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RESEARCH ARTICLE

Synthesis and characterization of rare earth metal complexes with novel Schiff base

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Abstract

Objectives. The work set out to synthesize Schiff base ligands containing a hydrazone moiety of (Z)-2-((E)-1-hydroxyethylidene)-hydrazineylidene)-2-phenylacetic acid, as well as their praseodymium, samarium, europium, and gadolinium complexes, and to study their structure.

Methods. The structure of ligands was identified by infrared (IR), ultraviolet (UV), and nuclear magnetic resonance (NMR) spectroscopy. The structure of the complexes was confirmed by elemental analysis, IR and UV spectroscopy, and thermogravimetric analysis.

Results. The Schiff base ligands containing a hydrazone moiety of (Z)-2-((E)-1-hydroxyethylidene)hydrazineylidene)-2-phenylacetic acid, as well as their praseodymium, samarium, europium, and gadolinium complexes, were synthesized using the authors' procedure.

Conclusions. NMR and IR spectroscopic data confirm that the Schiff base ligand is in the keto form. There are three absorption bands in the wavelength range of 205–306 nm in the UV spectrum of the ligand. A bathochromic shift is observed in the spectrum of all complexes. The molar ratio of ligand and metal in the complexes was 3 : 1.

Keywords

hydrazones, phenylglyoxylic acid, Schiff base, rare earth elements, complexing

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НАУЧНАЯ СТАТЬЯ

Синтез и характеристика комплексов металлов редкоземельных элементов на новом основании Шиффа

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Аннотация

Цели. Синтезировать лиганды основания Шиффа, содержащие гидразоновую часть (Z)-2-((E)-1-гидроксиэтилиден)гидразинилиден)-2-фенилуксусной кислоты, и их комплексы празеодима, самария, европия и гадолиния, а также исследовать их структуру.

Методы. Структура лигандов была идентифицирована с помощью инфракрасной (ИК) и ультрафиолетовой (УФ) спектроскопии и спектроскопии ядерного магнитного резонанса (ЯМР). Структура комплексов была подтверждена с помощью элементного анализа, ИК- и УФ-спектроскопии и термогравиметрического анализа.

Результаты. Лиганды основания Шиффа, содержащие гидразоновую часть (Z)-2-((E)-1-гидроксиэтилиден)гидразинилиден)-2-фенилуксусной кислоты, и их комплексы с празеодимом, самарием, европием и гадолинием были синтезированы по методике авторов.

Выводы. Данные ЯМР- и ИК-спектроскопии подтверждают, что лиганд на основе Шиффа находится в кето-форме. В УФ-спектре лиганда имеются три полосы поглощения в диапазоне длин волн 205–306 нм. В спектре всех комплексов наблюдается bathochromic сдвиг. Молярное соотношение лиганда и металла в комплексах составило 3 : 1.

Ключевые слова

гидразоны, фенилглиоксисовая кислота, основание Шиффа, редкоземельные элементы, комплексообразование

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INTRODUCTION

With the existence of an azomethine bond affecting biological activity [1–3], Schiff bases are a special type of ligands with a variety of donor atoms that exhibit remarkable ways of coordinating to transition metals [4–6]. The salen-type Schiff bases obtained by the condensation of salicylaldehyde (sal) and primary ethylenediamine (en) are thought to be workable ligands for coordination chemistry, due to the possibility of arranging their steric and electronic features by the primary amine and aldehyde. Due to the inclusion of donor centers for metal ions in their composition, which allow for the design of different geometries with other ligands, a significant number of metal complexes have been synthesized using salen-type Schiff bases [7]. Schiff bases, containing imine or azomethine groups,

and their metal complexes are used in encapsulation and separation processes, in industry, and in supramolecular chemistry as catalysts, corrosion inhibitors, pigments, and dyes [8–14]. Over the past few decades, numerous studies have been carried out on the use of this class of compounds, especially in medicine, due to the presence of diverse biological and pharmacological properties, including antimicrobial, anti-inflammatory, analgesic, antifungal, anti-tuberculosis, antiviral, anticancer, antiplatelet, antimalarial, anticonvulsant, cardioprotective, anthelmintic, antiprotozoal, antitrypanosomal, and antischistosomal [15–30].

Versatile applications of Schiff bases and their metal complexes stimulate interest in developing their synthesis methods. Hence the development of new effective, easy, and selective synthesis methods is still relevant for chemists. The aim of the work is to synthesize

Schiff base ligands, containing a hydrazone moiety of (Z)-2-((E)-1-hydroxyethylidene)hydrazineylidene)-2-phenylacetic acid, along with their praseodymium, samarium, europium, and gadolinium complexes.

EXPERIMENTAL

Materials and methods

All chemicals and solvents were purchased from commercial suppliers and were reagent grade. Salts were obtained from *Merck*, Germany, while solvents were procured from *Qualikems*, India. Phenyl glyoxylic acid and acetyl chloride purchased from *Sigma-Aldrich* (USA) were used without further purification.

Electronic absorption spectra were recorded on an Evolution 60S ultraviolet-visible (UV-VIS) spectrophotometer (*Thermo Fisher Scientific*, USA) in ethanol. Infrared (IR) spectra (KBr pellets) were recorded on a spectrophotometer Nicolet IS10 (*Thermo Fisher Scientific*, USA) in the range 500–4000 cm^{-1} .

^1H and ^{13}C nuclear magnetic resonance (NMR) spectra were recorded on a Bruker (300 MHz) spectrometer (*Bruker*, Germany) in CD_3O at room temperature (19–20°C). Thermogravimetric analysis was performed using a NETZSCH STA 449F3 derivatograph (*NETZSCH*, Germany). The elemental analyses were performed at the Tubitak analytical laboratory, in Ankara, Turkey, using a CHNS-932 analyzer (*Leco*, Germany).

Synthesis

of (Z)-2-(2-acetylhydrazineylidene)-2-phenylacetic acid (LH_2)

The starting hydrazide was synthesized according to the following procedure.

The mixture of 4 mL of triethylamine and 10 mL of anhydrous hydrazine was stirred with a magnetic stirrer. To this mixture, 14.4 mL of acetyl chloride was added with the rate of 1 drop in every 5 s. The reaction mixture was cooled with ice to avoid overheating. The resulting white slurry was separated and recrystallized from ethanol to give pure needle acetylhydrazine, having a melting temperature $T_{\text{m.p.}} = 70 \pm 3^\circ\text{C}$.

To obtain the target (Z)-2-(2-acetylhydrazineylidene)-2-phenylacetic acid (LH_2), phenylglyoxylic acid and

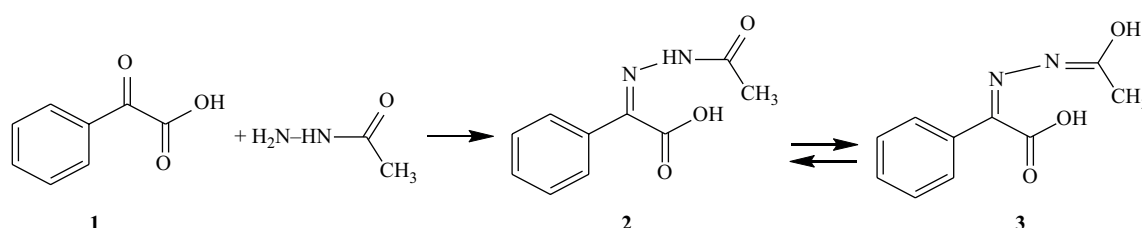
acetylhydrazine was taken in a molar ratio of 1 : 1. After dissolving 450 mg (5 mmol) of phenylglyoxylic acid in 5 mL of ethanol, 222 mg (5 mmol) of the acetylhydrazine solution in 5 mL water was added with stirring and heating to 70°C. From the resulting yellowish homogenous solution, bright yellow thin needle-like crystals precipitated within one day. The obtained crystals were filtered, recrystallized from ethanol, and dried over CaCl_2 in a desiccator. $T_{\text{m.p.}}$: $157 \pm 2^\circ\text{C}$. Yield was 525 mg (85%). Calculated for $\text{C}_{10}\text{H}_{10}\text{N}_2\text{O}_3$: C 58.25; H 4.85; N 13.59%. Found: C 58.35; H 4.98; N 12.95%. ^1H NMR (300 MHz, CD_3O): δ 7.91–7.48 (4d, t, 5H at benzene ring); 2.10 (s, 3H CH_3).

Preparation of metal complexes with LH_2

To obtain metal complexes with LH_2 , solutions of the corresponding metal salts in methanol and the ligand in ethanol were used. For this purpose, solutions of $\text{Pr}(\text{NO}_3)_3$, $\text{Sm}(\text{NO}_3)_3$, $\text{Eu}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, and $\text{Gd}(\text{ClO}_4)_3 \cdot 6\text{H}_2\text{O}$ salts in molar ratio of 1 : 3 to the ligand were used. Solutions of metal salts and the ligand were stirred upon mild heating. Following filtration, the obtained light yellow and yellowish-orange precipitates were washed with distilled water, and then with alcohol. The obtained complexes were dried over CaCl_2 in a desiccator until constant weight was reached. The melting points of praseodymium, samarium, europium, and gadolinium complexes were 200°C, over 260°C, 230°C and over 250°C. Yields were 172, 179, 197, and 173 mg, for 75, 72, 79, and 69%, respectively. Calculated for $\text{Me}(\text{C}_{10}\text{H}_9\text{N}_2\text{O}_3)_3$: C 47.65; H 3.59; N 11.11%; C 47.06; H 3.56; N 10.98%; C 46.96; H 3.55; N 10.95%; C 46.64; H 3.52; N 10.88%. Found: C 47.59; H 3.65; N 11.35%; C 46.95; H 3.65; N 11.05%; C 46.91; H 3.68; N 11.02%; C 46.72; H 3.59; N 11.01%, respectively, for praseodymium, samarium, europium, and gadolinium complexes.

RESULTS AND DISCUSSION

(Z)-2-(2-acetylhydrazineylidene)-2-phenylacetic acid is a potential tridentate dianionic ligand. Such a two-charged ligand can be realized at the expense of carboxylic and enolized acetyl groups (compound **3**) as presented in Scheme 1. However, NMR and IR spectroscopy data point to the presence of the keto form in ligand (compound **2**).



Scheme 1. Synthesis of the ligand and its tautomeric forms. **1.** Phenylglyoxylic acid. **2.** Keto form. **3.** Enol form

Electronic spectra

Electronic absorption spectra of compounds were recorded in ethanol in the UV region at room temperature (Fig. 1). There are three absorption bands in the wavelength range of 205–306 nm in the spectrum of the ligand.

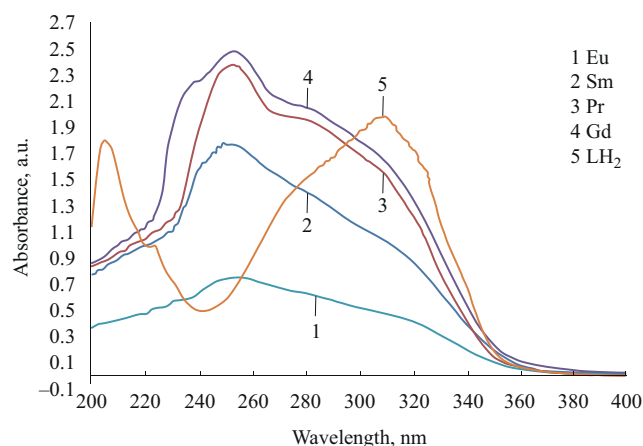


Fig. 1. UV-spectra of LH_2 and complexes

The band appearing at 306 nm and corresponding to low energy side is considered to be $n-\pi^*$ transition of conjugation between the lone pair of electrons of nitrogen atom in azomethine group and a conjugated π bond of benzene ring.

Other peaks appearing at higher energy sides of 205 and 223 nm are associated with $\pi-\pi^*$ and $n-\pi^*$ transitions of carbonyl moiety and aromatic ring [28, 31]. Complexation of ligand with metal ions results in a bathochromic shift in the spectra of all complexes. Thus, in the spectra of all complexes, a broad peak is observed in the wavelength range of 250–260 nm due to the coordination and the formation of chelate rings in the complexes [32].

IR spectroscopy

The IR spectra of ligand and its complexes are studied. It is known from the literature that the band of stretching vibrations of the non-ionized and non-coordinated

COO^- group lies in the range of $1750\text{--}1700\text{ cm}^{-1}$, while the ionized and coordinated COO^- group lies in the range of $1650\text{--}1590\text{ cm}^{-1}$ [33]. No ligand absorption band at 1745 cm^{-1} corresponding to the non-coordinated carboxyl group is observed in the spectra of complexes. On the contrary, peaks corresponding to the coordinated carboxyl group are observed in the spectra of all complexes at 1570, 1589, 1571 and 1507 cm^{-1} , for Pr, Sm, Eu, and Gd, respectively. These bands are observed because the hydroxyl group of the ligand is ionized to form a bond with the central ion.

As described in [33], bands of stretching vibrations of the $\text{C}=\text{N}$ group appear at $1590\text{--}1604\text{ cm}^{-1}$. Bands resulting from CN stretching vibration are observed in the spectra of complexes as shifted peaks (1603 , 1608 , 1604 , 1575 cm^{-1} , respectively, for Pr, Sm, Eu, and Gd complexes) comparatively with the ligand (1605 cm^{-1}) due to involvement of the iminic nitrogen atom in coordination [34]. Bands corresponding to the peptide bond ($1655\text{--}1630\text{ cm}^{-1}$) [35] are slightly shifted (1676 , 1669 , 1629 , and 1594 cm^{-1} , respectively, for Pr, Sm, Eu, and Gd) as compared with the bands in the spectrum of the ligand (1685 cm^{-1}) due to $\delta(\text{NH})+\nu(\text{CN})$ coupled vibrations. This indicates that oxygen atom in the amide group is involved in coordination. The spectra of both the ligand and the complexes have some medium peaks that may be attributed to stretching vibration of $\nu(\text{NH})$ $3000\text{--}3200\text{ cm}^{-1}$. It shows that NH group was not coordinated with metal. This band also proves that the coordination form of the ligand is the keto form. All bands characterizing the ligand and complexes are given in the Table.

NMR spectroscopic studies

The NMR spectrum of the obtained ligand is consistent with literature data. The ^1H NMR spectrum of the ligand exhibits some peaks in the range of $7.91\text{--}2.10\text{ ppm}$ (Fig. 2). Peaks in the range of $7.91\text{--}7.48\text{ ppm}$ are accepted as benzene ring protons. One s upfield peak observed at 2.10 ppm is suitable for representing the hydrogen atoms in the methyl group.

Table. Absorption bands of the ligand and its complexes

Substance	Non-coordinated carboxyl group	Coordinated carboxyl group	$\text{C}=\text{N}$ stretching vibration	$\delta(\text{NH})+\nu(\text{CN})$
Ligand	1745	–	1605	1685
Praseodymium	–	1570	1603	1676
Samarium	–	1589	1608	1669
Europium	–	1571	1604	1629
Gadolinium	–	1507	1575	1594

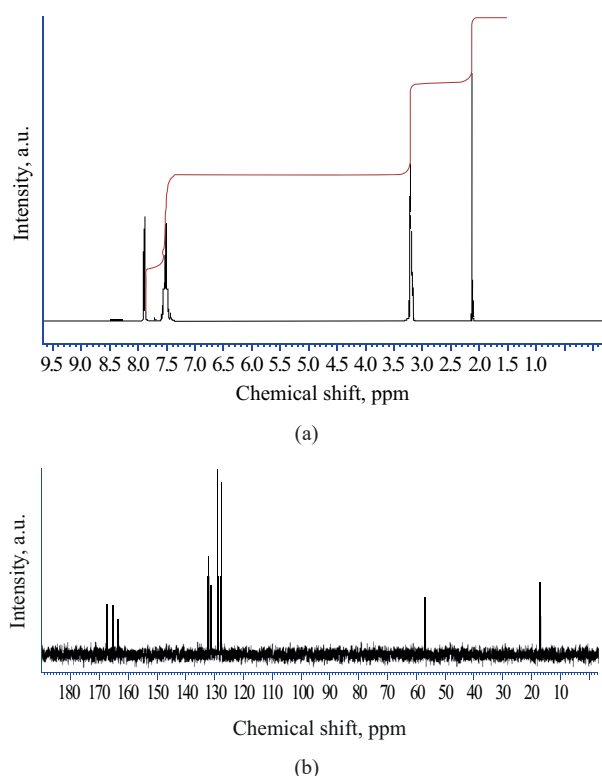


Fig. 2. (a) ^1H NMR spectrum of LH_2 in CD_4O ;
(b) ^{13}C NMR spectrum of LH_2 in CD_4O

Thermogravimetric analysis

The thermal decomposition of the obtained complexes was studied in the temperature range 25–995°C with a heating rate of 40.0°C/min, according to the data of

thermogravimetric analysis. Figures 3 and 4 depict thermogravimetry and differential thermal analysis curves for the samarium and europium complexes, respectively.

Thermogravimetric analysis data show that the decomposition proceeds in four stages. For the europium complex at the first stage (25–198°C), a weight loss of 6.861–23.652% occurs due to the removal of crystallization water. The second stage, which is observed with the weight loss of 16.381% in the temperature range of 198–322°C, occurs due to the elimination of carbonyl group and coordination bond between nitrogen and metal. At the next stage, decomposition starts at the temperature of 322°C, and lasts until 448°C; the corresponding weight loss (16.321%) is accompanied by breaking the bond of the carboxyl group to the metal. The last stage observed in the temperature range of 447–980°C, which involves a weight loss of 14.322%, corresponds to the complete destruction of the organic part. The remaining mass (~22.5%) corresponded to the europium content (19.8% theoreti.) in the sample.

In the case of samarium complex (Fig. 4), the first stage is accompanied by a weight loss of 6.467% at a temperature of 25–137°C. The second stage of decomposition occurs in a temperature range of 137–198°C with a weight loss of 28.656%, while the third stage is observed in the temperature range of 198–398°C. The significant weight loss observed during decomposition, which mainly occurs in stages 2 and 3, is due to the elimination of carbonyl group and coordination bond between nitrogen and metal. The last stage, which

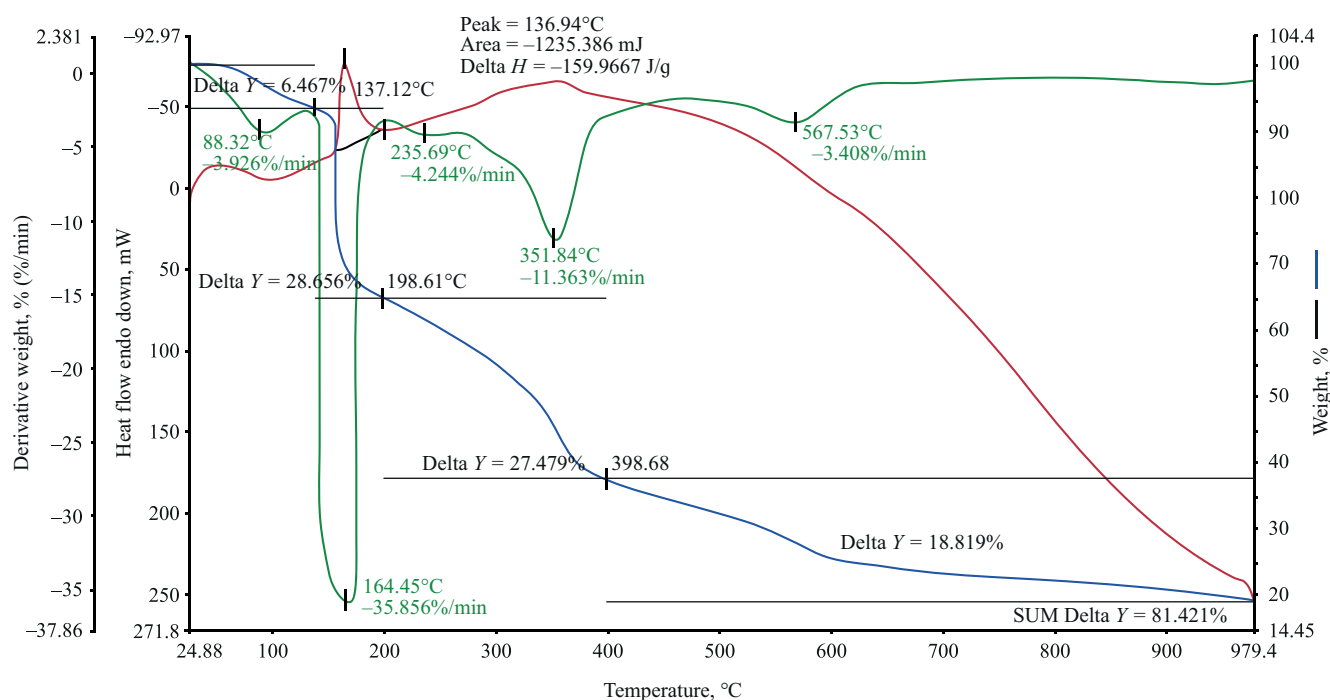


Fig. 3. Derivatogram of the complex of Sm with Schiff base

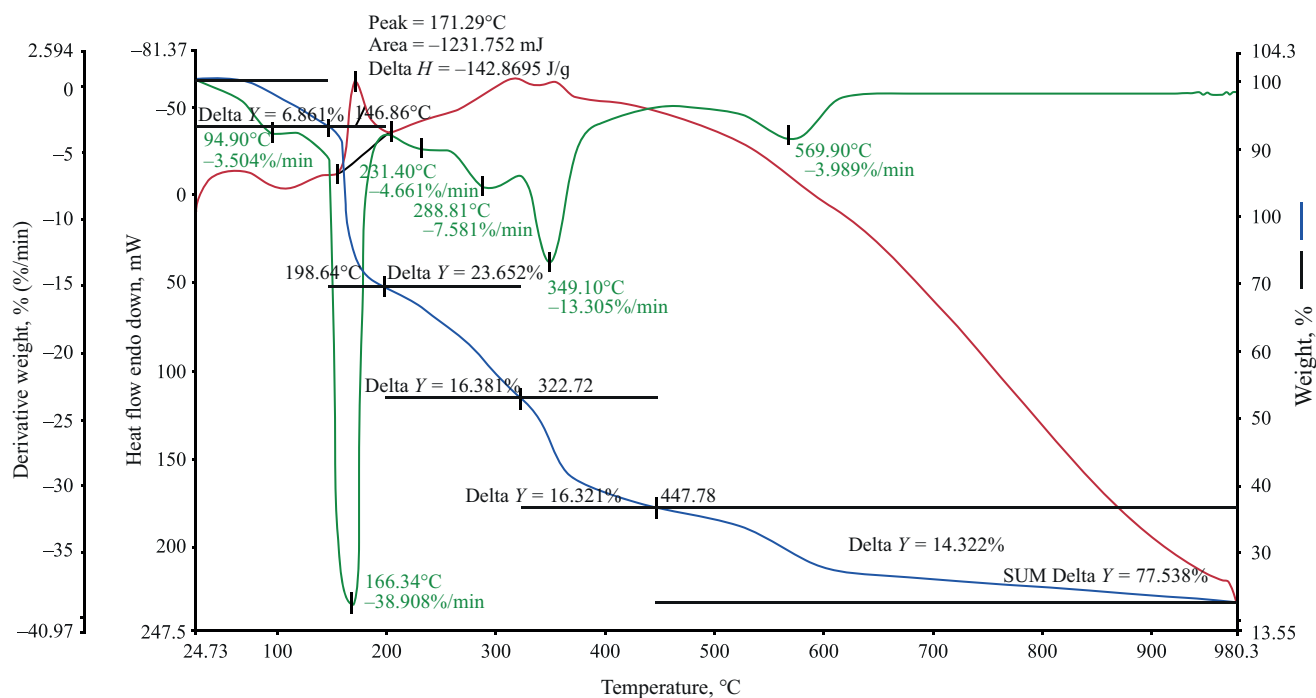
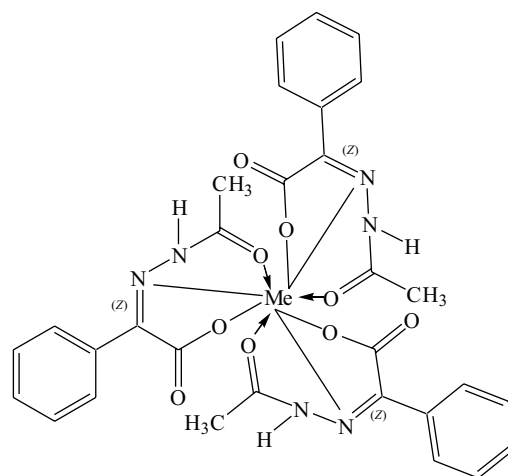


Fig. 4. Derivatogram of the complex of Eu with Schiff base

is observed in the temperature range of 398–980°C and involves a weight loss of 18.819%, corresponds to the fragmentation and complete destruction of the organic ligand. The remaining mass ~18.6% (19.6% theoreti.) following heating up to 980°C is predicted to be Sm_2O_3 . In all cases, metal percentages calculated from thermogravimetric curves are suitable for the results of elemental analyses.

Based on the results of physicochemical studies and the spectral data of the literature [28–35], the following proposed structure of the metal complexes is assumed (Scheme 2).



Scheme 2. Proposed structure of the metal complexes

Authors' contribution

All authors equally contributed to the research work.

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CONCLUSIONS

A new Schiff base ligand was synthesized together with its complexes with rare earth elements. The ligand and complexes were characterized by various physicochemical methods. Metal to ligand stoichiometry in the composition of complexes was found as 1 : 3.

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