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NOTE

3-Hydroxy-3-Phenyl-1-*o*-Chlorophenyltriazene: A New Reagent for Spectrophotometric Determination of Lead

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Lead(II) forms a grenish yellow complex with the reagent 3-hydroxy-3-phenyl-1-*o*-chlorophenyltriazene at 20–40°C. The complex is stable and does not change thereafter. The composition of the complex (Pb : R) is 1 : 1 with absorption maximum at 394 nm and optimum pH 7.0–7.5. The molar absorptivity and Sandell's sensitivity values are 7750 L/mole cm and 26.73 ng/cm^2 respectively at the working wavelength 406 nm. The Beer's law is obeyed in the concentration range 0.2×10^{-4} M–12×10^{-4} of lead. The standard deviation (10 measurements) for 8.28 ppm of lead was found as 0.002 ppm (0.60%). The interference studies with 22 diverse cations and anions have also been performed.

Hydroxytriazenes are a well known group of chelating agents and have been used for spectrophotometric determination of a number of transition metals in our laboratories[1–7]. Survey of literature reveals that no hydroxytriazene has been used for the spectrophotometric determination of lead. In view of this an attempt has been made to determine lead(II) using 3-hydroxy-3-phenyl-1-*o*-chlorophenyltriazene in the present investigation.

(a) Preparation of Solutions

(i) *Metal ion:* A stock solution of lead(II) (1.0×10^{-2} M) was prepared by dissolving the weighed quantity of lead(II) nitrate (BDH AR grade) in double distilled watter and a few drops of glacial acetic acid were also added to the solution to prevent hydrolysis. The solution was standardized against 1.0×10^{-2} M EDTA solution using xylenol orange as an indicator. Solutions of lower concentrations were prepared by proper dilution of stock solution with double distilled water.

(ii) *Reagent:* A fresh solution of 3-hydroxy-3-phenyl-1-*o*-chlorophenyltriazene of 1.0×10^{-2} M was prepared by dissolving the requisite quantity of the

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reagent in acetone. The stock solution was further diluted to appropriate dilution as and when required.

(iii) *Buffer:*

(a) A 1% (w/v) hexamine buffer solution was prepared by dissolving it in minimum quantity of double distilled water and then diluting with ethanol. A fresh solution was prepared as and when needed.

(b) An aqueous solution of 1% tris buffer was prepared by dissolving it in minimum quantity of double distilled water and then diluting with ethanol. A fresh solution was prepared as an when needed.

(b) Preparation of lead (ii)-3-hydroxy-3-phenyl-1-*o*-chlorophenyltriazene complex

The required volumes from the stock solution of lead and the reagent were taken in the ratio 1 : 6 in a 10 mL volumetric flask. The optimum pH of this mixture was then adjusted between 7.0–7.5 with hexamine and tris buffer. The volume was made up to 10 mL with acetone. A greenish yellow solution of the complex was formed. To ascertain completion of reaction, the absorbance of the complex was measured against reagent blank at 406 nm.

The absorbance measurements for the spectrophotometric determination were done on Systronics UV-Vis Spectrophotometer 108 instrument. A Systronics pH meter 324 was used for pH measurements.

The reagent 3-hydroxy-3-phenyl-1-*o*-chlorophenyl triazene forms a greenish yellow complex with lead(III) at 20–40°C in the pH range 7.0–7.5. The λ_{max} and the working wavelength of the complex were 394 nm and 406 nm respectively. The composition of the complex has been found to be 1 : 1 by three different methods, *viz.*, Job's method[8], Yoe and Jone's mole ratio method[9] and slope ratio method[10].

The stability constant were calculated by two different methods, *viz.*, Harvey and Mannings method[11] and Purohit's method[12] and the log β values obtained were 7.713 and 7.354 respectivley which are in good agreement. These are the values of conditional stability constants only.

Interference Studies

Interference studies in presence of equimolar concentration, fivefold molar excess and tenfold molar excess concentration of the following 22 cations and anions were performed: Sn^{2+}, Ca^{2+}, NO_3^-, NO_2^-, Cl^-, S^{2-}, SO_3^{2-}, PO_4^{3-} , Th^{4+}, I^-, CH_3COO^-, Cu^{2+}, Co^{2+}, Ni^{2+}, K^+, Hg^{2+}, Ag^{2+}, Br^-, CO_3^{2-}, F^-, Cd^{2+} and Ba^{2+}

It was found that in the determination of 8.28 ppm of Pb(II), following cations and anions did not interfere at equimolar concentration: Ca^{2+}, NO_3^-, Cl^-, I^-, Br^-, CO_3^{2-}, F^-, Cd^{2+}, K^+, Sn^{2+}, Th^{4+}, Hg^{2+}, CH_3COO^-, SO_3^{2-}, NO_2^-, Ba^{2+}, Ag^{2+}.

Further even at fivefold molar excess concentration, following cations and anions did not interfere in the determination of lead(II): NO_2^-, Cl^-, I^-, K^+, CO_3^{2-}, F^-, Cd^{2+}, Ba^{2+}, Ca^{2+}, NO_3^-.

Lastly, following cations and anions did not interfere in the determination of lead(II) at 10-fold molar excess concentration: NO_3^-, Ca^{2+}, Cl^-, CO_3^{2-}, Ba^{2+}.

It can be concluded that lead(II) at ppm levels could be determined even in the presence of up to 10-fold molar excess of many cations and anions proving the worthiness of the method.

Sandell's Sensitivity

The molar absorptivity of the complex (ε = 77501/mole cm) was calculated from the Beer's law graph and this value was used for determining Sandell's spectrophotometric sensitivity of the complex which was found to be 26.734 ng/cm^2. This value is comparable to the commonly used reagent *n*-butyl-xanthates and naphthalene (25.360 ng/cm^2).

Conclusion

It is therefore established that 3-hydroxy-3-phenyl-1-*o*-chlorophenyltriazene used in the present investigation is quite comparable to most of the spectrophotometric reagents being used in lead(II) determination. Its easy synthesis, higher yields and economic method of preparation further enhance its applicability as a spectrophotometric reagent for lead(II) determination. Thus the present study has introduced a new reagent for lead(II) determination spectrophotometrically. Further, the reagent can be of use even in presence of most of the commonly interfering species like Ca^{2+}, NO_3^-, Cl^-, Br^-, CO_3^{2-} and Ba^{2+} even up to 10-fold molar excess concentration. Conclusively the reagent is quite useful and its further usefulness can be explored.

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