

NOTE

Synthesis and Characterization of Iron(III)-Benzoin Complex

M.M. EL-AJAILY*, A.A. MAIHUB†, M.A. ABUZWIDA‡, M.M. ABOU-KRISHA,
A.A. AMAR‡ and E.A. ASAIH‡

Chemistry Department, Faculty of Science, Garyounis University, Benghazi, Libya
E-mail: melagaily@yahoo.com

Benzoin can form complexes with iron(III) in different ratios depending on the number of ligand moles used. The new metal complex of the formula $[\text{FeB}(\text{OH})_2(\text{H}_2\text{O})_2] \cdot 6\text{H}_2\text{O}$, in which B represents benzoin moiety, has been prepared and characterized by elemental analysis, infrared, electronic spectra and molar conductance. The elemental analysis data show the formation of 1 : 1 [M : L] ratio. Infrared spectral data confirm bonding through oxygen of the carbonyl and hydroxyl groups in benzoin. Electronic spectral data support the geometrical structure of the complex being an octahedral structure. The molar conductance measurement indicates the formation of non-electrolytic complex.

Key Words: Synthesis, Characterization, Complex, Iron(III), Benzoin.

Benzoin (Fig. 1) was known to have many applications in chemical industry, pharmaceutical products, etc.¹ The metal complexes of benzoin thiosemi-carbazone have been synthesis and characterized by different spectroscopic techniques². A number of complexes of the type ML_2X_2 and ML_2 , where M represents the metal ions of Co(II), Ni(II), Cu(II) or Zn(II), L is benzointhiosemi-carbazone and X is halide or nitrate ions, have been prepared and investigated on the basis of analytical analysis and electronic spectra³.

In this work we wish to prepare and investigate an iron(III)-benzoin complex.

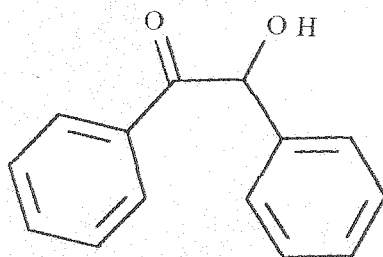


Fig. 1. Benzoin

†Chemistry Department, Faculty of Pharmacy, Al Fateh University, Tripoli, Libya.

‡The Advanced Laboratory of Chemical Analysis, Al-Fateh University, Tripoli, Libya.

All chemicals used in this study were of reagent grade of BDH or Aldrich including $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, benzoin, NH_4OH , $\text{C}_2\text{H}_5\text{OH}$, DMSO, CHCl_3 , EDTA and murexide indicator. All organic solvents used in this investigation were of pure grade and some were redistilled. Water was always used as doubly distilled.

Preparation of the complex: The complex was prepared by mixing 30 mL of ethanolic solution of 0.01 mol of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ with the same molar ratio of ethanolic solution of benzoin. The pH was adjusted to the desired range (pH = 6–8), with sodium hydroxide solution (1.25 M). The resultant metal-ligand solution was refluxed for 2 h, then kept overnight. The formed complex was collected by filtration, washed several times with hot ethanol to remove traces of unreacted materials. The solid product was then dried in air and stored in a desiccator over anhydrous CaCl_2 under vacuum (yield *ca.* 85%).

The complex was subjected to elemental analysis in the Micro-Analytical Center, Ras-Lanouf Company, Libya. Infrared spectrum was obtained by using IFS-25 DPUS/IR spectrometer (Bruker) 1998Y. Electronic spectrum was obtained by using Perkin-Elmer Lambda 4B spectrophotometer using 1 cm matched silica cell. Both infrared and electronic spectra were recorded at the Advanced Laboratory of Chemical Analysis, Al-Fateh University-Tripoli, Libya. Conductance measurement was carried out in DMSO using conductivity meter model CMD650 digital.

The elemental analysis of C and H of iron(III)-benzoin complex show that the found values (36.92% C and 3.23% H) are in good agreement with the theoretical values (37.65% C and 4.24% H) of the molecular formula of the complex formed in the ratio of 1 : 1 [M : L] of the type $[\text{FeB}(\text{OH})_2(\text{H}_2\text{O})_2] \cdot 6\text{H}_2\text{O}$.

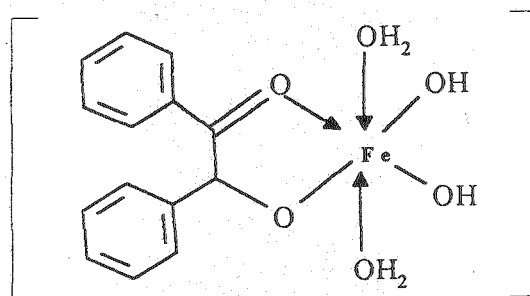
The infrared spectral band at 1600 cm^{-1} in the IR spectrum of the complex is attributed to the $\nu(\text{C}=\text{O})$ of the benzoin. The shifting of the band to the lower frequency in comparison with the free ligand by 90 cm^{-1} , indicates that the carbonyl-oxygen is involved in the coordination with Fe(III) ion⁴.

The bands at 3250 and 3590 cm^{-1} in the spectrum suggest the presence of $\nu(\text{OH})$ vibrations. These values indicate that the hydroxyl group is participating in the complexation with the metal ion (see the suggested structure)⁵. The appearance of a new band at 625 cm^{-1} which is absent in the free ligand is due to the $\nu(\text{Fe}-\text{O})$ vibration. This band supports the involvement of oxygen atom of hydroxyl group ions⁶. The spectral data of the complex display a broad band at 3400 cm^{-1} can be attributed to the presence of water molecules which are usually present as hydrated or coordinated molecules⁷. A bending motion of the coordinated water molecules⁸ is usually in the range of $820-750 \text{ cm}^{-1}$.

The electronic spectrum of the complex under investigation was scanned in chloroform solution. The data show a band at 29806 cm^{-1} (335.5 nm) due to the ${}^5\text{T}_{2g} \rightarrow {}^5\text{E}_g$ transition. The intensity of the absorption band suggests the octahedral geometry⁹.

Conductivity measurement was carried out in non-aqueous solution using DMSO to help in investigation of the geometrical structure of the complex. The conductance value $12.5 \text{ ohm}^{-1} \text{ cm}^{-1} \text{ mol}^{-1}$ shows the presence of non-electrolytic nature¹⁰.

On the basis of the present studies, we conclude that the complex geometrical structure is octahedral geometry as shown below:



Suggested structure of the complex

REFERENCES

1. A.M. El-Roudi, *Bull. Fac. Sci. Assiut Univ.*, **18**, 77 (1989).
2. O.E. Offiong and S. Martelli, *IL Farmaco*, **49**, 513 (1994).
3. B.K. Mohanty, R.M. Mohapatra, N. Mallick and B.K. Mohapatra, *Indian J. Chem.*, **26A**, 1069 (1987).
4. B.S. Garg, R. Dixit, A.L. Singh and R.K. Sharma, *J. Anal. Chem.*, **36**, 2541 (1991).
5. N.M. Samus, V.I. Sapkov and N.V. Kravehenko, *Russ. J. Gen. Chem.*, **67**, 1929 (1979).
6. I.M. Issa, A.L. El-Ansary, O.E. Sherif and M.M. El-Ajaily, *Transition Met. Chem.*, **22**, 441 (1997).
7. D.M. Boghaei and M. Lashanizadegan, *J. Sci. I.R. Iran*, **11**, 301 (2002).
8. K. Nakatomo, *Inorganic Spectra of Inorganic and Coordination Compounds*, John Wiley, New York (1970).
9. R.M. Awadallah, R.M. Issa, A.A.M. Belal and R.D. Peackoc, *Egypt. J. Chem.*, **36**, 79 (1993).
10. W.J Geary, *Coord. Chem. Rev.*, **7**, 81 (1971).

(Received: 30 December 2004; Accepted: 30 June 2006)

AJC-5008