

Thermal Decomposition Pathways of 5-Sulfosalicylic Acid–1,10-Phenanthroline Complexes of Thorium(IV) and Dysprosium(III): A Comparative Study

Rukhsana Jabeen Wani

SAM Degree College, Budgam, 191111

Govt. College for Women, Nawakadal Srinagar, 190002

Abstract

The thermal decomposition behaviour of two mixed-ligand rare-earth/actinide complexes containing 5-sulfosalicylic acid (SSA) and 1,10-phenanthroline (phen) is comparatively examined using previously reported thermogravimetric data for the thorium(IV)–SSA–phen and dysprosium(III)–SSA₃–phen systems. The Th(IV) complex was previously synthesized by coprecipitation from thorium(IV) nitrate tetrahydrate, SSA and 1,10-phenanthroline and characterized by elemental analysis, FTIR, UV–Visible spectroscopy, PXRD, TGA, DSC, SEM, magnetic measurements and photoluminescence. The Dy(III) complex, reported as Dy(SSA)₃Phen, was similarly characterized by elemental analysis, infrared spectroscopy and TGA. The comparison reveals distinct decomposition patterns despite the presence of the same two organic ligands. The Dy(III) complex exhibits a clearly resolved two-stage decomposition, with reported mass losses of 58.6% and 20.4% assigned predominantly to decomposition of SSA and phen, respectively, giving a total loss of 79.2%; the corresponding calculated loss for complete ligand decomposition was 80.9%. The first and second stages occur approximately over 273–325 and 557–669 °C, respectively. The Th(IV)–SSA–phen complex displays a progressive multistage thermal decomposition pattern, consistent with its chemically different ligand-to-metal stoichiometry and nitrate-containing coordination environment. The comparison indicates that thermal behaviour cannot be attributed solely to the identity of the metal ion. Rather, the decomposition pathway reflects the combined influence of metal centre, ligand-to-metal ratio, coordinated inorganic species and the stability of the resulting intermediate phases. In both systems, the major thermal events occur substantially above the initial heating region and involve progressive destruction of the organic coordination framework. The comparative analysis demonstrates that TGA/DSC provides a useful basis for distinguishing the thermal stability and decomposition characteristics of closely related SSA–phen complexes containing chemically different metal centres.

Keywords:

Thorium(IV); Dysprosium(III); 5-sulfosalicylic acid; 1,10-phenanthroline; thermogravimetric analysis; DSC; thermal decomposition; thermal stability; comparative thermal analysis.

1. Introduction

Thermal analysis is an important tool for investigating the stability and decomposition behaviour of coordination compounds because the sequence and magnitude of mass-loss events provide information about solvent retention, ligand degradation, coordinated inorganic groups and the formation of thermally stable residues. In metal–organic complexes, decomposition rarely occurs as a single event; instead, different ligand components and coordination environments may decompose over distinct temperature ranges.^{1–4} The thermal behaviour of actinide and lanthanide complexes is particularly interesting because these metal ions possess different electronic configurations, ionic sizes and coordination preferences. Thorium(IV) is a hard, highly charged actinide ion with a strong affinity for oxygen donor atoms and commonly forms complexes with relatively high coordination numbers.^{1–5} Dysprosium(III), in contrast, is a trivalent lanthanide ion whose coordination chemistry is dominated by predominantly ionic interactions with oxygen and nitrogen donor atoms.^{6–8} These differences can influence both the stability of the coordination framework and the nature of the products formed during thermal decomposition. 5-Sulfosalicylic acid (SSA) is particularly suitable for comparative coordination studies because it contains several oxygen-containing functionalities, including carboxylate, phenolic and sulfonate groups. 1,10-Phenanthroline (phen) is a rigid N,N-donor chelating ligand capable of forming stable metal–nitrogen interactions. The combination of SSA and phen therefore provides a useful mixed-donor system for comparing the thermal behaviour of complexes containing different metal centres.

A mixed-ligand Th(IV) complex involving SSA and 1,10-phenanthroline was reported by Wani *et al.* in 2023. The complex was synthesized by coprecipitation from Th(NO₃)₄·4H₂O and characterized using a broad range of techniques, including TGA and DSC. The authors reported an eight-coordinate Th(IV) complex in which SSA and phen participate in coordination and nitrate is also associated with the coordination environment.¹ The published work therefore provides a well-characterized Th(IV) system for thermal analysis. An especially relevant comparison is provided by the work of Sun *et al.*, who reported the synthesis and thermal characterization of **Dy(SSA)₃Phen**. In this system, three SSA ligands and one phen ligand are associated with Dy(III). The authors reported a two-stage thermal decomposition of the isolated Dy complex, with the first stage attributed mainly to SSA decomposition and the second to phen decomposition.⁹ The two compounds therefore offer an informative but **non-isostructural comparison**. They contain the same principal organic ligands but differ in metal ion, ligand-to-metal stoichiometry and coordination environment. This distinction is important because differences in thermal behaviour cannot be assigned exclusively to the metal centre.

The objective of the present work is consequently to compare the reported thermal decomposition behaviour of the Th(IV)–SSA–phen and Dy(III)–SSA₃–phen systems, with particular emphasis on decomposition stages, temperature ranges, ligand degradation, relative thermal stability and final residue formation. Structural information is considered only insofar as it assists in explaining the observed thermal behaviour.

2. Comparative Basis and Experimental Data

2.1 Synthesis of Thorium (IV)–SSA–phen system

The Th(IV) complex was synthesized by coprecipitation of thorium(IV) nitrate tetrahydrate with SSA and 1,10-phenanthroline. The original investigation employed elemental analysis, FTIR, UV–Visible spectroscopy, PXRD, TGA, DSC, SEM, magnetic measurements and photoluminescence. The reported complex was described as an eight-coordinate, amorphous, diamagnetic and air-stable material.¹ For the present comparative analysis, the published TG/DSC profile of this Th(IV) complex is considered as the Th reference system.

2.2 Synthesis of Dysprosium (III)–SSA₃Phen system

Sun *et al.* synthesized the ternary Dy(III) complex using SSA as the first ligand and 1,10-phenanthroline as the second ligand, with the composition **Dy(SSA)₃Phen**. The isolated complex was characterized by elemental analysis, IR spectroscopy and TGA.⁹ The published TGA data provide quantitative information that is particularly useful for the present comparison. The Dy(SSA)₃Phen complex undergoes two principal decomposition stages. The first stage extends approximately from 273 to 325 °C and accounts for 58.6% mass loss, whereas the second occurs approximately from 557 to 669 °C and accounts for a further 20.4% loss. The total observed mass loss is 79.2%, close to the calculated value of 80.9% for complete decomposition of the organic ligands and formation of a rare-earth oxide residue.⁹

2.3 Comparative approach

The comparison is based on the following thermal parameters:

- initial low-temperature stability;
- number of principal decomposition stages;
- temperature range of major decomposition;
- relative contribution of the ligand components;
- overall mass-loss behaviour;
- high-temperature residue formation; and
- relationship between thermal behaviour and chemical composition.

Because the two compounds were not synthesized and measured under an identical experimental protocol in the same study, the comparison is interpreted as a **literature-based comparative thermal analysis**, rather than as a strictly controlled head-to-head experiment.

3. Results and Discussion

3.1 Comparative thermogravimetric behaviour

The TG profiles of the two complexes reveal an important difference in the organization of their decomposition processes. The Dy(SSA)₃Phen complex exhibits a relatively well-resolved **two-stage decomposition pattern**, whereas the Th(IV)–SSA–phen system shows a more progressive multistage thermal degradation. The two-stage behaviour of Dy(SSA)₃Phen is particularly informative because

the published authors directly associated the first major loss with SSA and the second with phen. The first decomposition stage produces a 58.6% mass loss between approximately 273 and 325 °C, followed by a second 20.4% loss between approximately 557 and 669 °C.⁹ The Th complex, in contrast, exhibits a broader decomposition sequence rather than a simple two-step separation corresponding to complete removal of the two ligand types. This difference is chemically reasonable because the Th compound contains a different ligand-to-metal ratio and a nitrate-containing coordination environment.¹ The thermal events therefore reflect not only the intrinsic decomposition of SSA and phen but also their interactions with Th(IV) and the associated inorganic coordination environment.

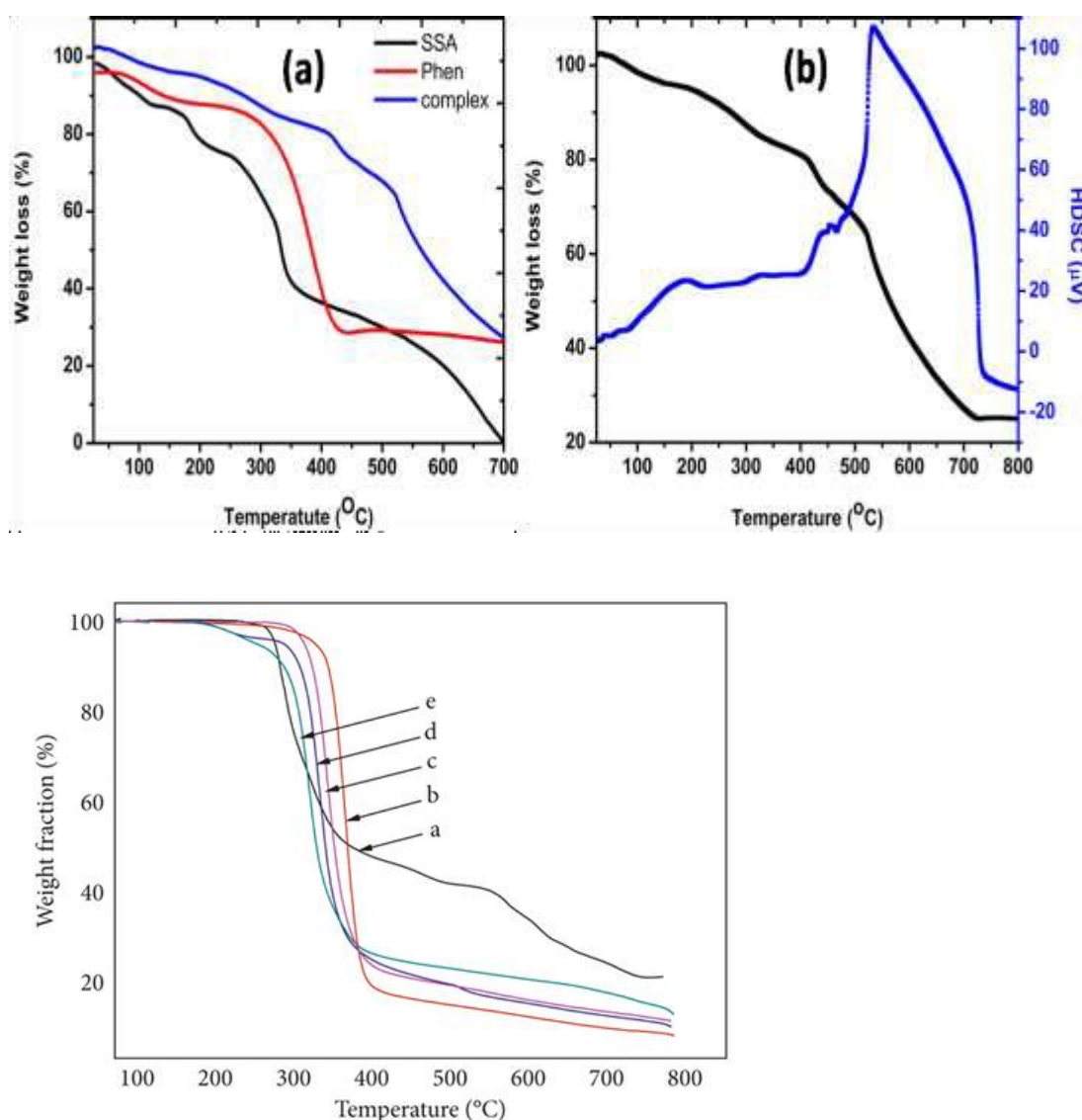


Figure 1. Comparative thermogravimetric profiles of the reported Th(IV)–SSA–phen and Dy(III)–SSA₃Phen complexes.

The Dy(III) data are reproduced/interpreted from Sun et al.⁹; the Th(IV) profile is from Wani et al.¹ The comparison is qualitative where numerical values were not explicitly reported in the respective publications.

3.2 Initial thermal stability

An important feature of the comparison is the behaviour in the initial heating region.

The Th(IV) complex does not show a dominant low-temperature dehydration event characteristic of a substantial amount of lattice or coordinated water. This observation is consistent with the previously reported characterization of the complex and suggests that the initial thermal region is comparatively stable.¹ The Dy(SSA)₃Phen system also does not show a large initial dehydration step. Instead, its first major decomposition begins at approximately 273 °C.⁹ Thus, both complexes remain comparatively stable through the lower-temperature region, but the onset of substantial decomposition in Dy(SSA)₃Phen is clearly defined in the published TGA data. This comparison suggests that the two complexes do not primarily undergo simple solvent-loss processes. Their major thermal behaviour is associated with degradation of the coordinated organic framework.

3.3 Major decomposition of the Dy(III) complex

The Dy(SSA)₃Phen complex provides the clearest quantitative reference for ligand-dependent decomposition. The first stage, from approximately 273 to 325 °C, produces a mass loss of 58.6%. The authors assigned this stage predominantly to decomposition of the SSA ligand.⁹ The second decomposition stage occurs at substantially higher temperature, approximately 557–669 °C, and results in an additional 20.4% mass loss attributed to decomposition of phen.⁹ The separation between the two decomposition regions is significant. It indicates that the two ligand components do not undergo complete simultaneous degradation. Instead, the thermogram reflects a sequential decomposition process in which the SSA-containing component is destabilized at lower temperature, while the phen-containing component persists to substantially higher temperatures. The total experimental mass loss of 79.2% agrees reasonably well with the calculated 80.9% expected for complete ligand decomposition and formation of a rare-earth oxide residue.⁹ This agreement provides strong support for the interpretation of the high-temperature residue as predominantly inorganic.

3.4 Thermal decomposition of the Th(IV) complex

The Th(IV)–SSA–phen complex exhibits a more complex decomposition profile than the two-stage Dy system. The TG/DSC data reported by Wani *et al.* indicate progressive decomposition of the complex rather than a simple separation into one SSA-loss event and one phen-loss event.¹ This behaviour can be understood in terms of the different chemical constitution of the Th complex. Unlike Dy(SSA)₃Phen, the Th system contains one SSA and one phen ligand together with nitrate associated with the Th(IV) centre.¹ The decomposition process therefore involves the breakdown of an organic–inorganic coordination framework rather than the sequential decomposition of three SSA ligands followed by one phen ligand. The broader decomposition behaviour should consequently not be interpreted as evidence of lower thermal stability by itself. A broad or multistage TG profile can arise because several chemically different components decompose over overlapping temperature ranges. Thus, the most appropriate description of the Th system is progressive multistage thermal decomposition, rather than a simple two-step ligand-loss mechanism.

3.5 Comparative decomposition stages

The major thermal characteristics of the two complexes are summarized in Table 1.

Table 1. Comparative thermal decomposition behaviour of Th(IV)–SSA–phen and Dy(III)–SSA₃Phen

Thermal characteristic	Th(IV)–SSA–phen	Dy(III)–SSA ₃ Phen
Metal ion	Th(IV)	Dy(III)
Ligand composition	SSA + phen + nitrate-containing environment	3 SSA + phen
Initial major thermal event	No pronounced dehydration event reported	No major low-temperature dehydration event
Main decomposition pattern	Progressive/multistage	Two major stages
First major decomposition	Progressive decomposition of coordinated framework	273–325 °C
First-stage assignment	Overlapping degradation of coordinated components	Mainly SSA
First-stage mass loss	Not stated here without direct numerical extraction from the published curve	58.6%
Second major stage	Further ligand/inorganic framework decomposition	557–669 °C
Second-stage assignment	Further degradation of ligand-containing framework	Mainly phen
Second-stage mass loss	Not stated here without direct numerical extraction	20.4%
Total reported mass loss	Profile-dependent	79.2%
Calculated loss	—	80.9%
High-temperature product	Thorium-containing oxide residue expected	Rare-earth oxide residue
Overall interpretation	Multistage degradation of an integrated coordination framework	Sequential ligand decomposition followed by inorganic residue formation

The table demonstrates that the two complexes cannot be ranked simply by counting the number of TG steps. The nature and temperature range of each decomposition stage are equally important.

3.6 Influence of ligand-to-metal stoichiometry

One of the most important factors distinguishing the two systems is their ligand-to-metal ratio. The Dy compound contains three SSA ligands and one phen ligand, whereas the Th compound contains one SSA and one phen together with nitrate.^{1,9} Consequently, the 58.6% first-stage mass loss observed for Dy(SSA)₃Phen is not directly comparable to a hypothetical SSA-loss percentage in the Th

compound. A larger contribution from SSA in the Dy thermogram is expected partly because **three SSA ligands are present per metal centre**. This is a critical point for comparative interpretation. The higher mass loss associated with the first Dy decomposition stage should therefore not automatically be interpreted as weaker Dy–SSA bonding or lower thermal stability.

Instead, the thermal profile reflects the combined effect of:

1. the number of SSA ligands;
2. their contribution to total molecular mass;
3. their coordination to the metal centre;
4. the presence or absence of nitrate; and
5. the thermal stability of the resulting intermediates.

The comparison therefore provides evidence for **composition-dependent thermal decomposition**, rather than a simple metal-dependent stability sequence.

3.7 Influence of the metal centre

The different metal ions nevertheless remain chemically important. Th(IV) possesses a higher charge density than Dy(III) and forms strong interactions with hard oxygen donor atoms.^{1–5} Dy(III), as a lanthanide ion, also has strong affinity for oxygen donors but typically forms more ionic coordination environments.^{6–8} These differences can influence the strength and distribution of metal–ligand interactions and therefore the temperatures at which ligand-containing fragments begin to decompose. However, the present comparison does **not** permit the effect of metal identity to be isolated quantitatively because the ligand stoichiometries and coordination environments are also different. Hence it is concluded therefore that the observed thermal differences arise from the **combined effects of metal ion, ligand stoichiometry and coordination environment**.

3.8 Stability of 1,10-phenanthroline within the complexes

The Dy thermogram provides particularly clear evidence that phen remains associated with a thermally persistent fraction of the complex after the first major decomposition event. The first Dy decomposition stage ends at approximately 325 °C, whereas the second major stage does not begin until approximately 557 °C.⁹ The approximately 230 °C separation between these temperature ranges indicates substantially different thermal decomposition behaviour for the SSA- and phen-containing components. This finding is consistent with the structural nature of phen as a rigid fused aromatic ligand. It also demonstrates why mixed-ligand complexes cannot always be described by a single thermal stability temperature. For the Th system, the corresponding ligand decomposition is less sharply separated, indicating that decomposition of the mixed coordination framework occurs through overlapping or sequential processes rather than the clearly resolved two-stage pattern observed for Dy(SSA)₃Phen.

3.9 DSC behaviour and its significance

DSC provides information complementary to TGA by indicating the energetic character of thermal events. For the Th complex, DSC was included in the original characterization and was interpreted together with the TG profile.¹ The major thermal events are associated with decomposition of the ligand-containing coordination framework rather than with simple melting of an intact molecular complex. This distinction is important because a genuine melting transition would not necessarily involve a large simultaneous mass loss, whereas decomposition is generally accompanied by a measurable decrease in sample mass. The Dy study did not provide an equivalent DSC analysis of the isolated complex; its thermal assessment was based primarily on TGA.⁹ Therefore, DSC cannot be used for a direct quantitative comparison between the two systems. Nevertheless, the combination of TG and DSC for the Th compound strengthens the interpretation that its major thermal events represent chemical decomposition rather than simple physical transitions.

3.10 High-temperature residue formation

Both systems ultimately approach an inorganic residue after destruction of the organic ligand framework. For Dy(SSA)₃Phen, the reported total mass loss of 79.2%, compared with a calculated 80.9%, was interpreted in terms of complete ligand decomposition and formation of a rare-earth oxide residue.⁹ The Th system similarly undergoes progressive destruction of the organic components, leaving a thermally stable thorium-containing residue. The formation of ThO₂ is chemically consistent with the established high-temperature behaviour of many Th(IV) compounds.^{10–13} The residue formation is therefore a common endpoint of the two systems, although the identity and exact composition of the final inorganic product depend on the metal and experimental atmosphere.

3.11 Comparative thermal stability

Dy(SSA)₃Phen begins its first major decomposition at approximately 273 °C, while a second major decomposition event occurs only at approximately 557 °C.⁹ This indicates that a substantial portion of the complex persists to relatively high temperature after the first ligand-decomposition event. The Th system displays progressive decomposition rather than the sharply separated two-stage pattern observed for Dy(SSA)₃Phen.¹ Therefore, its thermal behaviour is better described in terms of a **broad decomposition window** than by assigning a single decomposition temperature. Accordingly, it would be inappropriate to state categorically that either complex is "more thermally stable" without defining the stability criterion. We can say that **Dy(SSA)₃Phen** exhibits a distinctly separated two-stage decomposition with a high-temperature second stage, whereas Th(IV)–SSA–phen undergoes a more progressive multistage degradation of its mixed organic–inorganic coordination framework. This comparison is directly supported by the reported thermal data.

3.12 Mechanistic interpretation of the comparative decomposition

The thermal decomposition of the two complexes can be represented schematically as follows.

Th(IV)–SSA–phen

Th(IV)–SSA–phen/nitrate-containing framework

- progressive destabilization
- overlapping decomposition of SSA/phen/inorganic components
- Th-containing intermediates
- **thorium oxide residue**

Dy(III)–SSA₃Phen

Dy(SSA)₃Phen

- **SSA-dominated decomposition (273–325 °C)**
- Dy-containing intermediate
- **phen-dominated decomposition (557–669 °C)**
- **rare-earth oxide residue**

The Dy pathway is better resolved experimentally because the two principal decomposition regions are clearly separated.⁹ The Th pathway is more complex because of the different ligand-to-metal ratio and nitrate-containing environment. These schemes are conceptual rather than claims that specific intermediate compounds were isolated.

4. Comparative Interpretation

The comparison establishes several important features. First, both complexes show their principal decomposition at elevated temperature rather than undergoing dominant low-temperature solvent loss. Second, the Dy complex exhibits a particularly clear sequential decomposition of its two ligand classes. The first stage accounts for 58.6% mass loss and is associated mainly with SSA, whereas the second accounts for 20.4% and is associated mainly with phen.⁹ Third, the Th complex exhibits a more distributed decomposition pathway. This is consistent with the fact that its chemical composition differs substantially from Dy(SSA)₃Phen.¹ Fourth, the high-temperature persistence of the phen-containing fraction in the Dy complex demonstrates that the thermal behaviour of a mixed-ligand complex cannot be predicted simply from the thermal stability of the individual free ligands. Fifth, the comparison demonstrates why **mass-loss percentages must be interpreted together with molecular composition**. The Dy complex contains three SSA ligands per metal centre, whereas the Th complex contains only one. Thus, direct comparison of percentage mass loss without considering stoichiometry could lead to misleading conclusions.

Table 2. Factors governing the observed thermal decomposition behaviour

Factor	Th(IV)–SSA–phen	Dy(III)–SSA ₃ Phen	Thermal consequence
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Metal oxidation state	+4	+3	Different metal–ligand interactions
SSA content	One SSA	Three SSA	Strong effect on mass-loss contribution
Phen content	One phen	One phen	Common high-temperature organic component
Nitrate	Present in coordination environment	Not part of reported Dy(SSA) ₃ Phen formulation	Additional inorganic decomposition contribution in Th system
Decomposition pattern	Progressive/multistage	Two clearly separated stages	Different decomposition resolution
Principal lower-temperature process	Progressive ligand-framework degradation	SSA decomposition	Different ligand contribution
High-temperature process	Continued framework decomposition	Phen decomposition	Persistence of aromatic ligand
Final residue	Th-containing oxide	Rare-earth oxide	Metal-dependent inorganic endpoint

5. Relationship to Previously Reported Structural Characterization

The purpose of the present work is not to re-establish the complete molecular structures of the two compounds. Structural information is therefore considered only to the extent necessary for interpreting their thermal behaviour. The Th(IV) complex was previously reported as an eight-coordinate complex in which SSA and phen participate in coordination and nitrate is associated with the Th centre.¹ The Dy compound was reported as Dy(SSA)₃Phen.⁹ These established formulations are sufficient to explain why their thermograms should not be expected to be identical. In particular, the presence of three SSA ligands in the Dy complex provides a much larger SSA contribution to the total molecular mass, while the Th compound contains nitrate in addition to SSA and phen. Therefore, the thermal curves reflect different chemical compositions as well as different metal centres. This distinction strengthens rather than weakens the comparative analysis because it allows the thermal behaviour of two related but chemically distinct mixed-ligand systems to be examined within a common SSA–phen framework.

6. Overall Comparative Assessment

The principal thermal differences between the two complexes can be summarized as follows:

Th(IV)–SSA–phen:

A progressive, multistage decomposition of a nitrate-containing mixed-ligand coordination framework, followed by formation of a stable thorium-containing inorganic residue.

Dy(III)–SSA₃Phen:

A distinctly resolved two-stage decomposition, with the first stage at approximately 273–325 °C attributed mainly to SSA decomposition and the second at approximately 557–669 °C attributed mainly to phen decomposition.⁹ The most important comparative observation is therefore not simply that the curves have different shapes. Rather, the degree of separation of the ligand-decomposition processes differs markedly.

The Dy system shows relatively clear sequential decomposition, whereas the Th system displays more distributed degradation. This difference can reasonably be associated with the distinct metal centres, ligand-to-metal stoichiometries and inorganic coordination environments. The results therefore demonstrate that the thermal decomposition of SSA–phen complexes is governed by several mutually dependent variables and cannot be reduced to the identity of the metal ion alone.

7. Conclusions

The comparative thermal analysis of the reported Th(IV)–SSA–phen and Dy(III)–SSA₃Phen complexes demonstrates that both systems undergo substantial ligand-centred decomposition only after an initial region of comparatively limited mass change, but their subsequent decomposition pathways differ significantly. The Dy(III) complex exhibits a clearly resolved two-stage process, with 58.6% mass loss at approximately 273–325 °C attributed predominantly to SSA decomposition and a further 20.4% loss at approximately 557–669 °C associated mainly with phen decomposition, giving a total experimental loss of 79.2% that closely approaches the calculated value of 80.9%.⁹ The Th(IV)–SSA–phen complex, in contrast, displays a more progressive multistage decomposition of its mixed organic–inorganic coordination framework, consistent with its different ligand-to-metal stoichiometry and nitrate-containing environment. The comparison demonstrates that the thermal behaviour of these related complexes is governed not only by the identity and oxidation state of the metal centre but also by ligand-to-metal ratio, ligand composition and the nature of coordinated inorganic species. In particular, the three-SSA composition of Dy(SSA)₃Phen contributes substantially to its first mass-loss stage, while the Th system undergoes a more distributed decomposition process. The high-temperature residues in both cases are consistent with formation of thermally stable metal-containing oxide phases. Overall, the results show that thermal stability should be evaluated from decomposition onset, temperature range, stage separation and residue formation rather than from a single decomposition temperature, and that comparative TGA/DSC analysis provides a useful means of distinguishing the thermal behaviour of chemically related lanthanide and actinide complexes.

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