



Thermogravimetric Analysis of Rubidium Complex with P-bromoisonitrosoacetophenone

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Abstract: Complexes of alkali metals like Lithium, Sodium, Potassium and Rubidium [Li, Na, K and Rb] with ligand P-bromoisonitrosoacetophenone [P-BrINAP] have been synthesized and characterized by elemental analysis, conductivity measurement, magnetic susceptibility, different spectral studies and thermogravimetric analysis. Antimicrobial activities of complexes were screened by sensitivity test, minimum inhibition concentration and minimum bacterial concentration method. Thermogravimetric analysis 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. According to Duval³⁹ et al, information regarding thermal stability and composition of residue can be obtained from the examination of weight changes with increase in temperature at linear rate which is concerned with the analysis of sample weight curve.

Keywords: Alkali Metals, measurement, spectral, antimicrobial, thermogravimetric, function, Temperature, weight and curve.

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 including preparation of ligand, its purity 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, electronic, 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 system described by Muller and pechmann³. The basic principle used for this synthesis is that of claisen⁵, the reagent isoamyl nitrate¹ was prepared from

isoamylalcohol and sodium nitrate. Again 1:1 stoichiometric ratios of these salts and P-bromoisoinitrosoacetophenone were subjected to the method stated above. The change in colour of solution indicated the formation of complex. 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 acetate of rubidium was taken in round bottom flask with the ligand p-bromoisoinitrosoacetophenone in 1:2 molar proportions. This mixture was refluxed for 6-7 hours at boiling temperature with occasional shaking. The pH of mixture solution 5.5-6.5 was maintained by adding HCl or NH_4OH . The solid product obtained was immediately removed from the flask as soon as the reaction period was over then it kept in vacuum desiccators. On cooling the solid yellow crystalline complex was obtained. It was filtered, dried in air, recrystallized from ether and acetone respectively. Lastly the complex was analyzed for elements like 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 marked 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. The complexes synthesized were soluble in polar solvents like ethanol but insoluble in non-polar solvents like benzene, ether etc.

Thermogravimetric analysis

Thermogravimetric 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-bromoisoinitrosoacetophenone [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 inspection of thermogravimetric curves of all the chelates, it's observed that the sharp exothermic peaks are found in the temperature range between 400°C to 700°C and sudden weight loss in the 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.

[Rb (P-BrINAP) $_2$] Complex

The spectrum and experimental results obtained from thermogravimetric analysis of Rubidium complex with ligand P-bromoisoinitrosoacetophenone is discussed in the following manner.

The thermogravimetric (TGA) spectrum of $[\text{Rb}(\text{P-BrINAP})_2]$ complex shows number of steps in loss of weight below 400°C and the continuous weight loss can be observed. The steps in loss of weight at lower temperature could be caused by loss of moisture or solvent i.e. it set free from moisture on heating of which the calculated contents being approximately 18%. The thermogravimetric curve shows a drastic loss of weight between the temperatures 452.20°C to 510°C indicates the decomposition of compound in this region where the inflection point is 474.44°C . This interpretation is confirmed by differential thermal analytical curve which shows a strong exotherm at 478.21°C . Further after decomposition of ligand, oxide formation take place between the temperatures 450°C to 500°C at 478.21°C . This sudden loss in weight can be interpreted in terms of oxidation reduction reaction.

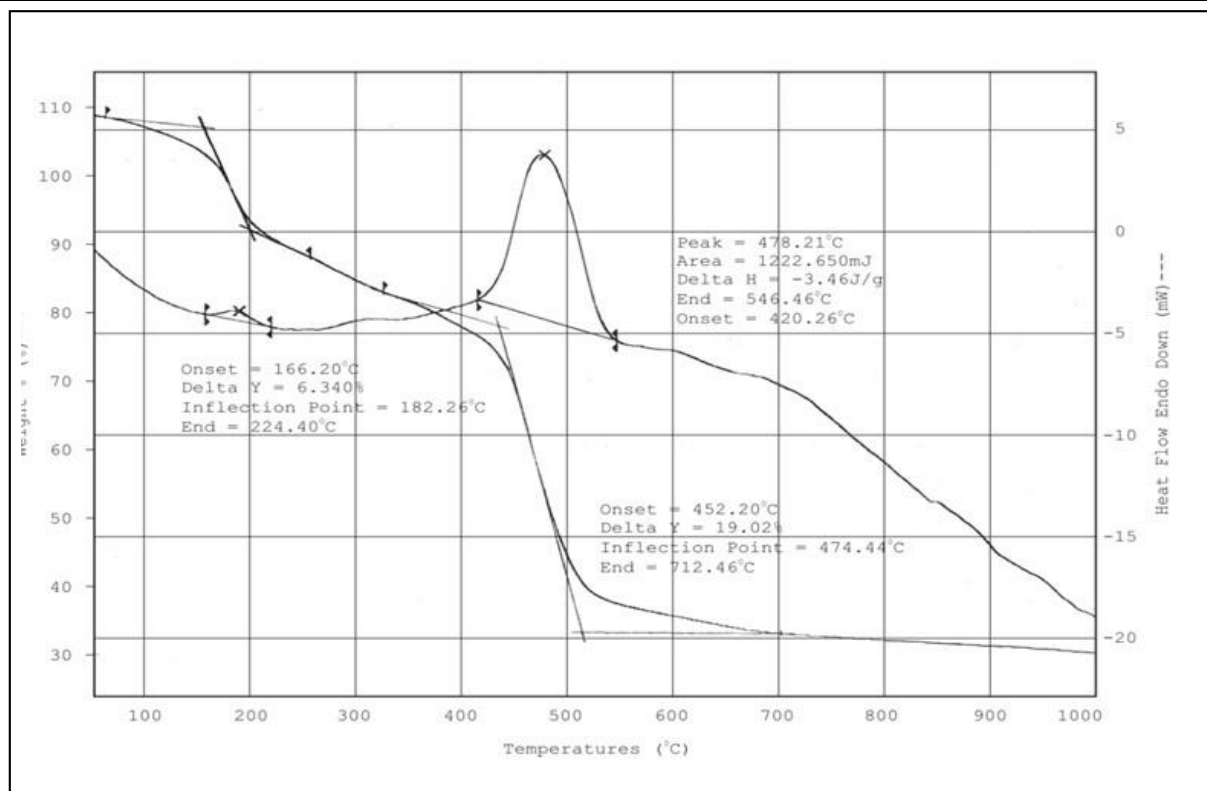
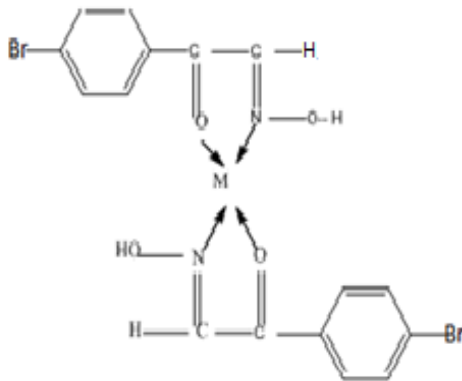


Fig. – Thermogram of $[\text{Rb} (\text{P-BrINAP})_2]$

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= Rubidium, the alkali metal

V. Conclusion

The Rubidium complex with ligand P-bromoisoinitrosoacetophenone has been synthesized by the above discussed condensation method is very simple and easily operable with minimum expenditure. The purity/transparency of newly synthesized complex has been experienced and established by special physico-chemical and experimental techniques.

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VII. References

1. Blatt: *Organic Syntheses, Coll.* Vol II, P. 108.
2. Lord R C and Merrifield R E, *J Chem Phys*, 21, 166 (1953).
3. Muller and Penchmann V, *Ber*, 22, 2560 (1888).
4. Natrajan and Hussain A nazeer, *Indian J Chem*, 20A, 307 (1981).
5. Victor Meyer, Zunlin J, *Ber*, 11, 695 (1978).
6. Honda K, TGA Sci. Report Tohoku University, 4, 97 (1951).
7. Duval C, "*Inorganic The TGA Analysis*" 2nd Edition Elsevier, Newyork.
8. Wendlandt W W, "*Differential Thermal Analysis*" 2nd Ed. Chard, Newdelhi (1971).
9. Bhawe N S and Kharat R B, *Jour Ind Chem Soc*, 27, 260 (1981).

