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*NOTE*

# Complexometric Determination of Chromium(III) from Binary and Ternary Metal Ion Mixture Using 3-Hydroxy-3-*p*-Tolyl-1-*p*-Nitrophenyltriazene

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A new indicator 3-hydroxy-3-*p*-tolyl-1-*p*-nitrophenyltriazene was developed for the simultaneous complexometric determination of Cr(III) from binary and ternary metal ion mixtures by kinetic masking. The method is simple and efficient for the complexometric determination of trace amount of Cr(III).

Hydroxytriazenes are although an important class of complexing agents[1–5] but so far no hydroxytriazene has been used as an indicator for the complexometric determination of chromium(III). In the present work synthesis and application of 3-hydroxy-3-*p*-tolyl-1-*p*-nitrophenyltriazene in the complexometric determination of Cr(III) has been attempted.

Stock solutions of $1.0 \times 10^{-2}$ M Cr(III), Fe(III), Ni(II) and Cu(II) were prepared by dissolving requisite amounts of AR grade salts in double distilled water which were standardised by usual methods. A 0.2% indicator solution was prepared by dissolving the requisite quantity of 3-hydroxy-3-*p*-tolyl-1-*p*-nitrophenyltriazene in acetone. Fresh solutions were prepared as and when needed. A fresh 1% hexamine buffer solution was also prepared by dissolving it in minimum quantity of double distilled water and then diluting it with ethanol.

## Synthesis of 3-Hydroxy-3-*p*-Tolyl-1-*p*-Nitrophenyltriazene

*p*-Nitrotoluene (0.2 mole) was reduced by $NH_4Cl$ (18.0 g) and zinc dust (50.0 g) in aqueous solution at temperature between 60–65°C in about 90 minutes. The *p*-tolylhydroxylamine so obtained was dissolved in ethanol and coupled with diazotized product of *p*-nitroaniline (0.2 mole) between 0–5°C using sodium acetate buffer at pH 5.0. The crude product was recrystallized twice with acetone to form yellow crystals of 3-hydroxy-3-*p*-tolyl-1-*p*-nitrophenyltriazene. Yield: 64%, m.p.: 182°C, m.f.: $C_{13}H_{12}N_4O_3$. Elemental anaylsis, % found (calcd.): C: 57.34 (57.35), H: 4.40 (4.41), N: 20.60 (20.58).

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Cr(III) in metal salt solutions could be satisfactorly determined in trace amount using hydroxytriazene indicator. For this Cr(III) solutions of different concentrations were prepared each of which was complexed with excess EDTA solution of corresponding concentrations between pH 3.0 to 4.0 with 1 M $H_2SO_4$ for about 30 min. The red violet Cr(III)-EDTA complex formed was buffered between pH 5.2 to 6.0 using hexamine buffer and diluted to 150 mL with acetone to remove any turbidity. The solution was titrated against standard $Fe(NO_3)_3$ solution with few drops of hydroxytriazene indicator to find excess EDTA. The colour change at the end point was from violet to yellow. The minimum concentration of Cr(III) determined was $2.54 \times 10^{-3}$ g and the amount of metal ions found was in good agrement with the amount of metal ions actually present; so the determinations were quite accurate.

## Interference Studies

It was found that 5.2 mg of Cr(III) could be successfully determined in presence of 10 mg of either of the following 16 cations and anions, *viz.*, $Na^+$, $K^+$, Ca(II), Mg(II), Ni(II), Cu(II), Ba(II), Zr(IV), $Cl^-$, $Br^-$, $I^-$, $NO_2^-$, $NO_3^-$, $SO_3^{2-}$, $SO_4^{2-}$, and $CO_3^{2-}$. However, 50 mg of Mg(II), Ni(II), Cu(II), Zn(IV) and $SO_3^{2-}$ ions interfered while the remaining eleven ions were tolerated up to 100 ppm.

## Complexometric Determinations of Chromium(III) in Binary and Ternary Metal Ion Mixtures

A procedure was developed for the selective complexometric determination of Cr(III) in binary mixtures containing ion pairs, *viz.*, Cr(III)-Fe(III), Cr(III)-Cu(III) and Cr(III)-Ni(II) using hydroxytriazene as indicator. The advantage of kinetic masking was taken in the determination of two metal ions from the mixture. When the solution was cold Cr(III) did not react with EDTA whereas other ions, *viz.*, Fe(III), Cu(II) or Ni(II) could be determined in the pH range of 5.2 to 6.0, 4.0 to 5.0 or 3.0 to 4.0 respectively in cold by the direct titration with EDTA using hydroxytriazene as indicator. Determination of Cr(III) in ternary mixtures containing any of the three ions, *viz.*, Cr(III)-Fe(III), Cr(III)-Cu(II) and Cr(III)-Ni(II) was done by taking advantage of kinetic masking as well as pH adjustments as described in the binary mixture. At the end point the colour changed from blue to yellow, yellow to green and green to greenish yellow for Fe(III), Cu(II) and Ni(II) resepectively. This solution was then used to determine Cr(III) by back titration after adding excess of EDTA and the same procedure as described earlier was adopted. The colour change at the end point was from violet to yellow. The volume of excess EDTA found was used to determine the volume of EDTA consumed to form Cr(III)-EDTA complex. Thus the difference in total volume of EDTA taken to form Cr(III)-EDTA complex and the volume of excess EDTA found gave the volume of Cr(III).

## Conclusion

Thus the recommended method can be used for the selective complexometric determination of Cr(III) in dilute solutions in presence of 20-fold excess of eleven diverse cations and anions using 3-hydroxy-3-*p*-tolyl-1-*p*-nitrophenyltrizene as

metallochromic indicator. The method has also been successfully applied to the determination of Cr(III) in binary and ternary mixtures. Thus a new metallochromic indicator for the complexometric determination of Cr(III) has been introduced.

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