J. Indian Inst. Sci. 62 (B), Oct. 1980, Pp. 173–176.

Short Communication

Titrimetric estimation of phosphate, molybdate and sulphate with lead nitrate solution, using 2-(2′-lepidyl azo)-1-naphthol-4-ammonium sulphonate (Lanas) as visual indicator

R. C. CHADHA, B. S. GARG*, SWARAN LATA AND R. P. SINGH
Department of Chemistry, University of Delhi, Delhi 110 007 (India).

Received on September 20, 1980.

Abstract

2-(2′-Lepidylazo)-1-naphthol-4-ammonium sulphonate (LANAS) forms a blue coloured complex with Pb(II) which is readily destroyed on addition of sulphate, phosphate and molybdate. Titrimetric procedures for the estimations of these ions have been developed. Large number of complexing anions do not interfere.

Key words : Titrimetric estimations, sulphate, phosphate and molybdate, LANAS as metallochromic indicator.

1. Introduction

Earlier LANAS has been found to be useful as a visual indicator in titration of metals[1–4]. Titrimetric estimation of molybdate, tungstate, phosphate and sulphate with lead nitrate solution using LANAS as indicator under specified conditions is rapid and sensitive. Sommer and Janoscova[5] have established the conditions under which PAR, TAR, TAMP and 1–PAN–4S can be used as indicators in titrimetric estimation of phosphate, molybdate, tungstate and sulphate. They have also mentioned the difficulty in the estimation of tungstate and sulphate. The present study shows that LANAS can be used successfully as visual indicator for rapid titrimetric estimation of these anions by titration with lead nitrate solution. The titration is easy and accurate under specified conditions which have been worked out in individual case.

* To whom all correspondence should be addressed to.

2. Experimental

Indicator solution, 0·01% (w/v) in doubly distilled water, hexamine buffers of pH 6·0 and 7·0. and sodium acetate acid buffer of pH 4·5–5·5 were prepared. Analytical grade reagents and doubly distilled water were used for the standard solutions.

LANAS was prepared[1] by the condensation of 1, 2-naphthoquinone-4-sodium sulphonate with 2-hydrozino lepidine in the presence of concentrated ammonia. The compound was isolated, purified and characterized by thin layer chromatography and elemental analysis.

3. Titration procedure

To a suitable aliquot containing 0·96 to 100·0 mg of sulphate or 0·90 to 100·0 mg of phosphate or 0·80 to 160 mg of molybdate in a 100 ml conical flask, add 4 drops of 0·01% (w/v) indicator, followed by a few drops of very dilute solution of nitric acid to get yellow colour. Then add buffer solution dropwise till the pH is 6·0–7·5 (hexamine/HNO_3) in the case of sulphate and phosphate; 4·5–5·2 (NaAc/HAc) in the case of molybdate. In the case of sulphate add the organic solvent (2·0 ml DMF and 2·0 ml isopropanol for 20·0 ml). Raise the volume to 20·0 ml and titrate with a standard Pb(II) solution till the end point, orange or pink to blue colour is obtained. The titrations can be done at room temperature in the case of sulphate and molybdate and above 70° C in the case of phosphate. Detection of end point is easier even in the case of sulphate at high temperature. Large amount of indicator obscure the end point in the case of molybdate. The relative error is generally less than 1%.

4. Effect of diverse ions

To ensure the suitability of the titration procedures, effects of diverse ions have been studied in detail. The tolerance limit (in ppm) of the ions which did not cause error of more than 1% in the determination of 0·96 mg of sulphate or 0·90 mg of phosphate or molybdate 1·6 mg per 20·0 ml are summarised in Table I.

Sulphate, phosphate and molybdate interfere with each other in the determinations. Other anions which interfere in all cases are tungstate, oxalate, chromate and sulphite. Metal ions, such as zinc(II) and manganese(II) which give coloured complexes with LANAS, also interfere in all cases[2,6].

LANAS thus serves as a good visual indicator for the precise titration of the above anions in the presence of large number of other ions. The lead : phosphate ratio in the precipitate is 5 : 3. The composition of the precipitate in the presence of chloride is $Pb_5(PO_4)_3Cl$ ($SP = 7·5 \times 10^{-80}$). In phosphate titration large amounts of chloride,

Table I

Effect of diverse ions

Foreign ion	Sulphate	Phosphate	Molybdate
Fluoride	90	400	95
Chloride	300	1000	400
Bromide	1000	1500	1000
Iodide	100	1800	100
Acetate	5000	5000	5000
Thiosulphate	110	200	1100
Nitrate	4000	4000	4000
Nitrite	60	450	2100
Borate	100	150	100
Citrate	Interferes	200	50
Tartarate	100	500	300
Thiocyanate	1400	4000	4000
Cyanide	2000	2000	2000
Thiourea	2000	2000	2000
Hydroxylamine	350	700	2100
Ascorbic acid	100	200	860
Cobalt* (II)	500	400	350
Nickel* (II)	150	125	125
Mercury* (II)	500	500	500
Copper* (II)	100	90	100
Magnesium(II)	400	400	1000
Barium(II)	Interferes	40	1370
Aluminium (III)	20	30	50
Bismuth** (III)	200	104	200
Tin (II)	Interferes	600	Interferes
Aluminium*** (III)	100	130	50

* = masked with cyanide, ** = masked with chloride, *** = masked with fluoride.

bromide, and acetate do not interfere like other known ligands. Molybdate could be titrated at low pHs and interference of number of metal ions thus could be avoided.

Acknowledgement

The authors are thankful to the University of Delhi, Chemistry Department, for providing necessary facilities and to Dr. S. K. Mehrotra for helpful discussions.

References

1. CHADHA, R. C., SINGH, I., GARG, B. S. AND SINGH, R. P. — Analytical potentialities of a new heterocyclic azo dye: 2-(2'-lepidylazo)-1-naphthol-4-ammonium sulphonate (LANAS) as a metallo-indicator for titrimetric determination of mercury (II) with EDTA, *Curr. Sci.*, 1976, **45**, 830–832.

2. CHADHA, R. C., GARG, B. S. AND SINGH, R. P. — Complexometric estimation of some transition elements (having $3d^5$ to $3d^9$ electronic configuration) using LANAS as visual metallochromic indicator, *Indian Nat. Sci. Acad.*, 1979, **45**, 20–24.

3. CHADHA, R. C., GARG, B. S. AND SINGH, R. P. — Ammonium 2-(2'-lepidylazo)-1-naphthol-4-sulphonate as indicator in mercurimetric determination of halides, *Talanta*, 1979, **26**, 329.

4. CHADHA, R. C., GARG, B. S. AND SINGH, R. P. — 2-(2'-lepidylazo)-1-naphthol-4-ammonium sulphonate (LANAS) as visual metallochromic indicator in complexometric determination of lead (II), *Curr. Sci.*, 1979, **48**, 15.

5. SOMMER, L. AND JANOSCOVA, L. — Contribution to the determination of some anions by titrating with lead (II) nitrate using heterocyclic azo dyes as indicator. *Coll. Czech. Chem. Commun.*, 1974, **39**, 101–109.

6. CHADHA, R. C., LATA, S., GARG, B. S. AND SINGH, R. P. — LANAS as a metallochromic indicator in complexometric estimation of zinc, cadmium and mercury, *Indian Chem. Soc.*, 1980 (communicated).