ESTIMATION OF SULPHIDE IN PRESENCE OF OTHER SULPHUR COMPOUNDS

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INTRODUCTION

There has been a growing interest in sulphur chemistry and accurate determination of sulphur compounds in mixures.

A number of methods have been used to determine and separate the S^{-2} ion from other anions and also in a mixture of other sulphur compounts[1]. Yoshimura Chozo [2] employed both conductometry and poentiometry for the estimation of S^{-2}. Zhustareva and others[3] adopted polarographic method by studing the electrochemical behaviour of S^{-2}.

Chromatographic methods have also been reported for the separation and identification of S^{-2}, S_2O_3 ^{2} etc.

Dooley and Hirsch[5] used mass-spectrophotometric techniques for the estimation of sulphur compounds in crude oil. Some workers have followed the spectrophotometric methods[6] for analyses of mixture of S^{-2}, SO_3^2, and $SO_2O_3^{-2}$, and SO_4^{-2} in aqueous solution. Aosi Itsuo [7] undertook the fluorimetric determination of S^{-2} ion with 7,4,6 — Tetrachloraffleurescien mercury compounds.

More recently ion selective electrodes have been used for S^{-2} determination. Thus Ochme, and other[8] constructed and applied sulphide — sensitive electrodes in the analysis of ground waters and waste waters.

Most of these methods, however, are rather complicated and need sophisticated instruments. The methods described in the present communication pertains to the conditions and facilities availbale in our laboratories.

EXPERIMENTAL

All the chemicals used were of reagent grade.

1. *Standard Lead Solutions*. 0.15 M: Dissolved 40 g of $PbAc^-$ in distilled water and made up the volume to one litre wth distilled water and a few drops of acetic acid. This was standardised against standard E.D.T.A. Solution.
2. Standard E.D.T.A. Solution — 0.01M. (4gl) in distilled water.
3. *Buffer — pH10*. Dissolved 67.5 g NH_4Cl in 570 ml of conc. NH_4OH and adding sufficient distilled water to make one liter of solution.
4. Erichrome-black-T —————————— 0.2 % in triethanolamine.
5. Tartaric acid/rochelle salt.

Procedure

The apparatus for the determination of S^{-2} ion consists of a round bottom flask (reactor) equipped with a carrier — gas inlet tube connected to the side arm and a delivery tube the end of which is connected to the reservoir (Trap for H_2S) containing standard lead accetate solution. 0.25-1.0 g of sodium sulphie was mixed with different ratios of sodium salts of $S_2O_3^{-2}$ and SO_3^{-2} and placed in the reactor. Nitrogen gas was passed to expell the air present in the reactor. The H_2S evolved was swept by nitrogen gas at the rate of 30-40 ml/min into absorber containing a known volume of standard lead acetate solution. The reaction completes in about half an hour. The apparatus was disconnected and the lead sulphide formed was then filtered. The filtrerate was analysed for Pb contents against std E.D.T.A. using Buffer pH10 with a few grams of tartaric acid or Rochelle salt and Erichrome black-T as indicator[9]. From this the equivalent amount of S^{-2} was estimated.

RESULTS AND DISCUSSION

Table 1 gives the various ratios in which the synthetic mixture of S^{-2}, SO_3^{-2} and $S_2^{-2}O_3$ was prepared and analysed for H_2S production. It is evident that the addition of sulphur compounds such as $S^{-2}O_3$ and $S_2^{-2}O_3$ in the mixture, in equimolar or in different ratios does not affect two much the evolution of H_2S, and this do not hinder the estimation of S^{-2}. The end point is very sharp and more stable. The proposed method also does not require any

Table 1. Analysis of mixtures of sulphur compounds

S.No.	Ratio of synthetic mixture (g)				Sulphide added as $Na_2S. 9H_2O$ (gm)	Sulphide found as $Na_2S. 9H_2O$ (gm)	Error (gm)	% Error
	$Na_2S.9H_2O$	Na_2SO_3	$Na_2S_2O_3$	$5H_2O$				
I)	1	–	1	–	1	1.0	0.999	−0.001	−0.1
II)	0.75	–	1	–	1	0.75	0.755	+0.005	+0.6
III)	0.50	–	1	–	1	0.50	0.50	0.50	–
IV)	0.25	–	1	–	1	0.25	0.248	−0.002	−0.8
V)	1	–	0.75	–	1	1.00	0.998	−0.002	−0.2
VI)	1	–	0.50	–	1	1.00	1.00	0.00	–
VII)	1	–	0.25	–	1	1.00	1.00	0.00	–
VIII)	1	–	1	–	0.75	1.00	1.006	+0.006	+0.6
IX)	1	–	1	–	0.50	1.00	1.00	0.00	–
X)	1	–	1	–	0.25	1.00	0.999	−0.001	−0.1

masking agent because the presence of other S compounds do not interfere with the estimation of sulphides.

The present method was also compared with the standard iodometric method[10]. The values obtained by both the methods were in agreement wtih each other. It shows that the present method is as good as the established standard method.

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