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NOTE

Analytical Application of 3-Hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene in the Spectrophotometric Determination of Molybdenum(VI)

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A new reagent 3-hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene has been used for the spectrophotometric determination of Mo(VI) 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 molar absorptivity and Sandell's sensitivity values are 7184.7 dm^3 mol^{-1} cm^{-1} and 13.35 ng cm^{-2}, respectively. The method is useful even in presence of several cations and anions.

Key Words: Hyroxytriazene, Spectrophotometric determination, Molybdenum(VI).

Survey of literature reveals that the hydroxytriazenes have been used for the determination of number of transition metals[1,2]. Herein, the spectrophotometric determination of 3-hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene for Mo(VI) is reported.

3-Hydroxy-3-*m*-tolyl-1-m-chlorophenyltriazene was prepared by using the method of Bamberger[3-5].

Stock solution: 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 (0.1 M) and making it up to the required volume with double distilled water. The solution was standardized with EDTA. A 1 × 10^{-2} M solution of the reagent was prepared in alcohol. Perchloric acid (1 %, v/v) was prepared.

Spectrum of 3-hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene was taken in the wavelength 380-460 nm region against solvent blank. Molybdenum and 3-hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene solutions were taken in 1:5 ratio and the spectrum of Mo(VI) complex was recorded against reagent blank in the range 387-460 nm. The working wavelength was found to be 408 nm. A set of solutions containing Mo(VI) and 3-hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene reagent in ratio 1:5 was prepared and pH is varied between 1 to 5. The pH range of constant maximum absorbance was found to be in between 2.0 to 3.0. Composition of the complex was

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determined by Job's method and moles ratio method of Yoe and Jones. The study revealed that the composition of Mo(VI) complex is 1:1 ratio. Absorbance of set of six solutions containing Mo(VI) to 3-hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene in ratio 1:5 was measured at corresponding working wavelength against reagent blank. Beer's law was obeyed in concentration range 1×10^{-5} to 6×10^{-5} M. Interference of 22 cations and anions in the determination of Mo(VI) was studied. To the set of solutions containing Mo(VI) to reagent 1:5 ratio, 10 ppm of different foreign ions were added at optimum conditions. Absorbance was measured against reagent blank. Those ions, which did not interfere at 10 ppm level their interference was again studied at 50 ppm level. In case no or little change in absorbance was seen as compared to the absorbance without any foreign ion, then for those ions interference was studied again at 100 ppm level. However tolerance of still higher concentration was not studied. A UV/Vis Systronic 108 spectrophotometer and a Systronic 324 pH meter were used.

TABLE-1
VALUE OF log β AND ΔG RESPECTIVELY BY TWO DIFFERENT METHODS

Name of methods	Name of reagents	Conc. of complex (M)	E_m	E_S	α	$K_{inst.}$	β	log β	ΔG_{27^oC} (k cal mol^{-1})
Harvey and Manning's method	3-hydroxy-3-*m*-tolyl-1-*m*-chlorophenyl triazene	2.5×10^{-5}	0.195	0.098	0.497	3.16×10^{-5}	3.15×10^{4}	4.49	-6142.2
Purohit's method	3-hydroxy-3-*m*-tolyl-1-*m*-chlorophenyl triazene	7.5×10^{-4}	0.500	0.300	0.400	0.20×10^{-4}	5.0×10^{4}	4.69	-6415.8
		1.5×10^{-4}	1.000	0.600	0.400	0.40×10^{-4}	2.5×10^{4}	4.39	-6005.4

Composition of complex = 1:1

Stability constants: Stability constants have been determined by Harvey and Manning's method[6,7] and Purohit's method[8]. Validity of the methods can be confirmed from the value of log β obtained from both the methods. The log β values agree quite well. Further, the precision studies were carried out by measuring the absorbance of 10 sets of solution containing 4.79 ppm of Mo(VI) and 3-hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene in 1:5 ratio, under optimum conditions. The absorbance was measured against reagent blank at working wavelength (408 nm). Molybdenum was successfully determined at 4.79 ppm level with good precision.

Interference of several cations and anions in the determination of molybdenum was studied at 10, 50 and 100 ppm level. Interference was studied using following cations and anions *viz.*, Na^+, K^+, Mg^{2+}, Pd^{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 at 10 ppm level Na^+, K^+, Ca^{2+}, Mg^{2+}, Ni^{2+}, Zn^{2+}, Co^{2+}, Cl^-, Br^-, I^-, NO_2^-, SO_4^{2-}, CO_3^{2-}, PO_4^{3-}, NO_3^-, CH_3COO^-, WO_4^{2-} did not interfere hence interference of these ions

was then studied at 50 ppm level. Here, Na^+, K^+, Ca^{2+}, Mg^{2+}, Zn^{2+}, Co^{2+}, Cl^-, Br^-, I^-, NO_2^-, SO_4^{2-}, CO_3^{2-}, PO_4^{3-}, NO_3^{2-}, CH_3COO^-, WO_4^{2-} did not interfere. Further it was seen that at even 100 ppm level the following ions did not interfere *viz.*, Na^+, K^+, Ca^{2+}, Mg^{2+}, Zn^{2+}, Co^{2+}, Cl^-, Br^-, I^-, NO_2^-, SO_4^{2-}, CO_3^{2-}, PO_4^{3-}, NO_3^-, CH_3COO^-, WO_4^{2-}. Thus, it can be seen that Mo(VI) can be determined even in presence of number of interfering species present at 100 ppm level. Thus from the above studies it can be concluded that 3-hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene can be used successfully for spectrophotometric determination of Mo(VI).

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