



Thermal Spectra of Sodium Metal Complex with Ligand P-bromoisonitrosoacetophenone

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Abstract: Complexes of alkali metals like Lithium, Sodium and Potassium [Li, Na and K] with ligand P-bromoisonitrosoacetophenone [P-BrINAP] have been synthesized and characterized by elemental analysis, conductivity measurement, magnetic susceptibility, nuclear magnetic, electromagnetic, infrared spectral studies and thermogravimetric analysis. Antimicrobial activities of ligands and its complexes with alkali metals were screened using sensitivity test, minimum inhibition concentration and minimum bacterial concentration method. Thermogravimetry is an experimental technique used for characterizing the complex by measuring the changes taking place in physical and chemical properties in the system as a function of increasing temperature with time.

Keywords: Alkali Metals, conductivity, spectral, antimicrobial, thermogravimetry, properties and temperature.

I. Introduction

P-bromoisonitrosoacetophenone has already been investigated for possible complex formation with transition metals⁴. Here in this communication we are reporting the neutral complexes of this ligand with alkali metals. The present work has been carried out in the above context and includes preparation of ligands. The purity of ligands has been confirmed by elemental analysis and melting point determination. Solid complexes of alkali metals have been synthesized and characterized by techniques such as elemental analysis, molecular weight determination, conductivity measurements, thermogravimetric analysis, magnetic susceptibility, nuclear magnetic resonance and infra-red spectra.

II. Materials and Method

The Experimental work has been carried out by using all the chemicals and solvents were of analytical reagent grade. Double distilled water was obtained by distilled water containing potassium permanganate and alkali in glass apparatus.

P-bromoisonitrosoacetophenone was synthesized by the procedure described by Muller and pechmann³. The basic principle used for this synthesis is that of claisen⁵, the reagent isoamylnitrate¹ was prepared from isoamylalcohol and sodium nitrate. Again 1:1 stoichiometric ratios of these salts and p-bromoisonitrosoacetophenone were subjected to the procedure stated above. The change in colour of solution indicated the formation of complexes. The chemicals used were of analytical grade. The precipitates so formed were filtered, washed with ethanol or ether as the case needed and subjected to melting point measurement and elemental analysis.

The mixture of aqueous solutions of nitrate salts of lithium and potassium were taken separately in round bottom flask with the ligand p-bromoisonitrosoacetophenone in 1:1 and 1:2 molar proportions.

This mixture was refluxed for 5-7 hours at boiling temperature with occasional shaking. The pH of mixture solution 6-6.5 was maintained by adding HCl or NH₄OH. The solid product obtained was immediately removed from the flask as soon as the reaction period was over and kept in vacuum desiccators. On cooling the solid crystalline complex was obtained. It was filtered, dried in air, recrystallized from ether and acetone respectively and finally analyzed for lithium, potassium, carbon, hydrogen and nitrogen.

III. Results and Discussion

Almost all the alkali metal salts and their respective complexes were found to be coloured and stable in air but stability decreased on exposure to moisture leading ultimately to decomposition, hence all the salts and complexes made were kept in a desiccators over solid anhydrous calcium chloride.

From the results it was evident that both the alkali metal salts and their complexes undergo transformation at a temperature which were considerably higher than the melting point of the ligand. Both the complexes were soluble in polar solvents such as ethanol but insoluble in non-polar solvents like benzene, ether etc.

Thermogravimetric analysis

Thermal analysis is an experimental technique used for characterizing the complex or compound by measuring the changes taking place in physical and chemical properties in the system as a function of increasing temperature with time. The technique of thermogravimetric analysis is concerned with the analysis of sample weight curve.

The alkali metal complexes with [P-BrHINAP] have been studied by thermogravimetric analysis and different thermal analytical techniques. All the complexes are found to be stable at room temperature and water of molecule is not associated with them. On heating, they start losing their weight with the loss of ligand by fragmentation and finally forming corresponding metal or metal oxides accordingly.

On examination of DTA curves of all the chelates, it's observed that the sharp exothermic peaks are found in the temperature range between 400⁰C to 700⁰C and sudden weight loss on the TGA curves indicates that, the complexes decompose slowly after initial decomposition in a number of steps till the formation of metal or metal oxide has been completed.

The experimental results with spectrum obtained from thermogravimetric analysis are discussed in the following compartment.

[Na (P-BrINAP) ₂]:

The thermogravimetric analytical spectrum of [Na (P-BrINAP) ₂] shows continuous loss of moisture with an increase in temperature up to 500⁰C. Almost 20% reduction in weight is observed. The sharp loss in weight is observed in TGA curve between the temperatures 476.41⁰C – 672.10⁰C. This significant loss in weight may be due to decomposition of the complex supported by differential thermal analytical curve. It is also pragmatic from the curve that, formation of oxides of sodium is takes place at 608.40⁰C and huge amount of heat is evolved during decomposition of the complex having peak area at 1176.56 MJ which is shown in the following figure...

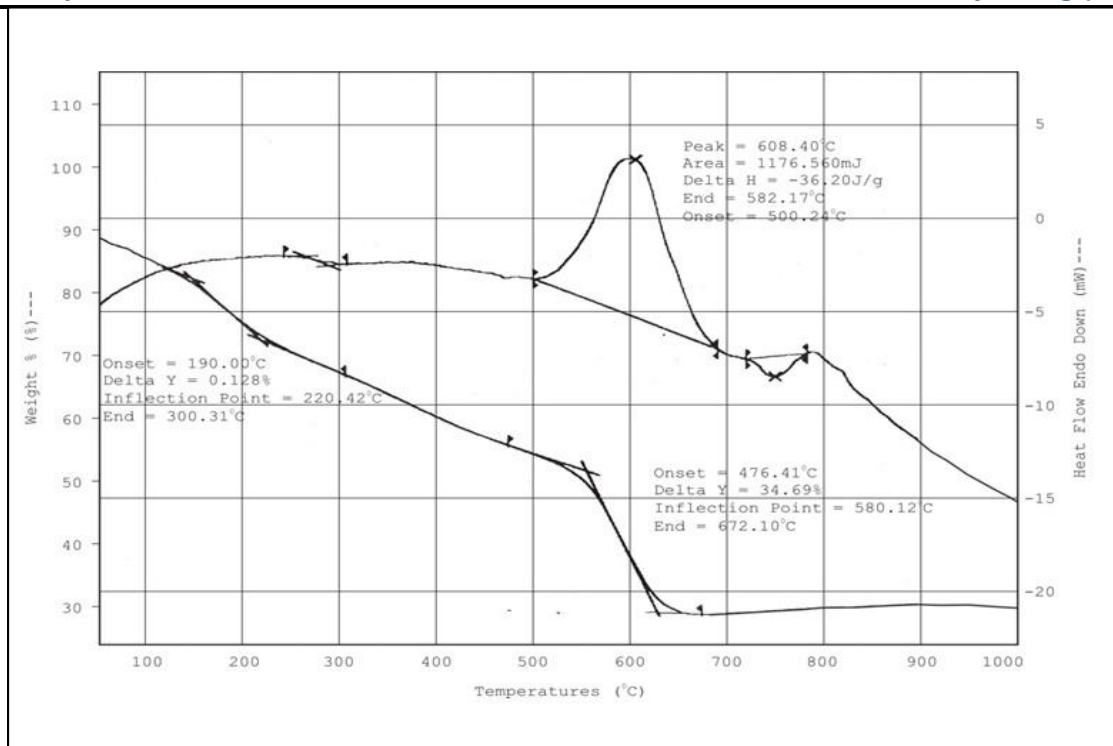
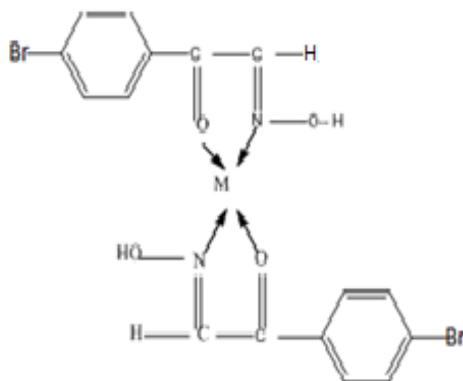


Fig. – Thermogram of [Na (P-BrINAP) ₂]

IV. Structure and bonding

On the basis of elemental analysis, molecular weight determination, molar conductivity measurement, spectral studies with thermogravimetric analysis following probable structure of the complex is possible.



M= Alkali metal sodium

V. Conclusion

The sodium complex with ligand p-bromoisonitrosoacetophenone has been synthesized by the above discussed condensation method is very simple. The purity of newly synthesized complex has been experienced and established by special physicochemical and experimental techniques.

VI. Acknowledgment

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VII. References

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