








Using of Benzotriazolium Iodide Ionic Liquids for Extractive Desulfurization of Model Diesel Oil

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Abstract: Benzotriazolium-based ionic salts have been prepared with an alkyl group (methyl and allyl), with iodine anion, the prepared compound (**[diMBt]I (1)**, **[AMBt]I (2)** and **[AMBt]I₃ (3)**) structure have determined by single crystal x-ray and they used for desulfurization of sulfur from model diesel oil in order to obtain reduced sulfur fuel with more environmental friendly properties. The prepared compounds were characterized using several techniques, and their efficiency in removing sulfur from the sample oil was evaluated. The results showed that the removal efficiency was acceptable and could be improved in future research.

Keywords: Ionic liquids, Benzotriazolium base, Desulfurization.

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1. INTRODUCTION

Liquids with just ions are generally referred to as anionic liquids. Nevertheless, any salt that is heated to the point of melting, like sodium chloride, is an ionic liquid with a very high melting point of 803°C. This term has another definition that differentiates it from the traditional definition of molten salts. Melted salts are typically considered extremely corrosive, highly viscous, and have a high melting point; however, ionic liquids are liquid at temperatures below 100°C and have a relatively low viscosity. The melting point temperature of 100°C is the relatively sharp boundary that separates molten salts from ionic liquids (1-3).

In theory, there are one trillion different types of ionic liquids. Ionic liquids are available for chemists to select from based on what their work requires. Organic solvents and ionic liquids are not the same thing. Gases cannot be formed from their evaporation. Thus, ionic liquids cannot produce new air pollution or hazardous gases in chemical experiments. Ionic liquids have been of interest to chemists because they are recyclable multiple times. Therefore, the design, development, and application of chemical products and processes aimed at

minimizing or completely doing away with using and producing substances harmful to the environment and human health is known as "green chemistry" (4-6).

Ionic liquids have many potential uses in the energy sector, from nuclear applications to the petroleum industry. Ionic liquids can potentially address several unresolved problems in the industry, including waste minimization, recycling, and recovering helpful hydrocarbon fuels from refractory sources. Although many of these ideas have been demonstrated in the lab, there have not been many reports or widespread attempts to scale them up to industrial proportions (3,7-9).

The increase in harmful emissions to the atmosphere is one of the effects of industrialized countries' rapid development. Elevated sulfur oxide levels are a primary historical source of air pollution because they can be converted to sulfuric acid when combined with water vapor. This is why high sulfur oxide levels cause unwanted acid rain. The combustion of fossil fuels containing sulfur is the primary source of these pollutant emissions, and environmental regulation efforts are concentrated on reducing emissions from transportation exhaust gases (10-12).

This work aims to determine how well benzotriazolium iodide de-sulfurizes model diesel oil.

2. MATERIAL AND METHODS

2.1. Preparation of Benzotriazolium iodide Salts (13-17)

1,3-dimethyl benzotriazolium iodide salt **[diMBt]I (1)**: The preparation of this compound involved dissolving 0.01 mol, 1.19 g of Benzotriazole in 10 mL of ethanol and adding 0.01 mol, 0.62 mL of methyl iodide to the mixture in a 50 mL round-bottom flask. Next, 10 mL of 10% KOH was added to the mixture. Refluxing the mixture lasted for one hour. The mixture was refluxed for one hour with five milliliters of methyl iodide added. The mixture was extracted three times with an equal volume of regular hexane to eliminate the extra methyl iodide. The solution was then set aside for a day. Diethyl ether was used to extract yellow crystals, filter them, and repeatedly wash them.

1-allyl -3-methyl benzotriazolium iodide salt **[AMBt]I (2)** It was prepared using the same method as in **(1)**, with the exception that, in order to produce brown-yellow crystals, the solution mixture was supplemented with 5 milliliters of allyl chloride in the second step rather than methyl iodide. Compounds **(1)** and **(2)** were characterized by NMR spectroscopy, mass spectroscopy, and other spectroscopic methods such as infrared spectroscopy. The results of these measurements are given in the attached supplementary file. With DMSO-d₆ as the solvent and 20°C, the proton nuclear magnetic resonance was measured using a BRUKER AVANCE DPX 400 MHz instrument. The Trio-1000 Mass Spectrometer was used to perform these measurements.

1-allyl -3-methyl benzotriazolium triiodide salt **[AMBt]I₃ (3)** was accidentally created while the second compound was being prepared, as its precipitated crystals were separated using the same preparation technique from the second compound's remaining solution.

2.2. X-Ray Diffraction

On a (Bruker D8) diffractometer, 1.5 kW graphite monochromated Mo radiations and an APEX CCD detector were used to gather single crystal X-ray data. The crystal's distance from the detector was 5.985 cm. During the data collection process, exposure times of 10 seconds per frame and scan widths of 0.3° were employed.

The crystals' faces were indexed for a numerical absorption correction, and their separations from the center were measured. Three ω scans with varying φ values were used to collect the data, which produced data with an average completeness of 90.2-99.7% in the 2.33 to 33.04° range. The SAINT v7.45a (150) was integrated with the frames. Using SADABS V2008-1 (151), a numerical absorption correction

based on the crystal's size and shape was performed. The structure was solved and improved using X-SEED, a graphical interface for SHELX (18,19).

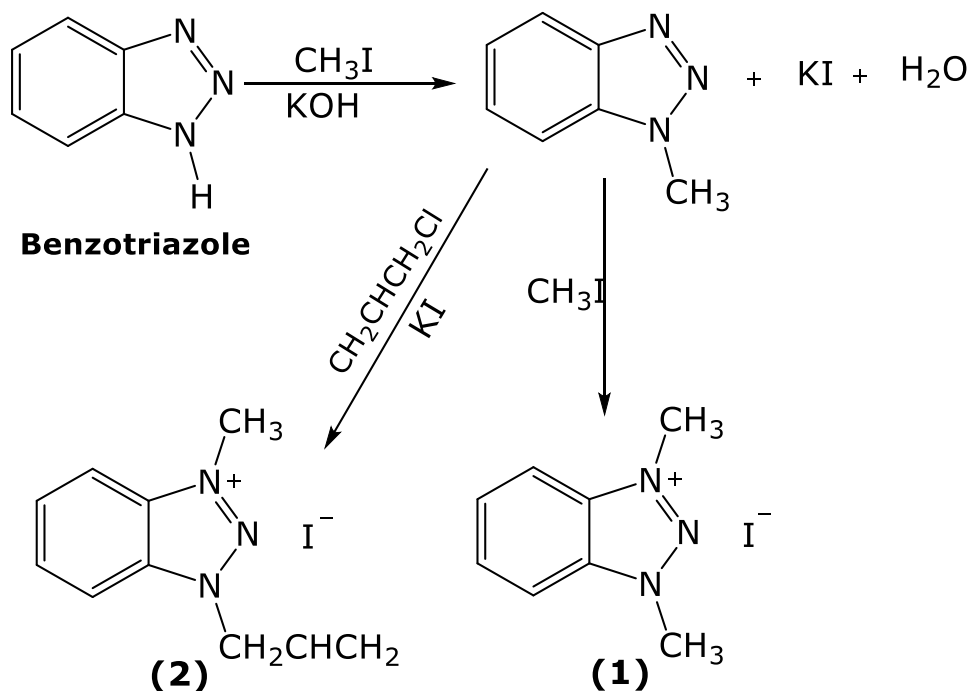
2.3. Desulfurization of Fuel

The model diesel oil was made by dissolving dibenzothiophene (DBT) by weight in n-hexane, resulting in a concentration of 3000 parts per million of DBT. In order to identify the best compound and conduct additional testing with a different concentration, time, and concentration, all of the prepared compounds were tested for extractive desulfurization using model fuel with the same concentration and time (10 g of model fuel, with 0.05 g of the compound dissolved in 5 ml of DMSO, 30 min of extraction). A flame ionization detector is used in gas chromatography (GC-FID). (CARLO ERBA STRUMENT-AZIONE 4200) It was used to perform the gas chromatography. The temperatures of the program are as follows: The temperature of the injector is 295°C, the detector is 273°C, and the oven is 130°C for two minutes before ramping up to 175°C for ten minutes. Details of gas flow rates: "Water:1kg/cm². Cosmetics (air): 1 kg/cm Helium: 2 kg/cm², air: 1.5 kg/cm². Column characteristics: Call sign: SE-30, 30 m in length, 0.53 mm in diameter, and 1.2 μ m in film thickness" (20,21).

3. RESULT AND DISCUSSION

The outcome is dependent on the reaction process, which involves adding methyl (allyl) group to the second nitrogen atom in Benzotriazole and then substituting the methyl group on nitrogen, as illustrated in scheme 1.

¹H-NMR of **[diMBt]I (1)**: DMSO-d₆ was used as a solvent to measure the proton nuclear magnetic resonance (NMR) spectra. The singlet signal at (4.63 ppm) with integration (6.11) is associated with two methyl groups on the 1 and 3 positions of the nitrogen atom. The four protons of the benzene aromatic ring in the benzotriazole are represented by the multiple signals in (7.99 - 8.37 ppm) with integrations of (4.20). The main mass fragments are *m/z*: 133 (100.0%), 132 (20%), 119 (10%). **[AMBt]I (2)**: singlet signal at (2.10 ppm) with integration (2.95) is associated with nitrogen atom methyl groups. The doublet signal in (4.64 ppm) with integrations of 1.84, its chemical shift, and its integration are associated with the protons in the allyl group's terminal (CH₂) group. The protons in the allyl group with the nitrogen atom in the benzotriazole's attached (CH₂) groups are responsible for the second doublet signals in (5.71 ppm) with integrations (1.78). With integrations of 1.04, the multiple signals in (5.46-5.55 ppm) correspond to the proton of (CH) in the allyl group. The four protons of the multiple signals in (8.00-8.02 ppm) and (8.34-8.39 ppm) with integrations of (1.80) and (1.85), respectively, belong to benzotriazole's aromatic ring.



3.1. Characterizations of Salts

The yellow crystals of compound **(1)** [diMBt]I, have been studied using a single crystal X-ray. An orthorhombic crystal system with a P_{nma} space group is present in the crystal size (0.52x 0.51x 0.40

mm³). A perspective drawing of compound **(1)** and the atom-numbering system are displayed in Figure 1. 50% of the time, displacement ellipsoids are depicted, and H atoms are displayed as tiny spheres with arbitrary radii.

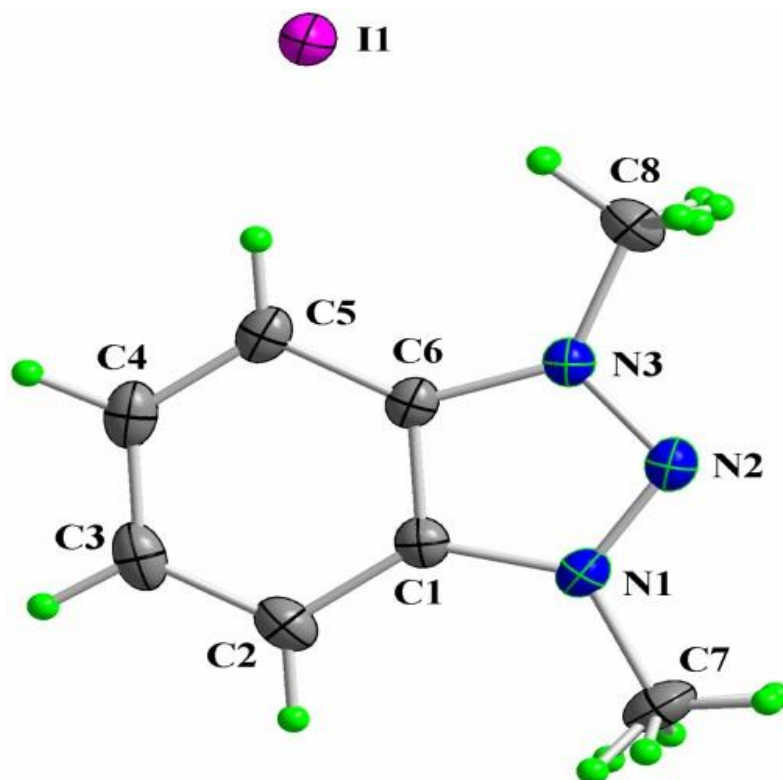


Figure 1: Crystal structure of compound **(1)** [diMBt]I.

Planar organic ions are those that are situated on the crystallographic mirror plane. 50% occupancy results in disordered methyl hydrogen. The positively charged portion of the corresponding cation is not directly connected to the anion I⁻. Instead, I⁻ holds

cations together via C-H...I1 (2.93 Å) and C(3)-H(3)...I1i (2.95 Å) are examples of I-hydrogen bonds. These findings are in good agreement with the literature (17,22). Table 1 contains a list of the crystal data.

Table 1: Crystal data of compound **(1)** [diMBt]I.

Empirical formula	(C ₈ H ₁₀ N ₃) ⁺ · I ⁻	Index ranges	-13 ≤ h ≤ 13, -9 ≤ k ≤ 9, -26 ≤ l ≤ 26
Formula weight	275.09	Reflections collected	16404
Temperature	153(2) K	Independent reflections	1922 [R(int) = 0.0606]
Wavelength	0.71073 Å	Completeness to theta = 32.00°	99.7 %
Crystal system	Orthorhombic	Absorption correction	multi-scan
Space group	P _{nma}	Max. and min. transmission	0.3597 and 0.2858
Unit cell dimensions	a = 8.8972(4) Å α = 90° b = 6.4646(3) Å β = 90° c = 17.0708(7) Å γ = 90°	Refinement method	Full-matrix least-squares on F ²
Volume	981.86(8) Å ³	Data/restraints/parameters	1922 / 0 / 76
Z	4	Goodness-of-fit on F²	1.005
Density (calculated)	1.861 Mg/m ³	Final R indices [I > 2σ(I)]	R1 = 0.0322, wR2 = 0.0773
Absorption coefficient	3.213 mm ⁻¹	R indices (all data)	R1 = 0.0418, wR2 = 0.0816
F(000)	528	Extinction coefficient	0.0398(18)
Crystal size	0.52 × 0.51 × 0.40 mm ³	Largest diff. Peak and hole	1.310 and -1.612 e.Å ⁻³

The Brown-yellow crystals of compound **(2)** [AMBt]I have been studied using a single crystal X-ray. Compound **(2)** crystallizes in the Triclinic space group P-1. A crystal size (0.39 × 0.40 × 0.10 mm³) was measured, and the substitution of methyl and

allyl groups on the 1,3 position on nitrogen atoms was clarified (14). Figure 2 shows the crystal structure of compound **(2)**. The crystal data are listed in Table 2.

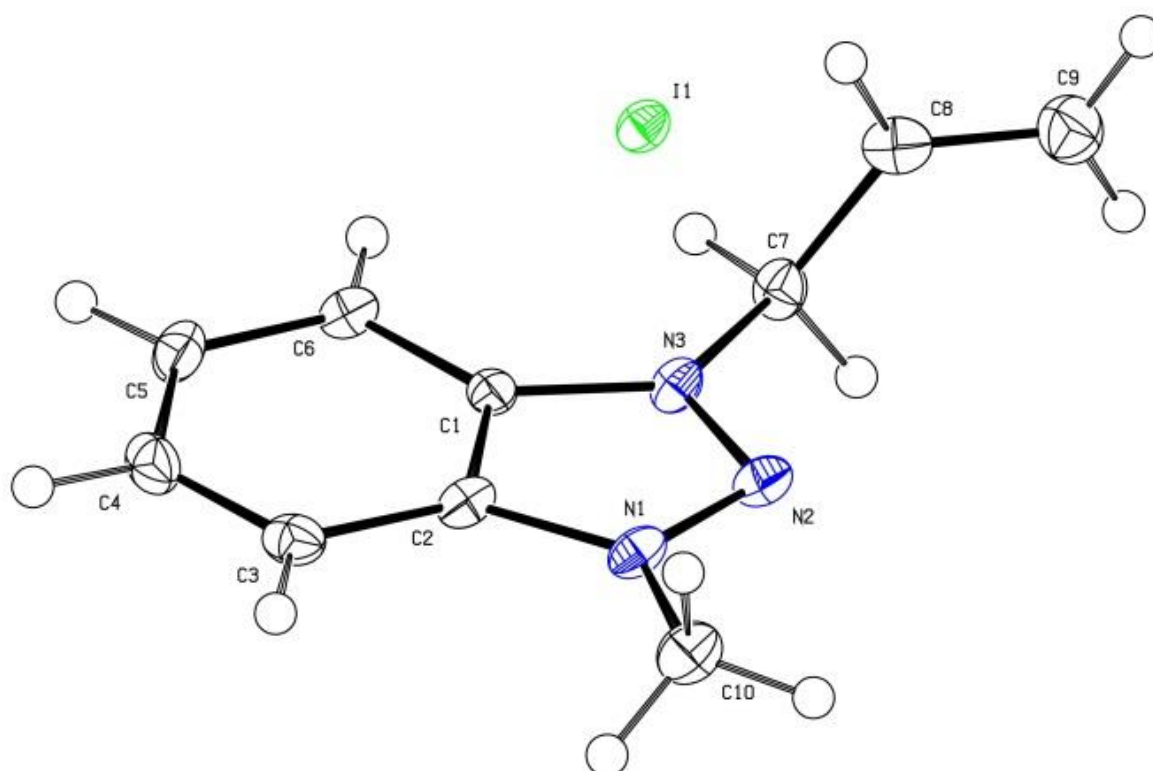
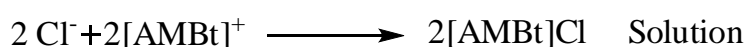
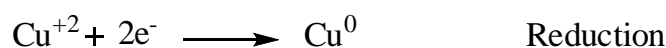
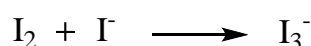
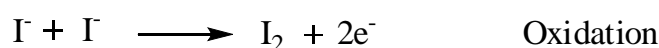
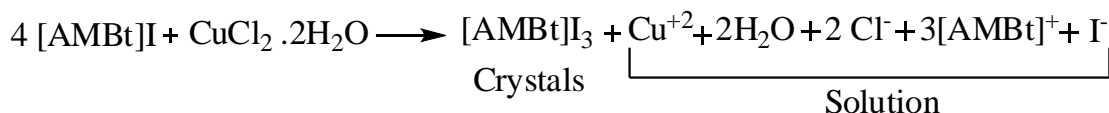
**Figure 2:** Crystal structure of compound **(2)** [AMBt]I.

Table 2: Crystal data of compound (2) [AMBt]I.

Empirical formula	(C ₁₀ H ₁₂ N ₃) ⁺ · I ⁻	Index ranges	2.33 to 28.31°.
Formula weight	301.13	Reflections collected	-10<=h<=10, -10<=k<=10, -13<=l<=13
Temperature	100(2) K	Independent reflections	7816
Wavelength	0.71073 Å	Completeness to theta = 32.00°	2798 [R(int) = 0.0894]
Crystal system	Triclinic	Absorption correction	Semi-empirical from equivalents
Space group	P -1	Max. and min. transmission	0.972 and 0.409
Unit cell dimensions	a = 7.8839(12) Å a = 114.093(2)° b = 8.2265(14) Å β = 104.033(15)° c = 9.9957(17) Å γ = 92.201(13)°	Refinement method	Full-matrix least-squares on F ²
Volume	567.20(16) Å ³	Data/restraints/parameters	2798 / 0 / 128
Z	2	Goodness-of-fit on F²	0.955
Density (calculated)	1.763 Mg/m ³	Final R indices [I>2sigma(I)]	R1 = 0.0349, wR2 = 0.0641
Absorption coefficient	2.790 mm ⁻¹	R indices (all data)	R1 = 0.0399, wR2 = 0.0661
F(000)	292	Extinction coefficient	1.581 and -1.354e.Å ⁻³
Crystal size	0.39 x 0.40 x 0.10 mm ³	Largest diff. peak and hole	2.33 to 28.31°.

The unexpected result is the preparation of the novel organic salt [AMBt]I₃ (3) isolated as a brown crystal through the preparation of the complex salt of the formula [AMBt]₂[CuCl₂I₂] of complex salt in the

attempt to prepare copper (II) chloride with the organic salt number (2) [AMBt]I. The suggested mechanism is shown in the equations below:



A single crystal x-ray characterized this new organic salt [AMBt]I₃ (3). A perspective drawing of compound (3) is shown in Figure 3. All the bond

lengths and angles are in the normal range and similar to those in the literature data (23,24).

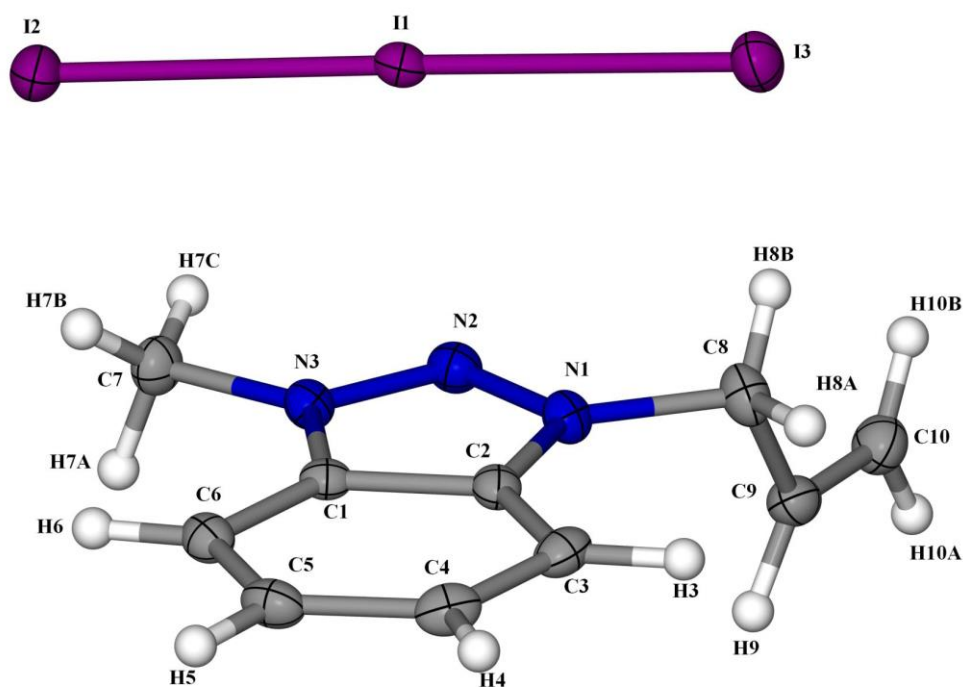


Figure 3: The crystal structure of compound **(3)** [AMBt] I_3 .

Different views of a fragment of the crystal structure of compound **(3)** [AMBt] I_3 are shown in Figure 4. Due to its large size, the linear triiodide anion is ideal for forming numerous weak C-H hydrogen bonds and

interacting with the electropositive phenyl rings of the cations. The overall positive charge of the cation is spread out over the hydrogen atoms.

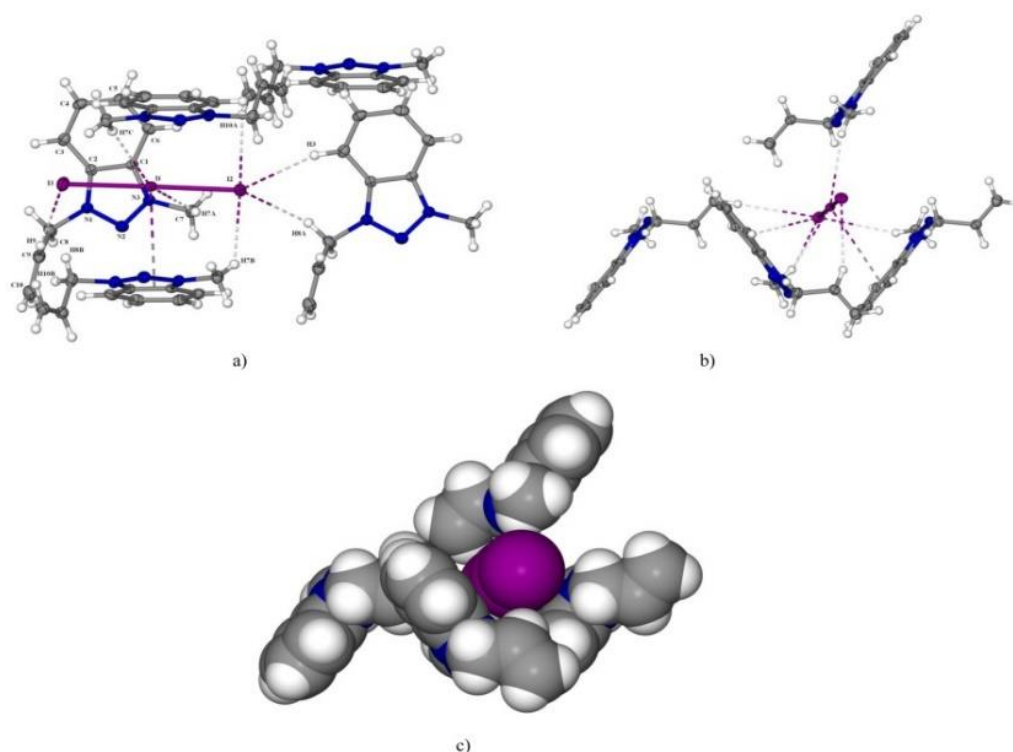


Figure 4: Different views of the crystal structure of the compound **(3)** [AMBt] I_3 .

Five cations surround every tri-iodide anion. The I_3^- ions are encircled by the organic molecules, creating a structure resembling a cage. The terminal I atom of the long bond (I_2) in the tri-iodide anion (I_1-I_2 , 2.9451(5) Å; I_1-I_3 , 2.8822(5) Å) accepts more

hydrogen bonds from the cations than the other I atoms in the anion. The H atoms are depicted as spheres with arbitrary radii, and displacement ellipsoids are drawn with a 50% probability. Table 3 contains a list of the crystal data.

Table 3: Crystal data of compound (3) [AMBt]I₃.

Empirical formula	C ₁₀ H ₁₂ I ₃ N ₃	Theta range for data collection	2.33 to 27.10°.
Formula weight	554.93	Index ranges	-12<=h<=12, -17<=k<=18, -14<=l<=15
Temperature	100(2) K	Reflections collected	10767
Wavelength	0.71073 Å	Independent reflections	3327 [R(int) = 0.0226]
Crystal system	Monoclinic	Absorption correction	Semi-empirical from equivalents
Space group	P21/n	Max. and min. transmission	0.8365 and 0.5773
Unit cell dimensions	a = 9.5684(13) Å α = 90° b = 14.2827(19) Å β = 111.566(2)° c = 11.9163(16) Å γ = 90°	Refinement method	Full-matrix least-squares on F ²
Volume	1514.5(4) Å ³	Data/restraints/parameters	3327 / 0 / 193
Z	4	Goodness-of-fit on F²	1.078
Density (calculated)	2.434 Mg/m ³	Final R indices [I > 2σ(I)]	R1 = 0.0199, wR2 = 0.0407
Absorption coefficient	6.173 mm ⁻¹	R indices (all data)	R1 = 0.0274, wR2 = 0.0423
F(000)	1008	Largest diff. Peak and hole	0.547 and -0.620 e.Å ⁻³
Crystal size	0.10 x 0.10 x 0.03 mm ³		

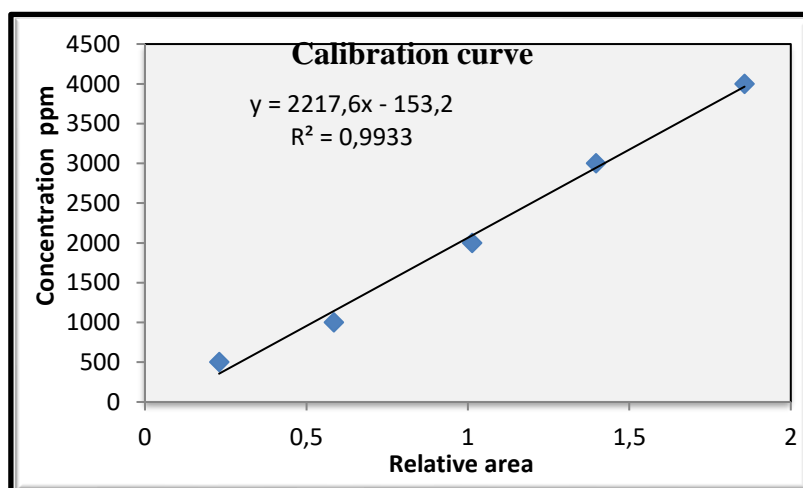
3.2. The Desulfurization of Model Diesel Oil

Model diesel oil has undergone treatment multiple times at various concentrations following the creation of a calibration curve using the gas chromatography method (25,26). As indicated in Table 4 Diagram 1,

the calibration curve diagram was created using the relationship between the concentration of the standard solution and the ratio of the standard Dibenzothiophene (DBT) peak's area over the celebrant's Hexadecane (C₁₆) peak's area.

Table 4: The relative area of (DBT) over (C₁₆) and the concentration of a standard solution.

Conc. ppm X1000	DBT / C ₁₆	C ₁₆ %	DBT %
4	1.8568	207314	384958
3	1.3963	226161	315797
2	1.0131	141492	143349
1	0.5847	135207	79067
0.5	0.2294	133305	30586

**Diagram 1:** Calibration curve of standard solution.

The prepared compounds (1-3) were all tested in the extraction process for 30 minutes at the same concentration (0.05g), which was the process' ideal condition. The outcomes are shown in Table 5. In

order to achieve the highest extraction efficiency, the optimal conditions for the extractive sulfur extraction process were predetermined and included 30 minutes at 25°C and a weight of ionic salts 0.05 g.

Table 5: The removal of sulfur % for 30 min and 0.05g extractive treatment.

No.	DBT %	C16 %	DBT/C16	DBT conc. ppm	S removal %
1	79602	138832	0.5734	1206.0249	22.4122
2	116492	182211	0.6393	1344.7557	13.4872
3	67591	125265	0.5396	1134.9612	26.9840

The extraction efficiency is good compared to previous studies (27, 28), with the observation that less salt is used; adding more weight will improve the extraction efficiency and the sulfur removal process. In conformity with the literature (15,27,28), it is also mentioned that the ionic liquids utilized are less effective than those that do contain Lewis acids, such as ferric chloride or other Lewis acids (28,29). Increasing the percentage of Lewis acids is anticipated to improve the extraction process' efficiency (30,31). The results indicate this. The rate at which sulfur is removed increases as iodine ions rise.

4. CONCLUSION

The prepared compounds have varying desulfurization efficiencies because the different anion or cation interacts differently with the organosulfur compounds in diesel oil. Because of the significant tri-iodide anion, salt (**3**) produced the best desulfurization.

5. ACKNOWLEDGMENT

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