It will be readily seen that the application of this bath is not confined to specific gravity determinations. At the same time that the one here described was built, another of the same surface area, but 22 inches deep, was constructed for use with the Roese fusel oil apparatus, which must be kept at constant temperature in order to get comparative results. Later, another, slightly smaller than the one described, was ordered for use with the Zeiss immersion refractometer. This has a basket of galvanized wire, bound with copper, instead of the perforated plate. The part of this basket next the ice-box is divided into compartments for holding four flasks. In front of these compartments is a strip of copper with holes for the small beakers used with the refractometer, under which is the strip of ground glass through which the light passes upwards. The wire frame for supporting the instrument is fastened to the sides of the basket, and the mirror for directing the light upward is attached below. The bath itself differs from those previously made in that the front side is of glass, to permit the entrance of light. This bath is kept at 20° C., and works just as perfectly as the one at 15.6° C.

The writer acknowledges suggestions from Messrs. Tolman, Howard and Munson, of this Bureau.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, No. 98.]

THE RAPID PRECIPITATION OF ANTIMONY IN THE ELECTROLYTIC WAY.

BY JULIA LANGNESS AND EDGAR F. SMITH.

Received August 28, 1905.

THE BEST electrolyte for the determination of antimony is sodium sulphide, containing a small amount of sodium hydroxide. The polysulphides, frequently present, have been destroyed in various ways, one of the most convenient being by the addition of potassium cyanide, first used by A. Fischer,[1] and subsequently and wholly independently by Exner,[2] when describing his experiences in the rapid determination of metals with the aid of a rotating anode. His results in the case of antimony were exceedingly good, both from the point of rapid and complete pre-

[1] *Ber.*, **36**, 2348.

[2] This Journal, **25**, 896.

cipitation of the metal as well as from the appearance of the deposit. Nevertheless, there seem to have been some difficulties encountered by other experimenters[1] upon repeating Exner's work with this metal, so that we have thought it only proper to communicate the observations made by us with the same metal during the past summer.

To begin, let it be noted that the antimony was present in solution both in its trivalent and quinquivalent form. Whichever compound it happened to be was weighed out and dissolved in a definite amount of sodium sulphide, diluting, of course, to a specified volume from which portions were removed for electrolysis. In this way a solution was prepared so that 10 cc. of it contained 0.2405 gram of antimony. Several such portions were treated as follows: to each were added 15 cc. of sodium sulphide (sp. gr. 1.18), 3 grams of potassium cyanide, 1 cc. of sodium hydroxide (10 per cent.), the solution was diluted to 70 cc. with water, heated almost to boiling and electrolyzed.

EXPERIMENTS.

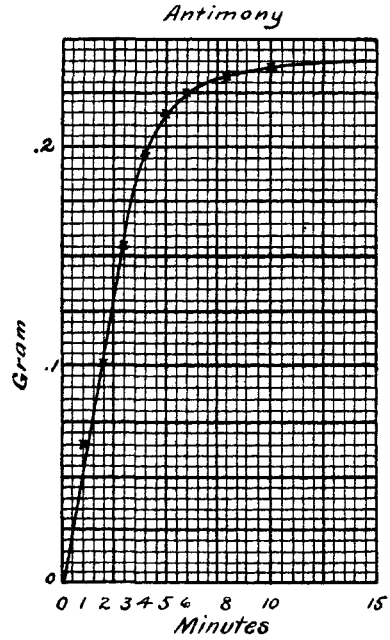
No.	Volts.	N. D_{100} Amperes.	Time. Minutes.	Antimony found. Gram.
1	3.5 to 4	6	15	0.2404
2	3.5 to 4	6	15	0.2409
3	3.5 to 4	6	15	0.2410
4	3.5 to 4	6	15	0.2402
5	3.5 to 4	6	15	0.2402

The precipitation was complete in each instance. The deposits of antimony were bright gray in color, like those of zinc. They were also perfectly adherent, exhibiting no signs of sponginess. Indeed, by employing roughened dishes as cathodes as much as 0.4851 gram and 0.4847 gram, and even 1 gram of metal were deposited in a beautiful and perfectly compact form in from twenty to twenty-five minutes. With conditions similar to those described above, experiments were conducted in a smooth platinum dish as cathode to ascertain the rate of precipitation. Thus, with a current of 6.5 amperes and 3.5 volts:

0.0652 gram of antimony was deposited in one minute,
0.1007 gram of antimony was deposited in two minutes,
0.1575 gram of antimony was deposited in three minutes,
0.1969 gram of antimony was deposited in four minutes,
0.2140 gram of antimony was deposited in five minutes,

[1] Fischer and Boddaert: *Z. f. Elektrochemie*, 10, 950.

0.2251 gram of antimony was deposited in six minutes,
0.2331 gram of antimony was deposited in seven minutes,
0.2369 gram of antimony was deposited in eight minutes,
and fifteen minutes for the entire amount of metal. These values gave the following curve:



Numerous experiments having been performed to subject the method to the most varying conditions, it was concluded to try it in the analysis of the mineral stibnite. Very pure samples of the latter were reduced to powder, and 0.5-gram portions were digested with 20 cc. (or more) of sodium sulphide (sp. gr. 1.18), filtered from the insoluble part, and after adding 3 grams of potassium cyanide and 1 cc. of sodium hydroxide to the solution the latter was heated to boiling and electrolyzed. Two results will suffice to show the applicability of the method.

Expt.	Mineral. Gram.	Volts.	Amperes.	Time.	Antimony. Gram.	Antimony. Per cent.
1.....	0.4241	3	7	25	0.3015	71.09
2.....	0.4488	3	7	20	0.3174	70.72

The deposits were all that could be desired. The residues contained no antimony. From a careful observation of the

method it may be added that the omission of the 1 cc. of sodium hydroxide from the electrolyte works no harm. The volume of sulphide may be reduced to 10 cc. without affecting the result; but then the quantity of alkaline cyanide should be reduced to 2 grams. The reduction of the cyanide to 1 gram without a corresponding reduction of sulphide is apt to give rise to a black border upon an otherwise most satisfactory deposit. If the volume of the alkaline hydroxide be increased to 10 cc., the antimony deposit will be black, powdery and non-adherent.

SEPARATION OF ANTIMONY FROM ARSENIC.

This separation can be carried out with perfect satisfaction when using the rotating anode. The conditions observed in getting the results given below were as follows: A solution of antimony oxychloride in sodium sulphide was made so that 10 cc. of it contained 0.1268 gram of antimony, and to each such portion were added 15 cc. of sodium sulphide (sp. gr. 1.18), 3 grams of potassium cyanide and water to increase the volume of liquid to 70 cc. On applying a current of 6 amperes and 4 volts the antimony was completely precipitated in from fifteen to twenty minutes.

RESULTS.

0.1268 gram of antimony.
0.1267 gram of antimony.
0.1269 gram of antimony.
0.1267 gram of antimony.
0.1269 gram of antimony.
0.1268 gram of antimony.

The deposit of metal was in each instance beautiful, light gray in color, metallic, and perfectly adherent. The arsenic existed in the solution as a sulpharsenate = (0.2000 gram of arsenic).

UNIVERSITY OF PENNSYLVANIA.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, No. 99.]

THE USE OF THE ROTATING ANODE AND MERCURY CATHODE IN ELECTRO-ANALYSIS.

BY LILY G. KOLLOCK AND EDGAR F. SMITH.

Received October 13, 1905.

(SECOND PAPER.)

IN THE previous communication upon this subject zinc, copper, nickel, cobalt, chromium and iron were the metals precipitated