Amperometric Determination of Metal Ions with Thio acids - V(V) and Mo(VI) With 2-Mercaptopropanoic acid

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2-Mercaptopropanoic acid (RSH) has often been used as a complexing agent [1-5] and sometimes as an analytical reagent for the amperometric determination of various transition metal ions [5-7]. The present work was undertaken with a view to examine it further.

EXPERIMENTAL

Stock solution of 2-mercaptopropanoic acid was prepared afresh daily and standardised using standard chloramin T solution [9]. NH_4VO_3 and $(NH_4)_6Mo_7O_{24}.4H_2O$ were employed as source compounds for the respective metal species. Stock solutions of V(V) were prepared and standardised [8]. Standard solutions of Mo(VI) were prepared by direct weighing of the source salt. Only analytical grade chemicals and doubly distilled water were used.

All titrations were performed at DME vs SCE using a manual polarograph (Toshniwaal Model CLO2A). Purified nitrogen was used for deaeration. All titrations were carried out thrice.

Titration voltages were selected in the limiting region of the anodic wave of RSH; metal solutions were employed as titrant (anodic titration).

RESULTS AND DISCUSSION

V(V) is reported to give irreversible wave in 0.1 M KCl [8]. Limiting region stretches from -1.55 to -1.70 V. Molybdenum also gets reduced but at

Table 1

Parameters of the titration

Parameter	V(V)	Mo(VI)
Medium	0.1 M KCl	2.5 M NaCl in 7% HAc [6]
Limiting current region of RSH in Volts	0.0 to -0.2	-0.02 to -0.08
Titrn. potl.	-0.10 V	-0.04 V
Conc.range ppm	255 - 6	480 - 9.6
Max error	1% (dil.sol)	1%

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Table 2

Safe Limit of Concentration wrt Metal Species in Molar tolerated

Foreign Ions	V(V)	Mo(VI)
Thiosulphate	x 1/5	x 1/20
Oxalate	equal	x 10
tungstate	> 20	x 5
Vanadium	-	< 1/20
Cr(III)	> 20	equal
Cr (VI)	<1/20	x 10
Fe(II)	x 1/20	x 1/20
Mo(VI)	<1/20	-
Cadmium	x 1/10	x 1/15

more negative potentials. In both cases, titrant was the metal solution and the titration potential was held at the anodic limiting current of RSH where no metal reduction current exists. The anodic current reduced with progress of titration and reached a constant current at the end of titration giving a reverse L shaped curve. However in the case of Mo(VI) titration, 50% current remains after the null point. Addition of aliquots of Mo(VI) solution produced yellow colour. Stoichiometry in both case is M:L = 1:2.

Results of the titration are presented in Table 1.

Interference Studies

These studies were carried out at 5.0, 1.0 and also 0.5 mM of each metal. The following foreign ions - Sulphate, nitrate, acetate, aluminium, divalent solutions of Mg, Mn, Co, Ni and Zn- were tolerated even when present in 20 fold excess of the metal ion. Hg(II), Fe(III) and Mn(VII) interfere seriously even if present to 1/20 times the metal. Intermediate cases are given in Table 2.

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