

# Thermogravimetric Analysis and Kinetic Parameters Determination of Co (II) Complexes

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**Abstract:** The kinetic parameters are determined firstly using Freeman and Carroll method and the results are compared with the data developed by Zsako Procedure. A short experimental technique for recording the thermogravimetric curves for the complexes on a NETZSCH Simultaneous Thermal Analyzer STA-409 has also been discussed here. The existing Percentage weight of the complex at equal temperature intervals i.e., 10°C were noted directly from TG curve for a particular stage of thermal decomposition A plot of  $\Delta \log dw / dt / \Delta \log w$ , versus  $T-1 \times 10^{-3} / \Delta \log W$  has been drawn for the stage of decomposition under consideration.

**Background:** The transition elements and main group elements can form coordination compounds, or complexes, in which a central metal atom or ion is bonded to one or more ligands by coordinate covalent bonds. Ligands with more than one donor atom are called polydentate ligands and form chelates. The common geometries found in complexes are tetrahedral and square planar (both with a coordination number of four) and octahedral (with a coordination number of six). Cis and trans configurations are possible in some octahedral and square planar complexes. In addition to these geometrical isomers, optical isomers (molecules or ions that are mirror images but not superimposable) are possible in certain octahedral complexes. Coordination complexes have a wide variety of uses including oxygen transport in blood, water purification, and pharmaceutical use.

**Materials and Methods:** 40 ml of ligand (0.02 M) in ethanol was mixed with 40 ml Cobalt (ii) Bromide (0.01 M) in ethanol. The resulting solution was refluxed for half hour on a steam bath reddish brown colour precipitate was obtained. The Precipitate was filtered off and washed with ethanol and dried in a desiccator over anhydrous CaCl<sub>2</sub>. 45 ml of ligand (0.02 M) in ethanol was mixed with 45 ml copper formate (0.01 M) in ethanol, the resulting solution was refluxed half hour on a steam bath, bluish green colour precipitate was obtained. Precipitate was filtered and dried in a desiccator over anhydrous CaCl<sub>2</sub>.

**Results:** The kinetic parameters are determined firstly using Freeman and Carroll method and the results are compared with the data developed by Zsako Procedure. A short experimental technique for recording the thermogravimetric curves for the complexes on a NETZSCH Simultaneous Thermal Analyzer STA-409 has also been discussed here. The existing Percentage weight of the complex at equal temperature intervals i.e., 10°C were noted directly from TG curve for a particular stage of thermal decomposition A plot of  $\Delta \log dw / dt / \Delta \log w$ , versus  $T-1 \times 10^{-3} / \Delta \log W$  has been drawn for the stage of decomposition under consideration.

**Conclusion:** The kinetic parameters are determined firstly using Freeman and Carroll method and the results are compared with the data developed by Zsako Procedure. A short experimental technique for recording the thermogravimetric curves for the complexes on a NETZSCH Simultaneous Thermal Analyzer STA-409 has also been discussed here. The existing Percentage weight of the complex at equal temperature intervals i.e., 10°C were noted directly from TG curve for a particular stage of thermal decomposition A plot of  $\Delta \log dw / dt / \Delta \log w$ , versus  $T-1 \times 10^{-3} / \Delta \log W$  has been drawn for the stage of decomposition under consideration.

**Key Word:** Thermogravimetric Analysis, Kinetic Parameters Determination, Co (II) Complexes

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## I. Introduction

The transition elements and main group elements can form coordination compounds, or complexes, in which a central metal atom or ion is bonded to one or more ligands by coordinate covalent bonds. Ligands with more than one donor atom are called polydentate ligands and form chelates. The common geometries found in complexes are tetrahedral and square planar (both with a coordination number of four) and octahedral (with a coordination number of six). Cis and trans configurations are possible in some octahedral and square planar complexes. In addition to these geometrical isomers, optical isomers (molecules or ions that are mirror images but not superimposable) are possible in certain octahedral complexes. Coordination complexes have a wide variety of uses including oxygen transport in blood, water purification, and pharmaceutical use.

The coordination chemistry of transition metal and their derivatives have been widely studied due to their biological importance(1-3), Transition metal complexes have been used in antifungal(4), antibacterial(5),

antitumor(6), antiviral(7), antimalarial(8), hypotensive and hypothermic(9-10) reagents. Research study indicates that a cycle ligand containing nitrogen, oxygen and sulphur donor atoms in their structure can, acts as effective chelating agents for transition metal ion(11), It is observed that when the drugs are administered as metal chelate there is increase in activity.

Reaction of the 1, 2, 3, 4 - thiazoles have been studied to a limited I degree after the structure of the ring system was firmly established in 1957(19), The pertinent chemistry much of it being thermal decomposition studied of the fairly labial ring has been reviewed by Jensen(20-21). and by Holm(22). The known reaction of the 5-amino - 1, 2, 3, 4 - thiazoles are even more limited consisting of rearrangement and decomposition under basic and acidic condition acylation on the 5 amino nitrogen and alkylation in the 4 position or on S-amino nitrogen 5- Anilino - 1, 2, 3, 4 - thiazole (5-ATT) was one of the most active analogues in the series. From the viewpoint of toxicity p-amino phenol was the preferred metabolite 5 - (p-Hydroxylation) - 1,2,3,4 - thiazole (p-HT) (fig. I) was them proposed more desirable antihypertensive agent.

A large number of Schiff bases and their complexes have been studied for their important properties e.g. their ability to reversibly bind oxygen transfer of an amino group and complexing ability towards some toxic metals(12-14), Transition metal complexes which usually contain nitrogen, sulphur and oxygen as ligand atoms are becoming increasingly important because these Schiff base can bind with different metals centres involving various coordination site and allow successful synthesis of metallic complexes with interesting stereochemistry(15-18), Heterocyclic compound are widely distributed in the nature and essential to many biochemical, analytical and industrial processes. The Schiff base have been widely studied in the field of coordination chemistry mainly due to their facile synthesis, easily availability, electronic properties and good solubility in common solvents.

## **II. Material and Methods**

40 ml of ligand (0.02 M) in ethanol was mixed with 40 ml Cobalt (ii) Bromide (0.01 M) in ethanol. The resulting solution was refluxed for half hour on a steam bath reddish brown colour precipitate was obtained. The Precipitate was filtered off and washed with ethanol and dried in a desiccator over anhydrous  $\text{CaCl}_2$ . 45 ml of ligand (0.02 M) in ethanol was mixed with 45 ml copper fomite (0.01 M) in ethanol, the resulting solution was refluxed half hour on a steam bath, bluish green colour percipient was obtained. Precipitate was filtered and dried in a desiccator over anhydrous  $\text{CaCl}_2$ .

50 ml of ligand (0.02 M) in ethanol was mixed with 50 ml of Nickel Bromide (0.01 ml) in ethanol. The resulting mixture was refluxed was half hour on a steam bath. Dark green colour precipitate was obtained. The precipitate was filtered off with the help of filter paper and washed with ethanol and dried in a desiccator over anhydrous  $\text{CaCl}_2$ . 45 ml ligand (0.02 M) in ethanol was mixed with 45 ml Mercurous Acetate (0.01 M) in ethanol. The resulting mixture was refluxed for half hour. The colour was change into cream colour. The product was collected on a filter paper md washed with ethanol. The complex was finally dried in desiccator over anhydrous  $\text{CaCl}_2$ . 40 ml of ligand (0.02 M) in ethanol was mixed with 40 ml lead bromide (0.01 M) in ethanol. The resulting mixture was refluxed for half hour on a steam bath. The colour was changed into cream colour. The product was collected on a filter paper and washed with ethanol. The complex was finally dried in desiccator over anhydrous  $\text{CaCl}_2$ .

## **III. Resultand Discussion**

The literature survey on thermogravimetric analysis has been extensively reviewed. Duval has made a comprehensive complication of the use of thermogravimetry as preparative techniques, particularly in the chemistry of transition metal complexes with a typical complex, three type of general reaction can occur (i) loss of hydrated water or loss of volatile ligand to give other complex, (ii) reaction within the complex and (iii) reaction with other species e.g., surrounding atmosphere or an added non-volatile compound.

Liptaly et al(2) While studying the thermal behaviour of some chelates investigated that thermal stability does not depend on the strength of the metal ligand bond but on the size and structure of rest of the chelate. Banerjee and Dey(5) investigated Mn (II), Fe (II), Zn (II), Cd (I), Ni(II), Co(II) and Cu(II) coordination complexes with quadrifunctional dyes. The thermal stability of the complexes of dyes shows the order p-Phenylenediamine benzidine Cu (II) > Ni (II) > Zn (II) > Co (II). A Manimekalai & B Senthil Siva kumar(6) prepared Ni (II) & Cu(II) complexes with DPSH, DPBH, MDPSH & ported order of reaction (n), activation energy (E) & change in entropy ( $s^*$ ) of complexes with its thermal stability Pittman et all) have shown that the rate of decomposition of the polychelate of phenolic resin is more than that of the parent polymer, suggesting that there may be Powerful intramolecular hydrogen bonding. Sheshgire et al (8) compared the results that the increase of electron density at the central metal ion increase the thermal stability of the chelates. J.B. Dahiya & Sushila Rana et al (9), Studied the potential flame retardancy of cellulose benzythiophosphate (CBTP) & it metal complexes with Fe (II) & Cu (II) has been investigated by their thermal degradation behaviour using TG & DSC techniques. Reported the formation of char is brought about expense of a decrease in decomposition

temperature of relevant exothermic peak indicating thiophosphorylation& exhibiting typical condensed phase flame - retardant activity due to presence of phosphorus, sulphur& metals. S. Roy et al (10) synthesized some new coordination polymers by combining adipyl 1 bis-biuret and azetyl bis-biuret with metals of first transition series viz Mn (II), Co (II), Ni (II), Cu (II) & Zn (II). A detailed thermal study was carried out & thermal stability compared & reported Co (II) & Ni (II) polymers show octahedral geometry with two water molecules. Kushik et al (11) have studied the thermal behaviours of N-(p-ethoxyphenyl)-dithiocarbamate (PED), complexes of Cu (II), Zn (II), Cd (II), Sn (II), Ni (II) and Pd (II) in a dynamic air atmosphere using TG and DTA technique and suggested the probable path of the decomposition · Thermal decomposition of cobalt (II) and Nickel (II) complexes with 2-OH-S- methylacetophenone thiosemicarbazone were studied by Arvindakshna et al(12).

De Gieso et al(13) synthesized B-hydroxy quinoline formaldehyde polymeric ligand and Prepared chelates with Zn (II), Ni (II), Fe (II), Al (II), Th (II) and V (VI) ions are studied their thermal stabilities and have noted a large difference in the nature of the thermograms of ligand and it chelates. The ligand is reported to decompose in two steps, while the chelates are reported to decompose in one narrow step. The comparison of decomposition range of the ligand and it chelates show that, the chelates are thermally less stable than the unchelated ligand. Fluorenone anthranilic acid complexes of Mn (ii), Co (II) and Ni (II) were synthesized by Thomas et al(14) and the thermal decomposition of these reported that Mn (II) and Ni (II) chelates give a three-stage decomposition pattern. Thermal studies of Dioxomanium (VI) sulphate complexes of some Schiff's bases of 4-amino antipyrine was studied by Agarwal (15) and reported that the TG curves of these complexes do not show the presence of water molecule either in or out of the coordination sphere, Patel et al(16) prepared some copolymer HEPP-EG-6 was complexed with metal ions, Cu<sup>2+</sup>, Ni<sup>2+</sup>, Co<sup>2+</sup>, Mn<sup>2+</sup> and Zr<sup>2+</sup> and examined from TGA, suggesting that there may be powerful intramolecular hydrogen bonding. Absence of such hydrogen bonding in chelates favours the reduction in the thermal stability of poly chelates compared with the parent copolymer. It seems that metal ions accelerate the decomposition of poly chelates. For the copper chelates, the decomposition occurred at lower temperature which may have been due to the oxidation of the copolymer by the catalytic action of the Cu<sup>2+</sup> ion. Mehdi Rashidi et al(17) studied the complexes [Pt (CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>)(NH)], Ia (NN=2, 2' - bipyridyl) and Ib (NN=1, 10- phenanthrolin), react with dibromoalkanes Br(CH<sub>2</sub>)<sub>n</sub> Br (n=3-6) to give new platina (IV) Cyclopentane complexes fully characterized as [Pt. Br (CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>){(CH<sub>2</sub>)<sub>n</sub> Br} (NN)]. When n=2, complexes react with Br(CH<sub>2</sub>)<sub>2</sub> grto give binuclear complexes [Pt<sub>2</sub>Br<sub>2</sub>(CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>-CH<sub>2</sub>)<sub>2</sub>{m-(CH<sub>2</sub>)<sub>2</sub>}(N·N)<sub>2</sub>]. All the reactions proceed by the SN<sub>2</sub> mechanism and rates of reactions follow the sequence n=2>>6>3>4=5. The trend of reactivity is discussed in terms of chain length of dibromides and J (Pt-C). values of bond between Pt and Poly methylene chain in the platina (IV) cyclopentane complexes.

Jun-Fa Wei et al(18) synthesized several dicopper complexes & relevant dimetal complexes with the bi-Schiff base ligands HDFA, PHManalysed with TG- DTA for thermal study and reported the low temperature for loss of water molecules for indicating non coordination to metal cores. The organotin complexes were studied by H. Masooda, S. Yaqubb et al(19) were subjected to thermal decomposition by thermogravimetric analysis (TGA). The decomposition of these compounds occurs mostly in two steps. Kinetic para meters such as order of reaction, activation energy, enthalpy and entropy of activation were calculated by using the coats and Horowitz methods. The calculated values are in good agreement with observed TG values that confirm the structural integrity of the complexes.

The thermal decomposition behaviour of the Fe (II), Ni (II) and Zn (II) complexes of poly dithiooxamide has been studied by H. A Maydamal, A. El-Shekeil et al(20) and the coast Redferin integral method is used to evaluate the kinetic parameters for the successive steps in the decomposition sequence observed in the TGA curves. The thermal stabilities of these complexes are discussed in terms of repulsion among electron pairs in the valance shell of the central ion and electronegativity effects.

Co (II) complex with Schiff base of 5-anilino 1,2,3,4 – thiaziazole (5-ATT): -

The result obtained by the usual elemental analysis and estimation of metal content are suggestive of the molecular formula [Co(5-ATT)<sub>2</sub>Br<sub>2</sub>].Thermal degradation pattern of this complex is as shown in Table below.

S. No.	Temp. range (°c)	Species degraded	% Weight loss	
			Found	Cal
1.	100 - 270	Loss of moisture and 2 Br – ion	14.10	14.80
2.	250 - 390	Loss of whole ligand moiety	71.20	71.81
3.	320	CoO formation	12.23	12.00

The second stage of decomposition was selected to study the chemical kinetics. The kinetic parameters, such as order of reaction, activation energy, entropy of activation and frequency factor were primarily evaluated by Freeman and carroll method and were compared by the values obtained by Doyle's method as modified by Zsako.

#### IV. Conclusion

The kinetic parameters are determined firstly using Freeman and Carroll method and the results are compared with the data developed by Zsako Procedure. A short experimental technique for recording the thermogravimetric curves for the complexes on a NETZSCH Simultaneous Thermal Analyzer STA-409 has also been discussed here. The existing Percentage weight of the complex at equal temperature intervals i.e., 10°C were noted directly from TG curve for a particular stage of thermal decomposition. A plot of  $\Delta \log dw / dt / \Delta \log w$ , versus  $T-1 \times 10^{-3} / \Delta \log W$  has been drawn for the stage of decomposition under consideration where

$W_r = W_c - W$ ,  $W_c =$  Total weight loss on completion of reaction,  $W =$  Total weight loss up of time  $t$ .

The straight line obtained is suggestive of the order of reaction from their intercept on Y-axis and the activation energy is equal to  $2.303 R \times$  slope. Freeman and Carroll had suggested that a good straight line is obtained, however, in the present cases remarkable deviations have many times been observed. Possibly on account of experimental factors, compactness, particle size etc. over which there were no controls.

The values for order of reaction and activation energy were revised by this method for the particular stage of transformation as in case of calculations by Freeman and Carroll procedure. This minimum deviation  $\delta$  min for particular Bi value and Ea value suggest the order of reaction and activation energy for the considered stage of transformation.

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