

Coulometric Determination of Arsenic(III) and Antimony(III)

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Abstract

The coulometric determination of tripositive arsenic and antimony by electrogenerated iodine in a NaHCO_3 buffered solution of pH 8 has been described. The end point can be determined visually using starch as an indicator. Quantities of As(III) and Sb(III) down to the microgram level can be determined within permissible limits of error.

The coulometric determination of As(III) by electrogenerated halogens has been described by several authors. EVERETT and REILLY¹⁾ used iodine as an oxidant and employed a spectrophotometric method for the detection of end point, while RAMSEY, FARRINGTON and SWIFT²⁾ detected the end point by amperometry. LINGANE and BARD³⁾ compared the results obtained by amperometry, potentiometry and visual methods and found that the use of physical methods for detecting the end point, yielded more accurate results. They also reported that the visual end point method was satisfactory when macro-amounts of arsenic were to be determined. BROWN and SWIFT⁴⁾ used electrogenerated bromine for the oxidation of Sb(III) and detected the end point by amperometry.

In all the above work the pH was adjusted to 8 by phosphate buffer.

In the present communication we have reported the determination of these cations by liberated iodine, in presence of NaHCO_3 (pH 8) using starch as a visual indicator. The method is applicable down to microgram concentrations of As(III) and Sb(III).

Experimental

A LEEDS and NORTHRUP coulometric analyser (Model 7960) operated on 220 V/50cycles a. c. mains, with the titration kit (7961) was used. The apparatus is designed to generate the titrant at constant current and the micro-equivalents of the titrant consumed, can directly

1) G. W. EVERETT and C. N. REILLY, *Analyt. Chem.* **26**, 1750 (1954).

2) W. J. RAMSEY, P. S. FARRINGTON and E. H. SWIFT, *Analyt. Chem.* **22**, 332 (1950).

3) J. J. LINGANE and A. J. BARD, *Analyt. Chim. Acta* **16**, 271 (1957).

4) R. A. BROWN and E. H. SWIFT, *J. Amer. chem. Soc.* **71**, 2717 (1949).

be read on the scale. The solution to be analysed was stirred with a magnetic stirrer during the reaction.

Potassium-antimonyl tartrate (B. D. H. AnalaR) and arsenious oxide (B. D. H. AnalaR) were used for preparing solutions of potassium antimonyl tartrate and sodium arsenite respectively, which were standardised by iodometry in the usual way.

In some earlier work, nitrogen was used to remove dissolved oxygen, but it was found unnecessary in these experiments, which supports the observation of LINGANE and BARD³).

Measured volumes of the solution to be analysed were taken in the titration cell, and 5 ml of a saturated solution of NaHCO_3 were added, to obtain a pH ca. 8 in the mixture. 5 ml of 5% KI were added and iodine was generated in the solution by electrolysis. The end point was detected by 1 ml of 1% starch solution added to the mixture, near the completion of the reaction. Blank reading was also taken in the absence of As(III) or Sb(III) and were accounted for in the observations. The results are given in the tables below:

Table 1
Determination of Arsenic (III)

As (III) taken μ gm	No. of Determinations	Average micro- equivalents consumed	Average As (III) found μ gm	Error of mean, %
23.3	7	0.6380	23.85 ± 0.15	+ 0.21
59.8	4	1.6050	59.77 ± 0.23	+ 0.06
119.5	5	3.1960	120.45 ± 0.95	+ 0.8
178.5	3	4.6550	176.60 ± 3.80	- 0.9
239.1	3	6.2930	236.85 ± 6.95	- 0.9
298.9	4	7.8730	295.90 ± 6.10	- 0.9
597.6	2	15.9840	598.65 ± 1.25	+ 0.2

Table 2
Determination of Antimony (III)

Sb (III) taken μ gm	No. of Determinations	Average micro- equivalents consumed	Average Sb (III) found μ gm	Error of mean, %
12.0	5	0.2014	12.05 ± 0.65	+ 0.41
24.0	5	0.3950	24.45 ± 1.85	+ 1.88
36.0	5	0.5900	36.00 ± 1.00	0.00
60.0	4	0.9640	58.85 ± 0.85	- 1.90
90.0	2	1.6020	91.00 ± 2.00	+ 1.10
150.0	4	2.5050	151.50 ± 0.50	+ 1.00
3000.0	3	4.9680	301.60 ± 0.60	+ 0.53

It may be seen that the error lies within permissible limits and the method is useful for micro-determination of As(III) and Sb(III). It is also convenient, because it does not require an auxiliary arrangement for the detection of the end point.

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Bei der Redaktion eingegangen am 7. Januar 1963.