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Research Article

Synthesis and Characterization of s-Triazine Cored Schiff Base Containing β-lactam and Its [M(Salen/Saloph)] (M= Cr^{3+} and Fe^{3+}) Capped Complexes

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ABSTRACT

This study consists of a tripodal 6-iminopenicylanic acid ligand synthesized based on the s-triazine group and its four trinuclear complexes. For this purpose, 2,4,6-tri(p-formylphenoxy)-1,3,5-triazine (III) (TRIPOD) was obtained from the reaction of 2,4,6-trichloro-1,3,5-triazine and 4-hydroxybenzaldehyde. Then, tripodal target ligand (V) was obtained from the reaction of TRIPOD and 6-aminopenicylanic acid. Finally, tripodal trinuclear (ML) capped target complexes were obtained from the reactions of the initial complexes $[(ML)_2O]$ (M= Cr^{3+} or Fe^{3+}, L= Salen or Saloph) with the target ligand in methanol media. The structures of all obtained ligands were elucidated using ^1H NMR, FT-IR, and elemental analysis methods. The structures of the complexes were characterized using FT-IR, magnetic susceptibility, ICP-OES, and TGA methods.

Araştırma Makalesi

β-laktam İçeren s-Triazin Merkezli Schiff Bazı ve Onun [M(Salen/Saloph)] (M= Cr^{3+} ve Fe^{3+}) Başlıklı Komplekslerinin Sentezi ve Karakterizasyonu

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ÖZ

Bu çalışma, s-triazin grubu ve onun dört trinükleer kompleksine dayalı olarak sentezlenen tripodal 6-iminopenisilanik asit ligandından oluşmaktadır. Bu amaçla 2,4,6-trikloro-1,3,5-triazin (III) (TRIPOD) ve 4-hidroksibenzaldehitin reaksiyonundan 2,4,6-tri(p-formilfenoksi)-1,3,5- triazin elde edildi. Daha sonra TRIPOD ve 6-aminopenisilanik asitin reaksiyonundan tripodal hedef ligandı (V) elde edildi. Son olarak, başlangıç komplekslerinin $[(ML)_2O]$ (M= Cr^{3+} veya Fe^{3+}, L= Salen veya Saloph) metanol ortamında hedef ligand ile reaksiyonlarından tripodal trinükleer (ML) başlıklı hedef kompleksleri elde edildi. Elde edilen tüm ligandların yapıları ^1H NMR, FT-IR ve elementel analiz yöntemleri kullanılarak aydınlatıldı. Komplekslerin yapıları FT-IR, manyetik duyarlılık, ICP-OES ve TGA yöntemleri kullanılarak karakterize edildi.

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1. Introduction

In the structures of heterocyclic compounds, s-triazine compounds are formed by the substitution of 3 nitrogen atoms in a symmetrical manner with carbon atoms. The simplest form of this substance in aromatic ring structure is 1,3,5-triazine. Due to their symmetric structure, 1,3,5-triazines were initially named sym-triazines, but nowadays they are simply referred to as s-triazines. The most used s-triazine is cyanuric chloride (2,4,6-trichloro-s-triazine), with a melting point in the range of 145-147 °C. This product, synthesized from cyanuric chloride and/or cyanuric acid, has a specific gravity of 1.32 g/cm^3 and a molecular weight of 184.41 g/mol. Ligands containing s-triazine remain stable even at temperatures above 150 °C. The discovery of the biological activity of triazine derivatives and their potential use in cancer treatments has given importance to these substances(De Hoog et al., 2002; Srinivas et al., 2005). They have many other applications such as bioinorganic chemistry, materials chemistry, coordination chemistry, and the plastic industry. In coordination chemistry, the ability of s-triazine derivatives to form complexes with transition metals is significant(Uysal and İsmet Uçan, 2009; Koç, 2011; Shiju et al., 2015). In the textile field, they are important due to their use as reactive dyeing agents in the production of wrinkle-resistant materials(Agathian et al., 2018; Wu et al., 2018). They undergo hydrolysis only when exposed to concentrated mineral acids thermally(Yuste et al., 2009).

The identification of 6-aminopenicillanic acid (6-APA) in 1957 and its subsequent adoption as the foundational constituent for the synthesis of all semi-synthetic penicillin represents a seminal advancement within the pharmaceutical domain. Characterized by its white hue and crystalline configuration, 6-APA assumes a pivotal role as the precursor in the manufacture of prevalent synthetic penicillin such as amoxicillin and ampicillin. This compound is derived through the enzymatic hydrolysis of Penicillin G, underscoring its foundational significance in the pharmaceutical production chain(Yabalak et al., 2017). The basic structure of penicillin contains a 6-APA. In other words, they are all derivatives of 6-APA. The chemical structure of 6-APA includes a thiazolidine ring, this ring is connected to a beta-lactam ring. It is attached to a (NH_2) group on the beta-lactam ring and to a carboxylic acid group on the thiazolidine ring. The synthesis of 6-APA started in the 1970s at the Gist-Brocades Company using harmful chemicals and solvents in a traditional synthesis method. The use of this method lasted for about 20 years and was replaced by the PA hydrolysis reaction. With this method, a high amount of 6-APA can be obtained from penicillin G(Hacıoğlu, 2014).

In the literature, there are many β-lactam substituted Schiff bases and some of their biological activity studies. However, there are no s-triazine cored β-lactam derivatives in the literature. As we mentioned above, 6-APA derivatives are known to have some biological activities. We hope that the ligands we obtained in this study will also show anticancer, antioxidant and antifungal activities.

In this study, we aim to synthesize and characterize s-triazine derivatives containing 6-APA and some of their [M(Salen/Saloph)] capped transition metal complexes. Therefore, the main purpose of this study is to produce thermally resistant original materials with paramagnetic properties.

2. Experimental Section

2.1. Materials and methods

All chemicals and solutions employed in this study were procured from Merck (Sigma-Aldrich) Company. ^{1}H-NMR analyses were performed on a Bruker Avance Core NMR spectrometer (400 MHz) device. Fourier-transform infrared (FTIR) spectrometry analyses were conducted with Thermo Scientific ID5 with ID7 ATR apparatus. Magnetic susceptibility measurements of the complexes were taken using the Gouy method with $Hg[Co(SCN)_4]$ as the calibrant, on the Sheerwood Scientific MX Gouy device. The effective magnetic moments, μ_{eff}, per metal atom were calculated from the expression: $\mu_{eff} = 2.84\sqrt{X_M}T$ B.M., where X_M is the molar susceptibility. The metal contents of each complex were determined on a Perkin Elmer Optima 7000 DV Model Inductively Coupled Plasma Optical Emission Spectrophotometer (ICP-OES). The mass spectra of the ligands were determined by ESI(+) method and a Thermo TSQ Quantum Access device was used. The melting points of the compounds were determined using a Thermo Scientific IA9000 device. Elemental analyses were performed using a LECO 932 CHNS instrument and the results were within ±0.4% of the theoretical values.

The following chemicals and solvents were purchased from the specified companies: cyanuric chloride (Merck), 4-hydroxybenzaldehyde (Sigma), o-phenylenediamine (Sigma), ethylenediamine (Sigma), salicylaldehyde (Sigma), 6-aminopenicillanic acid (Sigma), Chromium(III) chloride hexahydrate (Sigma), Iron(III) chloride (Sigma), N,N-diisopropylethylamine (DIPEA) (Sigma), solvents including ethanol (Merck), methanol (Sigma), and diethyl ether (Sigma). 2,4,6-tri(p-formylphenoxy)-1,3,5-triazine (TRIPOD), which are used in the formation reactions of cyanuric chloride and 4-hydroxybenzaldehyde, have been synthesized and utilized according to the literature(Tahmassebi and Sasaki, 1994). The starting complexes, which are used in the formation reactions of complexes and referred to as ligand complexes $[(ML)_2O]$ (M= Cr^{3+} or Fe^{3+}, L= Salen or Saloph), have been synthesized and utilized according to the literature(Kopel et al., 1998).

2.2. Synthesis of compound (V)

6-Aminopenisilanic acid (IV) (0.3244 g, 1.5 mmol) was dissolved in 50 mL of ethanol, and then 2,4,6-tri(p-formylphenoxy)-1,3,5-triazine (III) (0.22 g, 0.5 mmol) and 0.3 mL of DIPEA were added to the solution. The temperature was set to 85°C and stirred under a reflux for 90 minutes. After that, the solvent of the resulting mixture was evaporated under vacum. The solid residue was washed with diethyl ether to remove DIPEA. The product was recrystallized from ethanol. The structure of the product was elucidated using FT-IR, ^{1}H NMR, and mass spectrometry.

Compound (V): MA= 1036.12 g, m/z: 1035.30 (100%), 716.17 (44%), and 413.07 (28%); Color: Yellow solid; Yield: 0.3367 g (65%); Melting Point: 145-148°C; FT-IR (ATR, cm-1) δ (ppm): 2929 (C-H), 1603 (C=Nimine), 1557 (C=Ntriazine), 1297 (C-Ophen), 1684 (C=O), 1761 (C=Oacid), 1357 (COO-bending); ^{1}H NMR (400 MHz, DMSO-d_6): 1.20 (m,18H), 2.97 (d, 3H), 3.49 (s, 3H), 3.88 (d, 3H), 6.96 (d, 6H), 7.75 (d, 6H), 8.52 (s, 3H), 10.98 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): 182.38 (3C), 171.85 (3C), 170.58 (3C), 161.49 (3C), 155.53 (3C), 132.14 (3C), 130.83 (6C), 121.05 (6C), 79.50 (3C), 73.90 (3C), 68.50 (3C), 64.10 (3C), 29.80 (6C) Analyzed/Found: C: 55.64/55.61, H: 4.38/4.35, N: 12.17/12.14, O: 18.53/18.50, S: 9.28/9.25.

2.3. Synthesis of complexes (VI-IX)

Ligand (V) (1.0361 g, 1.00 mmol) is dissolved in 150 mL methanol, and [{(MSalen/Saloph)$_2$O}] ligand complexes (1.50 mmol) in 20 mL methanol are added to this solution. It is mixed under reflux at 80°C for 3 hours. At the end of the reaction, excess methanol is evaporated. The remaining solid is washed with diethylether, air dried.

Complex (VI): M_A = 1987.97 g, Dark green solid; μ_{eff}: 3.84 B.M.; Yield: 1.4313 g (72%); Melting Point: ≥200 °C (dec.); FTIR (ATR, cm^{-1}): 2970 (C-H), 1613 (C=N$_{im}$), 1595 (C=N$_{trz}$), 1305 (C-O$_{phen}$), 1684 (C=O), 1362 (COO$_{bend}$); Analyzed/Found: C: 57.96, H: 4.22, Cr: 7.81, N: 10.53, O: 14.45, S: 4.80.

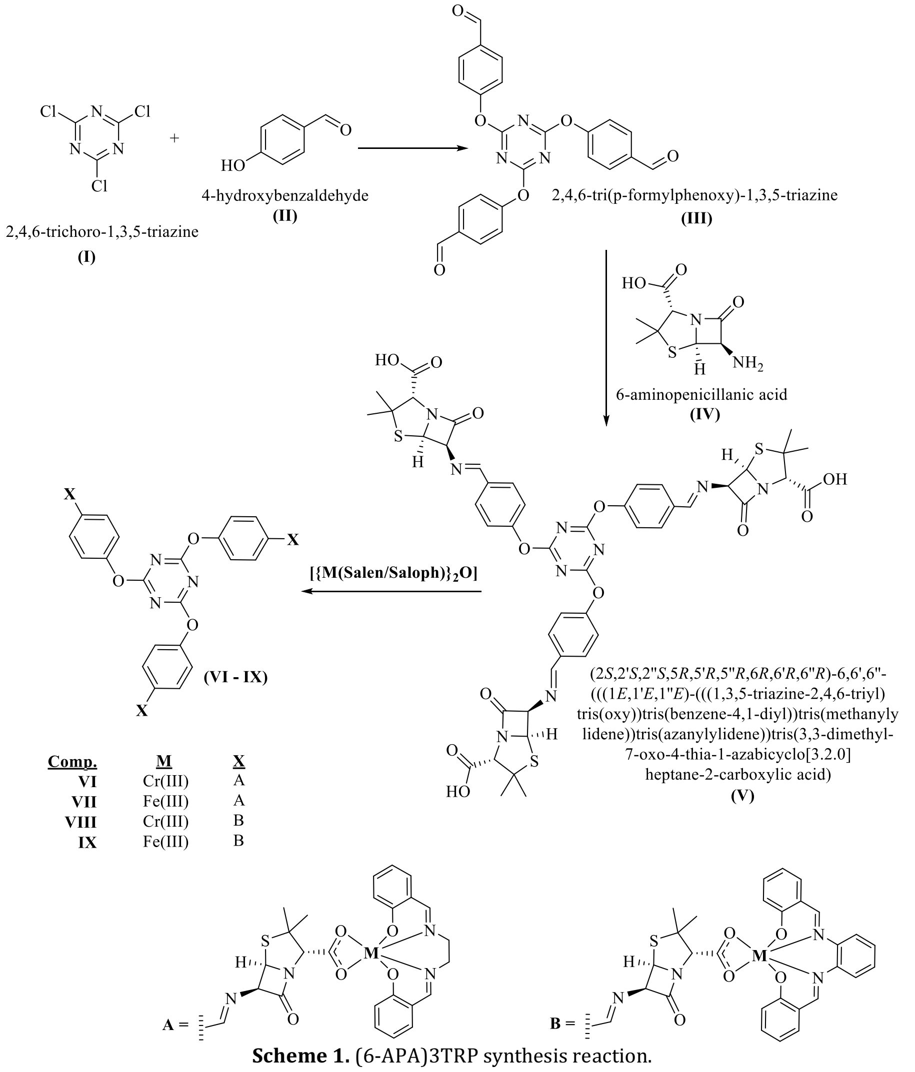
Complex (VII): M_A = 1999.51 g, Dark brown solid; μ_{eff}: 1.83 B.M.; Yield: 1.4796 g (74%); Melting Point: ≥200 °C (dec.); FTIR (ATR, cm^{-1}): 2898, 2979 (C-H), 1621 (C=N$_{im}$), 1596 (C=N$_{trz}$), 1297 (C-O$_{phen}$), 1679 (C=O), 1382 (COO$_{bend}$); Analyzed/Found: C: 57.63, H: 4.19, Fe: 8.34, N: 10.47, O: 14.36, S: 4.77.

Complex (VIII): M_A = 2132.10 g, Yellow solid; μ_{eff}: 3.87 B.M.; Yield: 1.4725 g (70%); Melting Point: ≥200 °C (dec.); FTIR (ATR, cm^{-1}): 2696 (C-H), 1603 (C=N$_{im}$), 1578 (C=N$_{trz}$), 1314 (C-O$_{phen}$), 1680 (C=O), 1364 (COO$_{bend}$); Analyzed/Found: C: 60.81, H: 3.94, Cr: 7.28, N: 9.82, O: 13.47, S: 4.48.

Complex (IX): M_A = 2143.64 g, Dark red solid; μ_{eff}: 1.84 B.M.; Yield: 1.6292 g (76%); Melting Point: ≥200 °C (dec.); FTIR (ATR, cm^{-1}): 2924, 3010, 3043 (C-H), 1603 (C=N$_{im}$), 1578 (C=N$_{trz}$), 1314 (C-O$_{phen}$), 1696 (C=O), 1376 (COO$_{bend}$); Analyzed/Found: C: 60.48, H: 3.92, Fe: 7.79, N: 9.80, O: 13.40, S: 4.46.

3. Results and Discussion

In this study, 2,4,6-trichloro-1,3,5-triazine (Cyanuric chloride) was used as the starting material. According to the literature, to obtain 2,4,6-tri(p-formylphenoxy)-1,3,5-triazine (TRIPOD), cyanuric chloride (CC) was reacted with 4-hydroxybenzaldehyde in a benzene environment(Tahmassebi and Sasaki, 1994). After this stage, TRIPPOD was used as the central compound. TRIPOD was reacted with 6-aminopenicillanic acid in the presence of diisopropylethylamine (DIPEA) in an ethyl alcohol media under reflux to obtain compound (V). Tripodal complexes (VI-IX) were obtained from the reaction of the tripodal ligand and [Cr/Fe(Salen/Saloph)]$_2$O initial complexes in an ethanol media (Scheme 1). The structures of the obtained ligands were elucidated by ^{1}H-NMR, FT-IR, elemental analysis, and mass spectroscopy methods. The structures of all obtained complexes were characterized by FTIR, magnetic susceptibility, elemental analysis, and ICP-OES methods.



Scheme 1. (6-APA)3TRP synthesis reaction.

3.1. Evaluation of ^{1}H and ^{13}C NMR spectra

When we examined the ^{1}H-NMR spectrum of the ligand (V) synthesized in our study, the ^{1}H NMR signal observed as a multiplet at 1.20 ppm corresponds to 18 protons, which are the methyl groups attached to the three thiazolidine rings (H_f). The observation of the protons H_e and H_d on the beta-lactam ring separately as doublets at 2.97 and 3.88 ppm, respectively, and the observation of three H_h protons in the acid group attached to the thiazolidine ring as a singlet at 10.98 ppm, the observation of three H_g protons as a singlet at 3.49 ppm, and most importantly, the observation of a singlet signal corresponding to three H_c protons at 8.52 ppm, and the absence of the singlet signal for TRIPOD at 10.00 ppm, as well as the observation of aromatic signals H_a and H_b as doublets at 6.96 ppm and 7.75 ppm, respectively, have shown that the targeted ligand has been successfully synthesized (Figure 1) (Yuste et al., 2009; Koç, 2011; Hacıoğlu, 2014; Yabalak et al., 2017; Agathian et al., 2018; İşçi and Uysal, 2018; Wu et al., 2018; Kiymaz and Uysal, 2023).

The ^{13}C NMR signals observed at 182.38 ppm (3C), 171.85 ppm (3C), 170.58 ppm (3C), 161.49 ppm (3C), 155.53 ppm (3C), 132.14 ppm (3C), 130.83 ppm (6C), 121.05 ppm (6C), 79.50 ppm (3C), 73.90 ppm (3C), 68.50 ppm (3C), 64.10 ppm (3C), and 29.80 ppm (6C) are also strong evidence that ligand V was successfully synthesized (Figure 2).

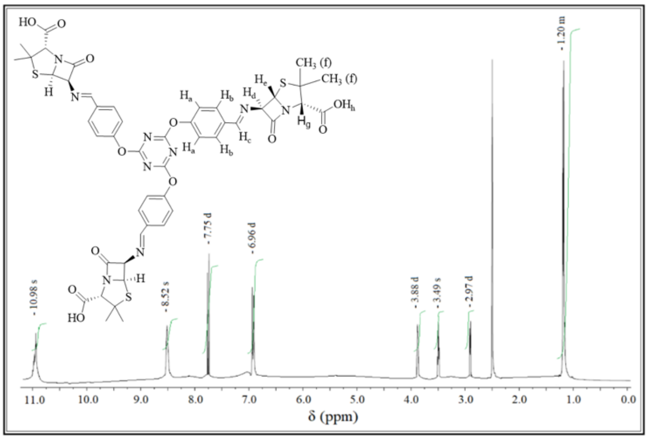


Figure 1. ^{1}H-NMR spectrum of compound (V).

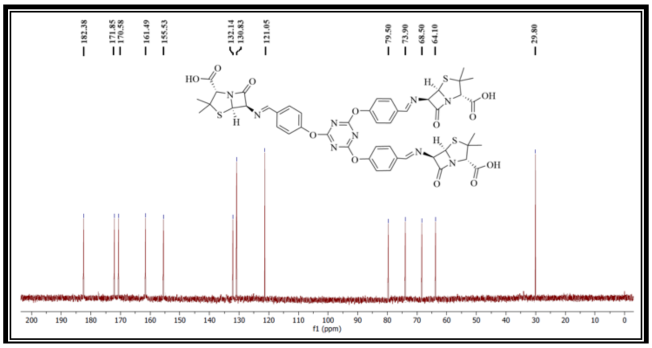


Figure 2. 13C-NMR spectrum of compound (V).

3.2. Evaluation of FT-IR spectra

In our study, when we examined the FT-IR spectrum of the ligand (V), the observation of the azomethine (-C=N-) band at 1604 cm^{-1} instead of the C=O band at 1695 cm^{-1} for TRIPOD (III), and the NH_2 bending band at 1620 cm^{-1} for 6-aminopenisilanic acid (III), along with the specific bands at 1684 cm^{-1} for the beta-lactam group and at 1761 cm^{-1} for the acid carbonyl of compound (V) demonstrate the successful synthesis of the target tripodal ligand (Figure 3)(Yuste et al., 2009; Koç, 2011; Hacıoğlu, 2014; Yabalak et al., 2017; Agathian et al., 2018; İşçi and Uysal, 2018; Wu et al., 2018; Kiymaz and Uysal, 2023).

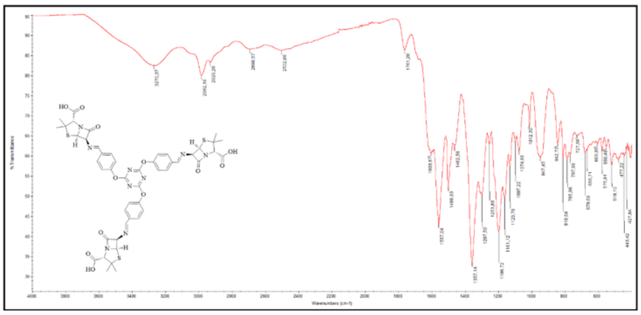


Figure 3. FT-IR spectrum of compound V.

[M(Salen/Saloph)] (M=Cr^{3+} and Fe^{3+}) capped tripodal trinuclear complexes were obtained from the reaction of the ligand and 1.5 equimolar amounts of [(MSalen/Saloph)$_2$O] in methanol medium. When we examined the FT-IR spectra of these tripodal homonuclear [M(Salen/Saloph)] capped complexes, the observed band at 1761 cm^{-1} for the carboxylic acid carbonyl group (C=O) shifted to the range of 1737-1741 cm^{-1}, demonstrating complexation between the caps and the ligand. FTIR spectra of all complexes were given in Figure 4 (Kopel et al., 1998; Uysal and İsmet Uçan, 2009; İşçi and Uysal, 2018; Kiymaz and Uysal, 2023).

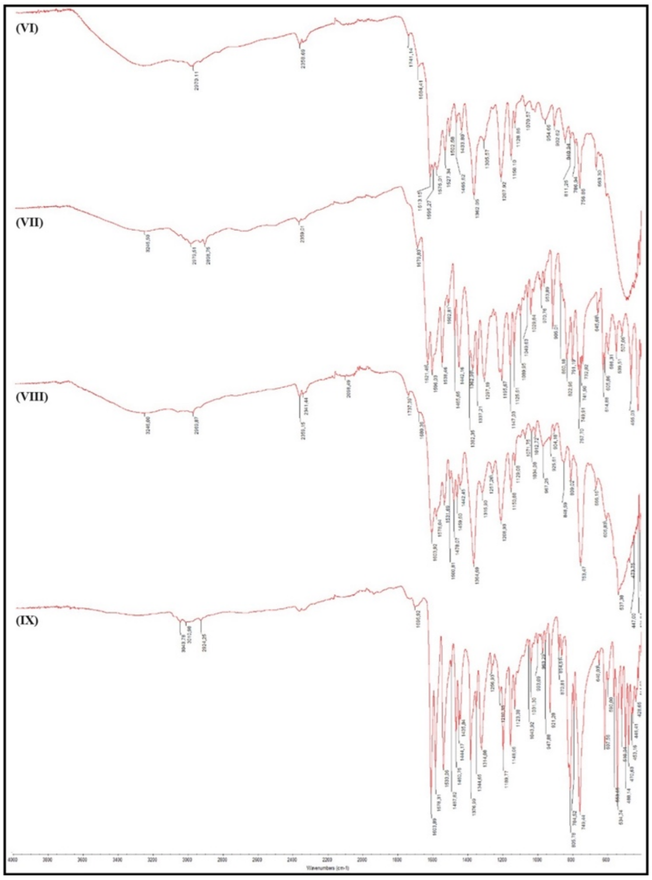


Figure 4. FTIR spectra of all complexes.

3.3. Evaluation of ESI-MS spectrum for ligand V

When the ESI-MS spectrum of ligand V is examined, the signal at m/z = 1035.30 (100%) is associated to the molecular ion peak. Since the device works with the electron spray ionization principle, the fragments formed as a result of the molecule decomposition are also associated to the signals at 716.17 (% 44), and 413.07 (% 28) in the spectrum. The predicted formulas of the corresponding molecules due to aforementioned signals are shown in Figure 5.

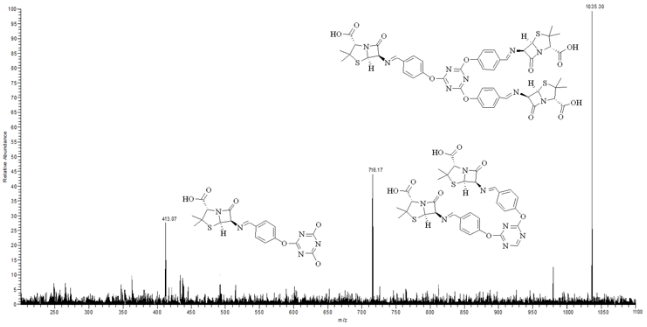


Figure 5. ESI-MS spectrum of compound V.

3.4. Evaluation of magnetic susceptibility results

In our study, magnetic susceptibility values of all synthesized tripodal trinuclear complexes were measured at room temperature using a Gouy balance to determine their molecular geometries. The effective magnetic moments, μ_{eff}, per metal atom were calculated from the expression: $\mu_{eff} = 2.84\sqrt{X_M}T$ B.M., where X_M is the molar susceptibility. The molar magnetic susceptibility (μ_{eff}) values of all complexes in terms of Bohr magneton (B.M.) are given below.

Complexes VI and VIII were measured paramagnetically, each having values of 3.84 and 3.87 B.M., respectively, with a $t_{2g}{}^3e_g{}^0$ electron configuration for each Cr(III) ion. Similarly, complexes VII and IX with d^5 electron configuration, were measured paramagnetically, each having values of 1.83 and 1.84 B.M., respectively, with a $t_{2g}{}^5e_g{}^0$ electron configuration for each Fe(III) ion. According to our literature research, $[M(Salen/Saloph)_2O]$ complexes are high spin complexes with a $t_{2g}{}^3e_g{}^2$ electron configuration, and it is known that another ligand can coordinate with them. Therefore, they are referred to as ligand complexes. When another monodentate ligand coordinates with these ligand complexes, their electronic configurations remain the same, but when a bidentate ligand coordinates with both teeth, it transitions to a low-spin electronic configuration. In other words, when other ligands coordinated to the centers of these ligand complexes force the metal ion into an octahedral geometry, low spin-capped complexes are formed. According to the results, [M(Salen/Saloph)] ($M=Cr^{3+}$ and Fe^{3+}) capped tripodal homonuclear complexes were evaluated as having octahedral geometry since they are low-spin complexes(Uysal and İsmet Uçan, 2009; Uysal and Koc, 2010; İşçi and Uysal, 2018; Uysal and Koç, 2018; Mahamoud Aouled and Uysal, 2021; Aouled and Uysal, 2023; Kiymaz and Uysal, 2023)

3.5. Assessment of TGA results of some complexes

In our study, one of the complexes we obtained (Compound VII), thermogravimetric analysis (TGA) was performed under an N_2 atmosphere with a heating rate of 10°C/min in the range of 50-800°C to determine the thermal behaviours (Figure 6). From the thermogravimetric analysis graph of the compound (VII), it is observed that the decomposition occurs in three steps. In the first step, with a 5.00% weight loss, the CO_2 group is detached from the molecule and the FeSalen caps are separated from the main body(Uysal and İsmet Uçan, 2009; Uysal and Koc, 2010; İşçi and Uysal, 2018; Uysal and Koç, 2018; Mahamoud Aouled and Uysal, 2021; Aouled and Uysal, 2023; Kiymaz and Uysal, 2023). The theoretical weight loss at this step is 6.60%. In the second step, with a 42.00% weight loss, the organic structure attached to the center is separated from the medium. The theoretical weight loss at this step is 41.16%. In the final step, a 34.00% weight loss indicates the decomposition of the salen group bound to iron. The theoretical weight loss at this step is 35.15%. Since the heating process was terminated at 800°C, further weight losses could not be observed. Upon FTIR and ICP-OES analysis of the remaining substances from the TGA analysis, it was determined that the remaining substances after decomposition were cyanide chloride center and Fe_2O_3. This experimental data are in good harmony with the theoretical data.

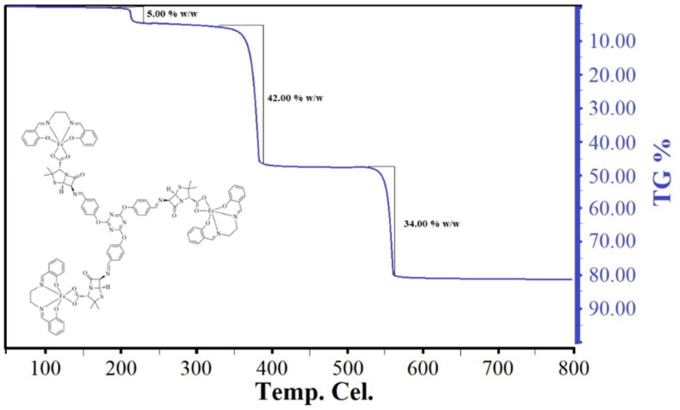


Figure 6. TGA curve of the complex (VII).

4. Conclusion

In this work, a novel s-triazine based tripodal 6-iminopenicylanic acid ligand (V) was adeptly synthesized and meticulously characterized through the application of elemental analysis, ^1H NMR, FTIR spectroscopy, and thermogravimetric analysis. [M(Salen/Saloph)] capped trinuclear complexes complexes (VI-IX) of this ligand were synthesized in ethanol media. Their structures were characterized using FTIR spectroscopy, metal concentration determination by ICP-OES spectroscopy, magneto chemical analysis, and thermogravimetric analysis. From the TGA result, it was understood that the resulting complexes showed high thermal stability. All Cr^{+3} complexes were found to be paramagnetic with three unpaired electron, and each Cr^{3+} ion was characterized as an inner orbital complex with a $t_{2g}{}^3e_g{}^0$ electron configuration. Similarly, the Fe^{3+} complexes were also paramagnetic with a low-spin 1 unpaired electron, and each Fe^{3+} ion formed an inner orbital complex with a $t_{2g}{}^5e_g{}^0$ electron configuration.

CRediT author statement

AC: Experimental study and structural characterization.
AHM: Experimental study and structural characterization. Writing–reviewing,
SU: Reviewing & editing, resources, supervision, project administration.

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