academicJournals

Vol. 7(16), pp. 1087-1088, 25 April, 2013
DOI: 10.5897/JMPR09.368
ISSN 1996-0875 ©2013 Academic Journals
http://www.academicjournals.org/JMPR

Journal of Medicinal Plants Research

Short Communication

Synthesis and elementary investigation of a new spectrophotometric reagent

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Accepted 15 April, 2011

The study is focused on the development of new spectrophotometric reagent for metal ion determination. The synthesis was through the condensation reaction between amino compounds and carbonyl compound. The spectrophotometric activities of the new reagent was studied using chromium (VI), cobalt (II), Hg (II), Cu(II) and Pb (II) respectively. The use of Jenway UV/VIS spectrophotometer (double beam) model 6505 was used to elicit the wavelength of maximum absorbance of the five metal complexes. Another Jenway UV/VIS spectrophotometer (single beam) model 6305 was employed for the determination of variation in absorbance at various concentrations of the metal ion at constant reagent's concentration to draw the calibration curve. The curve finally showed the efficiency of the reagent up to 5 x 10^{-4} mole dm^{-3} of the metal ion concentration.

Key words: Spectrophotometer, hydrazide derivatives, dioxallic thiophenyl azomethine hydrazine.

INTRODUCTION

With special regard to our continous search for a way out of heavy metal pollution in our environment (Rangnekar and Parekh, 1987) a new reagent for spectrophotometric use has just been synthesized.

The discovery of hydrazine marked the genesis of further studies on hydrazide derivatives (Michaylova and Kouleva, 1974) as spectrophotometric reagents for metal ions. Although, Hoffmann prepared sym-diphenyl hydrazine in 1875, the parent compound hydrazine was not known until 1887 (Pérez-Bustamante and Burriel-Martí, 1967). Current studies have confirmed the condensation between aromatic aldehyde and aromatic amine to be having high stable yield (Alfred and Francis, 1981).

$$Ar^1CHO + Ar^2NH_2 \rightarrow Ar^1CH(OH)NHAr^2 \xrightarrow{-H_2O} Ar^1CH{=}NAr^2$$

This product has proved to be highly sensitive chromogenic reagent for the determination of metal ions spectrophotometrically (Rangnekar and Parekh, 1987; Michaylova and Kuleva, 1980; Michaylova and Yuroukova, 1974; Alfred and Francis, 1981).

EXPERIMENTAL

All reagents and chemicals for these studies were of analytical grade purity and distilled water were used throughout. All glass wares were washed with detergent before rinsing with enough water and drying in the oven, all weighing were carried out on a Gallenkamp Mettler balance model H30.

Synthesis procedure of the spectrophotometric reagent

The method used was that of Schilt and Case (1980). The reaction proceeded readily by reacting Dioxallic Dihydrazide in ethanol with 2-thiophene carboxaldehyde. The whole reacting mixture was refluxed for 7 h. The resulting crystalline product was separated on cooling, filtered and recrystallized from aqueous alcohol to give crystal clear yellowish substance.

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Table 1. Wavelength of the maximum absorbance of metal-reagent complexes.

Metal ions	Max	Absorbance
Cr (VI)	360	0.541
Co (II)	390	0.2809
Hg(II)	385	0.390
Cu(II)	290	0.886
Pb (II)	395	0.245

Nitrogen content studies of the product

Preliminary investigation of the chemical and elemental constituents present in the product was carried out. It indicated the presence of Nitrogen in the elemental make-up of the product. This was carried out using the Kjeldal method as follows: 0.2 g of the sample was placed in Kjedal flask and digested with 4.0 ml concentrated sulphoric acid. 1.4 g of potassium sulphate and 0.08 g of manganese diode were added to the mixture. The mixture was then heated until solution became clear. The content was cooled and transferred quantitatively to a distillation apparatus, few anti bombing granules were added. Excess sodium hydroxide was then added and the mixture was heated to boiling, the end of the condenser was dipped into a flask containing 100 ml of 0.1 m hydrochloric acid, the distillation was continued until all the ammonia gas evolution ceased. The excess acid was titrated against standard 0.1 m sodium hydroxide, using phenolphthalein as indicator.

Spectrophotometric studies of the reagent with metals

To locate the wavelength of maximum absorbance of the resulting complexes, after the addition of the metal ions to the reagent separately. 8×10^{-5} m of the reagent was mixed with 1×10^{-5} mole per dm^{-3} of each metal ions in solution separately. The solution of the different metal complexes were now scanned on the Jenway uv/vis spectrophotometer (double beam) model 6505. The reagent alone was originally used as blank before scanning the reagent with metal ions.

The studies of variations in metal ion concentration with constant concentration of the reagent was equally out in order to ascertain the optimum concentration of the metal ion. 8×10^{-5} m of the reagent was kept constant, while the concentration of metal ions range from 1.0×10^{-5}, 2.0×10^{-5}, 6.0×10^{-5}, 8×10^{-5}, 2.0×10^{-6} (mole dm^{-3}), etc. The spectrophotometer, (single beam) model 6305, the reagent alone is equally used as blank here, while the various changes in concentration of each metal was scanned and results of maximum absorbance in each case recorded. These results were finally used to draw the calibration curve: absorbance against metal ion concentrations.

RESULTS AND DISCUSSION

Yellowish crystals abort 2.17 g (85%) and was finally crystallized out using ethanol as solvent with melting point 287°C.

Physical and spectrophotometric pase meters

Infra-red spectra analysis of the reagent had absorptions at frequencies 1567 cm^{-1} (C = N), 3328 cm^{-1} (N-H) and 1680 cm^{-1} (C = O). The “nitrogen” content determination gave 7.32% in the sample, while the calculated value is 6.98%. The sample had melting point of 287°C. The various metal complexes show the following wavelengths and maximum absorbance in (Table 1).

Conclusion

The reagent showed remarkable results for its potential use as spectrophotometric reagent (2000) for metal ion determination. The compound dioxallic thiophenyl azomethine hydrazine (DTAH) is a yellowish crystalline substance with melting point 287°C. Its metal-liquid – metal ratio is presently under investigation and comparative studies with some existing spectrophotometric reagent.

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