

FERROMANGANESE CONTAINING TUNGSTEN.

I. Residue Baked to Dust Dryness on the Stove.

Treatment.	HF1.	HF1 + H ₂ SO ₄ .	HF1 + H ₂ SO ₄ .	HF1.	HF1 + H ₂ SO ₄ .	HNO ₃ + HCl.
Silica regained by one evaporation.....	0.0040	0.0022	0.0059	0.0038	0.0035
Silica regained by two evaporations	0.0058	0.0024	0.0057	0.0049	0.0036
Silica regained by three evaporations	0.0065	0.0022	0.0055	0.0051	0.0036
Silica regained by four evaporations	0.0062	0.0055
Total silicon regained	0.0030	0.0010	0.0026	0.0026	0.0017
Per cent. total silicon.	21.7	7.0	19.0	18.2	12.8
Total silicon in the alloy.....	0.0139	0.0141	0.0137	0.0143	0.0133	0.0141

II. Residue Burnt over the Flame.

Treatment.	HF1.	HF1 + H ₂ SO ₄ .	HF1.	HF1.	HF1 + H ₂ SO ₄ .	HF1.
Silica regained by one evaporation.....	0.0008	0.0016	0.0014	0.0006	0.0022	0.0037
Silica regained by two evaporations	0.0010	0.0016	0.0020	0.0004	0.0024	0.0037
Silica reg'n'd by three evaporations	0.0010	0.0020	0.0024
Silica regained by four evaporations	0.0009	0.0022
Total silicon regained	0.0005	0.0007	0.0010	0.0003	0.0011	0.0017
Per cent. total silicon.	3.62	5.11	6.86	2.11	8.09
Total silicon in the alloy.....	0.0138	0.0137	0.0145	0.0142	0.0136

In both tables the figures refer to the silica recovered from the ignited tungstic oxide which has been "separated" by digestion with cold ammonia solution.

THE ELECTROLYTIC DETERMINATION OF CADMIUM.¹

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THE work here presented grew out of an effort to find a practical method for determining this metal. The usual methods often fail to give satisfactory results, and, while we have not succeeded in finding an ideal method, we hope that the experiments here given will be of interest.

¹ Read at the meeting of the Nebraska Section, March 19, 1897.

The double oxalate method presents the following difficulties: It is not always easy to get and keep a clear solution. The plate is apt to oxidize if not very carefully treated, and the last traces of cadmium are hard to eliminate from the solution. Several determinations were made by this method. In each one a solution containing 0.0641 gram of cadmium was taken. A small storage battery of four volts furnished the current, which varied from two-tenths to one-half ampère. The time of each was from fourteen to eighteen hours.

	Cadmium found, Gram.
No. 1.....	0.0615
No. 2.....	0.0658
No. 3.....	0.0613

We then tried the precipitation in acetic acid solution in the hope of finding a more serviceable method. The result was in complete harmony with the experiments of Heidenreich. He says,¹ "The separation of cadmium from acetic acid solutions did not give satisfactory results. By adding ten cc. of fifty per cent. acetic acid to 120 cc. of the solution, and using a current of four-tenths ampère at a tension of seven and a half volts, the cadmium separated out in the form of leaf-like crystals. Experiments conducted with less acetic acid and with less current at a lower tension (two to ten cc. acetic acid, one-tenth to four-tenths ampère, four to seven and a half volts) at various temperatures, also gave no serviceable results."

Solutions which contained either phosphoric acid or sulphuric acid were also found to be unsatisfactory.

The cyanide solution seems to us best adapted to the determination of cadmium where more than one-tenth gram of the metal is present (solution 150-200 cc. and area of electrode for plating 100-150 sq. cm.) The usual objections to a cyanide solution may be mentioned. The solution offers an enormous resistance, hence a strong current is necessary. The last traces of the metal are hard to precipitate, and finally these solutions are exceedingly unpleasant to manipulate. A solution containing 0.0452 gram of cadmium as the double potassium cyanide was placed in a current from a thermopyle for twenty-four hours. The thermopyle has a tension of four volts and shows

¹ *Ber. d. chem. Ges.*, 29, 1587.

an ampère of two when no resistance other than the ampère-meter is inserted. 0.0446 gram of cadmium was precipitated.

DETERMINATION OF CADMIUM IN FORMATE SOLUTION.

The excellent results that zinc gives when determined in a formate solution¹ suggested to us that cadmium might be satisfactorily determined in a similar manner. The results are perfectly satisfactory provided certain conditions are rigidly maintained. There should be not more than one-tenth gram of metal in 150 cc. of solution. This amount of metal should be deposited on a surface of not less than 100 sq. cm. The voltage should not exceed three and four-tenths, and the ampère 0.15-0.20.

The solution of cadmium was made as follows: Chemically pure cadmium nitrate was converted into the oxide, which, in turn, was reduced, in a current of hydrogen, to the metal. The latter was then placed in a hard glass tube bent into the shape of W. The metal was placed in the first bend and distilled into the second in a current of hydrogen. The distillate thus obtained showed no trace of silica or other impurities. A weighed quantity of this distilled cadmium was dissolved in dilute sulphuric acid, placed in a measuring flask, and diluted to the mark. Portions of this solution were measured out from a burette. The weights, measuring flask, and burette used were carefully calibrated.

The source of the current was the thermopile. The precipitation was made with platinum dishes or cones in the usual manner. The average time was twenty-four hours.

The following is given as a representative determination. To a solution of cadmium sulphate six cc. of formic acid, sp. gr. 1.20, was added. Potassium carbonate was then added until a slight permanent precipitate formed. This precipitate was just dissolved in formic acid, then one cc. excess of the latter added. Next the solution was diluted to 150 cc. The solution was then put in a weighed platinum dish and placed in the current generated by the thermopile. The meters in the first determination read as follows:

Time.	Ampère.	Volts.
5 P.M.	0.1	2.6
10 P.M.	0.1	2.6

¹ This Journal, 18, 654.

The next morning the resistance of the rheostat was diminished from time to time.

Time.	Ampère.	Volts.
9 A.M.	0.125	3.0
12 M.	0.105	3.1
5 P.M.	0.195	3.3

Determination.	Weight of cadmium taken. Gram.	Weight of cadmium found. Gram.
1	0.0454	0.0452
2	0.0454	0.0454
3	0.0642	0.0642
4	0.0642	0.0642
5	0.0642	0.0641

A large number of determinations were made in more concentrated solutions, also with stronger currents, but these often failed to give a good adherent plate of the metal. The plates formed in the determinations given showed no tendency to oxidize and they could be kept for several days in the desiccator without change of weight.

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THE NORMAL URINE.

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THE various compilations current as "Text-books of Urine Analysis," differ materially in their statements as to the average composition of a normal urine. In many cases, indeed, the authors have not even attempted to reconcile their "totals" with the figures given for individual constituents, but aside from this, which is, of course, the result of carelessness on the part of the compiler, we find great variations in the original figures, due not so much to errors of determination as to failure to secure representative samples for analysis. Normals determined for one nationality, or for one class of one nationality, are commonly applied indiscriminately to all without regard to fundamental differences in conditions. For instance, the average American's habit of life is not that of the German student, and yet it is a fact that the majority of figures given in our text-books have originated with the observations of German professors, working in conjunction with their student assistants.