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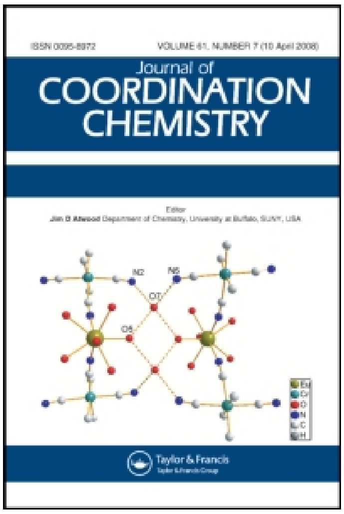

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Synthesis and characterization of cerium, thorium, and uranyl complexes with (*E*)-4-(4-methoxyphenoxy)-4- oxobut-2-enoic acid

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Synthesis and characterization of cerium, thorium, and uranyl complexes with (*E*)-4-(4-methoxyphenoxy)-4-oxobut-2-enoic acid

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(*E*)-4-(4-Methoxyphenoxy)-4-oxobut-2-enoic acid and its Ce(IV), Th(IV), and UO_2(II) complexes were synthesized and characterized by MS, elemental analysis, IR, UV, TG-DTA, and NMR. The complexes have composition $[CeL_2(OH)_2 \cdot 2H_2O] \cdot H_2O$, $[ThL_2(OH)_2 \cdot 2H_2O] \cdot H_2O$, and $[UO_2L_2 \cdot 2H_2O] \cdot H_2O$. Molar conductance data confirm that the three complexes are nonelectrolytes. The IR and NMR results show that the carboxylates coordinate to the metal ions bidentate, and the ester carboxylic groups do not take part in coordination. Luminescence spectra of the ligand and complexes in DMSO at room temperature were also studied showing strong luminescence of the metal ions.

Keywords: (*E*)-4-(4-Methoxyphenoxy)-4-oxobut-2-enoic acid; MS; NMR; Luminescence; Actinide coordination chemistry

1. Introduction

Actinide coordination chemistry is important for development of new technologies for safe separation and long-term storage of irradiated nuclear fuel [1–3]. Various ligands have been explored for these purposes and their extraction and coordination properties with lanthanide and actinide ions are well-documented [4, 5].

Spectroscopy of uranium compounds, particularly related to the uranyl ion, has been studied since 1940s because of the use of uranium as a nuclear fuel. Photoexcitation of the uranyl ion leads to a very strong oxidizer, and this fact allows the operation of a successful actinometer [6]. Other applications of uranyl photoprocesses include solar collectors [7], photochemical conversion of solar energy [8], and photocatalysts [9, 10]. Several reviews describe the spectroscopy [11], photophysics and photochemistry of this ion [12, 13]. Analytical applications of uranyl ion fluorescence resulted in very sensitive and selective methods for the determination of this ion in environmental and geological samples [14].

Ligands that have good coordination ability with lanthanide and actinide ions include Schiff-base [15], β-diketone [16], amino acid [17], carboxylate [18], and so on.

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Derivatives of 2-butenedioic acid, such as its alkyl esters [19, 20], possess a conjugated structure and have been widely applied as photoresist devices, semiconductors, potential catalysts [21], and can improve the stability of polymers [22, 23]. Some 2-butenedioic acid monoalkyl esters form quite strong coordination bonds with alkali and alkaline earth metals such as Li^+, Mg^{2+}, Ca^{2+}, etc, giving excellent anstatic and antibacterial actions [24]. The ability of 2-butenedioic acid and its derivatives to serve as coordinating ligands to lanthanide and actinide ions is of great interest [25]. Uranyl complexes with carboxylates are usually with the free carboxylate terminus binding to uranyl. The most common binding mode is via monodentate and/or bidentate chelation at uranium through the carboxylate oxygens [26], which tend to form strong bonds with the uranyl cation.

As part of our continuing investigation into the preparation of lanthanide and actinide compounds with derivatives of 2-butenedioic acid, we have synthesized cerium, thorium, and uranyl complexes with (*E*)-4-(4-methoxyphenoxy)-4-oxobut-2-enoic acid. The complexes are characterized by MS, elemental analysis, IR, UV-Vis spectroscopic data, TG-DTA, and NMR. We have also studied the luminescent properties of the ligand and complexes.

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2. Experimental

2.1. *Materials*

Maleic anhydride (99%) and *p*-hydroxyanisole (99%) were used as received from Chemical Reagent, Shanghai. Other chemicals (such as ethyl acetate, hexane, ethanol, hydrochloric acid, and so on) were of analytical reagent grade, used without purification.

2.2. *Methods*

The MS were performed on a HP-5988A spectrometer (EI at 70 eV); conductivity measurements were carried out with a DDS-11A type conductivity bridge using $10^{-3}\,\mathrm{mol\,dm^{-3}}$ solutions in DMSO at 25°C; the content of Ce and Th were determined by EDTA titration using xylenol orange as an indicator; the content of U was calculated as U_3O_8 when the UO_2^{2+} complex was heated to 800°C; nitrogen, carbon, and hydrogen analyses were determined using a Vario EL elemental analyzer; IR spectra were recorded on a Nicolet Avatar 360 FT-IR instrument using KBr discs in the 400–4000 $\mathrm{cm^{-1}}$ region; UV-Vis spectra were measured on a Agilent 8453E UV-Vis spectrometer; thermal analysis (TG-DTA) was carried out on a PCT-2A thermal balance; NMR spectra were measured on a Varian Mercury-400BB NMR spectrometer; fluorescence measurements were made on a Hitachi F-4500 spectrophotometer equipped with quartz curettes of 1 cm path length at room temperature. The excitation and emission slit widths were 10.0 nm.

2.3. *Synthesis of the ligand*

A mixture of *p*-hydroxyanisole (3.72 g, 30 mmol) and sodium (1.2 g, 30 mmol) in toluene (100 mL) was heated to reflux for 4 h. The reaction mixture was cooled to 85°C and

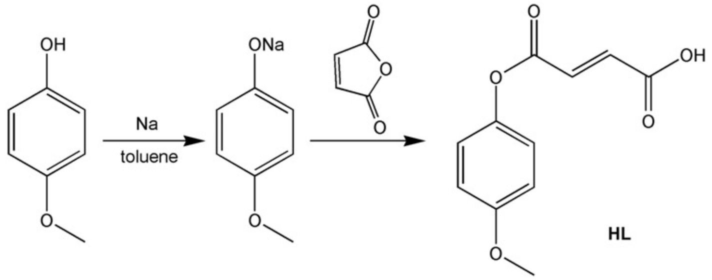

Figure 1. Scheme of the synthesis of the ligand.

maleic anhydride (2.94 g, 30 mmol) was added. After stirring at 85°C for 1 h, the reaction mixture was poured into ice-water, and the aqueous layer was collected. The aqueous layer was acidified with concentrated hydrochloric acid and extracted with ethyl acetate. The ethyl acetate layer was washed with water, dried ($MgSO_4$), and then treated with activated charcoal, after which the solvent was evaporated, to give (*E*)-4-(4-methoxyphenoxy)-4-oxobut-2-enoic acid as a white solid, which was then recrystallized from ethyl acetate–hexane to yield colorless needles. The structure of the ligand is as shown in figure 1. Yield: 82%; m.p.: 147.5149°C. ^{1}H-NMR (d-acetone, 400 MHz): 3.784 (S, 3H, OMe); 6.927–6.932 (d, 2H, CH); 6.945–7.136 (m, 4H, Ar–H). MS (EI, 70 ev): m/z (%) 222 (M^+, 8.99), 205 (0.35), 124 (100), 109 (27.41), 99 (19.74), 81 (10.50), 45 (15.25). The mass spectrum of the ligand (Supplementary material) shows its molecular ion at m/z 222, which coincides with the formula weight. All m/z peaks belong to the fragmentation of the ligand and the proposed fragmentation pattern of the ligand is provided in Supplementary material.

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2.4. *Synthesis of the complexes*

2.4.1. Ce(IV) and Th(IV) complexes. The HL (0.3072 g, 1.6 mmol) was dissolved in 15 mL ethanol and stirred at room temperature while an aqueous solution containing 0.4 mmol $(NH_4)_2Ce(NO_3)_6$ or $Th(NO_3)_4$ was added dropwise. The mixtures were kept stirring for 24 h, and a brown or white product was formed for the Ce(IV) or Th(IV) system, respectively. Th(IV) complex: ^{1}H-NMR (d-DMSO, 400 MHz): 3.716 (S, 3H, OMe); 6.626–6.891 (d, 2H, CH); 6.887–7.044 (m, 4H, Ar–H).

2.4.2. UO_2(II) complex. The HL (0.3072 g, 1.6 mmol) was dissolved in 15 mL ethanol and stirred at room temperature while an aqueous solution containing 0.2 mmol $UO_2(NO_3)_2$ was added dropwise, and the mixture solution was refluxed on an oil-bath for 10 h with stirring. After cooling to room temperature, the mixture was kept stirring for 24 h in air, and a yellow product formed as the UO_2(II) complex.

Solid precipitates were isolated from solution by centrifugation and then washed with distilled water three times, and finally washed with ethanol. Further drying in a vacuum afforded a powder, yield: 70 (thorium complex), 40 (cerium complex), and 55% (uranyl complex).

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3. Results and discussion

3.1. *Properties of the complexes*

The complexes are air stable for extended periods and soluble in DMF and DMSO. The molar conductance values of the complexes are in the range 12.1–19.5 $\mathrm{S\,cm^{-1}\,mol^{-1}}$ in DMSO (table 1). It is clear from the conductivity data that the complexes are non-electrolytes in DMSO [27, 28]. The elemental analyses and the content of metal ion show that the formulas of the complexes are $[ML_2(OH)_2 \cdot 2H_2O] \cdot H_2O$ (M = Ce, Th) and $[UO_2L_2 \cdot 2H_2O] \cdot H_2O$.

3.2. *IR spectra*

The IR spectra of the complexes are obviously different from the ligand, but resemble each other. The main stretching frequencies of the IR spectra of the ligand and its complexes are tabulated in table 2.

The ligand shows strong bands at 1743 and 1685 $\mathrm{cm^{-1}}$, which are assigned to ν(C=O) of ester carbonyl and carboxylic group, respectively. In the complexes, the band for ν(C=O) of ester carbonyl group is shifted by 0–4 $\mathrm{cm^{-1}}$ towards lower wavenumber; it is clear that the ester carbonyl does not take part in coordination. The peaks at *ca* 2838 and 1246 $\mathrm{cm^{-1}}$ are assigned to $\nu_{as}(CH_3)$ and ν(C–O–C) [29]; the two bands indicate Ar–O–CH_3 group in the ligand and complexes. Two strong absorption bands lying at 1405–1434 and 1531–1545 $\mathrm{cm^{-1}}$ are observed in complexes, which are attributed to the symmetric $\nu_s(COO^-)$ and asymmetric $\nu_{as}(COO^-)$ of carboxylate. Kakihana and co-workers showed that the nature of carboxylate coordination to a metal is conveniently obtained from a comparison of $\Delta\nu_{as\text{-}s}$ [30]. The determined $\Delta\nu_{as\text{-}s}$ for the three complexes show a bidentate chelate to the lanthanide or actinides [30–32].

Table 1. Elemental analyses and molar conductance data for the ligand and complexes.

Complexes	C (%)	H (%)	M (%)	Λ_m ($\mathrm{S\,cm^{-1}\,mol^{-1}}$)
HL	59.42 (59.46)*	4.511 (4.505)		
$[CeL_2(OH)_2 \cdot 2H_2O] \cdot H_2O$	39.88 (39.40)	3.659 (3.881)	21.23 (20.90)	12.1
$[ThL_2(OH)_2 \cdot 2H_2O] \cdot H_2O$	34.86 (34.65)	3.271 (3.412)	31.09 (30.45)	19.5
$[UO_2L_2 \cdot 2H_2O] \cdot H_2O$	35.81 (34.46)	2.749 (3.133)	31.59 (31.07)	14.8

*The values in parentheses are the theoretic values.
L: the HL (or NaL) which lost the H of the COOH.

Table 2. IR spectral data of the free ligand and its complexes ($\mathrm{cm^{-1}}$).

Complexes	$\nu_{O–H}$	$\nu_{C=O}$		ν_{sCH_3}	$\nu_{asC–O–C}$	$\nu_{sC–O–C}$	ν_{asCOO^-}	ν_{sCOO^-}	$\Delta\nu_{as\text{-}s}$	$\nu_{M–O}$
HL	3440	1743	1685	2838	1235	1143				
NaL	3448	1738		2836	1251	1144	1596	1400	196	450
$[CeL_2(OH)_2 \cdot 2H_2O] \cdot H_2O$	3395	1740		2836	1246	1141	1545	1405	140	457
$[ThL_2(OH)_2 \cdot 2H_2O] \cdot H_2O$	3467	1739		2838	1246	1141	1544	1409	135	459
$[UO_2L_2 \cdot 2H_2O] \cdot H_2O$	3256	1743		2839	1249	1145	1531	1434	97	458

The absorptions due to UO_2 are observed at 946 and 836 cm^{-1}, corresponding to asymmetric and symmetric stretching vibrations of U=O, respectively [31]. The weak bands at *ca* 457 cm^{-1} are assigned to v(M–O) [33].

Two absorptions at 830 and 575 cm^{-1} are assigned to in-plane and out-of-plane bending vibrations of coordinated water, respectively [34]. The broad continuous absorption (*ca* 3400 cm^{-1}) indicates crystal water is present [35, 36].

3.3. *UV spectra*

The UV absorption spectra of free ligand and its complexes in DMSO are listed in table 3. The free ligand and all its complexes exhibit absorptions in the ultraviolet region (200–400 nm). The ligand has an intense band at $\lambda_{max} = 328$ nm, attributed to the n–π^* transition, but in the complexes the band at 328 nm shifts to 276 (Ce and Th complexes) and 277 nm (UO_2^{2+} complex), attributed to the π–π^* transition. The uranyl absorption at 277 nm is blue shifted compared to UO_2^{2+}. These results suggest that conjugation of the ligand changes after coordination with the metal. Vulpius *et al.* showed that uranium(VI) forms weak carboxylate 1 : 1 complexes with monohydroxybenzoic acids including benzoic acid, in which the carboxylate is bidentate [37], as for the UO_2^{2+} complex in our result.

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3.4. *TG-DTA analysis*

Thermal behaviors of the ligand and Th(IV) complex are shown in Supplementary material. Samples of about 10 mg were placed in a crucible and heated to 800°C at a rate of 10°C min^{-1} in air, using α-Al_2O_3 as reference material.

The DTA curve of free ligand has an endothermic peak at 153°C, but there is no weight loss on the corresponding TG curve, showing that this is a phase transition, the melting point of the ligand (148°C). In the range 273–285°C, the free ligand has an obvious weight loss, exceeding 85%.

There are two endothermic peaks at 88–105 and 163–185°C for the Ce(IV) complex, 76–92 and 178–192°C for the Th(IV) complex, and 86–105 and 158–164°C for UO_2(II) complex. The corresponding TG curves show that the weight losses are equal to one and two water molecules, indicating the presence of one lattice water and two coordinated waters in accord with the elemental analyses and IR study. For the three complexes, two or three exothermic peaks appear from 330 to 550°C. The Th(IV) complex commences decomposition at 356°C, and the Ce(IV) complex at 332°C, suggesting that the Th(IV) complex is more stable than the Ce(IV) complex [38].

Table 3. UV-Vis data of the ligand and its complexes.

Compounds	λ_{max} (nm)	ε (dm^3 M^{-1} cm^{-1})	Assignment
HL	328	11,000	n–π^*
$UO_2(NO_3)_2 \cdot 6H_2O$	260	10,050	π–π^*
$[CeL_2(OH)_2 \cdot 2H_2O] \cdot H_2O$	276	10,700	π–π^*
$[ThL_2(OH)_2 \cdot 2H_2O] \cdot H_2O$	276	7900	π–π^*
$[UO_2L_2 \cdot 2H_2O] \cdot H_2O$	277	12,200	π–π^*

Table 4. Chemical shifts (ppm) in ^{13}C NMR (400 MHz) spectra of HL and $[ThL_2(OH)_2 \cdot 2H_2O] \cdot H_2O$.

	1	2	3	4	5	6, 10	7, 9	8	11
HL	165.93	135.99	133.45	164.40	144.86	123.10	115.17	158.48	55.84
$[ThL_2(OH)_2 \cdot 2H_2O] \cdot H_2O$	171.00	144.20	130.02	165.14	143.24	123.08	114.93	157.59	56.04
$\Delta\delta$*	−5.07	−8.21	3.43	−0.74	1.62	0.02	0.24	0.89	−0.18

*$\Delta\delta = \delta HL - \delta[ThL_2(OH)_2 \cdot 2H_2O] \cdot H_2O$.

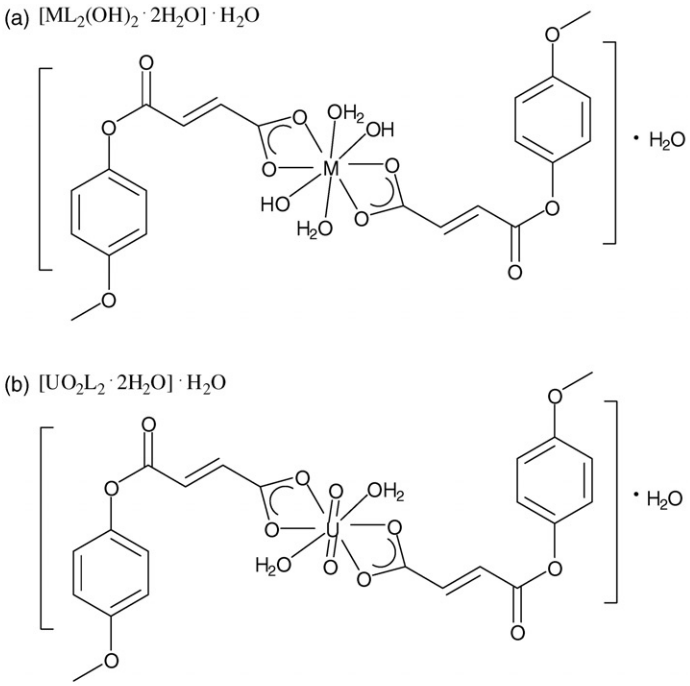

Figure 2. Suggested structure of the complexes.

Total weight losses for complexes of Ce, Th, and UO_2 were 76.46 (Calcd 74.33%), 66.84 (Calcd 65.35%), and 64.51% (Calcd 63.36%), respectively, suggesting that the final decomposition products for the three complexes are CeO_2, ThO_2, and U_3O_8 when the temperature is above 800°C.

3.5. ^{13}C-NMR

The ^{13}C-NMR of free ligand and Th(IV) complex were measured in d-acetone and d-DMSO at room temperature, respectively. The ^{13}C-NMR data are shown in table 4, and the ^{13}C-NMR spectra of the HL and the Th(IV) are provided in Supplementary material.

There are resonances at 165.93 and 164.40 ppm, which can be assigned to C1 and C4, respectively. C1 shifts about 5.07 ppm downfield in the Th(IV) complex, but the change

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Table 5. Fluorescence data of the complexes.

Compounds	λ_{ex} (nm)	λ_{em} (nm)	RLI*
HL	334	375.2	228
		414.8	168.3
		430.2	164.6
$[CeL_2(OH)_2 \cdot 2H_2O] \cdot H_2O$	388	442.8	751.7
$[ThL_2(OH)_2 \cdot 2H_2O] \cdot H_2O$	398	452.6	405.3
$[UO_2L_2 \cdot 2H_2O] \cdot H_2O$	379	499.8	830.6
		521.6	730.5
		545.4	355.7
		571.8	170.1
$UO_2(NO_3)_2 \cdot 6H_2O$	472	491.4	151.2
		511.4	391.6
		534.6	204.3
		559.8	62.42

*RLI: Relative Luminescence Intensity.

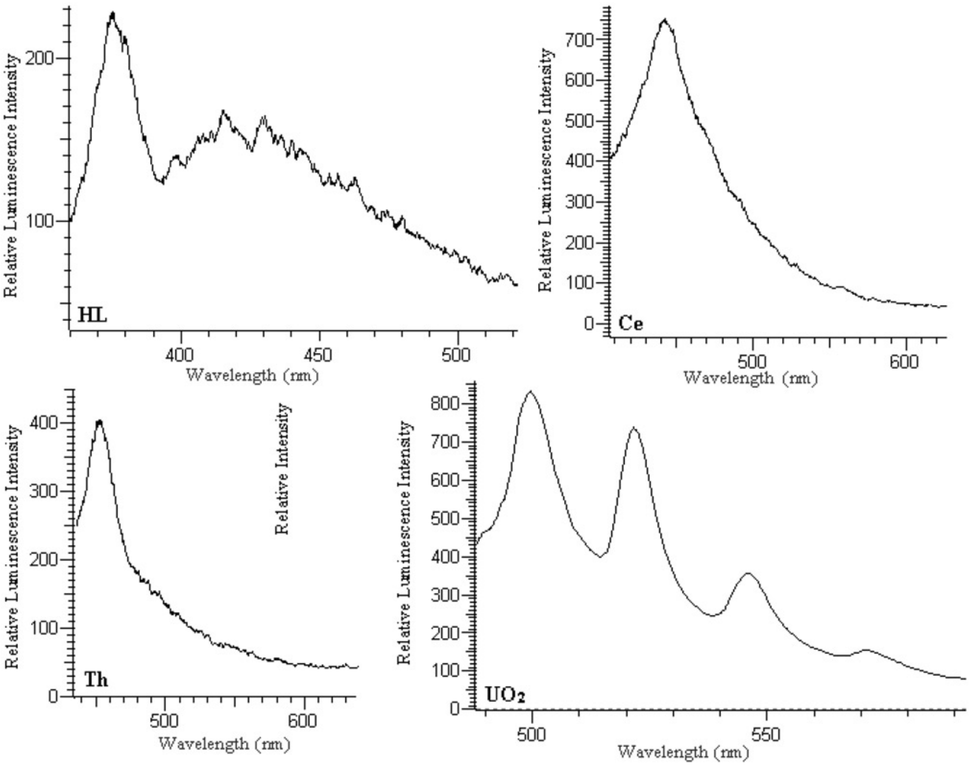

Figure 3. The emission spectra of the complexes.

of C4 is not significant, indicating that the oxygen attached to C1 takes part in coordination and the oxygen attached to C4 does not.

For the Th(IV) complex, the ^{13}C chemical shifts for C5–C11 are all found at rather normal positions, similar to those in ligand.

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On the basis of above evidence and analyses, the possible structure of the complexes is shown in figure 2.

3.6. *Luminescence studies*

The excitation and emission spectra, as well as the relative luminescence intensities, were measured in DMSO at room temperature at $10^{-4}\,\mathrm{mol\,L^{-1}}$. The major luminescence spectral data are summarized in table 5.

The luminescence analysis shows that the ligand exhibits three peaks, the strongest centered at 375 nm. The complexes exhibit strong characteristic luminescence of the metal ions. Complexes of Ce(IV) and Th(IV) show only one fluorescence band [Ce(IV) at $\lambda_{\max} = 442.8$ nm and Th(IV) at $\lambda_{\max} = 453.6$ nm]. Compared with the ligand, the complexes of Ce(IV) and Th(IV) turn deeply red, from ligand-to-metal charge transfer [39] and intraligand transitions [40], respectively. In comparison to the peak positions of uranyl free ions (491.4, 511.4, 534.6 and 559.8 nm), the emission spectra at room temperature of the UO_2^{2+} complex (499.8, 521.6, 545.4 and 571.8 nm) are shifted to longer wavelengths. The shifts vary from ~9 to 11 nm. Furthermore, the relative luminescence intensity increases for the UO_2^{2+} complexes. Figure 3 contains the emission spectra of the ligand and complexes. The emission spectra of uranyl compounds come from an excited state in which an electron has been transferred from a UO_2(II) molecular orbital to a 5*f* U atomic orbital [41, 42].

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4. Conclusion

According to the data, it is obvious that the ligand forms complexes with Ce(IV), Th(IV), and UO_2(II). Obvious changes in the IR spectra and NMR were observed when the Ce(IV), Th(IV), and UO_2(II) complexes formed, indicating that the metal ions were coordinated to oxygens of carboxylate; the ester carboxylic groups do not coordinate, as observed in complexes containing ester as ligands. The three complexes exhibit characteristic luminescence of the metal ions. Future work will include designing new 2-butenedioic acid derivatives to synthesize complexes.

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