When the pressure is equal to 760 mm. the boiling point is indicated. Other applications of the moving drop of mercury are being planned.

I am greatly indebted to Mr. Edward O. Heuse for assistance rendered in connection with this investigation.

URBANA, ILLINOIS April 25, 1907.

## THE USE OF TIN AS A CATHODE FOR THE RAPID QUANTITA-TIVE ELECTROLYTIC DEPOSITION OF ZINC, COPPER, SILVER, CADMIUM AND NICKEL.

By LAURENCE T. SHERWOOD AND GELLERT ALLEMAN, Received May 21, 1907.

The high price of platinum, together with other minor considerations, led to an investigation in this laboratory in order to determine if metallic cathodes, other than platinum, could be used for the quantitative electrolytic deposition of carious metals, and, if so, to determine the exact conditions productive of the best results. While it is intended to continue this investigation using other metallic cathodes, especially tungsten, this article has to deal entirely with the tin cathode, and the deposition on it of the five elements named in the title.

Attention is directed to the fact that all the results obtained are published in the tables—none having been rejected.

While not so generally applicable as platinum, it was found that for the estimation of the metals enumerated, tin could be substituted for the more expensive material. The attempt has not yet been made to use this cathode in determining any other of the metals but judging from the work thus far it seems quite possible that it may be used for all of those which are deposited in the metallic state.

Platinum has been regarded as about the only suitable cathode material because of its insolubility and immunity from corrosion, and, of course, these were anticipated as the chief defects of tin. The solvent action of electrolytes was especially feared, but with certain precautions which will be mentioned under the special considerations there was no loss of tin during the determination even with such powerful solvents as sulphuric acid and potassium cyanide. Corrosion of other natures gave no trouble whatever.

The following general facts regarding the care of the dishes may be worth mentioning:

Solutions which have not a solvent action may during the progress of an analysis be heated in them upon a sheet of asbestos (thickness exceeding a quarter inch) or upon an electric stove without fear of oxidizing the tin.

On account of the low melting point of tin the dish cannot, of course,

be held in the free flame nor set upon hot asbestos or an electric stove. if empty.

The surface should be kept bright by an occasional scouring with fairly coarse sea sand.

For reasons which will appear later it is preferable, but not necessary, to keep separate dishes for depositions of each particular metal.

The first dishes were made of Kahlbaum's tin, cast in an iron mold of the shape represented by cross-section diagram No. 1, and cut down on a lathe to a suitable weight, (70-80 grams). The dimensions were: diameter of top, 8 cm.; diameter of bottom, 7 cm.; height, about 3.3 cm. The total inside surface was approximately 115 sq. cm. which was equivalent to about 100 sq. cm. when 100 cc. of liquid was rotating inside. Most of the work on zinc was done with these dishes, but they were unsatisfactory both in their form and on account of pores in their surfaces due to imperfections in the castings. The dish which has thus far given the best results has a form indicated by cross-section diagram No. 2.



These were spun by J. Bishop and Son, from sheet block tin (No. 18 B. & S. gauge) bought of Messrs. Eimer & Amend. The diameter of the top was 9 cm.; the total surface, about 125 sq. cm. which was equivalent to about 100 sq. cm. when 100 cc. of liquid was rotating within. They weighed about 65 grams. Owing to the softness of tin, the edge of the dish where it is grasped by the forceps in weighing should be as thick as consistency will permit.

To economize time the rotating anode was used in practically all the investigations. The forms used consisted of platinum wire respectively 1.4 mm, and 1.1 mm, in diameter, (numbers 17 and 19, B. & S. gauge), bent into spirals. The former size was slightly preferable. The spirals were depressed in the center, conforming somewhat to the shape of the dish. Their diameter was 3.5 cm. The anodes were inserted in the motor shaft. The motors supplied by the Electro-Dental Manufacturing Co., of Philadelphia, were wound to run on a 110 volt lighting circuit and had a speed of 500-700 revolutions per minute. While they answere l practically all purposes it is recommended if this form of rotator is used that the maximum speed be at least 1000 revolutions per minute. A lamp resistance could be put in the circuit to diminish this speed to

any desired. Within the range of speeds used there was hardly a perceptible difference in the character of the metallic deposits.

Methods outlined by Exner 1 were adapted for this metal, Zinc. first using a platinum dish and later tin. With a few exceptions, probably due to some slightly different conditions, his methods served very well. A few experiments showed also that Classen's method, using ammonium oxalate and tartaric acid electrolytes<sup>2</sup>,  $\mathbf{as}$ could probably be used with the tin cathode. However, not enough tests were made to decide conclusively.

Table I records a series of determinations with a platinum dish. The results in Table II were obtained by almost the same method on the tin cathodes described in diagram No. 1. Sodium hydroxide dissolves tin slowly when no current is passing through it and the following procedure is therefore recommended. Prepare the sodium zincate by adding the specified amount of sodium hydroxide (in solid form) to the zinc sulphate solution in a beaker. (It is perhaps worth mentioning that lena glass should be used in preparing the zincate solution since the strong alkali is apt to dissolve material from the ordinary glass beaker<sup>3</sup>). Place the dish on its support and add thirty or forty cc. of hot water; close the circuit, having the rheostat adjusted so that about one ampere will flow as soon as the electrolytes are added; introduce the hot zincate solution; dilute to a suitable volume; adjust the electrodes; start the rotator, and allow the full current to pass in. If this procedure is followed, tin will not be dissolved. With the sodium acetate method it is unnecessary to observe the precaution of having the circuit closed before the electrolyte is added.

The deposits on tin exactly resembled those on platinum. Thev were crystalline but could easily be washed in the usual manner and weighed. After weighing, the loose material was wiped and brushed out and another determination made by depositing on the remaining coating. Moreover, when once coated with zinc, no precautions were necessary to protect the tin from the solvent action of the sodium hydroxide. The adherent coating was very thin and several depositions could be made before cleaning all the zinc off. To remove the adherent zinc, use dilute hydrochloric or sulphuric acid, or nitric acid with a specific gravity, 1.52. Nitric acid more dilute than this attacked the tin rapidly. After cleaning, the tin surface usually had to be scoured bright with sea sand. Of course the dishes lost a little weight in cleaning but with reasonable care they last indefinitely. Dishes in constant use for a year lost less than two grams.

<sup>1</sup> This Journal, 25, 900.

<sup>2</sup> Ber., 12, 1622. <sup>3</sup> It must be remembered that Jena glass contains zinc, however, and is some-what attacked by alkaline solutions. Porcelain would probably be safer.—Editor.

LAURENCE T. SHERWOOD AND GELLERT ALLEMAN

ZuSO <sub>4</sub> .7H <sub>2</sub> O =Zu in gms.	NaOH in gms. i	Volume in cc. when diluted	Approx- imate temp. at start	$\begin{array}{l} Current \\ N.D_{Iiki} \\ = A \end{array}$	Volts	Time in minutes	Zn deposited in gms.	Errer in pet. of Zn iu ZnSO <sub>4</sub> .7H <sub>2</sub> O
0.2496	8	100	65°	5	5	30	0,2490	0.05
	••	4.4		,,			0.2495	<b>0.</b> 01
••	• •	• •	••	۰,	••	٠.	0.2501	0.05
**	••	• •	• •	• •	••	25	0.250 <b>2</b>	0.06
			т	ABLE I	I.			
0.2496	8	001	6 <b>;°</b>	3	7	30	0,2494	0.02
**	• •	••	••		i.		0.2501	0.05
••		••		•••	٠.	44	0.2492	0.03
••	• •		۰.	••	•••	••	0.2494	0.02
•••	••			۰.	• •	••	0.2494	0.02
••	۰.	••	٠.	۰.	••	25	0.2582	0.13
		••			•••	-0	0.2536	0.37
		<u>:</u> •	• •	۰.	4.4		0.2515	0.18
	••		• •		• •		0.2507	0.10
		••	۰.		••	• •	0.2505	0.08
0.2500	••	••	۰.	••	٤,	••	0.2508	0.07
	••			۰.	•••		0.2405	0.02
	٠,	* *			••	• •	0.2517	0.16
• •				• 4	• •		0.2.105	0.04
• •	• •		4.1	• •	• •	. (	0.2502	0.02
		••				• •	0.2.117	0.75
• •	••			••	••	••	0.2118	0.17
• •	÷ .		· •	٠.	• 1	• •	0.2475	0.22
••	••	•••	••	• •	••	"	0.2486	0.13

TABLE I.

Note: Speed of rotator was 700 R. P. M.

In all the determinations of Table II only two dishes were used and neither of them was cleaned of the adherent zinc after the series was begun. It is quite possible that by depositing upon the porous zinc surface which was formed after the first few determinations a source of error was introduced which may account for the inconsistent results in the latter part of the series and throughout Table IV. This point was not thoroughly investigated but the better results in the first determinations seemed to indicate that that was the case. The irregular results in both Tables II and IV are also probably due to the pores and imperfections in the dishes used. Series V was run on the better form of dish described by diagram No. 2 and the results are consistent. Four or five determinations were made without cleaning off the adherent zinc. It is not advisable to run more than this number in succession.

The time set down by Exner for these depositions was ten to fifteen minutes.<sup>1</sup> Failure to comply with some conditions made it necessary to take a longer time with the sodium hydroxide electrolyte. The bulk of the metal came down in the first few minutes, but traces remained until the time stated

<sup>1</sup> See also paper by Ingham, This Journal, 26, 1272.

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had expired. The time required on tin was the same as that on platinum. As rapidity was not a special object in this investigation no great effort was made to reduce the time although it should not be difficult to do so. Five amperes, the current used on platinum, gave too loose a deposit on the tin, but no longer time was required with three amperes.

Tables III and IV record series on platinum and tin respectively with the sodium acetate electrolyte. This method was adapted with practically no change from Exner's conditions. The deposits in both cases were crystalline and rather loose but there was no difficulty in washing and weighing them.

				1					
$ZnSO_4.$ $7H_2O$ =Zn in gms.	CH <sub>3</sub> .COONa in gms.	30 per cent. acet- ic acid in cc.	Volume in cc. when diluted	Approxim- ate temp. at start	Cur- rent $N.D_{100} = A.$	Volts	Time in minutes	Zn de- posited in gms.	Error in pct. of Zn in ZnSO <sub>4</sub> . 7H <sub>2</sub> O
0.2500	3	0.2	100	20°	3	18.5-11.5	15	0.2509	0,08
		* *	۴.	• •	••	14	4.4	0.2496	0.03
"		* *	• •	• •	• •	• 6	4.6	0.2499	0.01
" "	"				" "	" "	44	0.2489	0.10
••		" "	" "		"	" "		0.2491	0.08
				TABLE	IV				
0.2500	3	0.2	001	20°	2	12.5- 9.5	15	0.2494	0.05
••=0==			• •		"			0.2483	0.15
		44		" "	<b>6</b> 6	<b>ć •</b>		0,2505	0.05
• •		64	"	" "	• •	**	4.4	0.2491	0.08
• 6	• •	**	44	• •	"	**		0.2504	0.04
• •	• •			" "		4.4	* *	0.2474	0.23
				" "	6.6	"	* *	0.2488	0.11
• •		• •			" "	" "		0.2509	0.08
• •	* *		63	**	" "	4.6	"	0.2484	0.14
• •	••	" "	• •		**		"	0.2483	0.15
"		••		••			* *	0.2497	0.03
• •		• •	+ 4	• •	* *	**	4.6	0.2513	0.12
"	• •	• •	• •	**	**	" "	• •	0.2490	0.09
• •			• •	• •	1.5	6.6	"	0.2489	0.10
"		• •	• •	* *	**		* *	0.2517	0.16
	* *	"	۰.	••	••		• •	0.2496	0.03
	4.	"			* *	• •		0.2516	0.15
	• •	••	• •	* 1	4.4		••	0.2480	0.18

Note: Speed of rotator was 700 R. P. M.

The series recorded in Table V was run by Mr. R. L. Hill of this laboratory. He used the tin dishes represented in diagram No. 2 and was careful not to run more than four or five determinations on a particular dish without cleaning off the adherent coating of zinc. He also used a slower rotator (550 R. P. M. instead of 700 R. P. M.) and a slightly smaller anode, which may account for the extra time required.

$ZnSO_4.$ $7H_4O =$ Zn in gms.	CH <sub>3</sub> . COONa in gms.	30 per cent. acetic acid in cc	Volume in cc. when diluted	Approxim- ate temp. at start	$\begin{array}{c} \text{Cur-}\\ \text{rent}\\ \text{N}.\text{D}_{1^{(0)}}\\ = \text{A} \end{array}$	Volts	Time in min.	Zn de- posited in gms,	Error in pet. of Zn in ZnSO <sub>4</sub> 7H <sub>2</sub> O
0.2500	3	0.2	100	20°	2	18-13	17	0,2500	0.00
••		۰.	••	65°	2	14-11	• •	0.2493	0.06
••			٠.	• •			••	0.2500	0.00
••	• 6	**		••	• •	••	6 1	0.2497	0.02
* *	• 6	**	" "	• •	••	6 N	16	0.2496	0.03
• •	**	••	••	• •	••		••	0.2490	0.09
" (	• 6		4.5	í •	65	• •	18	0.2498	0.02
	**	" "	44	••	••	٠.	16	0.2496	0.03
ю.	6	**	٠.	••	••		17	0.2495	0.04

TABLE V

Silver. When silver nitrate solution comes in contact with tin a black deposit forms on the tin. This action is of course objectionable; but if potassium cyanide were first added and the double cyanide formed, it did not take place. However when potassium cyanide was used alone as an electrolyte the silver deposits were dark and loose. The addition of a suitable amount of oxalic acid afforded a reducing action and this difficulty was also overcome, the deposit being the characteristic milk white silver. Unless rather low currents were used the deposits had somewhat more tendency to sponginess than on platinum but if a little clear gelatine solution were added, one and a half amperes could be used.

Potassium cyanide in the dish when no current is passing of course dissolves a small amount of tin, (from one to three milligrams if allowed to stand a minute), but when the tin surface is absolutely clean of oxide or any foreign material the dish does not lose appreciable weight in silver determinations if the circuit is closed before the addition of the cyanide. A number of experiments were made with the potassium cyanide alone in the dish and also with the cyanide and oxalic acid together. Hot water was first put in the dish and the circuit closed, after which the respective amounts of cyanide or mixture of the two reagents that were used for the silver determinations were added. The remaining operations were the same as in the course of a regular analysis.

		TABLE	VI.		
Weight of dish	KCN in grams	Saturated solution of oxalic acid in cc.	Am- peres	Time in minutes	Loss of weight
59.8008	2	10	0.6	20	0
65.1525	4.4	6 x	••	**	0
63.1403	" "	**		* 6	0.0001
64.8920	**		· •	* *	0.0003
63.0190	• •	• •	1.0	6.6	0
64.8203		4.4	1.0	* *	0

When silver is being deposited it is improbable that tin ever dissolves at all, for the determinations were fully as accurate as any made on the platinum dishes. To prevent possible solution of tin the following pro-

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cedure was observed: the cathode was placed upon its support and hot water added; the circuit was closed, having the rheostat adjusted so that about one ampere would flow when the electrolytes were introduced; oxalic acid, gelatine<sup>1</sup>, and the cyanide solution of silver were added in the order named: the volume was diluted to 100 cc.; the electrodes were adjusted, the rotator started and the current regulated. It was not thought that oxalic acid would dissolve tin appreciably when the current is not passing, except on standing. It is certain that it does not when the current is on.

There was no trouble in depositing one layer of silver upon another indefinitely if each time the loose metal were wiped out and the remaining coating well brushed, but it is probably safer not to deposit more than four or five layers without cleaning. When desirable the firm deposit may be cleaned off with potassium cyanide. The action may be hastened by passing a current of about 0.2 amp. through the cyanide, making the dish the anode. The deposit could also be scoured off without the aid of a solvent.

				TAF	BLE VI	I				
$\begin{array}{l} \operatorname{AgNO}_{3} \\ = \operatorname{Ag} \\ \operatorname{in} \\ \operatorname{grams} \end{array}$	KCN in gms.	Oxalic acid in cc. of saturated solution	Gelatine (1 gm =1000 cc.) in cc.	Vol- ume when diluted	Approx- imate temp. at start	Cur- rent N.D <sub>1(8)</sub> =A	Volts	Time in min- utes	Ag depos- ited in gms.	Error in pct. of Ag in AgNO
0.2500	2	01	I	100	65°	1.0	6	25	0.2500	0.00
* 4	• •	• •	s 4	• •	••			• 4	0.2499	0.0 <b>2</b>
	• •	••	••	6.4	۰.	4.4	• •	+ 4	0.2503	c.o8
••	• •					4.4	•••		0.2501	0.03
· ·			••	• •		• •		• •	0.2497	0.07
	÷ 1	" "	<b>6 1</b>		• •	I.5	7	20	0.2502	0.05
**	• •	• (		" "		۰. ّ	7	* 1	0.2504	0.10
		• •	••	" "		14	6.5	••	0.2497	0.07
۰,		• •	• •	• •	**	••	6	• •	0.2499	0.02
* 4	4 é		4.4	**		۰.	6.5	• •	0.2495	0.12
••		" "	* 4	" "	• •	* *	6	÷ •	0.2498	0.05

Note: Speed of rotator in first five determinations was R. P. M.; in the last six run by Mr. Hill the speed was 550 R.P.M.

*Cadmium*. Exner's condition for cadmium<sup>2</sup> applied to the tin cathode with little alteration. Sulphuric acid was the electrolyte and this of course dissolved tin unless the dish was protected by the current. The circuit therefore had to be closed before adding the acid. Experiments showed also that only a small quantity could be used. With more than I cc. of acid, specific gravity, I.II5 (conc. acid diluted I to IO), the solvent action was more powerful than the tendency of the current to keep the metal at the cathode. But regardless of the solvent action, I cc. of the electrolyte gave the best deposits of cadmium. The experi-

<sup>1</sup> The gelatine should be pure and the solution clear. "Isinglass" was the material used.

<sup>2</sup> This Journal, 25, 903.

ments recorded in Table VIII were made to show the solvent action of different amounts of acid of the specific gravity noted above. The dishes were filled with hot water; the circuit closed: the rotator started, and the acid added from a pipette. The regular procedure of a determination was then followed.

TABLE VIII										
Weight of dish	H <sub>2</sub> SO <sub>4</sub> . Sp. Gr. 1.115 in ec.	Amperes	Time in minutes	Loss of Weight						
62.9874	3	5	IO	0.0009						
64.7832	2	5	10	0.0003						
62.9865	1	5	10	0.00						

In the experiments of Table IX, made by Mr. Hill, all the reagents were put in the dish before the circuit was closed, except the acid, the addition of which was the final detail of the manipulation. The deposits were crystalline, closely resembling zinc. The adherent coating was cleaned out once in four or five determinations, only the loose material being removed after the other depositions. Sulphuric acid aided by a feeble current dissolved the coatings, or else they were scoured off without using any solvent.

			1 A	15LE 1	А			
3CdSO4.8H2O Cd in gms.	H <sub>2</sub> SO <sub>4</sub> , Sp. Gr. 1.115 in ce.	Volume when dil- uted in cc.	Approxim- ate temp. at start	Cur- rent N.D <sub>100</sub>	Volts	Time in minutes	Cd de- posited in grams	Error in per cent Cd in 5CdSO4.8H2O
0.2500	I	100	65°	5	24-15	10	0.2502	0.04
64	2	<b>6</b> 4	••	••	**	۰.	0.2495	0.09
	1.5	• •		••	• •	••	0.2504	0.07
6.6	I		••	÷ •		••	0.2503	0.05
	÷ .	• •	• •	••	• •	• •	0.2499	0.02
**	**	• •	6.0	••		• •	0.2501	0.02
••	44	• •	4.4	••	۰۰	••	0.2503	0.05
• •	••	••		÷ •	• •	÷ •	ം <b>.250</b> 0	0.00

Note: Speed of the rotator was 550 R.P.M.

*Copper.* Copper, although usually most readily determined by electrolytic methods presented considerable difficulty. When a solution of the neutral or slightly acid sulphate was put in a tin dish a black deposit came down. Also, with the common acid electrolytes, loose black deposits resulted in the attempt to separate the metal by a current. Additions of ammonium hydroxide to the sulphate solution prevented the immediate formation of the black coating until the current was introduced but results from this electrolyte were not at all promising, the deposits being dark and loose.

An adaptation of E. Wagner's oxalate method<sup>1</sup> gave fairly good results although considerable manipulation was required. The dish, first of all, should be absolutely free from oxide of any sort, although a deposit may be made upon a surface of bright adherent copper. Then by observance of the following conditions good <sup>1</sup>Z. Electrochem., 2, 613. results may be obtained: Mix in a beaker 55 cc. of saturated ammonium oxalate solution, 5 cc. of saturated oxalic acid solution, and 1 cc. clear solution of pure gelatine (I gram of gelatine = 1000 cc.). Place the dish on its support, introduce this mixture, start the rotator and add the copper sulphate solution. Without delay close the circuit and dilute to a suitable volume. At the end of ten minutes add from a pipette 10 cc. of oxalic acid solution, at fifteen minutes add 5 cc., and at eighteen minutes, 5 cc. The deposition will then usually be complete after twenty minutes. The solution must be cold at the start, for otherwise the deposit will be black and loose. It is important too that the anode be rotating when the copper solution is added, since occasionally a very adherent blue material formed on it which could not be washed off. If the oxalic acid was not added at the stated times the results were invariably low, slight amounts of copper remaining in solution until the end of about thirty minutes. The deposits were dark red and crystalline but adherent enough to wash and weigh. They were removed by a hot potassium evanide solution. Series X was run by Mr. Hill.

			1.	ABLE V	L .				
CuSO <sub>4</sub> . 5H <sub>2</sub> O= Cu in grams	Saturated solution (NH <sub>4</sub> ) <sub>2</sub> C <sub>2</sub> O <sub>4</sub> in cc.	Saturated sclu- tion of H <sub>2</sub> C <sub>2</sub> O <sub>4</sub> at start, at 10, 15 & 18 minutes respectively	Vol- ume in cc. when dilut- ed	Approx- imate temp. at start	Cur- rent N.D <sub>100</sub> =A	Volts	Time in min- ntes	Cu de- posi'ed in grams	Erro T in pet. Cu in CuSO <sub>4</sub> 5H <sub>2</sub> O
<b>0.25</b> 00	55	5 - 10 - 5 - 5	100	20°	5	19-10	24	0.2498	0.02
••	••	+ <b>k</b>	• •	••	••	••	21	0.2495	0.05
- 6	••		• •	• •		• •	20	0.2502	0.02
× -	•	• •	* *	••	••	••	20	0.2492	0.08
	•	••	* *	• •	• •	••	20	0.2502	0.02
. 4			••	<b>6 1</b>	••	**	22	0.2502	0.02
		**	٤,	**	**	• •	2 I	0.2487	0.14
•	•	••	••	* 4	• •	**	22	0.2498	0.02
5 <b>6</b>	••	• 6	• •		• •	• •	20	0.2487	0.14
	••	••	* *	**	••	¥ 4	21	0.2490	O. I I
••	• •	* *	• •	• •	• *	4.4	20	0.2492	0.08

TABLE X

Note: Speed of the rotator was 550 R.P.M.

This method while fairly accurate if the conditions are closely observed, requires more manipulation than the methods on platinum. It was observed, too late for an investigation to be undertaken, that from cyanide solutions copper, with a high current density, comes down on tin as a bright and extremely adherent deposit just as it does on platinum. Since the cyanide was a satisfactory electrolyte for the quantitative determination of silver on tin, it is quite probable that it may also be used for copper. A thesis written in 1906 by Miss Anna L. Flanigan at the University of Pennsylvania describes some recent cyanide methods for copper deposition on platinum and it is intended to try and adapt them in this laboratory for use with tin cathodes. *Nickel.* Exner's conditions for nickel <sup>4</sup> served very well for the determination of this metal. There was no decided preference in the use of animonium sulphate and animonium hydroxide; or animonium acetate and animonium hydroxide as the electrolytes. The strong alkaline solutions should not stand longer than momentarily in the tin, unprotected by a current, but it is not necessary to have the circuit closed before putting in the reagents. The deposits were gray and very compact. A slight amount of loose material may be brushed out and the dish used for another determination, but if too thick a coating of nickel is deposited it will be found very difficult to remove. The nickel was dissolved with potassium cyanide aided by the current or by nitric acid, specific gravity 1.52.

TABLE XI

				I A DL P	-21				
NiSO <sub>4</sub> . 7H <sub>2</sub> O = Ni in grams	30 pet, acet- ic acid in cc,	NH4OH Sp. Gr. 0.90 in cc.	Volume in cc. when diluted	Approx- imate temp. at start	Current N.D <sub>100</sub> TrA	Volts	Time in min- utes	Ni deposit- ed in gms,	<ul> <li>Error in pct.</li> <li>of Ni in NiSO<sub>4</sub>.</li> <li>7H<sub>2</sub>O</li> </ul>
0.2500	10	30	100	65°	5	9	13	0.2497	0.03
• •	••	5.5	4.4	**	••	* 5	14	C.2502	0.02
• •	• •	š.	÷ 1	• •	۰.	* *	13	<b>0.2</b> 496	0.03
• •	• •	**	• •		••		• -	0.2500	0.00
••	• •	• •	1.5	•••	• •	11.1	× .	0.2504	0.03
x 1	k •	* *	••	• •	14	••	۰.	0.2491	0,08
۰.	•••	۰.	• •	• •	<b>6</b> -		4.5	0.2495	0.04
		÷.	• •		6.	• •		0.2501	0.01

Note: Speed of the rotator was about 700 R. P. M.

TABLE XII

NiSO <sub>p</sub> 7H <sub>2</sub> O= Ni in grams	(NH <sub>4</sub> ) <sub>2</sub> SO, in grams	NH,0H Sp. Gr. 0.90 in cc.	Volume in cc. when diluted	Approxim- ate temp. at start	Cur- reut N.Dpu- -=A	Volt+	Time in minutes	Ni deposi ed in gran	t- Error ns in pet. of Ni in NiSO4. 7H2O
0.2500	1.5	2,5	100	65°	5	18-10	15	0.2500	0.00
••	•	• •	÷ .	• •	۰.	• •	• •	0.2497	0.03
••	× i	**	* 4	• •	• •		16	0.2504	0.03
4.4	( I	4.1	6 1	÷+	i 1	• •	15	0.2500	0,00

Note: This series was run by Mr. Hill. Speed of the rotator was about 550 R.P.M. DEPARTMENT OF CHEMISTRY, SWARTHMORE COLLEGE May 18, 1997.

## THE DETERMINATION OF MANGANESE IN WATER.

BY ROBERT SPURE WESTON. Received April 20, 1907.

The need for a convenient method of determining manganese in water has been recognized by many analysts. In the ordinary practice of the water analyst either the determination is made gravimetrically or it is omitted.

Manganese together with iron occurs in many ground waters, and very often its presence complicates or vitiates the processes used for the re-

<sup>1</sup> This Journal, 25, 899.