ELECTROLYTIC DETERMINATION OF MINUTE QUANTITIES OF COPPER¹

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The determination of quantities of copper less than one milligram cannot be carried out satisfactorily by the ordinary procedure of electrolytic analysis because of the impossibility of weighing with sufficient accuracy the dishes or other cathodes usually employed. Phelps² avoids this difficulty by precipitating the copper electrolytically, dissolving the deposit and estimating it colorimetrically. By the use, however, of a small electrode the colorimetric determination may be dispensed with and the copper weighed directly as deposited, a fair degree of accuracy being obtainable with quantities of copper as small as 0.05 milligram. The electrode is made from a small piece of platinum wire bent into a spiral with one end extending along its axis. It is supported in the bath by wrapping this free end four or five times about a hook of heavy platinum wire. If the hook be straight and slightly tapered toward the point the electrode may be easily attached and removed and makes perfect contact with the hook. The electrode used by the writer weighed about 0.3 gram.

The extraction and concentration of the copper are carried out by the usual methods. The electrolysis may be made in either nitric or sulphuric acid solution, low currents being used on account of the small cathode area. The writer uses 2 percent to 4 percent nitric acid and a few drops of sulphuric acid in about 25 cc solution and electrolyzes over night with about 1.8 volts and 0.01 ampere. The containing dish serves as anode. When the deposition is finished the electrolyte is washed out with a siphon or sucking-bottle without breaking the current. The electrode is grasped with a pair of small forceps, removed from the hook, washed successively in water, alcohol and ether, dried over sulphuric acid and weighed with great accuracy on a button balance.

¹ Read at New York meeting of American Electrochemical Society.

² Jour. Am. Chem. Soc., 28, 368 (1906).

The copper is then removed by nitric acid and the electrode washed, dried and weighed as before. The difference gives the weight of the copper. Following are some results, the first four obtained by electrolyzing a solution of known strength, the last two by redissolving the copper from one precipitation and reprecipitating on another electrode.

No.	Copper present, Mgs	Copper found, Mgs
1	0.529	0.509
2	0.492	0.498
3	0.556	0.558
4	0.499	0.500
Copper found		
No.	1st precipitation, Mgs	2nd precipitation, Mgs
5	0.092	0.104
6	0.348	0.344

Uniformly good deposits are obtained from solutions containing nothing but copper salts and acids. If, however, compounds of carbon or of platinum (derived from platinum dishes during the preliminary treatment) are present in the electrolyte the deposit will be black and loosely adherent, owing to contamination with these elements,¹ and the results will be high. Organic matter dissolved in the disintegration of a filter paper by nitric acid will in this way seriously interfere with the obtaining of good deposits. The difficulty may be avoided by making a preliminary (electrolytic) precipitation on a platinum dish in the usual way, dissolving the deposit and reprecipitating on the spiral electrode as above. The carbon and platinum remain undissolved on the first dish, and the electrolyte for the second precipitation is quite pure.

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¹ The precipitation of carbon during electrolysis has recently been investigated by Blum & Smith. *Proceedings Am. Philosophical Soc.*, 46, 60 (January-March, 1907).