$	5	$9^{\frac{1}{2}}$	II	20	0.4000
0.4045	I	5	5	8	II	20	0.4000
	Na ₂ CO ₃ . Gram.						
0.4045	I	5	5	$10\frac{1}{2}$	IV	15	0.4046
0.4045	I	5	5	$9\frac{1}{2}$	IV	$2\frac{1}{2}$	0.3387
0.4045	1	5	5	$9\frac{1}{2}$	IV	5	0.4032
0.4045	I	5	5	II	IV	7	0.4032
0.4045	I	5	5	10	IV	IO	0.4042

TABLE XI.

In all these experiments a trace of cadmium was found in the liquid from the deposits.

(J) AMMONIUM SUCCINATE ELECTROLYTE.

No mention has ever been made of the deposition of cadmium from a succinate electrolyte, so that the excellent deposits of cobalt from such a solution led to its use for cadmium. The results were good, the deposits being similar to those from a formate or an acetate electrolyte. A small excess of acid was found to be desirable to insure complete precipitation. The ammonium succinate was of such a strength that $1 \text{ cc.} = \bigcirc = 0.2 \text{ gram succinic acid.}$

Τa	BLE	\mathbf{XI}	Ι.

0480	nium eu						
=C= Cd. Gram.	cinate.	NH₄OH. сс.	N.D. ₁₀₀ amperes.	Volts.	Speed.	Time. Minutes.	Cd found.
0.3996	5		5	9	II	10	
0.3996	5	5	5	$11\frac{1}{2}$	I	12	0.3997
0.3996	5		5	$11\frac{1}{2}$	I	15	0.3995
	5 per c	ent. acid.					
0.3996	5	12	5	9ł	V	15	0.3995
0.3996	5	10	5	10	V	12	0.3997
0.4040	5	15	5	12	IV	2	0.2830
0.4040	5	15	5	12	IV	5	0.4032
0.4040	5	15	5	111	IV	7	0.4040
0.4040	5	15	5	11	IV	10	0.4043

(K) SODIUM SUCCINATE ELECTROLYTE.

In this electrolyte the presence of free acid was found to be undesirable. I cc. of the sodium succinate $= \bigcirc = 0.2$ gram acid. The rate of precipitation is shown in Fig. 2 to be identical with

. . . .

that in the ammonium succinate electrolyte. Five experiments gave the following results:

			IABLE	am,			
$CdSO_4$ = \bigcirc = Cd. Gram.	Sodium succinate. cc.	5 per cer acid, cc.	nt. N.D. ₁₀₀ amperes.	Volts.	Speed,	Time. Minutes.	Cd found.
0.4040	5	15	5	11	\mathbf{IV}	15	0.4036
0.4040	5	• •	5	11	IV	15	0.4044
0.4040	5	• •	5	10	IV	2	0.2885
0.4040	5		5	10	\mathbf{IV}	5	0.4041
0.4040	5		5	9	IV	10	0.4043

The deposits were like those from ammonium succinate.

SEPARATIONS.

By the use of the high current densities, which are possible with a rotating anode, the separations, based upon differences of voltage, cannot be effected. Separations possible under these conditions are based upon the deposition or non-deposition of the separate metals from the electrolyte in which they are present together.

Ashbrook¹ described the conditions for the separation of cadmium from several metals in mineral acid electrolytes. A separation of cadmium from magnesium in a formate solution was thought to be possible, as cobalt had been separated from magnesium in this way. The following results indicate that the separation is easily accomplished.

(1) CADMIUM FROM MAGNESIUM.

			TABLE .	XIV.				
CdSO ₄ =C= Cd. Gram.	MgSO₄ =⊖= Mg. Gram.	Na2CO3. Gram.	HCOOH (sp. gr. 1.06). cc.	N.D. ₁₀₀ am- peres.	Volts.	Speed.	Time. Minutes.	Cd found.
0.2660	0.1000	0.2	12	5	7	II	10	0.2662
0.2660	0.1000	0.2	12	5	6	II	10	0.2658
0.3339	0.2500	0.2	12	5	6	II	10	0.3338

It is probable also that this electrolyte would offer a separation of cadmium from the alkaline earth metals.

(2) CADMIUM FROM IRON.

The separation of these metals in a potassium cyanide electrolyte was described by Stortenbeker.² This method was adopted in the experiments following. Pure ferrous sulphate

¹ Loc. cit. ² Z. Elektrochem., 4, 409.

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was added to the cadmium solution, acidified with sulphuric acid, potassium cyanide in excess added, and then a small quantity of sodium hydroxide necessary to prevent creeping and consequent loss. The results were as follows:

TABLE	\mathbf{x}_V
LADLE	-ΛV,

CdSO₄ =⊂= Cd. Gram.	FeSO ₄ . =⊖= Fe. Gram.	KCN. Grams.	NaOH. Grams.	n.D. ₁₀₀ am- peres.	Volts.	Speed.	Time. Minutes.	Cd found.
0.2650	0.2500	12	I	5	5	IV	20	0.2600
0.2650	0.2500	12	2	5	5	IV	20	0.2648
0.2650	0.2500	12	2	5	5	IV	25	0.2649
0.2650	0.2500	14	2	5	5	IV	25	0.2652
0.2650	0.2500	14	2	$5\frac{1}{2}$	5	IV	25	0.2652

In the first experiment some loss was occasioned by creeping. In the last four experiments the current was increased to 6 amperes at the end. In each of the above determinations no cadmium was found in the filtrate, and no iron in the deposit.

(3) CADMIUM FROM NICKEL.

The fact that a double cyanide solution of nickel gave no deposit even with a strong current gave rise to the hope that this separation was possible, using a rotating anode. The presence of cadmium, however, in the electrolyte caused a difference in the behavior of the nickel, and some of the latter metal was always found in the cadmium deposit. Much cadmium was always discovered in the liquid siphoned off, even after twenty minutes.

(4) CADMIUM FROM COBALT.

This separation in a double cyanide solution is mentioned,¹ and since cobalt as cobalticyanide is not deposited, even with a very strong current, the possibility of its separation from cadmium, using a rotating anode, was suggested. To a solution of potassium cobalticyanide was added a cadmium solution together with sodium hydroxide, and a current of 5-6 amperes passed through the same for twenty minutes. The resulting deposit was dark, and when dissolved off the dish with nitric acid, was found to contain no cobalt but a comparatively large quantity of insoluble particles which dissolved in aqua regia and proved to be platinum. During this experiment the platinum dish lost about 7 mg., though in a blank experiment without the cobalt no loss was sustained. In the next experiments a silvered dish was used, Am. Chem. J., 12, 104; Z. physikal. Chem., 12, 112. with the result that the platinum was not attacked, but the cadmium was not completely precipitated even after allowing the current to pass for thirty-five minutes. This incomplete precipitation was observed in the previous experiments.

UNIVERSITY OF PENNSYLVANIA.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEM-ISTRY, NO. 94.]

THE RAPID DETERMINATION OF LEAD IN THE ELEC-TROLYTIC WAY.¹

BY RALPH O. SMITH. Received August 28, 1905.

IN MAKING this study it was not proposed to determine the lead as metal but as dioxide, adopting, however, the course pursued by Exner.²

At the very beginning care was taken to use perfectly pure lead nitrate, that there might be no question as to its metal content.

After the solution of the salt had been made, definite portions of it were removed and evaporated to dryness with sulphuric acid, and after carefully heating, the residue was weighed as lead sulphate.

Thus, 25 cc. of the stock solution of the lead nitrate gave 0.3635 gram of lead sulphate, equivalent to 0.2438 gram of metallic lead, or 0.2867 gram of lead dioxide.

To 25 cc. of this solution 20 cc. of nitric acid (sp. gr. 1.4) were added and then diluted with water to 115 cc. The solution was heated to 70° before the current was allowed to pass.

These were the conditions in each of the following experiments. The rotating electrode, which in this case was the cathode, performed about 450 revolutions a minute.

			TAB	LE I.		
Experi- ment,	Time. Minutes.	PbO ₂ found.	Pb calculated.	Pb present.	Volts.	N.D 100 amperes.
I	3	0.2478	0.2146	0.2483	3.7	11.6-11.0
2	6	0.2679	0.2320	0.2483	3.9-3.8	10 -10.8
3	9	0.2807	0.2431	0.2483	3.8	11
4	12	0.2858	0.2475	0.2483	3.7-3.8	10.8-11
5	15	0.2873	0.2488	0.2483	3.7-3.8	II
6	18	0.2873	0.2488	0.2483	3.9	11.2-11
7	15	0.2874	0.2489	0.2483	4.0	13

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

² This Journal, 25, 896.