

Thermal Decomposition Kinetic Studies of Nickel 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 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.

Materials and Methods: 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 CaCl₂.

Results: 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 x 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 were there no controls.

Conclusion: 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 x 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 were there no controls.

Key Word: Thermogravimetric Analysis, Kinetic Parameters Determination

Date of Submission: 11-10-2021

Date of Acceptance: 25-10-2021

I. Introduction

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.

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.

Reaction of the 1, 2, 3, 4 - thiaziazoles 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 - thiaziazoles 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 - thiaziazole (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 - thiaziazole (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

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 CaCl₂. 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 CaCl₂. 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 CaCl₂.

A weighted amount of nickel complex was taken and decomposed by repeated evaporation with perchloric acid and concentrated nitric acid mixture. The decomposed product was treated with concentrated Hydrochloric acid and evaporated almost to dryness. It was diluted with water and the Nickel content was precipitated as Nickel dimethylglyoximate in ammoniacal medium. The bright red precipitate was filtered, washed with Water, dried at 105°-110°C in air oven and weighted as Ni(C₄H₇N₂O₂)₂. The amount of Nickel was calculated by multiplying the weight of the precipitate with the chemical factor 0.20314.

An accurately weighted quantity (0.5-0.209) of (the powdered complex was placed in 500 ml capacity round bottom flask containing 2 ml of liquid bromine and 20-30 ml. concentrated nitric acid. The mouth of the flask was covered by conical funnel. The contents of flask were strongly refluxed for an hour over steam bath to a small volume. This process was repeated twice by the addition of the same amount of bromine and nitric acid and heating was continued until the solution was clear. Insoluble residue was removed by diluting the solution with excess of water and filtering the solution while hot. The solution was cooled and transferred into a beaker. Sulphate formed on account of oxidation was precipitate as barium sulphate. The precipitate was filtered, washed till free from ionic impurities, dried and weighed as barium sulphate. The gravimetric factor for sulphate is 0.13737.

The Bromine content of the complexes was determined as silver chloride on addition of silver nitrate to solution of the compound dissolved in Bromide free dilute HNO₃. In case compound were insoluble in dilute nitric acid the complex was initially fused with fusion mixture, extracted with hot water and then acidified with hot water and then acidified with dil. HNO₃ the conversion factor for evaluating Bromide is 0.24734.

III. Result and Discussion

The value obtained by the elemental analysis and by the conventional gravimetric estimation for the metal content in the complex compound are suggestive of the molecular Formula [Ni(L₂)Cl₂] and molecular weight 487.11 mg.

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Thermal degradation pattern of this complex is as shown in Table-1

Table-1: Thermoanalytical data and decomposition temperature for [NiL₂Br₂]: -

| S. No. | Temp. range (°c) | Species degraded | % Weight loss | |
|--------|------------------|---------------------------------|---------------|-------|
| | | | Found | Cal |
| 1. | 100 - 120 | Loss of moisture and 2 Br – ion | 14.10 | 11.78 |
| 2. | 250 - 310 | Loss of whole ligand moiety | 74.17 | 71.56 |
| 3. | 320 | NiO formation | 12.05 | 12.20 |

Freeman and Carroll graphical method was primarily employed to evaluate the order of reaction and activation energy for the first stage of decomposition. In the selected stage of thermal decomposition, the existing weights of the complex at equal temperature interval 10°C were noted as well as $\frac{\Delta T^{-1} \times 10^{-3}}{\Delta \log W_r}$ were calculated and tabulated in Table-II.

Where $W_r = W_c - W$

W_c = Weight loss at completion of reaction

W = Total weight loss up to time T.

Table- II: Data obtained by Freeman and Carroll method.

| S. No. | Temp. | Weight | $\frac{\Delta \log (dw/dt)}{\Delta \log W_r}$ | $\frac{\Delta T^{-1} \times 10^{-3}}{\Delta \log W_r}$ |
|--------|-------|---------|---|--|
| 1 | 150 | 3.67197 | 3.41925 | 1.79492 |
| 2 | 160 | 3.64645 | 4.29935 | 2.07683 |
| 3 | 170 | 3.60597 | -2.99586 | 1.43727 |
| 4 | 180 | 3.53758 | -2.81142 | 0.94889 |
| 5 | 190 | 3.42591 | -1.08142 | 0.35171 |
| 6 | 200 | 3.26904 | -0.42427 | 0.13124 |
| 7 | 210 | 3.14530 | 0.06858 | 0.03999 |

Initial weight at 130°C = 3.77142 mg.

Final weight at 220°C = 3.14128 mg.

The plot $\frac{\Delta \log (dw/dt)}{\Delta \log W_r}$ versus $\frac{\Delta T^{-1} \times 10^{-3}}{\Delta \log W_r}$ gave straight line with an intercept at 0.2 and applying $E_a = 2.303 R \times$ Slope gave the values of activation energy to be equal to 12.81 Kcal / mole.

The same values for different weights taken at different temperature were subjected to the Zsako method. The weight of the compound at different temperatures as noted from the TG curves in the earlier procedure were used for the calculation with the help of following relations.

$$\alpha = \frac{W_0 - W_t}{W_0 - W_f}$$

Where W_0 = Initial weight

W_f = final weight

W = Actual Weight

The log F(a) values are tabulated in Table - III

Table- III: Data of log F(α) values for the complex [Ni₂L₂Br₂] at different temperatures.

| S. No. | Temp. | Weight | $\alpha = \frac{W_0 - W_t}{W_0 - W_f}$ | log α | Log (ln $\frac{1}{1-\alpha}$) | Log ($\frac{\alpha}{1-\alpha}$) |
|--------|-------|---------|--|----------|--------------------------------|-----------------------------------|
| 1 | 140' | 3.71886 | 0.08340 | -1.07870 | -1.06000 | -1.04095 |
| 2 | 150 | 3.67797 | 0.14830 | -0.82885 | -0.79446 | -0.75914 |
| 3 | 160 | 3.64645 | 0.19832 | -0.70263 | -0.65551 | -0.60663 |
| 4 | 170 | 3.60597 | 0.26256 | -0.58077 | -0.51631 | -0.44849 |
| 5 | 180 | 3.53758 | 0.37109 | -0.43052 | -0.33370 | -0.22910 |
| 6 | 190 | 3.42591 | 0.54830 | -0.26098 | -0.09977 | 0.08416 |
| 7 | 200 | 3.26904 | 0.79725 | -0.09840 | 0.20297 | 0.59463 |
| 8 | 210 | 3.14530 | 0.99362 | -0.00277 | 0.70368 | 2.1923 |

The β_0 values for different order of reaction were calculated by the following equation.

$$\beta_0 = \log a - \log p(x)$$

$$\beta_1 = \log (\ln 1 / 1-\alpha) - \log p(x)$$

$$\beta_2 = \log (\alpha / 1-\alpha) - \log p(x)$$

The value of - log p(x) was noted from Zsako table for temperature from 100-430°C for energy of activation from 10-66 kcal / mole.

The value of log f(a) was used to evaluate the value of β_0 , β_1 and β_2 for different activation energies at all the temperatures. The calculated values of

ultimately used to get the 6 min. values for all three presumed order of reaction. All the data obtained are given in the Table-IV, V and VI.

Table - IV: Calculation of β_0 for different activation energies and δ_0 , values at different temperature for $[\text{Ni}_2\text{L}_2\text{Br}_2]$:-

| S. No. | Temp. | 10 k cal | 12 kcal | 14 k cal |
|--------|-----------------------------------|----------|---------|----------|
| 1 | 140 | 6.44430 | 7.65230 | 8.83830 |
| 2 | 150 | 6.55015 | 7.73615 | 8.89615 |
| 3 | 160 | 6.53837 | 7.69937 | 8.83437 |
| 4 | 170 | 6.52923 | 7.66323 | 8.77623 |
| 5 | 180 | 6.55348 | 7.66548 | 8.75548 |
| 6 | 190 | 6.60102 | 7.69202 | 8.76402 |
| 7 | 200 | 6.64560 | 7.71960 | 8.76560 |
| 8 | 210 | 6.62923 | 7.68023 | 8.71423 |
| | Average ($\bar{\beta}_0$) | 6.56192 | 7.68854 | 8.79304 |
| | Standard Deviation (δ_0) | 0.05984 | 0.02719 | 0.05464 |

Table- V: Calculation of β_1 , for different activation energies and δ_1 , values at different temperature for $[\text{Ni}_2\text{L}_2\text{Br}_2]$ complex.

| S.No. | Temp. | 16 k cal | 18 kcal | 20 k cal |
|-------|-----------------------------------|----------|----------|----------|
| 1 | 140 | 10.03000 | 11.18300 | 12.33100 |
| 2 | 150 | 10.07054 | 11.20254 | 12.32354 |
| 3 | 160 | 10.00349 | 11.10949 | 12.20449 |
| 4 | 170 | 9.94168 | 11.02569 | 12.09869 |
| 5 | 180 | 9.92730 | 10.99430 | 12.04630 |
| 6 | 190 | 9.97823 | 11.01823 | 12.05323 |
| 7 | 200 | 10.10297 | 11.12597 | 12.13797 |
| 8 | 210 | 10.43368 | 11.43468 | 12.43068 |
| | Average ($\bar{\beta}_1$) | 10.06098 | 11.13673 | 12.20323 |
| | Standard Deviation (δ_1) | 0.15164 | 0.13358 | 0.13455 |

Table- VI: Calculation of β_2 , for different activation energies and δ_2 , values at different temperature for $[\text{Ni}_2\text{L}_2\text{Br}_2]$ complex.

| S.No. | Temp. | 30 k cal | 32 kcal | 34 k cal |
|-------|-----------------------------------|----------|----------|----------|
| 1 | 140 | 17.98205 | 19.09505 | 20.20705 |
| 2 | 150 | 17.86886 | 18.95686 | 20.04286 |
| 3. | 160 | 17.64537 | 18.70837 | 19.76937 |
| 4 | 170 | 17.44151 | 18.48151 | 19.52551 |
| 5 | 180 | 17.31590 | 18.33490 | 19.34990 |
| 6 | 190 | 17.29816 | 18.29516 | 19.28916 |
| 7 | 200 | 17.49063 | 18.47063 | 19.44263 |
| 8 | 210 | 18.78439 | 19.74239 | 20.69639 |
| | Average ($\bar{\beta}_2$) | 17.72835 | 18.76060 | 19.79035 |
| | Standard Deviation (δ_1) | 0.46100 | 0.45782 | 0.46029 |

A comparison of the 6 min, values evidently indicated that the least value 0.02719 corresponding to, activation energy $E_a = 12$ kcal/mol. order of reaction $b=0$ and $\bar{\beta}_0 = 7.688854$.

Table- VII

| b = 0 | | b = 1 | | b = 2 | |
|----------|------------|----------|------------|----------|------------|
| Ea | | Ea | | Ea | |
| Kcal/mol | δ_0 | Kcal/mol | δ_1 | Kcal/mol | δ_2 |
| 10 | 0.05984 | 16 | 0.15164 | 30 | 0.46100 |
| 12 | 0.02719 | 18 | 0.13358 | 32 | 0.45782 |
| 14 | 0.05464 | 20 | 0.13455 | 34 | 0.46029 |

These observed values suggested the order of reaction is zero and the activation energy equal to 12 kcal/mol for the step of reaction under consideration.

With the help of above values, the apparent frequency factor $Z = 1.35590 \times 10^3 \text{ sec}^{-1}$ and apparent entropy of activation $\Delta S^\ddagger = -188.52836 \text{ e.u.}$ Where calculated for the considered stage of transformation using the equation.

$$\log z = \bar{\beta} + \log Rq - \log E_a$$

and

$$\Delta S^\ddagger = 2.303 R \log Zh /KT$$

Where T is the absolute temperature i.e., 483 k at which the weight lost is half of total weight lost.

Thus, resemblance of the value for kinetic terms obtained for the same stage of decomposition evaluated by two different methods is comparable and is given in the Table - VIII.

Table-VIII

| | Methods | Order of reaction | activation energy |
|----|---------------------|-------------------|-------------------|
| 1. | Freeman and Carroll | 0.2 | 12.819 kcal/mole |
| 2. | J. Zsako | 0 | 12.000 kcal/mole |

Where T is the absolute temperature.

IV. Conclusion

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 were there no controls.

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Ghanshyam Kumar. "Thermal Decomposition Kinetic Studies of Nickel Complexes. *IOSR Journal of Applied Chemistry (IOSR-JAC)*, 14(10), (2021): pp 01-06.