

# THERMAL STUDY OF SOME NOVEL BENZOTRIAZOLE IONIC LIQUIDS AND THEIR COMPLEX SALTS WITH SOME TRANSITION METALS

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**Abstract:** Investigations were carried out to evaluate the thermal stability of a series of benzotriazole-based ionic liquids synthesized via sequential alkylation of benzotriazole with methyl iodide, followed by ethyl iodide to yield the organic salt [EtMeBT]I (S1), or with allyl iodide to form [AMeBT]I (S2). These ionic liquids were subsequently reacted with Lewis acids, specifically transition metal chlorides, leading to the formation of complex salts in which the metal centers are coordinated in a tetrahedral environment. Nuclear magnetic resonance (NMR), mass spectrometry, elemental analysis, electron spectroscopy, infrared spectroscopy, atomic absorption spectroscopy, molar conductivity, and other methods were used to characterize the produced compounds. The produced organic salts can be used in industrial settings at temperatures up to 150°C, according to thermal studies.

**Keywords:** ionic liquids, complex salts, thermal stability.

## Introduction

Molten salts made of ions, ionic liquids (IL), have a melting point lower than 100°C [1, 2]. They consist of both organic and inorganic negative ions, as well as organic positive ions. Coulomb forces, also called electrostatic forces, hold these ions together. The selection of positive and negative ions greatly influences their physical characteristics. Depending on the ions required for the preparation process, we can adjust the melting point, density, viscosity, solubility, and water affinity of both positive and negative ions [3, 4].

Because of their distinctive properties, such as non-flammability, thermal and chemical stability, and non-volatility, ionic liquids derived from benzotriazole nitrogen bases have achieved a strong scientific standing in both the past and present literature [5]. Additionally, these ionic liquids remain liquid over a wide temperature range, typically above 200°C, due to their high thermal stability. Along with these benefits, they can also be recovered and reactivated through post-use recycling and purification, which lowers production costs [6].

Ionic liquids based on benzotriazole have a wide range of applications, especially in industry, including batteries, lubricants, desulfurization, photocells, and others [7]. Benzenesulfonic acid and 2-naphthalenesulfonic acid (NSA) are anions, while benzotriazole (BTA) and benzimidazole are cations. These ILs were applied to brass specimens in a nitric acid solution as green corrosion inhibitors. FT-IR, <sup>13</sup>C-NMR, and <sup>1</sup>H-NMR spectroscopy were used to characterize the structure of the protic ILs. The weight-loss method, in conjunction with SEM and EDS spectroscopy, was used to examine the effects of IL structure, IL concentration, acid concentration, and corrosion time on the surface morphology of brass specimens, as well as the inhibition efficiency of ILs [8]. To produce reduced-sulfur fuel with more environmentally friendly properties, Benzotriazolium-based ionic salts containing alkyl groups (methyl and allyl) and an iodine anion have been developed. The compound's structure has been determined by single-crystal X-ray diffraction, and it is used to desulfurize model diesel oil. Several methods were used to characterize the produced compounds, and their effectiveness in removing sulfur from the extracted oil was investigated. The findings indicated that although the removal efficiency was satisfactory, it could be further improved in future research [9].

When choosing ionic liquids for some specific industrial application, it is important to understand their thermal stability. The choice of ionic liquid depends on thermal stability, which is

essential for studying thermodynamic parameters and determining an appropriate application. Thus, this investigation aimed to test the thermal stability of various benzotriazole ionic liquids and to examine their thermodynamic parameters [9-11].

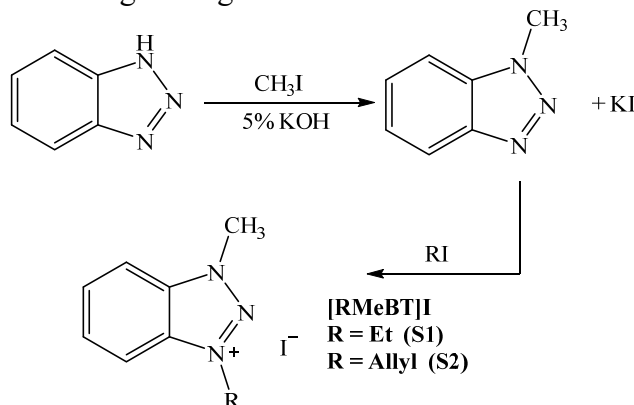
The novelty of this study lies in using different alkyl halides than those used in previous studies to determine the effect of varying the alkyl group on the physical properties of the resulting compound and, consequently, its suitability for different applications.

## Experimental part

**Synthesis of Benzotriazolium Iodide Ionic Liquids.** *3-ethyl-1-methylbenzotriazolium iodide* ( $C_9H_{12}N_3I$ ) [EtMeBT]I S1

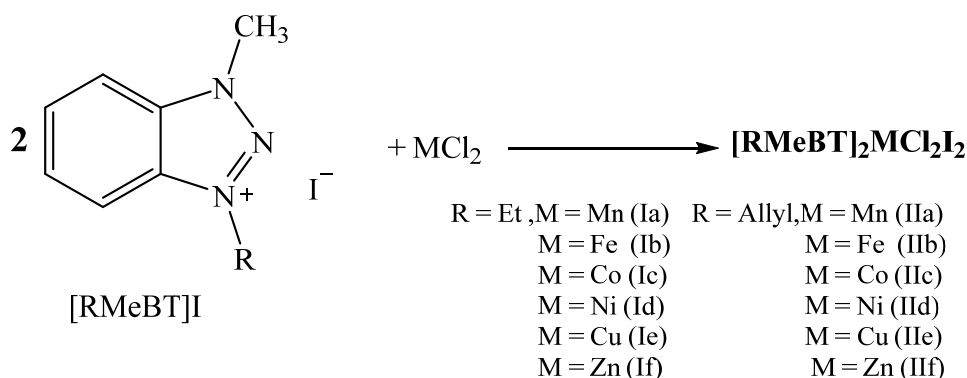
*3-allyl-1-methylbenzotriazolium* ( $C_{10}H_{12}N_3I$ ) [AMeBT]I S2. The literature-standard techniques were used to synthesize the first benzotriazole ionic liquid [12, 13]. Benzotriazole and methyl iodide were reacted in the presence of a 5% potassium hydroxide solution to carry out the N-alkylation reaction. 10 mL of ethanol was used to dissolve 0.01 mol (1.19 g) of benzotriazole in a 50 mL round-bottom flask. The solution was then supplemented with 0.01 mol (0.62 mL) of methyl iodide. The reaction mixture was then supplemented with a 5% KOH solution (5 mL). For one hour, the mixture was refluxed.

An addition reaction was performed at the nitrogen atom (N3) in the second step, which was used to prepare the ionic liquid. This step involved a 1:1 molar reaction between 1-methylbenzotriazole and ethyl iodide for S1 (or allyl iodide for S2). An excess of the alkyl halide was used to speed up the reaction and increase the yield of the product. By repeatedly washing the finished product with n-hexane until all unreacted reagent was removed, the excess reagent was eliminated. Using the same reaction conditions, allyl iodide was substituted for ethyl iodide to create the second ionic liquid. The process of making the organic benzotriazole salts is shown in Scheme 1.



**Scheme 1.** Preparation of organic salts S1 and S2

**Synthesis of complex salts of Benzotriazolium iodide ionic liquids.** In the second process of production, one mole of Lewis acid (the chlorides of several transition metals) is reacted with two moles of benzotriazole ionic liquids (S1, S2) to create complex salts. Suitable physical and spectroscopic techniques determine the elements in complex salts. Scheme 2 shows the preparation of complex salts:



**Scheme 2.** Preparation of complex salts 1-12

**Physical and spectroscopic techniques for identifying prepared compounds.** The prepared benzotriazole ionic liquids were characterized by mass spectrometry, elemental analysis, proton and carbon<sup>13</sup> nuclear magnetic resonance (NMR), and other spectroscopic measurements. To determine the metallic element content of the complex salts, these included atomic absorption measurements, electrical conductivity, magnetic susceptibility, and analysis of electronic and infrared spectra. Table 1 presents characteristic data for the prepared compounds.

**Table 1.** Some of the characteristics of the compounds prepared

No.	Compound	Cond.	m.p °C	Color	M %
S1	[EtMeBT]I	42	290	white	....
S2	[AMeBT]I	40	185	hony brown	....
Ia	[EtMeBT] <sub>2</sub> MnCl <sub>2</sub> I <sub>2</sub>	68	172*	bright orange	54.0
Ib	[EtMeBT] <sub>2</sub> FeCl <sub>2</sub> I <sub>2</sub>	72	200*	brown	56.11
Ic	[EtMeBT] <sub>2</sub> CoCl <sub>2</sub> I <sub>2</sub>	75	180*	dark brown	57.26
Id	[EtMeBT] <sub>2</sub> NiCl <sub>2</sub> I <sub>2</sub>	80	270*	dark brown	57.07
Ie	[EtMeBT] <sub>2</sub> CuCl <sub>2</sub> I <sub>2</sub>	78	261*	light brown	61.58
If	[EtMeBT] <sub>2</sub> ZnCl <sub>2</sub> I <sub>2</sub>	83	300*	offwhite	63.71
IIa	[AMeBT] <sub>2</sub> MnCl <sub>2</sub> I <sub>2</sub>	78	180*	bright brown	53.55
IIb	[AMeBT] <sub>2</sub> FeCl <sub>2</sub> I <sub>2</sub>	77	300*	black	53.75
IIc	[AMeBT] <sub>2</sub> CoCl <sub>2</sub> I <sub>2</sub>	66	137-140	dark purple	57.29
IId	[AMeBT] <sub>2</sub> NiCl <sub>2</sub> I <sub>2</sub>	70	85*	dark green	57.89
IIe	[AMeBT] <sub>2</sub> CuCl <sub>2</sub> I <sub>2</sub>	80	266*	black	62.16
IIf	[AMeBT] <sub>2</sub> ZnCl <sub>2</sub> I <sub>2</sub>	75	242*	yellow	65.0

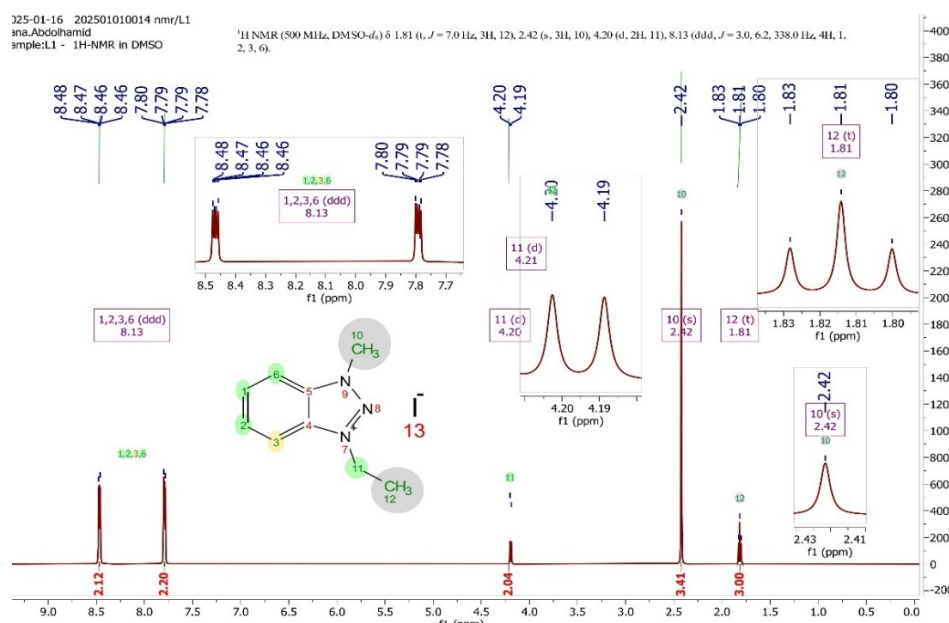
\*=decomposition, Cond. AM(DMF)

## Results and Discussion

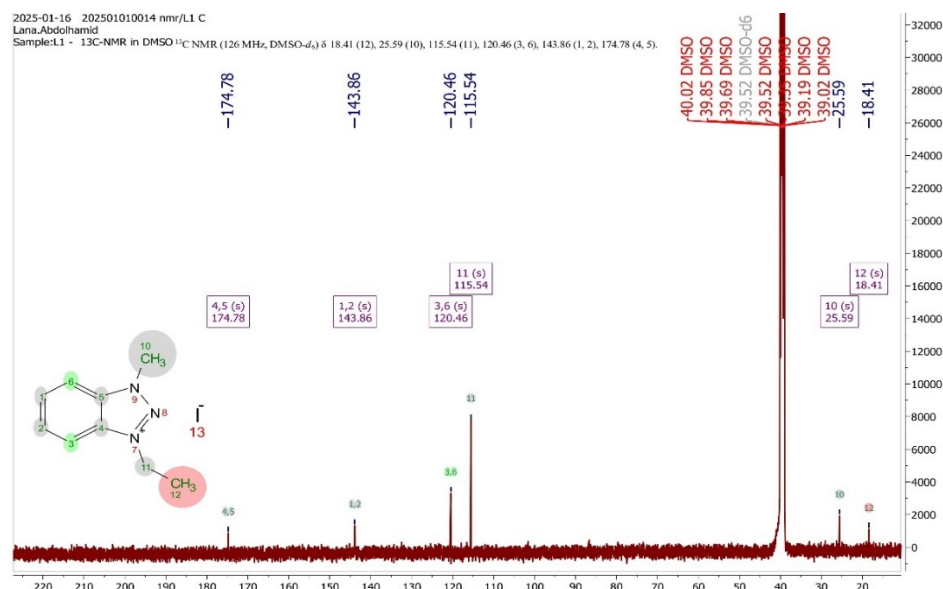
Numerous physical and spectral techniques were employed; a Bruker Avance DPX 500 MHz spectrometer was used for <sup>1</sup>H-NMR spectroscopy with DMSO-d<sup>6</sup> as the solvent. Mass spectrometry was performed using Trio-1000 equipment, and elemental analysis was performed using Thermo Electron Corporation's Flash EA 1112 Series. The SensAA GCB scientific equipment system measures FT-IR using the Shimadzu instrument and Avanta 2.02 software for atomic absorption. With a ramping heat rate of 10 °C/min in air, the METTLER Toledo TGA\DSC is used for thermal analysis at temperatures between 35 and 600 °C, using STARe evaluation software 19.00.

**Characterization data of organic salts.** [EtMeBT]I S1: <sup>1</sup>H-NMR (500 MHz, DMSO) δ 1.81 (t, J = 7.0 Hz, 3H, 12) belongs to CH<sub>3</sub> in the ethyl group, 2.42 (s, 3H, 10) refers to the methyl group, 4.20 (d, 2H, 11) for CH<sub>2</sub> in the ethyl group, and 8.13 (ddd, J = 3.0, 6.2, 338.0 Hz, 4H, 1, 2, 3, 6) is assigned to protons of the aromatic benzene ring. Fig. 1 shows the signal details and the atoms numbered. <sup>13</sup>C-NMR (126 MHz, DMSO): δ 18.41 (12) for methyl in ethyl group, 25.59 (10) for

methyl group, 115.54 (11) belongs to CH<sub>2</sub> in ethyl group; the last signals for aromatic carbon 120.46 (3, 6), 143.86 (1, 2), 174.78 (4, 5). Fig. 2 shows this signal's details and numbered atoms. m/z (Intensity %): 167 (12%), 149 (50%), 133 (15%), 105 (17%), 85 (5%), 69 (100%), 53 (20%). Elemental analysis: C, 37.39 (38.24); H, 4.18 (4.05); N, 14.53 (14.66). IR cm<sup>-1</sup>: 393, 511, 752, 987, 1201, 1404, 1610, 3599.



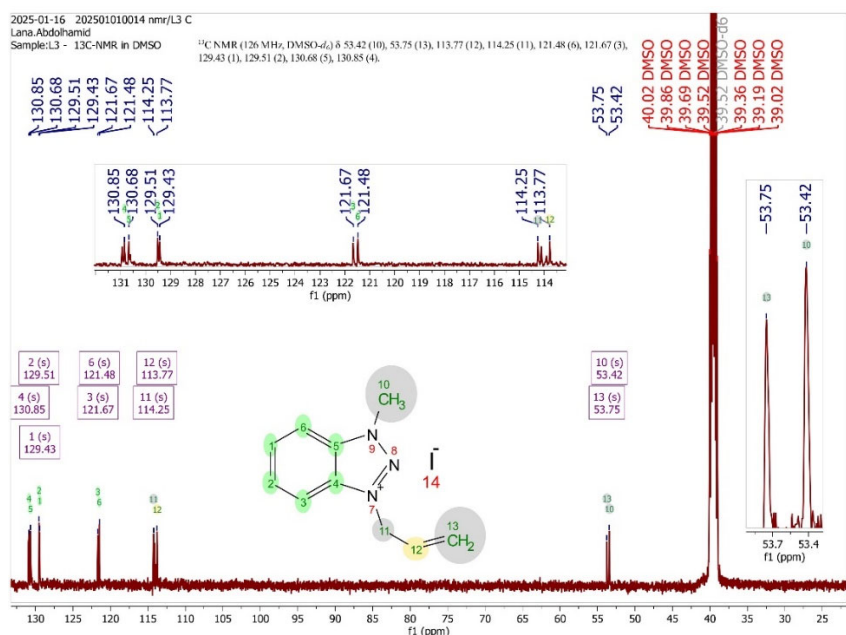
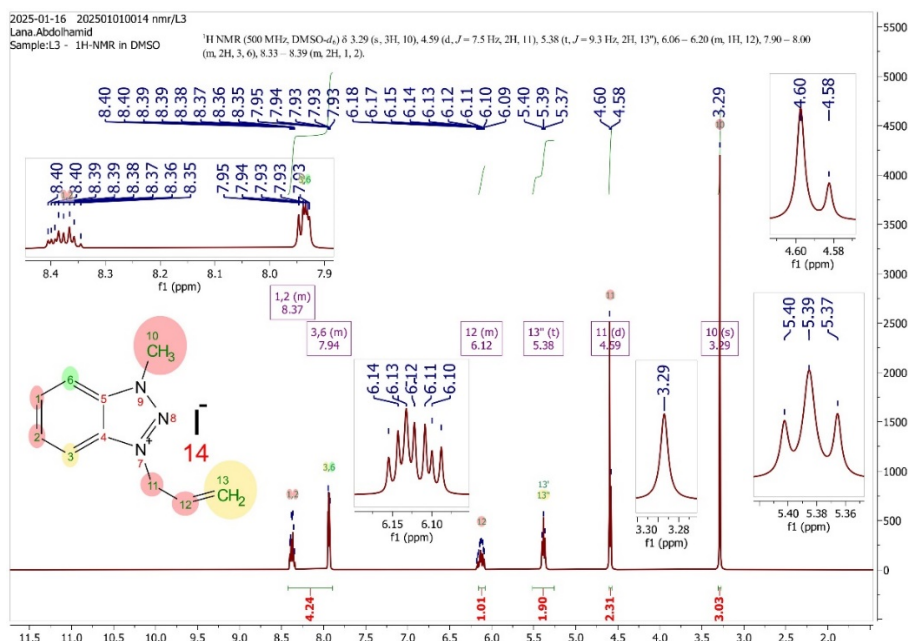
**Fig. 1.** The <sup>1</sup>H-NMR spectrum of [EtMeBT]I S1

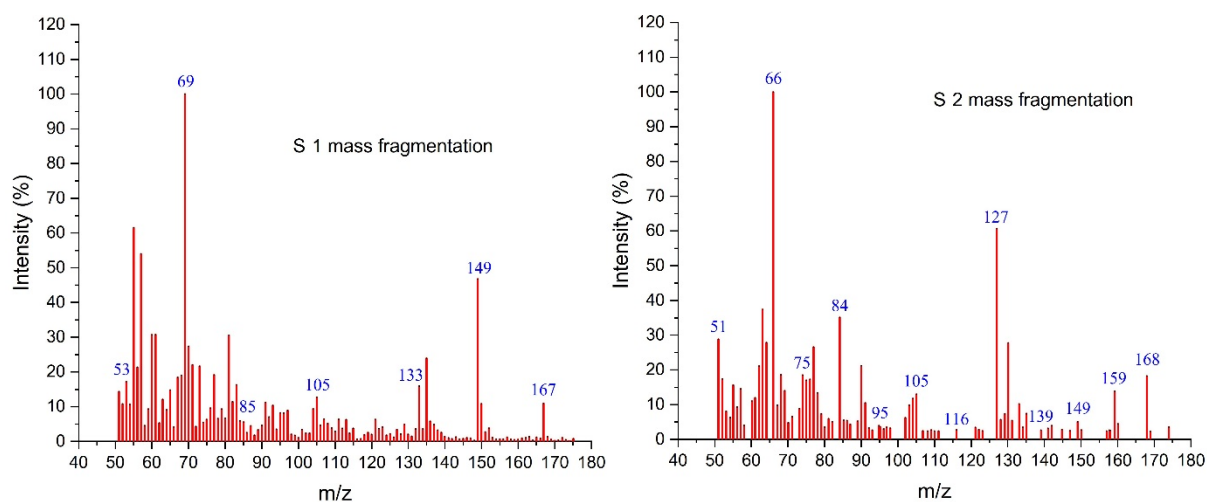


**Fig. 2.** The <sup>13</sup>C-NMR spectrum of [EtMeBT]I S1

[AMeBT]I S2: <sup>1</sup>H-NMR (500 MHz, DMSO): δ 3.29 (s, 3H, 10) refers to the CH<sub>3</sub> group; 4.59 (d, J = 7.5 Hz, 2H, 11) for the attached CH<sub>2</sub> of the allyl group; 5.38 (t, J = 9.3 Hz, 2H, 13) belongs to the terminal CH<sub>2</sub> of the allyl group; 6.06 – 6.20 (m, 1H, 12) sings to the CH of the allyl group. Aromatic protons appear in 7.90 – 8.00 (m, 2H, 3, 6) and 8.33 – 8.39 (m, 2H, 1, 2). Fig. 3 shows these signal details and numbered atoms. <sup>13</sup>C-NMR (126 MHz, DMSO) δ 53.42 (10) refers to the methyl group; 53.75 (13) belongs to the terminal CH<sub>2</sub> of the allyl group; 113.77 (12) is a single to the CH of the allyl group; 114.25 (11) belongs to the attached CH<sub>2</sub> of the allyl group; other signals are for the carbon of the aromatic ring: 121.48 (6), 121.67 (3), 129.43 (1), 129.51 (2), 130.68 (5), and 130.85 (4). Fig. 4 shows these signal details and numbered atoms. m/z (Intensity %): 168 (20%), 159 (18%),

127 (60%), 105 (20%), 84 (40%), 66 (100%), 51 (30%). Elemental Analysis: C, 39.89 (40.84); H, 4.02 (3.87); N, 13.95 (13.56). IR cm<sup>-1</sup>: 383, 451, 754, 987, 1267, 1400, 1610, 3593.





**Fig. 5.** The mass spectra of S1 and S2

Additionally, all complex salts were identified using the proper spectroscopic and physical techniques. To examine the geometrical shapes of complex salts, the magnetic susceptibility was measured at 25 °C. Using DMSO as a solvent at 10<sup>-3</sup> M, the results demonstrated the tetrahedral shape of the complex salts and used conductivity to confirm the ionic state of the prepared salts at the same temperature. The atomic absorption method was used to measure the percentage of metal in the complex salts, and the results were in good agreement with theoretical values and comparable to those of other studies [10, 14-17]. Table 2 provides information on magnetic susceptibility, spectral data, and other data.

**Table 2.** Some of the characteristics of the compounds prepared

Comp.	Electronic spectra bands cm <sup>-1</sup>	Suggested Translation	$\mu_{\text{eff}}$
S1	21142, 35336, 38760	G.T	---
S2	21739, 26882, 35211	G.T	---
Ia, IIa	9497, 10000, 11223, 11561, 13774, 41667	<sup>6</sup> A <sub>1</sub> → <sup>4</sup> T <sub>1</sub> G.T	5.80, 5.75
Ib, IIb	9488, 9990, 11223, 11574, 13141, 13774	<sup>5</sup> E → <sup>5</sup> T <sub>2</sub> G.T	4.88, 4.90
Ic, IIc	9488, 9980, 11211, 11561, 13774, 15015, 40486	<sup>4</sup> A <sub>2</sub> → <sup>4</sup> T <sub>2</sub> <sup>4</sup> A <sub>2</sub> → <sup>4</sup> T <sub>1</sub> <sup>4</sup> A <sub>2</sub> → <sup>4</sup> T <sub>1</sub> G.T	3.54, 3.24
Id, IId	9488, 9990, 11211, 11574, 13774, 40323	<sup>3</sup> T <sub>1</sub> → <sup>3</sup> T <sub>2</sub> <sup>3</sup> T <sub>1</sub> → <sup>3</sup> A <sub>2</sub> <sup>3</sup> T <sub>1</sub> → <sup>3</sup> T <sub>1</sub>	3.22, 3.26
Ie, IIe	9488, 9990, 11211, 11561, 13774, 40323	<sup>2</sup> T <sub>2</sub> → <sup>2</sup> E G.T	1.92, 1.88
If, IIIf	9488, 9990, 11211, 11561, 13774, 40486, 43860	<sup>6</sup> A <sub>1</sub> → <sup>4</sup> T <sub>1</sub> G.T	Dia

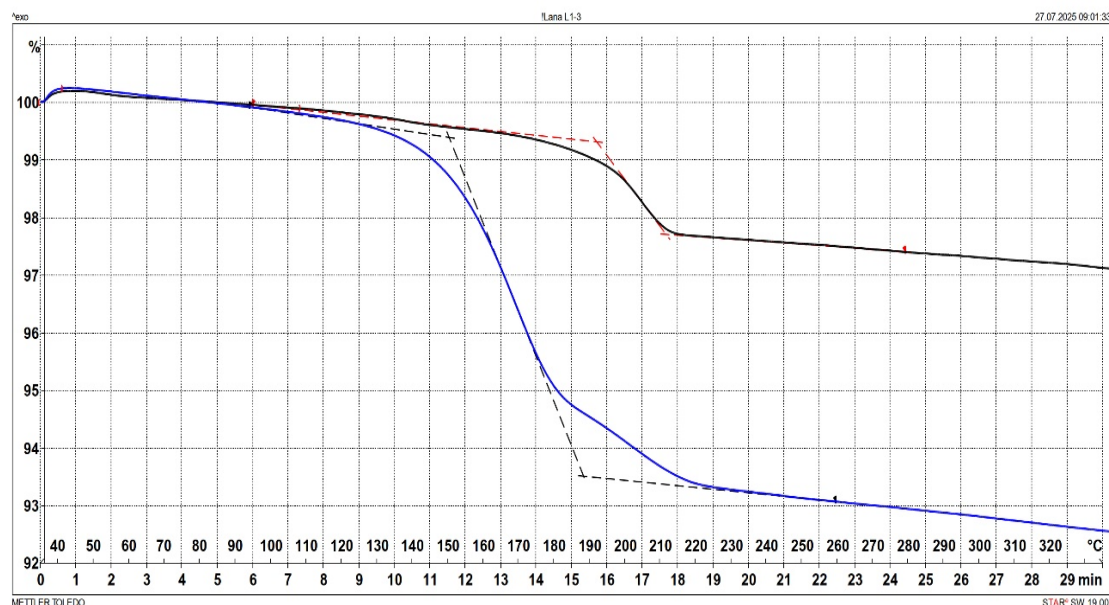
The tetrahedron is the expected geometric shape for the complex compounds, as seen in the electronic spectral bands of the complex salts and in the magnetic susceptibility values that support them, from which the effective magnetic moment values are determined. Because the values of the electronic transition wavelengths and magnetic moment are consistent with those found in the literature [18, 19], the results of previous studies that determine the structure of complex salts similar to those prepared are in close agreement with these results. Furthermore, earlier research accurately

identified the tetrahedral structure formed by the bonding of mixed halogens to the central ion in the complex salt using single-crystal X-ray diffraction [20, 21].

The KBr disk of the 400–4000  $\text{cm}^{-1}$  region was used for the infrared measurements, whereas the CsI disk of the 200–400  $\text{cm}^{-1}$  region was used. The  $\nu(\text{C}=\text{C})$  group was found to occur in the region of (1595-1652  $\text{cm}^{-1}$ ) in the stretch frequencies of the aromatic ring of organic salts and their complex salts [22]. The  $\nu(\text{C}-\text{H})$  bands were found in the (3520-3605  $\text{cm}^{-1}$ ) region, whereas the  $\nu(\text{C}=\text{N})$  and  $\nu(\text{C}-\text{N})$  bands were found in the (1400-1448  $\text{cm}^{-1}$ ) and (985-1016  $\text{cm}^{-1}$ ) regions, respectively. The metal-halide bond  $\nu(\text{M}-\text{X})$  ( $\text{X} = \text{Cl}, \text{I}$ ) was observed at (358-516  $\text{cm}^{-1}$ ), and the new active group  $\nu(\text{N}^+-\text{R})$  was observed at (2400-2420  $\text{cm}^{-1}$ ). Both of these values are consistent with the literature [23-25]. FT-IR data are listed in Table 3.

**Table 3.** FT-IR data of some functional groups in prepared compounds ( $\text{cm}^{-1}$ )

No.	Compound	$\nu(\text{C}=\text{C})$	$\nu(\text{C}-\text{H})$	$\nu(\text{C}=\text{N})$	$\nu(\text{C}-\text{N})$	$\nu(\text{M}-\text{X})$
S1	[EtMeBT]I	1610	3599	1404	987	---
S2	[AMeBT]I	1610	3593	1400	987	---
Ia	[EtMeBT] <sub>2</sub> MnCl <sub>2</sub> I <sub>2</sub>	1622	3570	1406	987	385,426
Ib	[EtMeBT] <sub>2</sub> FeCl <sub>2</sub> I <sub>2</sub>	1614	3571	1425	989	362,449
Ic	[EtMeBT] <sub>2</sub> CoCl <sub>2</sub> I <sub>2</sub>	1647	3605	1406	999	378,445
Id	[EtMeBT] <sub>2</sub> NiCl <sub>2</sub> I <sub>2</sub>	1604	3593	1423	985	360
Ie	[EtMeBT] <sub>2</sub> CuCl <sub>2</sub> I <sub>2</sub>	1614	3520	1402	989	372,435
If	[EtMeBT] <sub>2</sub> ZnCl <sub>2</sub> I <sub>2</sub>	1618	3573	1440	993	358,441
IIa	[AMeBT] <sub>2</sub> MnCl <sub>2</sub> I <sub>2</sub>	1612	3554	1442	991	389,476
IIb	[AMeBT] <sub>2</sub> FeCl <sub>2</sub> I <sub>2</sub>	1631	3589	1444	1001	358,516
IIc	[AMeBT] <sub>2</sub> CoCl <sub>2</sub> I <sub>2</sub>	1600	3540	1442	1000	395,445
IId	[AMeBT] <sub>2</sub> NiCl <sub>2</sub> I <sub>2</sub>	1622	3520	1448	1016	360,433
IIe	[AMeBT] <sub>2</sub> CuCl <sub>2</sub> I <sub>2</sub>	1595	3572	1402	995	395,445
IIf	[AMeBT] <sub>2</sub> ZnCl <sub>2</sub> I <sub>2</sub>	1652	3520	1409	1006	360,433



**Fig. 6.** Thermal graphic TGA of S1 and S2

**Thermal Analysis.** The thermal stability of the produced ionic liquids was confirmed. The ionic liquid's thermal curve and thermal differential curve are displayed in Fig. 6. The ionic liquids [EtMeBT]I (S1) and [AMeBT]I (S2) were found to be thermally stable up to 150°C and to degrade in a single main phase, suggesting the presence of a single substance. The thermal analysis scheme for ionic liquids mixed with Lewis acids, on the other hand, shows that the mixture consists of two

components (ionic liquid and metal chloride) because dissociation occurs in two main stages [26, 27]. Table 4 displays the mass-loss percentages and the corresponding heat ranges.

TGA data of [EtMeBT]I (S1): Onset 193.72 °C, Endset 212.24 °C, Inflect. Pt. 205.41 °C, Inflect. Slp.  $-8.27 \times 10^{-3} \text{ \% min}^{-1}$ , Left Limit 100.54 °C, Right Limit 277.98 °C, Heating Rate 10.00 °C  $\text{min}^{-1}$ , n-th order Kinetics:  $\ln(k_0)$  1.72 +/- 0.65, EA 33.52 +/- 2.30 kJ/mol, n -1.22 +/- 85.79e-03.

TGA data of [AMeBT]I (S2): Onset 153.68 °C, Endset 189.80 °C, Inflect. Pt. 171.69 °C, Inflect. Slp.  $-15.44 \times 10^{-3} \text{ \% min}^{-1}$ , Left Limit 101.15 °C, Right Limit 258.56 °C, Heating Rate 10.00 °C  $\text{min}^{-1}$ , n-th order Kinetics:  $\ln(k_0)$  -0.13 +/- 0.64, EA 26.86 +/- 2.27 kJ/mol, n -1.45 +/- 87.84e-03.

**Table 4.** Thermal degradation steps for organic salts S1 and S2

No.	Compounds	1 <sup>st</sup> degradation Dehydration		Mass loss%	2 <sup>nd</sup> degradation Dicompsition		Mass loss%
		Onset °C	Endset °C		Onset °C	Endset °C	
1	[EtMeBT]I (S1)	80	110	2	193	212	3
2	[AMeBT]I (S2)	90	105	4	153	189	7

The thermal decomposition curves of the organic salts indicate that they contain trace amounts of water, as shown in Table 4. The compounds' thermal stability is also evident, and their decomposition results in a minimal mass loss of no more than 7% of their initial weight—a beneficial outcome when compared to previous studies [28, 29]. We observe that the ionic liquids based on benzotriazole are more thermally stable than those based on imidazole and pyridine [11, 30]. This could be explained by the crystalline structure of the nitrogen bases formed by benzotriazole and its compounds [20].

## Conclusion

It is evident from physical and spectroscopic data that the complex salts of benzathriazole form a tetrahedral geometry around the central ion and that the organic salts are of good purity. According to thermal studies, the produced organic salts can be used in industrial settings at temperatures up to 150°C, for example, in processes that form complex salts during desulfurization or heavy-metal removal. To evaluate their effectiveness, these ionic liquids may be tested in such applications in future research.

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