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# Calorimetric study and thermal analysis of $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$ and $[ErY(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$ (Gly: glycin)

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**Abstract**

Two solid-state coordination compounds of rare earth metals with glycin, $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$ and $[ErY(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$ were synthesized. The low-temperature heat capacities of the two coordination compounds were measured with an adiabatic calorimeter over the temperature range from 78 to 376 K. $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$ melted at 342.90 K, while $[ErY(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$ melted at 328.79 K. The molar enthalpy and entropy of fusion for the two coordination compounds were determined to be 18.48 kJ mol$^{-1}$ and 53.9 J K$^{-1}$ mol$^{-1}$ for $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$, 1.82 kJ mol$^{-1}$ and 5.5 J K$^{-1}$ mol$^{-1}$ for $[ErY(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$, respectively. Thermal decompositions of the two coordination compounds were studied through the thermogravimetry (TG). Possible mechanisms of the decompositions are discussed.

*Keywords:* $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$; $[ErY(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$; Heat capacity; Thermal decomposition; Adiabatic calorimeter; TG analysis

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## 1. Introduction

The anticancer effect of the coordination compound of $LaCl_3$ with glycin was reported by Anghilexi [1] in 1975. From then the coordination compounds of rare earth metals with amino acid have attracted increasing interest. Many applications of these coordination compounds have been discovered [2,3]. In recent years, about 200 coordination compounds of rare earth metal with amino acid have been synthesized and studied, and structures of over 40 of these coordination compounds have been determined [4]. Few studies on thermodynamic properties, especially low-temperature thermodynamic properties of these coordination compounds have been reported in the literature.

In this paper, low-temperature heat capacities of $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$ and $[ErY(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$ were measured with an adiabatic calorimeter over the temperature range from 78 to 376 K.

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## 2. Experimental

### 2.1. Sample preparation and characterization

$Gd(ClO_4)_3$, $Y(ClO_4)_3$ and $Er(ClO_4)_3$ were prepared by reaction of corresponding oxides with aqueous solution of $HClO_4$ [5,6]. $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6\cdot 5H_2O$ was synthesized by mixing mole ratio of 2:1:9 of $Gd(ClO_4)_3$, $Y(ClO_4)_3$ and glycin, and $[ErY(Gly)_6(H_2O)_4](ClO_4)_6\cdot 5H_2O$ was synthesized by mixing mole ratio of 1:1:6 of $Er(ClO_4)_3$, $Y(ClO_4)_3$ and glycin, respectively. The mixed solutions were stirred in water bath at 80 °C for several hours, and concentrated by evaporation at 60 °C. Afterwards, the solutions were cooled and filtered. The filtrates were kept at room temperature until crystalline products appeared. The crystals were filtered out and washed with absolute alcohol three times. Finally, the collected crystals were desiccated in a desiccator to prevent the coordination compounds from deliquescence in air.

The purities of the two coordination compounds were determined by EDTA titrimetric analysis, and the purities were 99.76 and 99.88% for $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6\cdot 5H_2O$ and $[ErY(Gly)_6(H_2O)_4](ClO_4)_6\cdot 5H_2O$, respectively.

### 2.2. Adiabatic calorimeter

Heat capacity measurements were performed with a small sample automatic adiabatic calorimeter. The calorimeter consists mainly of a sample cell, an inner and outer adiabatic shields, a platinum resistance thermometer, an electric heater, two sets of differential thermocouples and a high vacuum can. The principle and structure of the calorimeter were described previously in detail elsewhere [7–9]. Liquid nitrogen was used as the cooling medium. The mass of the samples for heat capacity measurements were 2.4648 g for $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6\cdot 5H_2O$ and 1.2536 g for $[ErY(Gly)_6(H_2O)_4](ClO_4)_6\cdot 5H_2O$, which are equivalent to 1.668 and 0.856 mmol, based on their molar mass 1478.13 and 1465.29 g $mol^{-1}$, respectively. Heat capacity of the sample is derived from the total heat capacity subtracting from the total heat capacity subtracting the heat capacity of the calorimeter cell determined in a separate experiment.

To verify the reliability of the adiabatic calorimeter, the molar heat capacities for the reference standard material $\alpha$-$Al_2O_3$ were measured. The deviations of our experimental results from the recommended values of the former National Bureau of Standards (NBS) [10] were within ±0.2% in the entire temperature range of 80–400 K.

### 2.3. Thermogravimetry (TG) analysis

A thermogravimetric analyzer (Model TGA/SDTA 851e, METTLER TOLEDO, Switzerland) was used for TG measurements under high purity (99.999%)

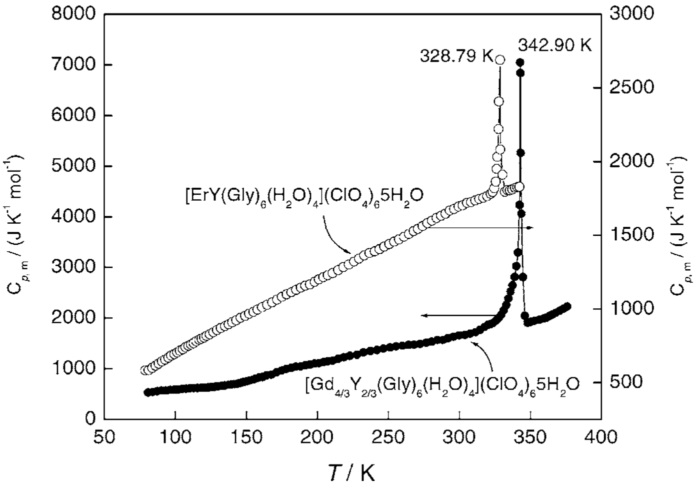

Fig. 1. Experimental molar heat capacity curves of $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6\cdot 5H_2O$ and $[ErY(Gly)_6(H_2O)_4](ClO_4)_6\cdot 5H_2O$.

nitrogen atmosphere with a flow rate of 60 ml $min^{-1}$. The heating rate was 10 °C $min^{-1}$.

## 3. Results and discussion

### 3.1. Heat capacity

The experimental molar heat capacities of the two solid-state coordination compounds of rare earth metal with amino acid are shown in Fig. 1, and tabulated in Tables 1 and 2. The molar heat capacities are fitted to the following polynomials.

For $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$ over the temperature range of 80–314 and 348–376 K:

$$C_{p,\mathrm{m}}\ (\mathrm{J\,K^{-1}\,mol^{-1}}) = 1285.7944 + 896.9624X - 381.3883X^2 - 209.6530X^3 + 482.1186X^4 + 134.0361X^5 \quad (1)$$

where $X = (T - 228)/148$, and $T$ is the absolute temperature. The correlation coefficient of the fitted curve, $R^2 = 0.9986$.

Table 1
Experimental molar heat capacities of $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$ [$M = 1478.13\ \mathrm{g\,mol^{-1}}$]

| $T$ (K) | $C_p$ ($\mathrm{J\,K^{-1}\,mol^{-1}}$) | $T$ (K) | $C_p$ ($\mathrm{J\,K^{-1}\,mol^{-1}}$) | $T$ (K) | $C_p$ ($\mathrm{J\,K^{-1}\,mol^{-1}}$) |
|---|---|---|---|---|---|
| 80.750 | 530.0 | 174.879 | 961.7 | 314.697 | 1768.7 |
| 84.061 | 536.3 | 178.306 | 997.4 | 317.955 | 1822.6 |
| 87.294 | 555.4 | 181.701 | 1015.6 | 320.000 | 1860.0 |
| 90.424 | 562.9 | 185.050 | 1038.5 | 321.970 | 1878.8 |
| 93.481 | 564.6 | 188.356 | 1044.4 | 323.939 | 1899.9 |
| 96.446 | 580.6 | 191.632 | 1063.4 | 325.891 | 1953.8 |
| 99.356 | 586.7 | 194.865 | 1081.0 | 327.794 | 1999.7 |
| 102.198 | 591.9 | 198.075 | 1099.1 | 329.640 | 2063.8 |
| 104.980 | 596.8 | 201.240 | 1111.6 | 331.416 | 2154.7 |
| 107.707 | 601.5 | 204.395 | 1129.2 | 333.109 | 2260.4 |
| 110.381 | 606.7 | 207.512 | 1146.2 | 334.694 | 2386.1 |
| 113.005 | 610.8 | 211.007 | 1163.8 | 336.190 | 2519.0 |
| 115.592 | 616.6 | 214.868 | 1196.0 | 337.615 | 2649.3 |
| 118.135 | 620.4 | 218.680 | 1224.8 | 338.961 | 2810.5 |
| 120.642 | 625.9 | 223.014 | 1254.7 | 340.215 | 3024.6 |
| 123.114 | 630.7 | 227.879 | 1274.4 | 341.372 | 3293.9 |
| 125.550 | 636.9 | 232.672 | 1325.1 | 342.517 | 4230.0 |
| 127.958 | 644.0 | 237.424 | 1351.7 | 342.905 | 7046.4 |
| 130.335 | 650.1 | 242.045 | 1365.7 | 343.018 | 6839.7 |
| 132.682 | 658.9 | 246.660 | 1398.6 | 343.286 | 5261.2 |
| 135.005 | 666.5 | 251.212 | 1421.2 | 343.810 | 4059.5 |
| 137.298 | 677.0 | 255.690 | 1443.4 | 344.741 | 2806.1 |
| 139.572 | 683.2 | 260.076 | 1450.1 | 346.150 | 2043.2 |
| 141.815 | 694.7 | 264.242 | 1468.8 | 348.039 | 1903.3 |
| 144.039 | 709.4 | 268.258 | 1471.2 | 350.306 | 1921.0 |
| 146.238 | 721.4 | 272.468 | 1491.7 | 352.661 | 1941.0 |
| 148.414 | 737.2 | 276.590 | 1512.0 | 356.591 | 1967.8 |
| 150.569 | 753.6 | 280.639 | 1534.7 | 359.394 | 1988.9 |
| 152.703 | 768.5 | 284.608 | 1562.2 | 361.364 | 2007.6 |
| 154.819 | 783.9 | 288.488 | 1563.4 | 363.712 | 2040.4 |
| 156.917 | 802.8 | 292.252 | 1605.3 | 365.682 | 2070.9 |
| 158.995 | 826.5 | 295.911 | 1629.8 | 368.030 | 2103.7 |
| 161.531 | 834.6 | 300.000 | 1653.9 | 370.399 | 2140.0 |
| 164.529 | 867.8 | 303.864 | 1665.6 | 373.333 | 2183.3 |
| 167.813 | 881.7 | 307.727 | 1691.4 | 375.985 | 2227.8 |
| 171.382 | 930.1 | 311.439 | 1719.5 | | |

Table 2
Experimental molar heat capacities of $[ErY(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$ [$M = 1465.29\,g\,mol^{-1}$]

| $T$ (K) | $C_p$ ($J\,K^{-1}\,mol^{-1}$) | $T$ (K) | $C_p$ ($J\,K^{-1}\,mol^{-1}$) | $T$ (K) | $C_p$ ($J\,K^{-1}\,mol^{-1}$) |
|---|---|---|---|---|---|
| 78.929 | 582.4 | 158.018 | 993.9 | 274.378 | 1561.4 |
| 81.266 | 582.8 | 161.257 | 1009.2 | 277.633 | 1580.4 |
| 83.707 | 599.0 | 164.791 | 1025.8 | 280.856 | 1596.8 |
| 86.082 | 614.1 | 168.280 | 1043.2 | 284.046 | 1616.4 |
| 88.398 | 627.6 | 171.728 | 1062.0 | 287.178 | 1631.8 |
| 90.659 | 641.5 | 175.134 | 1080.5 | 290.356 | 1645.3 |
| 92.873 | 653.7 | 178.503 | 1097.4 | 293.389 | 1666.3 |
| 95.038 | 667.4 | 181.832 | 1111.7 | 296.422 | 1676.8 |
| 97.165 | 680.7 | 185.128 | 1129.1 | 299.456 | 1693.3 |
| 99.252 | 692.1 | 188.393 | 1143.8 | 302.489 | 1700.9 |
| 101.305 | 703.5 | 191.630 | 1155.9 | 305.522 | 1718.9 |
| 103.323 | 714.0 | 194.838 | 1164.0 | 308.411 | 1723.4 |
| 105.310 | 725.3 | 198.013 | 1183.4 | 311.300 | 1735.4 |
| 107.267 | 738.6 | 201.158 | 1199.3 | 314.189 | 1745.9 |
| 109.200 | 748.1 | 204.279 | 1215.0 | 316.933 | 1753.4 |
| 111.104 | 757.7 | 207.370 | 1227.7 | 319.678 | 1763.9 |
| 112.986 | 766.8 | 210.833 | 1241.3 | 322.278 | 1780.5 |
| 114.844 | 778.3 | 214.656 | 1262.6 | 323.433 | 1792.5 |
| 116.682 | 788.5 | 218.443 | 1281.5 | 324.733 | 1818.0 |
| 118.499 | 796.6 | 222.189 | 1299.8 | 325.744 | 1863.1 |
| 120.296 | 805.0 | 225.894 | 1320.6 | 326.467 | 1948.7 |
| 122.073 | 815.9 | 229.544 | 1340.4 | 326.756 | 2031.3 |
| 124.142 | 825.9 | 233.156 | 1361.4 | 327.622 | 2219.0 |
| 126.714 | 839.6 | 236.767 | 1377.9 | 328.001 | 2407.1 |
| 129.468 | 855.3 | 240.378 | 1390.0 | 328.794 | 2689.1 |
| 132.187 | 868.0 | 243.844 | 1403.5 | 328.905 | 2081.1 |
| 134.865 | 880.8 | 247.321 | 1421.6 | 330.421 | 1909.7 |
| 137.509 | 894.8 | 250.798 | 1438.8 | 332.244 | 1794.0 |
| 140.123 | 907.5 | 254.244 | 1456.7 | 333.978 | 1806.0 |
| 142.972 | 920.9 | 257.670 | 1470.7 | 335.711 | 1803.0 |
| 146.049 | 937.5 | 261.067 | 1489.5 | 337.444 | 1810.5 |
| 149.093 | 951.0 | 264.439 | 1506.9 | 339.178 | 1819.5 |
| 152.101 | 966.1 | 267.781 | 1525.9 | 340.880 | 1824.8 |
| 155.076 | 979.2 | 271.096 | 1542.2 | 342.508 | 1826.5 |

For $[ErY(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$, over the temperature range of 78–324, and 332–343 K:

$$C_{p,\mathrm{m}}\,(J\,K^{-1}\,mol^{-1}) = 1243.1840 + 630.6306X + 65.4433X^2 + 86.0700X^3 - 113.0539X^4 - 91.1114X^5 \quad (2)$$

where $X = (T - 210.5)/132.5$, $R^2 = 0.9999$.

Melting was observed in the range of about 314–348 K with peak temperature 342.90 K for $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$; in the range of about 324–332 K with peak temperature 328.79 K for $[ErY(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$, respectively. But we considered that the melting should be a incongruent melting, and a dissociation reaction took place in the transition, so the liquid state was a mixture of some components different from the solid-state in structures.

### 3.2. Melting points, molar enthalpies and entropies of fusion

The melting temperatures of the two samples can be determined according to the $C_p$–$T$ data listed in Tables 1 and 2, and corresponding $C_p$–$T$ curves are shown in Fig. 1. The initial melting temperature is 314 K and the final melting temperature is 348 K, the melting peak temperature taken as

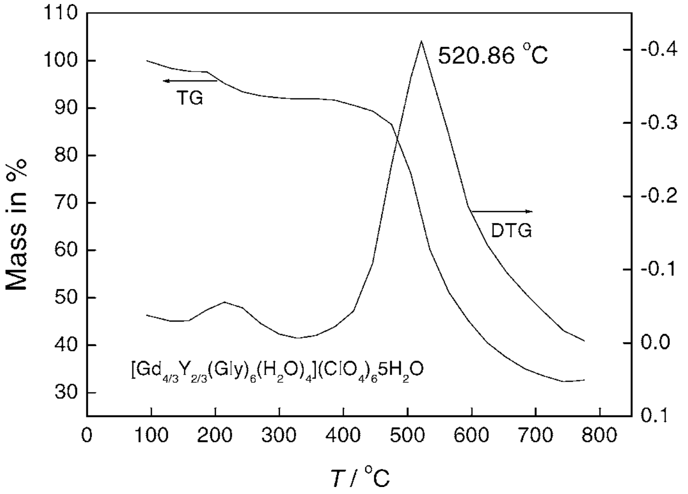

Fig. 2. TG/DTG curve of $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$ under nitrogen atmosphere.

the melting point is 342.90 K for $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$; the initial melting temperature is 324 K and the final melting temperature is 332 K, the melting peak temperature taken as the melting point is 328.79 K for $[ErY(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$. The two samples exist in two-phase state in the temperature range of 314–348 K for $[Gd_{4/3}Y_{2/3}(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$, and of 324–332 K for $[ErY(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$.

The molar enthalpies, $\Delta_{\mathrm{fus}}H_{\mathrm{m}}$ and molar entropies, $\Delta_{\mathrm{fus}}S_{\mathrm{m}}$ of the transition can be calculated based on the following equations:

$$\Delta_{\mathrm{fus}}H_{\mathrm{m}} = \left[Q - n\int_{T_{\mathrm{i}}}^{T_{\mathrm{m}}} C_{p,1}\,\mathrm{d}T - n\int_{T_{\mathrm{m}}}^{T_{\mathrm{f}}} C_{p,2}\,\mathrm{d}T - \int_{T_{\mathrm{i}}}^{T_{\mathrm{f}}} H_0\,\mathrm{d}T\right]/n \qquad (3)$$

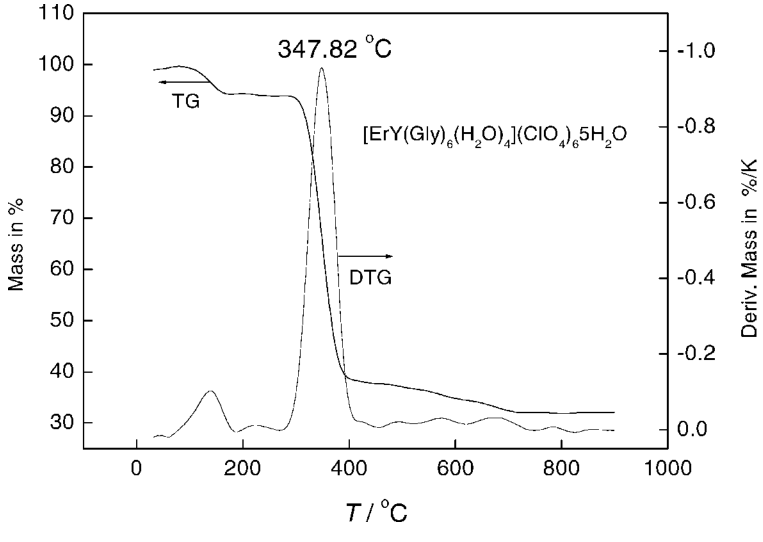

Fig. 3. TG/DTG curve of $[ErY(Gly)_6(H_2O)_4](ClO_4)_6{\cdot}5H_2O$ under nitrogen atmosphere.

$$\Delta_{\mathrm{fus}}S_{\mathrm{m}} = \frac{\Delta_{\mathrm{fus}}H_{\mathrm{m}}}{T_{\mathrm{m}}} \tag{4}$$

where $T_{\mathrm{i}}$ is the temperature a few degrees lower than the initial melting temperature, $T_{\mathrm{m}}$ the melting peak temperature, $T_{\mathrm{f}}$ the temperature slightly higher than the final melting temperature, $Q$ the total energy introduced into the sample cell from $T_{\mathrm{i}}$ to $T_{\mathrm{f}}$, $H_0$ the heat capacity of the sample cell from $T_{\mathrm{i}}$ to $T_{\mathrm{f}}$, $C_{p,1}$ the heat capacity of the sample in solid phase from $T_{\mathrm{i}}$ to $T_{\mathrm{m}}$, $C_{p,2}$ the heat capacity of the sample in liquid phase from $T_{\mathrm{m}}$ to $T_{\mathrm{f}}$, and $n$ the molar amount of the sample. The molar enthalpies and molar entropies of fusion for the two coordination compounds were determined to be 18.48 kJ mol$^{-1}$ and 53.9 J K$^{-1}$ mol$^{-1}$ for $[\mathrm{Gd}_{4/3}\mathrm{Y}_{2/3}(\mathrm{Gly})_6(\mathrm{H_2O})_4](\mathrm{ClO_4})_6{\cdot}5\mathrm{H_2O}$, and 1.82 kJ mol$^{-1}$ and 5.5 J K$^{-1}$ mol$^{-1}$ for $[\mathrm{ErY}(\mathrm{Gly})_6(\mathrm{H_2O})_4](\mathrm{ClO_4})_6{\cdot}5\mathrm{H_2O}$, respectively.

### 3.3. TG analysis

The TG curve of $[\mathrm{Gd}_{4/3}\mathrm{Y}_{2/3}(\mathrm{Gly})_6(\mathrm{H_2O})_4](\mathrm{ClO_4})_6{\cdot}5\mathrm{H_2O}$ is shown in Fig. 2. It can be seen clearly from the mass-loss curve that the most of the activities occur in the temperature range of 263–750 °C. The solid-state coordination compound was stable below 90 °C, and started decomposition at 93 °C. The total mass-loss (%) is 57.9%. We consider that the residue should be 4/3 $\mathrm{GdCl_3}$ and 2/3 $\mathrm{YCl_3}$ because the theoretically calculated mass-loss (%) is 56.8% if the final residues are 4/3 $\mathrm{GdCl_3}$ and 2/3 $\mathrm{YCl_3}$.

Fig. 3 shows that the structure of $[\mathrm{ErY}(\mathrm{Gly})_6(\mathrm{H_2O})_4](\mathrm{ClO_4})_6{\cdot}5\mathrm{H_2O}$ is stable below 100 °C, it started mass-loss at 112 °C. The experiment result of final mass-loss was 61.9%, which suggested that the residual products should be $\mathrm{ErCl_3}$ and $\mathrm{YCl_3}$, because the theoretical mass-loss (%) of the decomposition is 61.9% when the final residues are $\mathrm{ErCl_3}$ and $\mathrm{YCl_3}$. According to the mass-loss in each step, the possible mechanisms of the thermal decompositions may be deduced as follows:

$$[\mathrm{Gd}_{4/3}\mathrm{Y}_{2/3}(\mathrm{Gly})_6(\mathrm{H_2O})_4](\mathrm{ClO_4})_6\cdot 5\mathrm{H_2O}$$
$$\xrightarrow[9.5\%\ (10.96\%)]{93\text{–}320\,^{\circ}\mathrm{C}} [\mathrm{Gd}_{4/3}\mathrm{Y}_{2/3}(\mathrm{Gly})_6(\mathrm{H_2O})_4](\mathrm{ClO_4})_6$$
$$\xrightarrow[57.9\%\ (56.8\%)]{386\text{–}776\,^{\circ}\mathrm{C}} \tfrac{4}{3}\mathrm{GdCl_3} + \tfrac{2}{3}\mathrm{YCl_3}$$

$$[\mathrm{ErY}(\mathrm{Gly})_6(\mathrm{H_2O})_4](\mathrm{ClO_4})_6\cdot 5\mathrm{H_2O}$$
$$\xrightarrow[6.0\%\ (6.1\%)]{112\text{–}189\,^{\circ}\mathrm{C}} [\mathrm{ErY}(\mathrm{Gly})_6(\mathrm{H_2O})_4](\mathrm{ClO_4})_6$$
$$\xrightarrow[61.9\%\ (61.9\%)]{304\text{–}746\,^{\circ}\mathrm{C}} \mathrm{ErCl_3} + \mathrm{YCl_3}$$

The mass-loss percentages in the brackets are the calculated theoretical values of the corresponding thermal decomposition reaction.

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