

Short Communication

2-(α -benzoylmethylbenzylideneimino)ethane sulphonic acid as an analytical reagent for Cu(II) and Ni(II)

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Received on December 22, 1982; Revised on August 13, 1983.

Abstract

2-(α -benzoylmethyl benzylideneimino)ethane sulphonic acid (H_2BE) has been used as a reagent for gravimetric determination and separation of Cu(II) and Ni(II) in solution. The standard deviation is found to be $\pm 0.23\%$. Interference by foreign ions like Mg^{2+} , Ca^{2+} , Sr^{2+} , Ba^{2+} , Mn^{2+} , Zn^{2+} , Cd^{2+} , SO_4^{2-} , S^{2-} and Fe^{2+} has been studied.

Key words: Reagent for Cu(II) and Ni(II), gravimetric.

1. Introduction

Despite the existence of many organic reagents¹⁻⁵ for the separation and estimation of Cu(II) and Ni(II) there is a need for a new reagent with high selectivity and sensitivity. 2-(α -benzoylmethylbenzylideneimino)ethane sulphonic acid (H_2BE) satisfies some of these requirements and the present study describes the investigations of H_2BE .

2. Experimental

2.1. Preparation of reagent

Freshly prepared equimolar ethanolic solutions of dibenzoyl methane and taurine were mixed and refluxed for 2 h on a water-bath. On cooling the product, crystals

cf H₂BE obtained were recrystallised from ethanol. The yield was found quantitative m.p. 218.0° C. Found : C, 61.51 ; H, 5.07 ; N, 4.11 and S, 9.49 ; calculated for C₁₇H₁₇NSO₄ : C, 61.63 ; H, 5.14 ; N, 4.23 and S, 9.66%. H¹-NMR, CDCl₂/TMS, δ (ppm) 1.4, -CH₂- ; 6.8, >C=C<'' ; 7.4, Ar-H ; 11.3, -SO₃H ; 15.4, >C=C<''.

2.2. Estimation and separation of copper (II) nickel (II)

From a solution containing Cu(II) and Ni(II), Cu(II) is quantitatively separated first at the pH 3.0-4.0. The Ni(II) in the filtrate is estimated at pH 6.5-7.0 (neutral or faintly acidic medium).

The solutions containing copper(II) (16-70 mg/500 ml) and nickel(II) (15-60 mg/500 ml) were diluted in a 500 ml beaker and its pH adjusted at 3.0-4.0 with sodium acetate buffer. One per cent solution (W/V) of the reagent prepared in water was added with constant stirring to ensure precipitation. On completion of precipitation a brown mass was obtained. This was digested on a steam-bath, cooled, filtered, washed with water and dried at 113°, as found from the thermograms and weighed as [CuC₁₇H₁₅NSO₄ · 3H₂O]. Found Cu, 14.24 ; N, 3.03 and S, 7.08 [CuC₁₇H₁₅NSO₄ · 3H₂O] requires Cu, 14.32 ; N, 3.13 and S, 7.16%.

The filtrate containing nickel(II) was concentrated to half of its initial volume and the pH was adjusted at 6.5-7.0 with phosphate buffer (0.01 M KH₂PO₄ + 0.01 M Na₂HPO₄). Addition of one per cent reagent solution results in the formation of a green precipitate. It was digested, cooled, filtered, washed, dried at 104°, as found from the thermograms and weighed as [Ni C₁₇H₁₅NSO₄ · 3H₂O]. Found Ni, 13.33 ; N, 3.10 and S, 7.11. [Ni C₁₇H₁₅NSO₄ · 3H₂O] requires Ni, 13.35 ; N, 3.17 and S, 7.24%. The results are summarised in Table I.

Table I

Results of estimation and separation of copper (II) and nickel (II)

Sl. No.	Copper (II) (mg)			Nickel (II) (mg)		
	Found	Taken	Error %	Found	Taken	Error %
1.	65.21	55.36	-0.26	54.18	54.30	-0.21
2.	40.92	40.82	+0.24	48.37	48.52	-0.29
3.	35.15	35.28	-0.35	34.97	34.91	+0.17
4.	20.78	20.70	+0.38	28.67	28.59	+0.27
5.	18.49	18.55	+0.32	17.21	17.28	-0.38

2.3. Effect of diverse ions

Appropriate masking agents are needed to prevent the precipitation of foreign ions in solution only when their amounts exceed the tolerance limit. The masking agents (0.0-1.5 gm/dm³) consist of citrates, oxalates and tartrates. The tolerance limit for various ions was found to be as follows :

(Con. in mg/dm³) Mg²⁺, Ca²⁺, Sr²⁺, Ba²⁺, Mn²⁺, Zn²⁺ (1000), SO₃²⁻, CO₃²⁻, S²⁻ (50), Fe²⁺ (10), Pd²⁺ (8) and PO₄³⁻ (50).

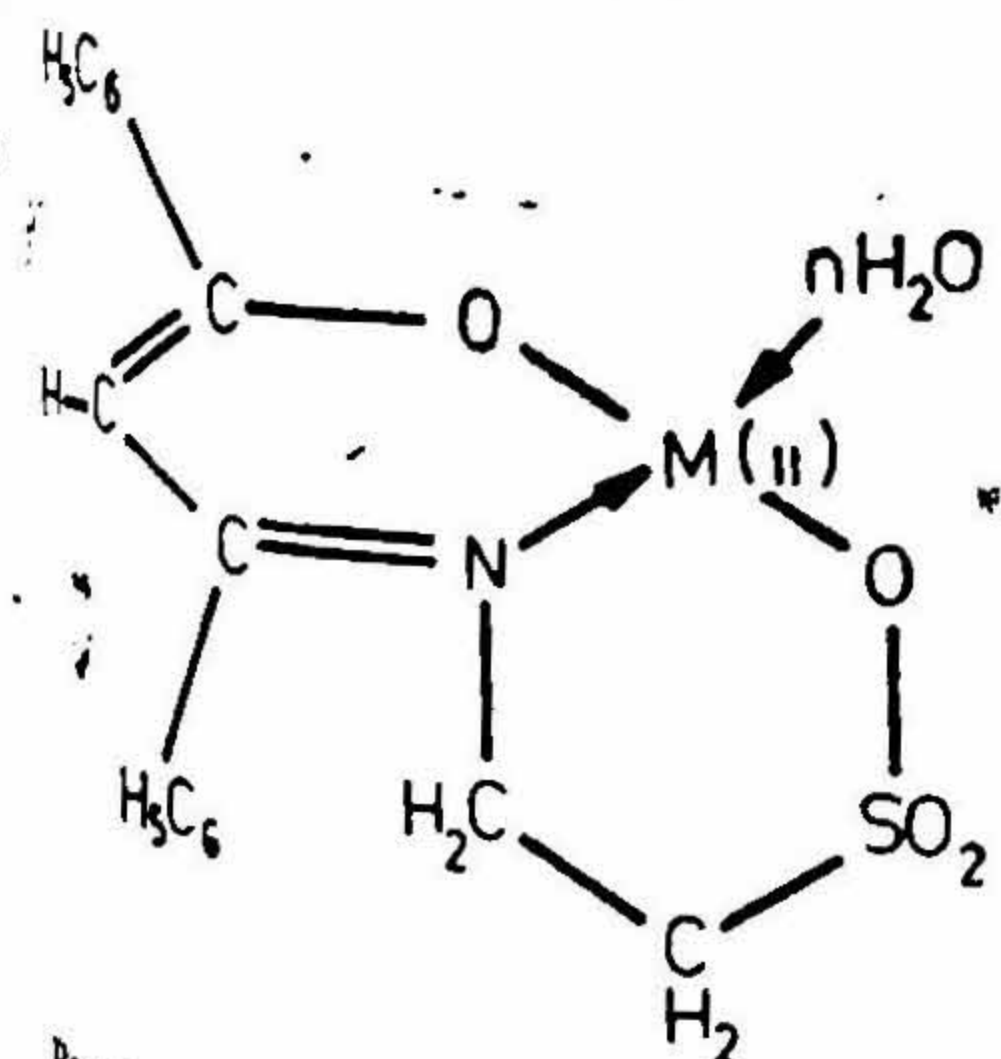
3. Results and discussion

The copper(II) and nickel(II) complexes are soluble in DMSO, DMF and acetonitrile but insoluble in chloroform, benzene and isopropylalcohol. Both the chelates are quite stable and decompose on heating above 180°.

3.1. Stoichiometry and structure

The analytical and molecular weight data of the chelates show 1 : 1 (metal-ligand) stoichiometry, besides the presence of three water molecules. The magnetic moment studies suggest the presence of 1 and 2 unpaired electrons in Cu(II) and Ni(II) complexes, respectively. The electronic spectra of copper(II) chelate exhibits one peak only at 12900 cm⁻¹ assignable to ²E_g → ²T_{2g} indicating distorted octahedral or tetragonal stereochemistry. Nickel(II) chelate spectra consist of two peaks at 13700 cm⁻¹ and 26100 cm⁻¹ assignable to ³A_{2g} → ³T_{1g} (F) and ³A_{1g} → ³T_{1g} (P) respectively indicating an octahedral geometry of the complex.

The IR spectra of H₂BE show four bands at 3365, 1685, 1600 and 1090 cm⁻¹ assignable to ν OH, ν C=O, ν C=N and ν SO₃H, respectively. In Cu(II) and Ni(II) chelates the ν C=N is lowered (from 1600 cm⁻¹ to 1580 cm⁻¹) suggesting the involvement of azomethine nitrogen in chelation. The spectra of metal chelates display two new bands at 415 and 525 cm⁻¹ arising from ν (M-N) and ν (M-O)⁷. The broad band observed at 3280 cm⁻¹ may be due to the presence of ν OH of co-ordinated water molecules.



where $n = 3$, $M(II) = Cu$ or Ni

FIG. 1. Bivalent metal chelates of 2-(α -benzoylmethylbenzylideneimino)ethane sulphonic acid (H₂BE).

Based on the above data structure as shown in fig. 1 is assigned to Cu(II) and Ni(II) chelates.

Acknowledgement

Thanks are due to UGC, New Delhi, India for financial assistance.

References

1. MALISSE, H. AND GOMISEEK, S. *Anal. Chem. Acta*, 1962, 20, 402.
2. MIESSLER, G. L., STUK, G. S., SMITH, T. P., GIVEN, K. W., PALAZZOTTO, M. C. AND PIGNOLET, L. H. *Inorg. Chem.*, 1976, 15, 1982.
3. ABDULLAH, M. I., EL RAJIS O. A. AND RILEY, J. R. *Anal Chim. Acta*, 1976, 84, 363.
4. LIU SHAV-LING AND YIN T'AN-CHENG *Acta. Chem. Sincia*, 1962, 28, 20.
5. WELCHER, F. J. *Org. Anal. Reagent*, Van Nostrand, New York, 1947, 4.
6. SCHEINMANN, F. *An introduction to spectroscopic methods for the identification of organic compounds*, Pergamon Press, 1970, p. 186.
7. NAKAMOTO, K. *Infrared spectra of inorganic and coordination compounds*, John Wiley, New York, 1962, p. 157.
8. BELLAMY, L. S. *Advances in infrared group frequencies*, Methuen and Co. Ltd. U.K. 1969, p. 246.