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NOTE

Analytical Application of 3-Hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene as Metallochromic Indicator for Complexometric Determination of Molybdenum(VI)

SABA KHAN* and REHANA KHANAM†
Department of Chemistry, Vidhya Bhawan Rural Institute, Udaipur-313 001, India
E-mail: masoom_75@rediffmail.com

A new reagent 3-hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene has been used for the spectrophotometric and complexometric determination of Mo(VI). Spectrophotometric determination is carried out at 408 nm, in the pH range 2.0-3.0. Beer's law is obeyed in the range (1 to 6) × 10^5 M. The values of molar absorptivity and Sandell's sensitivity are 7184.7 dm^3 mol^{-1} cm^{-1} and 13.35 ng cm^{-2}, respectively. It has also been used as metallochromic indicator in complexometric titration of Mo(VI), where the colour change is from light greenish yellow to colourless at the end point. The best results are obtained in pH range between 2-3. Satisfactory results have been obtained in the entire studied range.

Key Words: Hydroxytriazene, Molybdenum(VI), Metallochromic indicator.

Survey of literature reveals that hydroxytriazenes have been used as metallochromic indicators for various transition metals[1-7]. Hydroxytriazenes have also been reported to be used as spectrophotometric reagents for Mo(VI)[8,9]. Herein, the application of 3-hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene for complexomertic determination of Mo(VI) has been reported.

Standard solution of Mo(VI): A 1.0 × 10^{-2} M stock solution of Mo(VI) was prepared by dissolving appropriate quantity of AR grade molybdic acid in minimum quantity of sodium hydroxide (0.1M)and making it up to the required volume with double distilled water. The solution was standardized with EDTA.

Standard solution of EDTA: A 1.0 × 10^{-2} M stock solution of EDTA was prepared by dissolving appropriate quantity of disodium salt of EDTA(BDH; AR grade) in double distilled water and standardized using $ZnSO_4$ solution and xylenol orange[10] as indicator.

Indicator solution: 0.2% indicator solution was prepared by dissolving required quantity of hydroxytriazene in ethanol.

Method: For preliminary study 10 mL of ethanol was added to 10 mL of Mo(VI) and pH was then adjusted between 2.0 to 3.0 with perchloric acid. 4 drops of 0.2 % indicator was added. A light greenish yellow colour developed. This solution

†Department of Chemistry, M.L. Sukhadia University, Udaipur-313 001, India.

was then titrated against 1.0×10^{-2} M EDTA. A sharp colour change from light greenish yellow to colourless is observed at the end point. The effect of pH was studied by carrying out a number of titrations between 10 mL of 1.0×10^{-2} M Mo(VI) solution and equimolar EDTA solution adjusting the pH between 1 to 5. Most accurate results were obtained between 2-3 pH range. For the study of effect of temperature 10 mL of Mo(VI) solution was diluted to 20 mL with ethanol, keeping the pH between 2-3 this flask was kept in large water bath of selected temperatures range and titrated with standard EDTA solution.

For determining the optimum concentration of Mo(VI) different concentrations, 2×10^{-2} M, 1.0×10^{-2} M, 2.0×10^{-2} M, 1.0×10^{-2} M and 5.0×10^{-4} M were used for titrations against EDTA. For the sake of comparison, the whole process was repeated using sulfosalicylic acid[11] as indicator.

For interference studies, 10 mL of 1.0×10^{-2} M Mo(VI) solution (9.59 mg) was titrated with equimolar EDTA solution using the given reagent as indicator. Titration was then repeated in presence of desired quantity of foreign ion whose interference was being studied. Thus 10 mL of 1.0×10^{-2} M Mo(VI) solution was mixed with 9.59 mg of particular foreign ion and then titrated with equimolar EDTA solution. If 9.59 mg of foreign ion didn't interfere then same titration was repeated with increasing the quantity of ion to 47.5 mg in second level and 95.9 mg in third and highest level of the interference study. However tolerance of still higher level was not studied.

3-Hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene which has been used as spectrophotometric reagent is also used as metallochoromic indicator in complexometric determination of Mo(VI).

The reagent gives a sharp and perceptible colour change at the end point *i.e.* from light greenish yellow to colourless.

Effect of pH: The most accurate result is obtained in the pH range 2-3, where intensity of colour of Mo(VI) solution with the indicator is maximum and colour change at end point is sharpest.

TABLE- 1
COMPLEXOMETRIC DETERMINATION OF Mo(VI) USING
3-HYDROXY-3-*m*-TOLYL-1-*m*-CHLOROPHENYLTRIAZENE
Volume of Mo(VI) in each titration = 10.0 mL, pH range = 2.0 to 3.0
Colour change at end point = light greenish yellow to colourless

Concentration of Mo(VI) solution	Volume of EDTA of corresponding conc. consumed (mL) when		Change in colour at end point using reagent as indicator
	Hydroxytriazene is used as indicator	Sulfosalicylic acid is used as indicator	
2×10^{-2} M	10.0	10.0	Light greenish yellow to colourless
1×10^{-2} M	10.0	10.0	Light greenish yellow to colourless
2×10^{-3} M	9.9	10.0	Light greenish yellow to colourless
1×10^{-3} M	9.7	10.0	Light greenish yellow to colourless
5×10^{-4} M	–	–	End point is not perceptible

Effect of temperature: Three temperature ranges are selected *viz.*, 20-30, 30-40 and 40-50 ºC. Titration between 10 mL of Mo(VI) and EDTA at any temperature between are 20-50 ºC gives satisfactory results indicating that reagent can be used as metallochromic indicator at room temperature.

Optimum concentration range of Mo(VI): The effect of Mo(VI) was studied to find minimum concentration of Mo(VI) that could be satisfactorily titrated using the given indicator. The results showed that even low concentration such as 1.0×10^{-3} M of Mo(VI) solution could be titrated with fairly excellent results. Result is comparable with standard method using sulfosalicylic acid as indicator.

Interference of 24 common cations and anions in the complexomertic determination of molybdenum using the given indicator has been studied at three levels. These ions are Na^+, K^+, Mg^{2+}, Pd^{2+}, Pb^{2+},Cd^{2+}, Fe^{3+}, Co^{2+}, Ca^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, F^-, Cl^-, Br^-, I^-, NO_2^- NO_3^-, SO_4^{2-}, CO_3^{2-}, $C_2O_4^{2-}$, PO_4^{3-}, CH_3COO^-, WO_4^{2-}. It was seen that 20 ions, *viz.*, Na^+, K^+, Mg^{2+}, Pb^{2+}, Cd^{2+}, Co^{2+}, Ca^{2+}, Ni^{2+}, Zn^{2+}, Cl^-, Br^-, I^-, NO_2^- , NO_3^-, SO_4^{2-}, CO_3^{2-}, $C_2O_4^{2-}$, PO_4^{3-}, CH_3COO^-, WO_4^{2-} were tolerated when added in equivalent amount of Mo. Out of these, following 19 ions were tolerated when added in 5 fold excess to Mo solution: Na^+, K^+, Mg^{2+}, Pb^{2+}, Cd^{2+}, Co^{2+}, Ca^{2+}, Ni^{2+}, Zn^{2+}, Cl^-, Br^-, I^-, NO_2^-, NO_3^-, SO_4^{2-}, CO_3^{2-}, PO_4^{3-}, CH_3COO^-, WO_4^{2-}. The 18 ions tolerated at still higher level *i.e.*, 10 fold excess to molybdenum solution were Na^+, K^+, Mg^{2+}, Pb^{2+}, Cd^{2+}, Co^{2+}, Ca^{2+}, Ni^{2+}, Zn^{2+}, Cl^-, Br^-, I^-, NO_2^-, NO_3^-, SO_4^{2-}, CO_3^{2-}, PO_4^{3-}. CH_3COO^-. Thus from the above studies it can be concluded that 3-hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene can be successfully used for complexometric determination of Mo(VI) even in presence of many fold excess of interfering ions.

REFERENCES

1. A.M. Golwalkar and D.N. Purohit, *Ciencia E. Cultura (Brazil)*, **38**, 552 (1986).
2. D.N. Purohit, J.P.C. Jaimini and N.C. Sogani, *J. Inst. Chemists (India)*, **48**, 123 (1966).
3. A.K. Manjumadar and D. Chakraborti, *Anal, Chem Acta*, **55**, 450 (1971).
4. R.C. Sharma, R.S. Chauhan, A.K. Goswami and D.N. Purohit, *Asian J. Chem.*, **54**, 207 (1995).
5. R. Bhatnagar, Ph.D. Thesis, Hydroxytriazene in Cobalt Determination, M.L.S. University, Udaipur, India (1988).
6. D.N. Purohit and A.M. Golwalkar, *Ciencia E. Cultura (Brazil)*, **37**, 777 (1985).
7. R. Singh, Ph.D. Thesis, Spectrophotometric and Complexomertic Determination of Copper with Hydroxytriazenes, M.L.S. University, Udaipur, India (1989).
8. J.K. Humad, Ph.D. Thesis, Hydroxytriazenes in the Determination of Zinc, Cadmium and Molybdenum(VI), M.L.S. University, Udaipur, India (1984).
9. T. Babel, S. Khan, A. Mehta, R.S. Chauhan and A.K. Goswami, *J. Indian Chem. Soc.*, **81**, 799 (2004).
10. Complexometric Assay Methods with Tritriplex, Pub. E. Merk, Darmstadt, edn. 3, pp. 38-39.
11. G.H. Jeffery, J. Bassitt, J. Mendham and R.C. Denney, A.I. Vogel's, Text Book of Quantitative Chemical Analysis, ELBS, edn. 15 (1994).

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