

## ELECTROLYTIC DETERMINATIONS.

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### I. URANIUM.

SOME years ago one of us found that it was possible to completely precipitate uranium<sup>1</sup> from an acetate solution by means of the electric current. The deposit consisted of the hydrated protosquioxide of uranium; which was ignited and weighed as  $U_3O_8$ . Indeed, the procedure was adopted in the separation of uranium from the alkali metals when they were associated in certain rare minerals. It has frequently been used since that time by the author and its reliability has been fully confirmed. However, Heidenreich<sup>2</sup> evidently experienced some difficulty in obtaining satisfactory results, for he remarks: "Versuche, Uran aus seiner Acetatlösung quantitativ abzuschneiden führten zu keinem Ergebniss, selbst bei 50-stündiger Einwirkung des elektrischen Stromes." A method, requiring fifty hours, would scarcely be regarded as having value even if the final results were really good. The following experiments, however, show that not only can satisfactory results be obtained, but they furthermore leave little doubt as to the time-factor, which is of prime importance in all determinations.

A solution of uranium acetate was prepared, which contained 0.1185 gram of urano-uranic oxide ( $U_3O_8$ ) in ten cc. of the liquid. To the latter volume were added five-tenths cc. of concentrated acetic acid and the whole was then diluted to forty cc. with water, when it was electrolyzed with a current  $N. D_{40} = 0.18$  A.  $V = 3$ . The temperature of the liquid during the decomposition was  $70^\circ$  C. The uranium was completely precipitated in six hours. The filtrate or solution poured from the deposit showed no traces of uranium, when it was evaporated and the residue tested for that metal.

<sup>1</sup> *Am. Chem. J.*, 1, 329; Smith's *Electrochemical Analysis*, p. 94.

<sup>2</sup> *Ber. d. chem. Ges.*, 29, 1587.

## RESULTS.

U <sub>3</sub> O <sub>8</sub> present in grams.	U <sub>3</sub> O <sub>8</sub> found in grams.
0.1185	0.1187
0.1185	0.1184
0.1185	0.1182

## 2. CADMIUM.

Those interested in electrolysis will probably remember that the writers have at various times proposed methods and conditions not only for the electrolytic determination, of this metal, but also for its separation from the more frequent associates. So often have we ourselves carried out these separations that we were inclined to believe other chemists would meet with like success. Such, however, seems not to have been the case, for in a very recent issue of this Journal<sup>1</sup> we felt called upon to publish very carefully outlined conditions for the complete deposition of cadmium from acetic and nitric acid solutions in order to again confirm our early statements. Heidenreich<sup>2</sup> mentions that the separation of cadmium from solutions containing sodium phosphate and free phosphoric acid gave results which were "ebenfalls nicht quantitativ; die letzten Reste von Kadmium sind nicht zu entfernen, selbst wenn der Strom bis 1-Ampère verstärkt wird." We regret exceedingly that this chemist has met with so little success in his efforts to repeat our work, but as in former instances of disagreement we again present new experimental evidence showing that our original suggestions were undoubtedly correct.

To ten cc. of a cadmium sulphate solution, containing 0.1656 gram of metallic cadmium, were added an excess of disodium hydrogen phosphate (1.0358 sp. gr.) and one and one-half cc. of phosphoric acid (1.347 sp. gr.). The liquid was then diluted to 100 cc., heated to 50° C., and electrolyzed with a current  $N. D_{100} = 0.06$  A. Volt = 3. The precipitation was finished in seven hours. The metal deposits were bright and adherent. They did not show the slightest trace of sponginess. At the expiration of four hours the current was increased to  $N. D_{100} = 0.35$  A. Volt = 7. The acid liquid was siphoned out without the interruption of the current. The pole separation, during the

<sup>1</sup> This Journal, 19, 870.

<sup>2</sup> *Ber. d. chem. Ges.*, 29, 1587.

entire precipitation, equaled one and one-fourth inches. The cadmium was washed with hot water and then carefully dried. The filtrates were examined but did not reveal a trace of unprecipitated metal. This is a direct contradiction of Heidenreich's statement that "die letzten Reste von Kadmium sind nicht zu entfernen."

## RESULTS.

	Cadmium present in grams.	Cadmium found in grams.
I .....	0.1656	0.1654
	0.1656	0.1658
	0.1656	0.1657

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**PRELIMINARY REPORT OF THE COMMITTEE ON  
COAL ANALYSIS.**

*To the President and Members of the American Chemical Society :*

At the Cleveland meeting of the American Chemical Society a Committee, consisting of W. F. Hillebrand, C. B. Dudley and W. A. Noyes, was appointed to consider the methods of proximate analysis of coal with a view to the adoption, if possible, of uniform methods for this country. After discussion among themselves, the committee have formulated the following outline of methods for analysis which they herewith submit, rather as a means of securing further information than as a final report. The committee very earnestly request all chemists interested in the matter, who may see this report, to send to the chairman of the committee a criticism of the methods proposed and a statement of any objections they see, and of any modifications which they think desirable.

Since the literature of the subject is widely scattered and much of it is not easily accessible, the committee request that all who have published papers bearing on the subject will, if possible, send to the committee reprints of such papers, or, if that cannot be done, a statement of where such papers can be found and the points covered.

Without the full and hearty cooperation of others the labors of the committee will be greatly increased and the conclusions finally arrived at will be less representative of general experience than it is desired they should be.