

**THERMAL STABILITY AND DECOMPOSITION OF
POLY[ETHYLENEDIAMINETETRAACETATO-AQUA-DISODIUM-
NICKEL(II)]: A TGA-DTA INVESTIGATION****U.U. Ruziev., Kh.Kh. Turaev., A.B. Ibragimov., K.B. Kholduraev**

Department of Chemistry, Termez State University, 43 Barkamol Avlod Street,
Termez, 190111, Uzbekistan

Institute of General and Inorganic Chemistry of Uzbekistan Academy of

Abstract

The thermal stability and decomposition behavior of poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)] were investigated using simultaneous thermogravimetric analysis (TGA) and differential thermal analysis (DTA) in the temperature range of approximately 25-800 °C. The thermogram revealed a multistep decomposition process involving dehydration, partial degradation of coordinated organic fragments, and complete decomposition of the EDTA framework. Three major mass-loss stages were observed. The first decomposition stage occurred between 88.0 and 316.9 °C with a mass loss of 40.82%, corresponding to the removal of coordinated water molecules and the initial degradation of organic functional groups. The second stage, between 316.9 and 439.2 °C, exhibited a mass loss of 29.62%, attributed to the decomposition of the EDTA ligand backbone and cleavage of metal-ligand coordination bonds. The third stage occurred between 441.4 and 753.0 °C with an additional mass loss of 9.97%, corresponding to carbonaceous residue oxidation and formation of thermally stable nickel-containing inorganic residues. The DTA curve displayed thermal events associated with these decomposition processes, confirming the multistep thermal degradation mechanism. The results demonstrate that the complex possesses good thermal stability up to approximately 300 °C and decomposes progressively at higher temperatures.

Keywords: Nickel(II), EDTA, Coordination Polymer, Thermogravimetric Analysis, Thermal Stability, TGA, DTA.

Introduction

The thermal stability of coordination compounds is an important parameter influencing their practical applications in catalysis, materials science, environmental technologies, and coordination chemistry. Thermogravimetric analysis (TGA) is widely employed to investigate decomposition pathways, hydration behavior, ligand stability, and the formation of final metal oxide residues. Nickel(II)-EDTA coordination compounds represent an important class of metal chelates due to their

high stability constants and diverse structural properties. Ethylenediaminetetraacetic acid (EDTA) acts as a hexadentate ligand, coordinating metal ions through four oxygen atoms and two nitrogen donor atoms. The presence of coordinated water molecules and organic ligand fragments significantly affects the thermal decomposition pathway of these compounds. Simultaneous thermogravimetric and differential thermal analyses provide valuable information concerning dehydration processes, ligand degradation mechanisms, and the thermal stability of coordination polymers. Therefore, the present work aims to investigate the thermal decomposition behavior of poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)] through TGA-DTA measurements.

Results and Discussion

Thermogravimetric Analysis

The TGA-DTA curves of poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)] indicate a multistep decomposition process extending from room temperature to approximately 800 °C. The decomposition occurs through three distinct stages accompanied by characteristic thermal effects observed on the DTA curve. The gradual mass loss observed throughout the heating process reflects the sequential removal of water molecules, decomposition of the organic ligand framework, and formation of thermally stable inorganic residues.

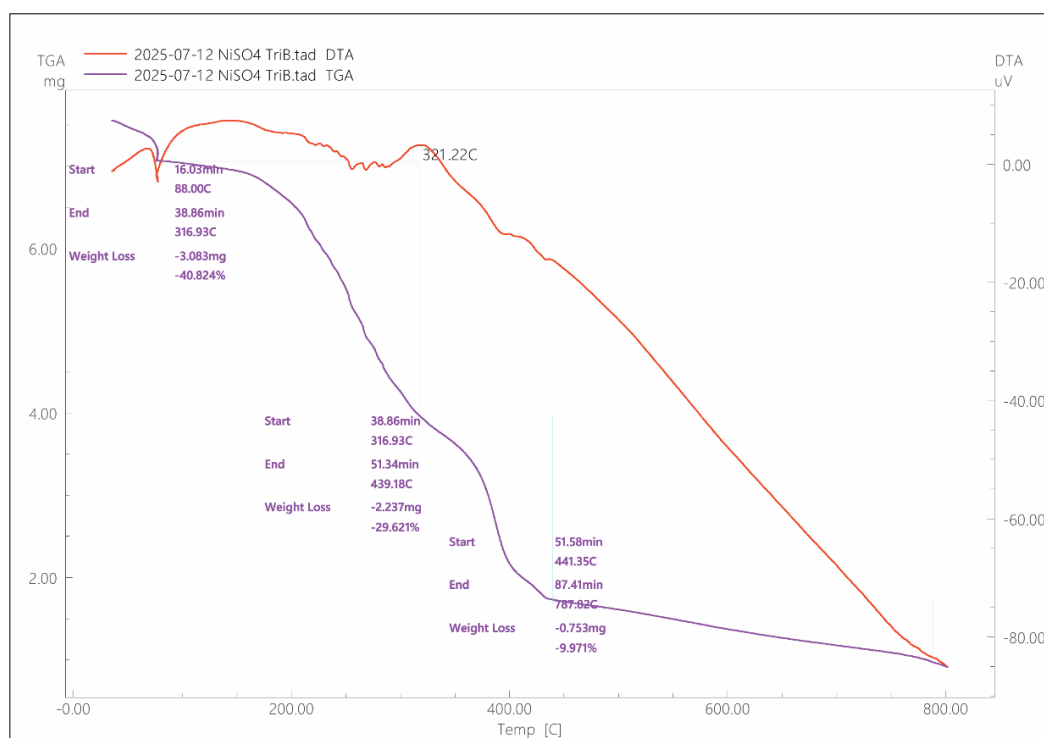


Figure 3. Thermogravimetric (TGA) and differential thermal analysis (DTA) curves of poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)] showing the multistep thermal decomposition process.

First Decomposition Stage (88.0–316.9 °C)

The first major weight-loss stage begins at approximately 88.0 °C and ends at 316.9 °C, resulting in a mass loss of 40.82%.

This stage is attributed primarily to:

- Removal of physically adsorbed moisture.
- Elimination of coordinated water molecules.
- Initial decomposition of carboxylate groups.
- Partial degradation of the EDTA coordination environment.

The DTA curve exhibits a thermal event within this temperature range, indicating that dehydration and initial ligand decomposition occur simultaneously. The relatively large mass loss suggests that coordinated water molecules and thermally labile organic fragments contribute significantly to the overall decomposition process. The absence of significant decomposition below approximately 80 °C demonstrates that the complex possesses good thermal stability under ambient conditions.

Second Decomposition Stage (316.9–439.2 °C)

The second decomposition stage occurs between 316.9 °C and 439.2 °C, producing an additional mass loss of 29.62%.

This region corresponds to:

- Breakdown of the EDTA carbon skeleton.
- Cleavage of C-N and C-C bonds.
- Destruction of the chelating framework.
- Progressive rupture of Ni–EDTA coordination interactions.

The DTA curve displays a pronounced thermal effect near 381 °C, indicating an intense decomposition process. This thermal event can be assigned to the combustion and degradation of the major organic component of the ligand. The large mass reduction observed during this stage confirms that most of the organic ligand is removed within this temperature interval.

Third Decomposition Stage (441.4–753.0 °C)

The final decomposition stage extends from approximately 441.4 °C to 753.0 °C and is associated with a mass loss of 9.97%.

This stage is attributed to:

- Oxidation of residual carbonaceous material.
- Decomposition of intermediate metal-organic fragments.

- Formation of thermally stable inorganic nickel-containing residues.

The relatively smaller weight loss compared with previous stages suggests that only residual organic matter remains after the second decomposition process. At temperatures above 750 °C, the mass becomes nearly constant, indicating completion of thermal degradation and formation of stable inorganic products.

Residual Mass and Final Decomposition Product

The total mass loss observed during the three decomposition stages is: $[40.82 + 29.62 + 9.97 = 80.41\%]$ Thus, the remaining residue corresponds to approximately: $[100 - 80.41 = 19.59\%]$

The residual mass is attributed mainly to thermally stable nickel-containing inorganic products, most likely nickel oxide (NiO) together with sodium-containing inorganic species formed during thermal decomposition. The formation of such residues is typical for nickel coordination compounds undergoing thermal degradation under oxidative conditions.

Table 1. Thermal Decomposition Stages of Poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)]

Stage	Temperature Range (°C)	Weight Loss (%)	Proposed Process
I	88.0-316.9	40.82	Dehydration and initial ligand decomposition
II	316.9-439.2	29.62	Major EDTA framework degradation
III	441.4-753.0	9.97	Oxidation of residual organic fragments
Residue	>753	19.59	Nickel-containing inorganic residue

Conclusion

Thermogravimetric and differential thermal analyses demonstrated that poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)] undergoes thermal decomposition through three distinct stages. The first stage (88.0-316.9 °C) corresponds to dehydration and initial degradation of the ligand framework, resulting in a mass loss of 40.82%. The second stage (316.9-439.2 °C) involves extensive decomposition of the EDTA backbone and accounts for a further 29.62% mass

reduction. The third stage (441.4-753.0 °C) is associated with oxidation of residual organic fragments and contributes an additional 9.97% weight loss. The total mass loss of 80.41% leaves a stable residue of approximately 19.59%, attributed primarily to nickel-containing inorganic products. The results indicate that the synthesized complex exhibits good thermal stability below 300 °C and undergoes gradual decomposition at elevated temperatures, confirming the formation of a thermally stable nickel(II)-EDTA coordination polymer.

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