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Data Article

Synthesis and characterization data of monocationic and dicationic ionic liquids or molten salts

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ABSTRACT

Data presented in this article are related with the research paper entitled "Ecotoxicity assessment of dicationic versus monocationic ionic liquids as a more environmentally friendly alternative" [1]. The present article describes the synthesis steps and characterization data of a set of twenty-six imidazolium, pyrrolidinium and pyridinium-based ionic liquids (ILs) or molten salts: nine monocationic and seventeen dicationic. Specifically, the chemical structure of the compounds was confirmed by ¹H NMR, ¹³C NMR and ¹⁹F NMR spectroscopy and mass spectrometry (MS). Other data such as physical state at room temperature, melting point temperature (for solids at room temperature) and thermal decomposition temperature (when melting was not reached before decomposition) of the ILs or molten salts are also reported here.

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Specifications Table

Subject area	Chemistry
More specific subject area	Synthesis and characterization of ionic liquids or molten salts
Type of data	Tables, figures

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How data was acquired	^1H , ^{13}C and ^{19}F NMR spectra were recorded on a Jeol model EX270 instrument. MS was recorded on a Bruker MicroTOF 61 spectrometer. Melting point temperature was recorded on a Büchi Melting Point B-540 instrument. Thermal decomposition temperature was acquired by Differential Scanning Calorimetry on a DSC 2920 (TA instruments) instrument.
Data format	<i>Raw and analyzed data</i>
Experimental factors	<i>Samples were dried under vacuum overnight before their characterization.</i>
Experimental features	<i>Relevant data on the characterization of the ILs or molten salts were determined.</i>
Data source location	<i>University of Murcia, Murcia, Spain, Europe</i>
Data accessibility	<i>The data are provided with this article.</i>
Related research article	<i>M.G. Montalbán, G. Villora, P. Licence, Ecotoxicity assessment of dicationic versus monocationic ionic liquids as a more environmentally friendly alternative, Ecotox. Environ. Safe. 150 (2018) 129–135.</i>

Value of the Data

- All the steps for the synthesis of the ILs or molten salts here described and the methods can be followed by other researchers.
- The chemical synthesis of some of these ILs or molten salts had not been reported before.
- NMR spectra and MS data of the ILs or molten salts synthesized are useful for structural characterization of these and other similar ILs or molten salts.
- Data on melting point and decomposition temperature of these ILs or molten salts can be valuable for the design of their applications.

1. Data

The abbreviations, molecular weights and structures of the ILs or molten salts are presented in [Table 1](#). The synthesis steps necessary for the preparation of the ILs or molten salts are described then in detail. After the report of the chemical synthesis of the ILs or molten salts, their characterization (NMR spectra and MS) is included. [Figs. 1–20](#) show the ^1H NMR and ^{13}C NMR spectra of the novel compounds. Finally, [Table 2](#) collects the physical state of the ILs or molten salts at room temperature, their colour, melting point (for solids at room temperature) and decomposition temperature (when melting is not reached before thermal decomposition).

2. Experimental design, materials, and methods

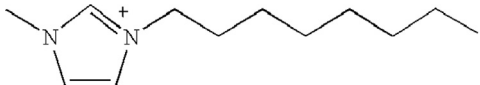
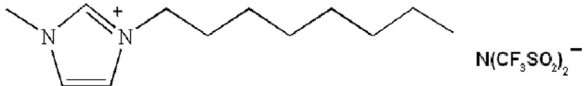
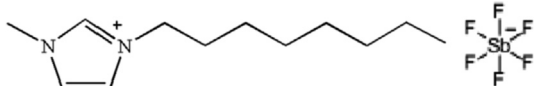
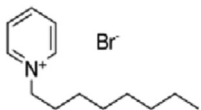
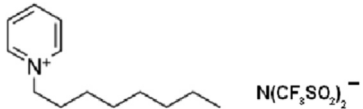
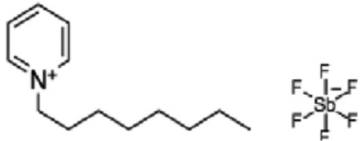
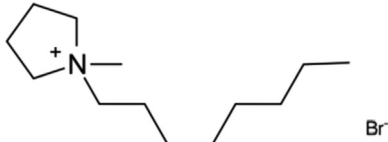
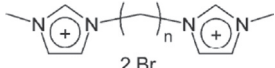
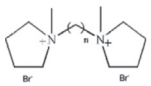
See [Table 1](#)

3. Synthesis and characterization of ILs

$\text{C}_8(\text{MIm})\text{Br}$ was synthesized following procedures described in the literature with modifications [\[2,3\]](#). 100 mmol of 1-methylimidazole was placed in a round bottom flask fitted with a reflux condenser and an additional funnel under a static atmosphere of Ar. 110 mmol of 1-bromooctane was added dropwise via a pressure equalising addition funnel while the mixture was stirred at 60°C under inert atmosphere. The additional funnel was removed and the reaction mixture was stirred at

Table 1.

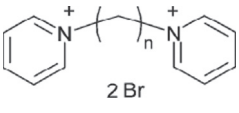
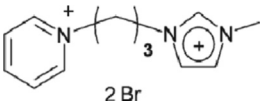
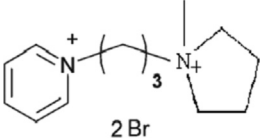
Abbreviations and structures of the studied ILs or molten salts.

Abbreviation	Molecular weight (g mol ⁻¹)	Structure
C₈ (MIm) Br	275.23	
C₈ (MIm) NTf₂	475.47	
C₈ (MIm) SbF₆	431.07	
C₈ (Pyr) Br	272.22	
C₈ (Pyr) NTf₂	472.47	
C₈ (Pyr) SbF₆	428.07	
C₈ (MPyrr) Br	278.27	
C₈ (MPyrr) NTf₂	478.51	
C₈ (MPyrr) SbF₆	434.12	
C₂(MIm)₂ Br₂	352.07	
C₃(MIm)₂ Br₂	366.07	
C₄(MIm)₂ Br₂	380.12	
C₆(MIm)₂ Br₂	408.18	
C₈(MIm)₂ Br₂	436.23	
C₃(MPyrr)₂ Br₂	372.21	
C₄(MPyrr)₂ Br₂	386.21	
C₆(MPyrr)₂ Br₂	414.26	
C₈(MPyrr)₂ Br₂	442.32	

n = 2, 3, 4, 6, 8

n = 3, 4, 6, 8

Table 1. (continued)

Abbreviation	Molecular weight (g mol ⁻¹)	Structure
C₂(Pyr)₂ Br₂	346.06	 2 Br
C₃(Pyr)₂ Br₂	360.09	
C₄(Pyr)₂ Br₂	374.09	
C₆(Pyr)₂ Br₂	402.09	
C₈(Pyr)₂ Br₂	430.09	
C₁₂(Pyr)₂ Br₂	486.09	
C₃(Pyr)(MIm) Br₂	363.12	 2 Br
C₃(Pyr)(MPyrr) Br₂	366.14	 2 Br

60 °C for 72 h. The crude product recovered as a honey coloured oil was dissolved in 20 mL of acetonitrile and washed with 250 mL of ethyl acetate to remove unreacted 1-bromooctane. The upper layer (ethyl acetate containing 1-bromooctane) was decanted set aside, residual acetonitrile was removed from the product by automated rotary evaporator. The oil was dried under high vacuum ($p < 10^{-2}$ mbar) at 60 °C for 24 h and eventually recovered as a colourless liquid. Data in agreement with the literature [4].

C₈(MPyrr)Br and **C₈(Pyr)Br** were synthesized according to procedures described in the literature [5]. 240 mmol of 1-methylpyrrolidine or pyridine was placed in a round bottom flask fitted with a water condenser topped with a drying tube (CaCl₂) to avoid moisture penetration. 288 mmol of 1-bromooctane was added dropwise while stirring at 70 °C. The reaction time was 48 hours. The desired product was recrystallised in acetonitrile/ethyl acetate ($\approx 1:3$ v:v) and dried first using a rotary evaporator and then under high vacuum ($p < 10^{-2}$ mbar) at 60 °C for 12 h to yield a white solid. Data in agreement with the literature [6,7].

C₈(MIm)NTf₂, **C₈(MPyrr)NTf₂** and **C₈(Pyr)NTf₂** were synthesized according to procedures described in the literature [5,8,9]. 5 mmol of C₈(MIm)Br, C₈(MPyrr)Br or C₈(Pyr)Br were transferred to a round bottom flask and dissolved in 10 mL of ultrapure water while stirring at room temperature. Aqueous lithium bis(trifluoromethane)sulfonylimide (6 mmol in 6 mL of ultrapure water) was added dropwise. The mixture was stirred at room temperature for 24 hours. Then the mixture was transferred to a funnel washing with ethyl acetate; the aqueous layer was separated and the ionic liquid dissolved in 30 mL of ethyl acetate and washed with ultrapure water (4×30 mL). Finally, the ethyl acetate was removed in a rotary evaporator and the ionic liquid was dried under vacuum ($p < 10^{-2}$ mbar) at 60 °C for 24 hours. Data in agreement with the literature [8,10,11].

C₈(MIm)SbF₆, **C₈(MPyrr)SbF₆** and **C₈(Pyr)SbF₆** were synthesized according to procedures described in the literature [12]. In a single necked round bottom flask with a magnetic stirring bar, 5 mmol of C₈(MIm)Br, C₈(MPyrr)Br or C₈(Pyr)Br was dissolved in 20 mL of dichloromethane. Then 6 mmol of sodium hexafluoroantimonate (V) was added. The mixture was stirred for 24 hours at room temperature while observing the formation of a white solid (NaBr). This solid was filtered off and the filtrate was washed with ultrapure water several times (5×50 mL). The solvent was removed in a rotary evaporator and the ionic liquid was dried under high vacuum ($p < 10^{-2}$ mbar) at 70 °C for 24 hours. Data in agreement with the literature [13].

C₂(MIm)₂ Br₂, **C₃(MIm)₂ Br₂**, **C₄(MIm)₂ Br₂**, **C₆(MIm)₂ Br₂** and **C₈(MIm)₂ Br₂** were synthesized following the same procedure [8,14,15]. A three-necked round bottom flask fitted with reflux condenser and pressure equation funnel was filled with a solution of 30 mmol of 1,2-dibromoethane, 1,3-dibromopropane, 1,4-dibromobutane, 1,6-dibromohexane or 1,8-dibromooctane in 12 mL of

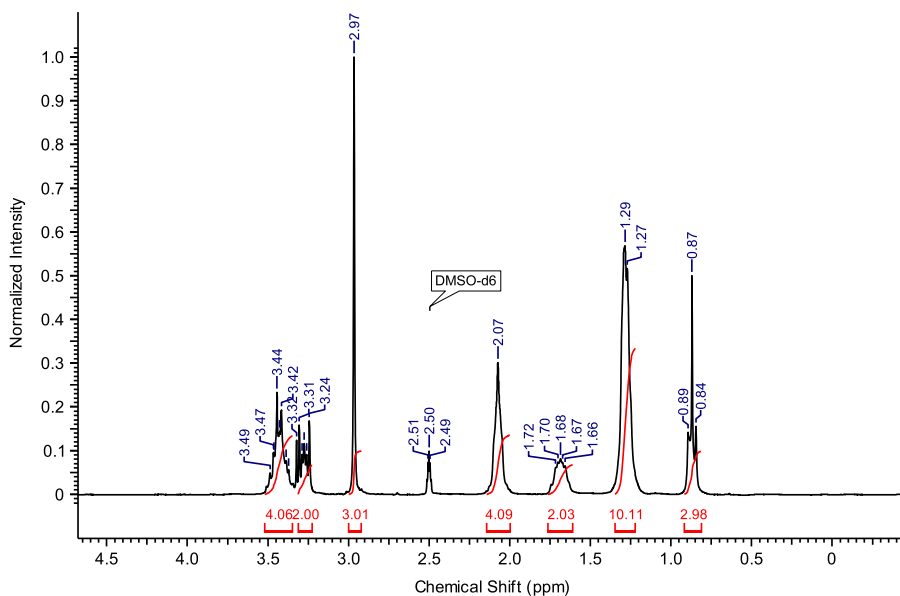


Fig. 1. ^1H NMR spectra of $\text{C}_8(\text{MPyrr})\text{SbF}_6$.

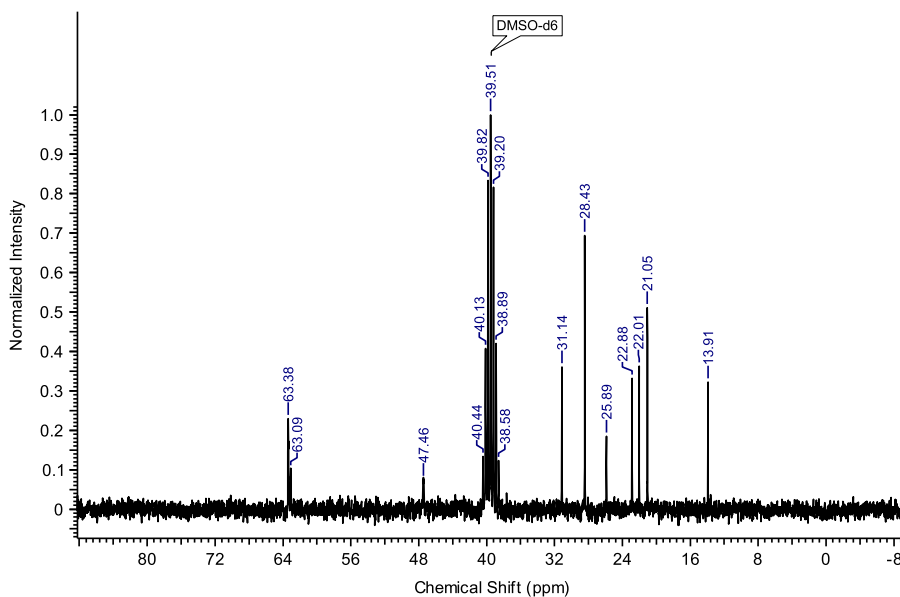


Fig. 2. ^{13}C NMR spectra of $\text{C}_8(\text{MPyrr})\text{SbF}_6$.

methanol. Then 60 mmol of 1-methylimidazole was added dropwise while stirring at room temperature. The resulting mixture was further heated and stirred at 40–50 °C for 48 hours. The product was isolated by filtration and purified by recrystallization. The resulting product was transferred to a single-necked round-bottomed flask, washing with methanol. The solvent was then removed under reduced pressure using a rotary evaporator. Data in agreement with the literature [15–18].

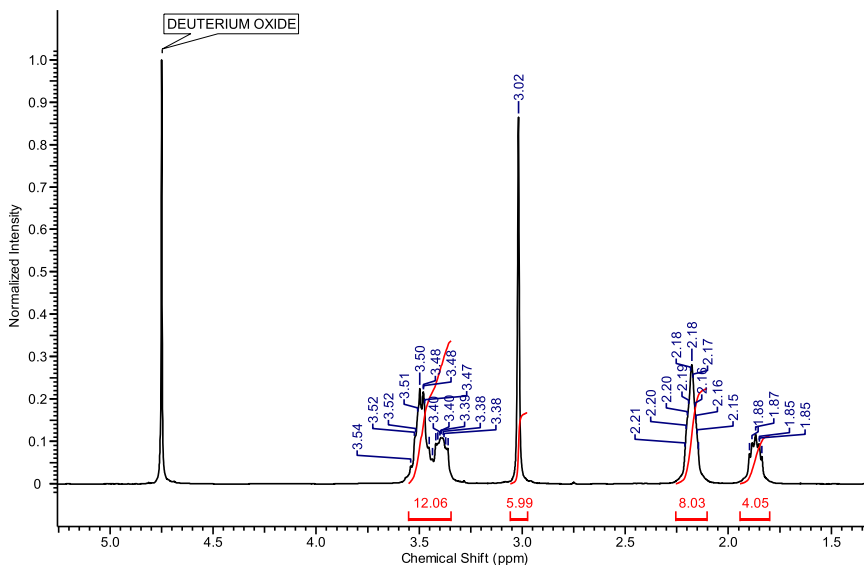


Fig. 3. ^1H NMR spectra of $\text{C}_4(\text{MPyrr})_2\text{Br}_2$.

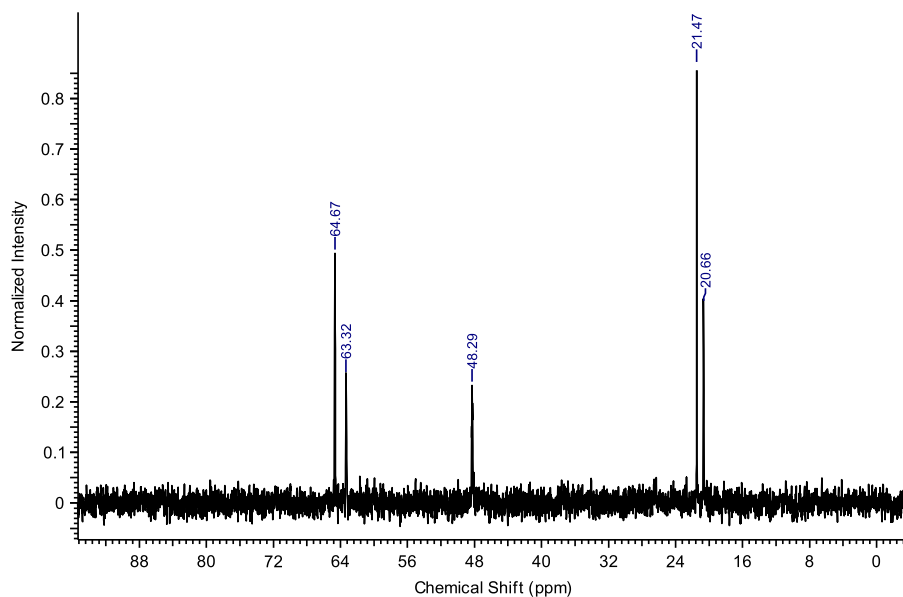


Fig. 4. ^{13}C NMR spectra of $\text{C}_4(\text{MPyrr})_2\text{Br}_2$.

$\text{C}_3(\text{MPyrr})_2\text{Br}_2$, $\text{C}_4(\text{MPyrr})_2\text{Br}_2$, $\text{C}_6(\text{MPyrr})_2\text{Br}_2$ and $\text{C}_8(\text{MPyrr})_2\text{Br}_2$ were synthesized with the same procedure [15,19]. A three-necked round-bottomed flask fitted with a reflux condenser and pressure-equalised funnel was charged with a solution of 30 mmol of 1,3-dibromopropane, 1,4-dibromobutane, 1,6-dibromohexane or 1,8-dibromooctane in 10 ml of methanol. Then 63 mmol of 1-methylimidazole was added dropwise while stirring at room temperature. The resulting mixture was further heated and stirred at 40–50 °C for 48 hours. The product was isolated by filtration and

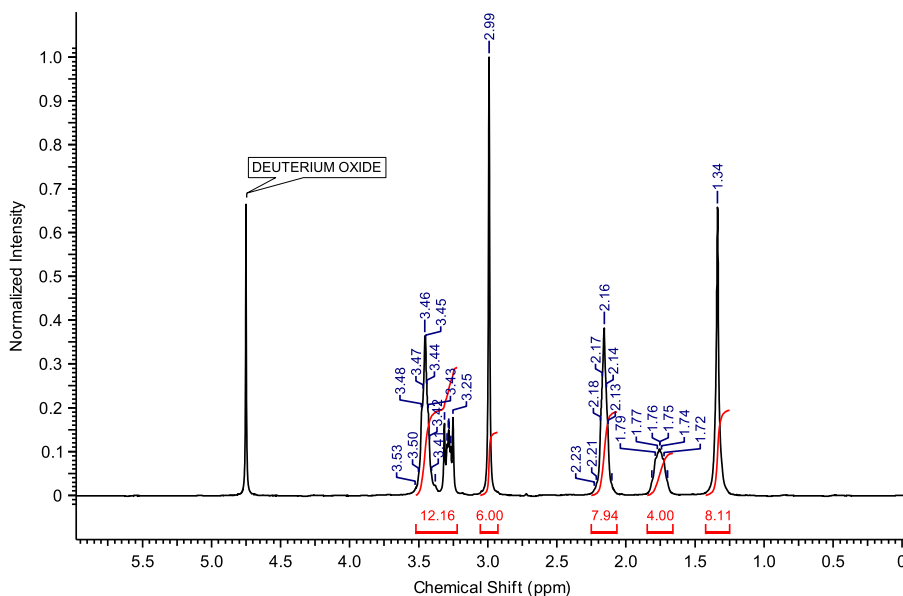


Fig. 5. ^1H NMR spectra of $\text{C}_8(\text{MPyrr})_2\text{Br}_2$.

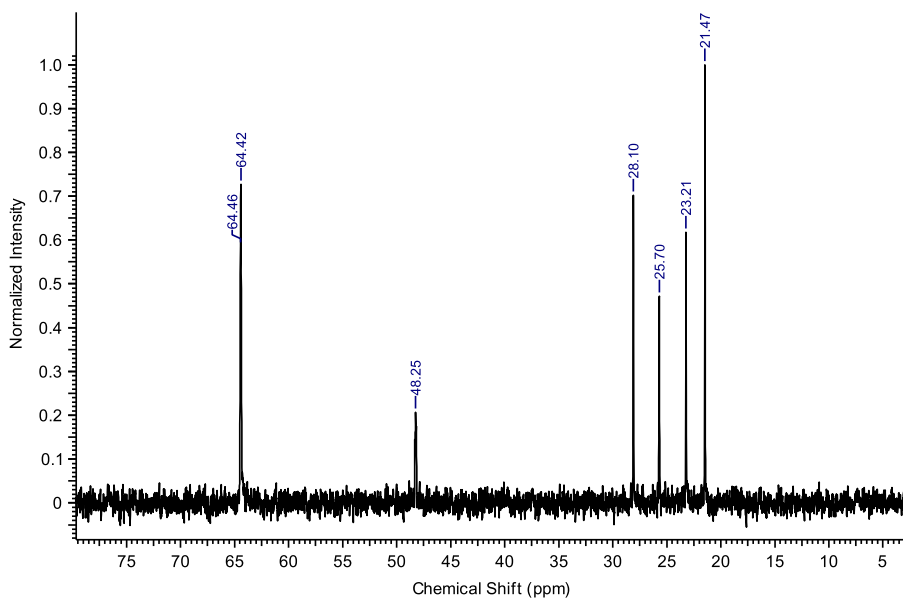


Fig. 6. ^{13}C NMR spectra of $\text{C}_8(\text{MPyrr})_2\text{Br}_2$.

purified by recrystallization in methanol/ethyl acetate ($\approx 1:3$ v:v). The resulting product was transferred to a single-necked round-bottomed flask, washing with methanol. The solvent was then removed under reduced pressure using a rotary evaporator. Data in agreement with the literature [15,18].

$\text{C}_2(\text{Pyr})_2\text{Br}_2$, $\text{C}_3(\text{Pyr})_2\text{Br}_2$, $\text{C}_4(\text{Pyr})_2\text{Br}_2$, $\text{C}_6(\text{Pyr})_2\text{Br}_2$, $\text{C}_8(\text{Pyr})_2\text{Br}_2$ and $\text{C}_{12}(\text{Pyr})_2\text{Br}_2$ were synthesized with the same procedure [15]. A three-necked round bottomed flask fitted with a reflux

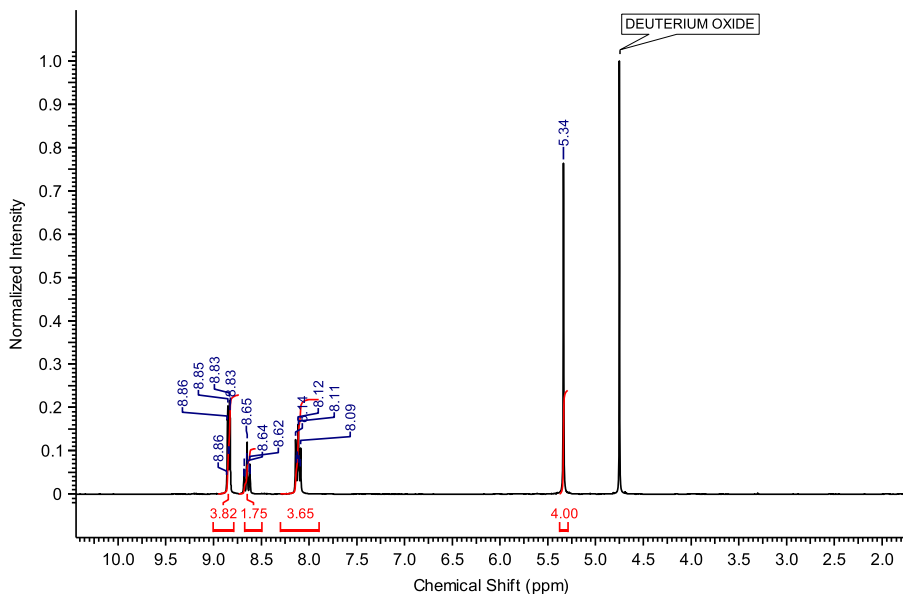


Fig. 7. ^1H NMR spectra of $\text{C}_2(\text{Pyr})_2\text{Br}_2$.

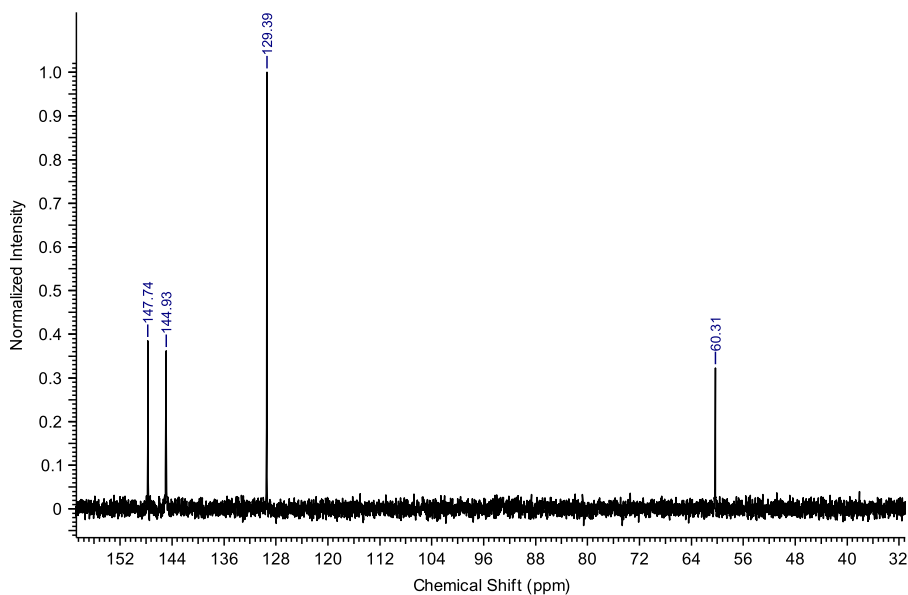


Fig. 8. ^{13}C NMR spectra of $\text{C}_2(\text{Pyr})_2\text{Br}_2$.

condenser and pressure-equalised addition funnel was charged with a solution of 30 mmol of 1,2-dibromoethane, 1,3-dibromopropane, 1,4-dibromobutane, 1,6-dibromohexane, 1,8-dibromooctane or 1,12-dibromododecane in 5 mL of methanol. Then 75 mmol of 1-methylimidazole was added dropwise while stirring at room temperature. The resulting mixture was further heated and stirred at 50 °C for 48 hours. The product was isolated by filtration and purified by recrystallization in methanol/ethyl acetate ($\approx 1:3$ v:v). The resulting product was transferred to a single-necked round-

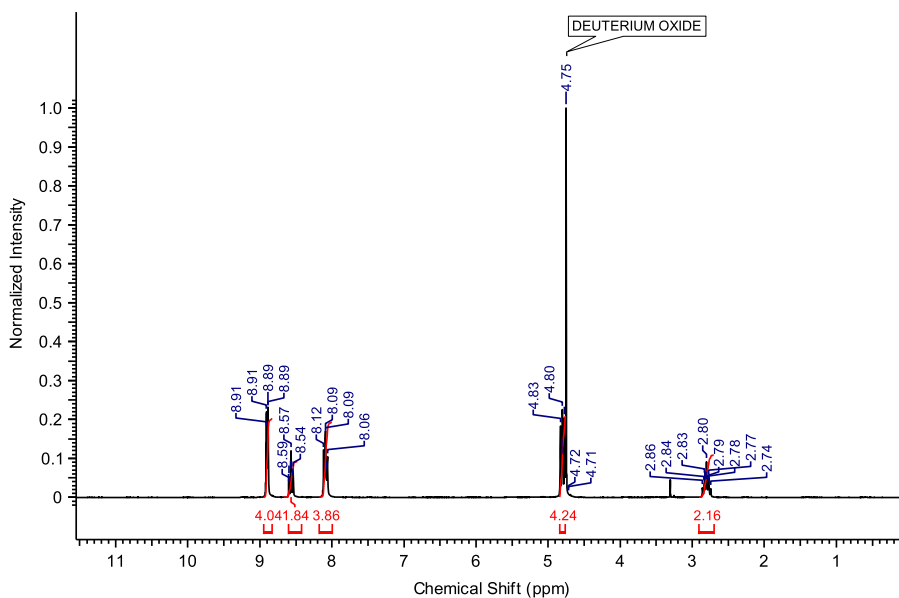


Fig. 9. 1H NMR spectra of $C_3(Pyr)_2Br_2$.

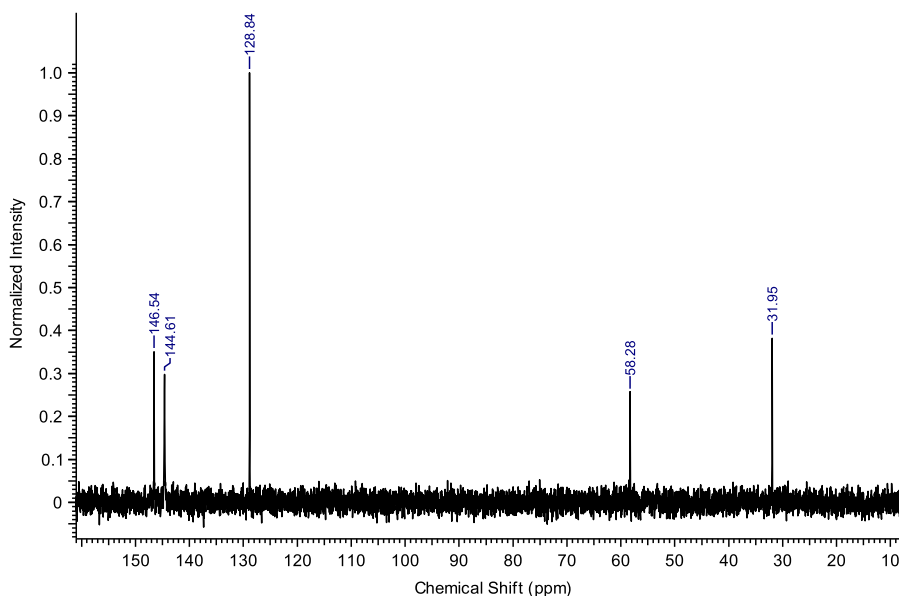


Fig. 10. ^{13}C NMR spectra of $C_3(Pyr)_2Br_2$.

bottomed flask washing with methanol. The solvent was then removed under reduced pressure using a rotary evaporator. Data in agreement with the literature [15].

1-(3-bromopropyl)pyridinium bromide was synthesized following the procedure described in the literature [20]. 100 mmol of pyridine was transferred to a round bottom flask. 150 mmol of 1,3-dibromopropane was added and the resulting mixture was stirred for three days at room

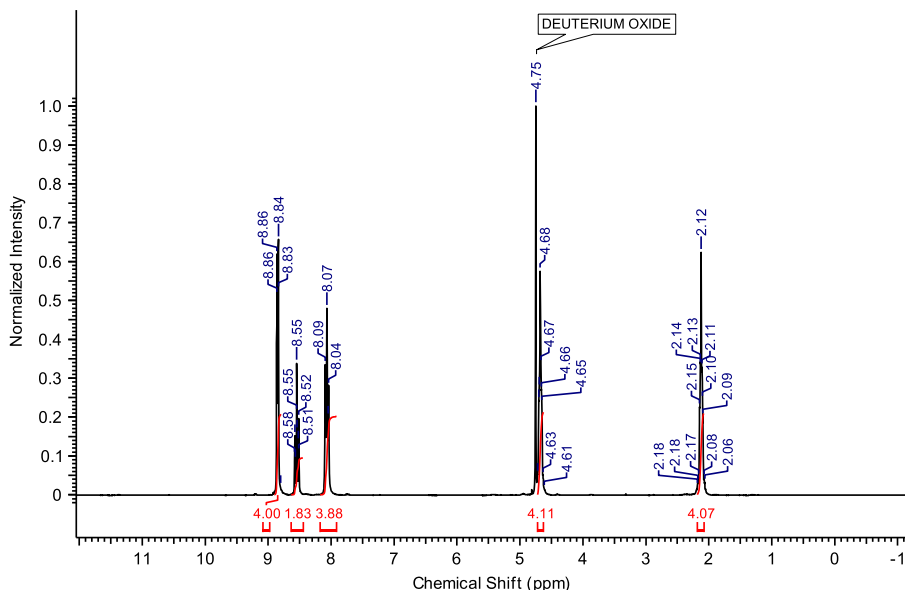


Fig. 11. ^1H NMR spectra of $\text{C}_4(\text{Pyr})_2\text{Br}_2$.

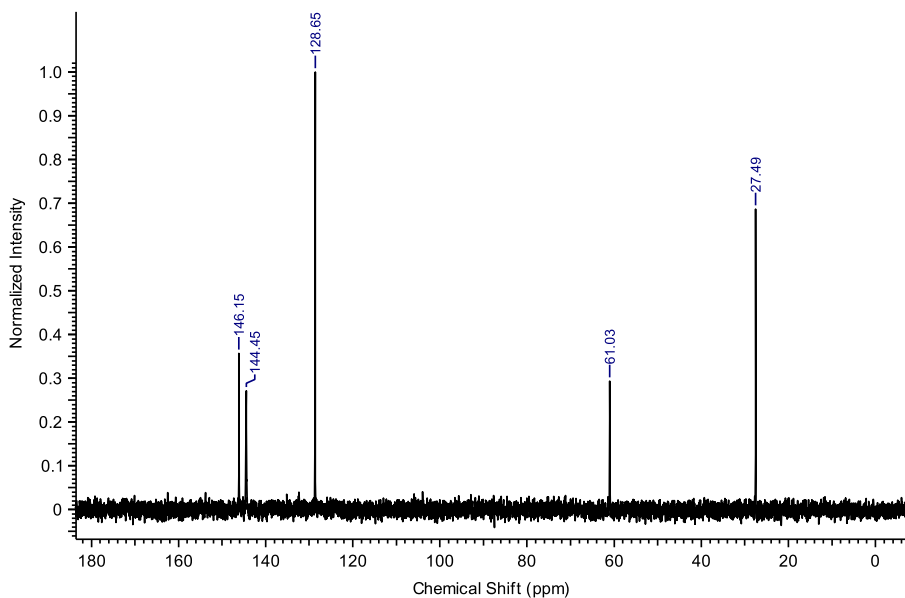


Fig. 12. ^{13}C NMR spectra of $\text{C}_4(\text{Pyr})_2\text{Br}_2$.

temperature. Then, the mixture was washed with ethyl acetate to remove any unreacted reactants and filtered to obtain a white precipitate.

C₃ (Pyr) (MIm) Br₂ and **C₃ (Pyr) (MPyrr) Br₂** were synthesized with the same procedure [20]. A three-neck round bottom flask fitted with a reflux condenser was charged with a solution of 15 mmol of (3-bromopropyl)pyridinium bromide in 20 mL of methanol. Then 18 mmol of 1-methylimidazole or

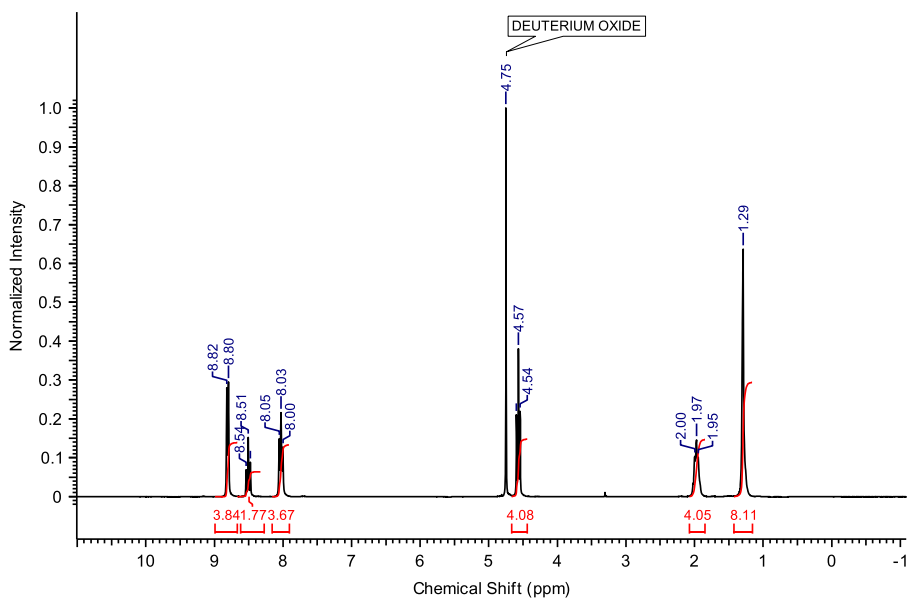


Fig. 13. ¹H NMR spectra of $C_8(Pyr)_2Br_2$.

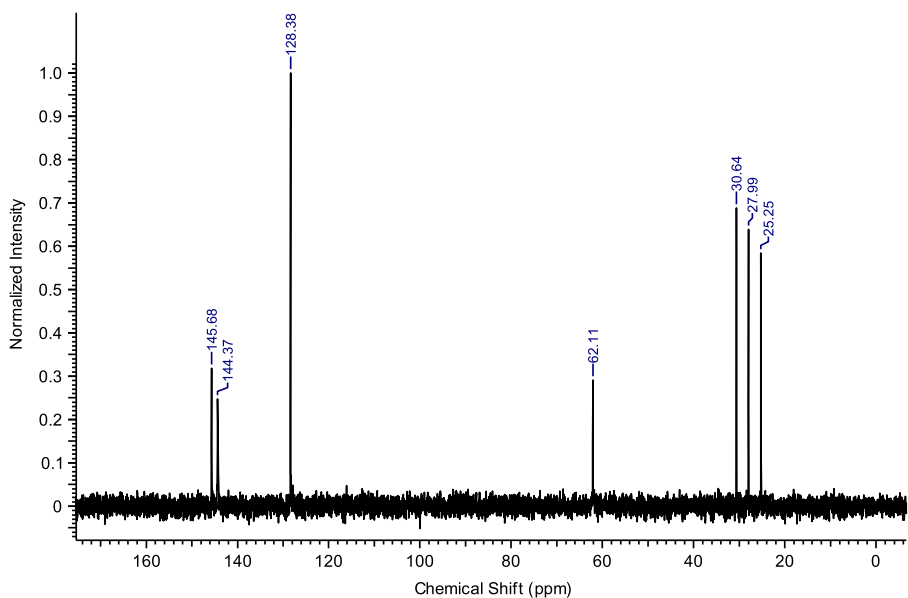


Fig. 14. ¹³C NMR spectra of $C_8(Pyr)_2Br_2$.

19.5 mmol of 1-methylpyrrolidine was added dropwise while stirring at room temperature. The resulting mixture was refluxed while stirring at 50 °C for 48 hours. The resulting solution was recrystallised directly from methanol/ethyl acetate ($\approx 1:5$ v:v) and the product was isolated by filtration.

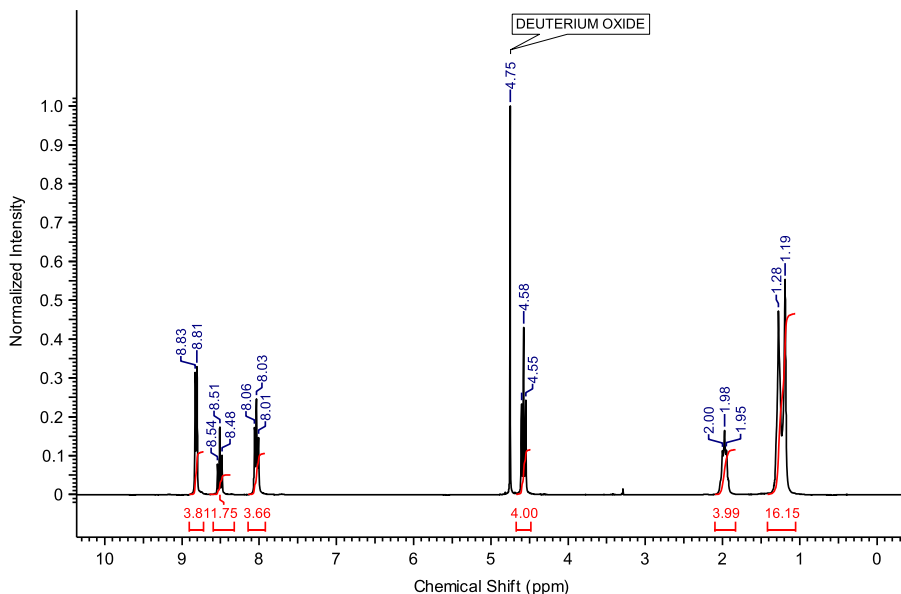


Fig. 15. ^1H NMR spectra of $\text{C}_{12}(\text{Pyr})_2\text{Br}_2$.

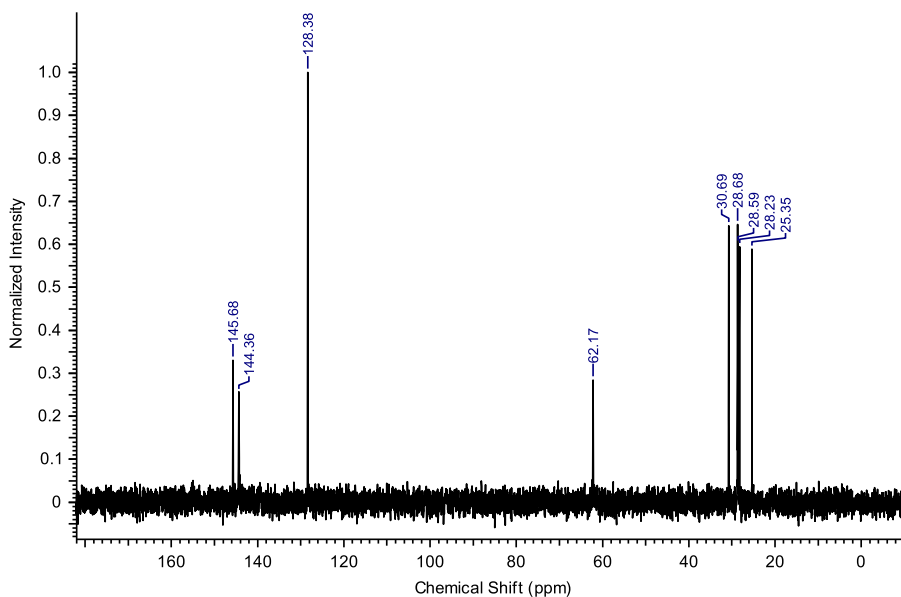


Fig. 16. ^{13}C NMR spectra of $\text{C}_{12}(\text{Pyr})_2\text{Br}_2$.

The structures of the resulting ILs were confirmed by ^1H , ^{13}C and ^{19}F NMR spectroscopy (recorded generally at room temperature on a Jeol model EX270) and mass spectrometry (Bruker MicroTOF 61 spectrometer).

$\text{C}_8(\text{MIm})\text{Br}$: 1-methyl-3-octylimidazolium bromide. $\text{C}_{12}\text{H}_{23}\text{BrN}_2$; from 8.21 g (100 mmol) of 1-methylimidazole and 21.24 g (110 mmol) of 1-bromooctane, 22.98 g (83.49 mmol) of $\text{C}_8(\text{MIm})\text{Br}$ was obtained (yield: 84%); ^1H NMR (270 MHz, CDCl_3): δ 10.29 (s, 1H, N- CH -N), 7.62 (s, 1H, N- CH -CH-

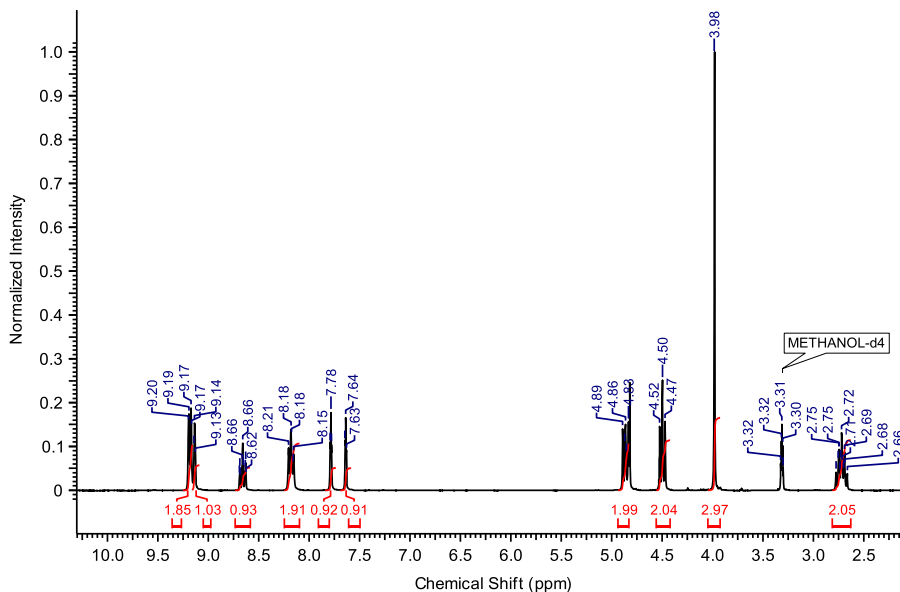


Fig. 17. ^1H NMR spectra of $\text{C}_3(\text{Pyr})(\text{MIm})\text{Br}_2$.

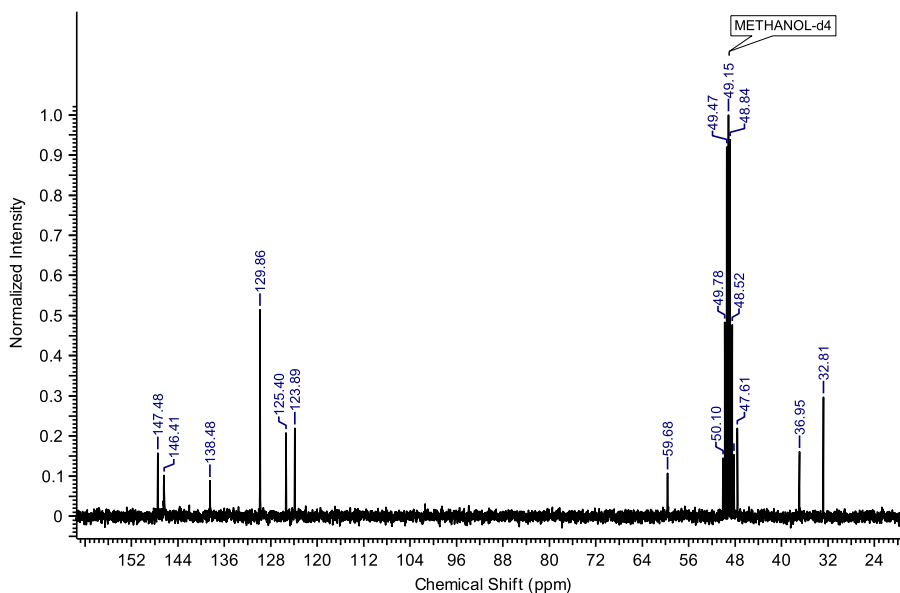


Fig. 18. ^{13}C NMR spectra of $\text{C}_3(\text{Pyr})(\text{MIm})\text{Br}_2$.

N), 7.43 (s, 1H, N-CH-CH-N), 4.27 (t, 2H, N-CH₂-CH₂), 4.08 (s, 3H, N-CH₃), 1.91–1.80 (m, 2H, N-CH₂-CH₂-C₅H₁₀-CH₃), 1.31–1.15 (m, 10H, N-CH₂-CH₂-C₅H₁₀-CH₃), 0.80 (t, 3H, N-CH₂-CH₂-C₅H₁₀-CH₃). ^{13}C NMR (68 MHz, CDCl₃): δ 137.49 (N-CH-N), 123.97 (N-CH-CH-N), 122.21 (N-CH-CH-N), 50.31 (N-CH₂-CH₂), 36.91 (N-CH₃), 31.83 (N-C₅H₁₀-CH₂-CH₂-CH₃), 30.50 (N-CH₂-CH₂-C₅H₁₀-CH₃), 29.18 (N-C₄H₈-CH₂-C₂H₄-CH₃), 29.11 (N-C₃H₆-CH₂-C₃H₆-CH₃), 26.41 (N-C₂H₄-CH₂-C₄H₈-CH₃), 22.73 (N-C₆H₁₂-CH₂-CH₃), 14.24 (N-C₇H₁₄-CH₃). MS m/z molecular ion: calcd. 195.1856; found 195.1864 (cation).

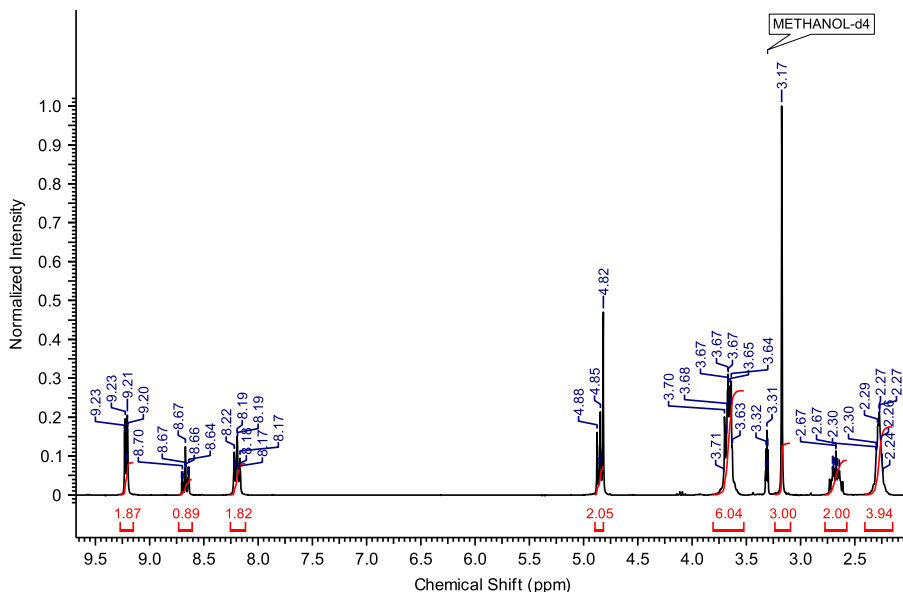


Fig. 19. ^1H NMR spectra of $\text{C}_3(\text{Pyr})(\text{MPyrr})\text{Br}_2$.

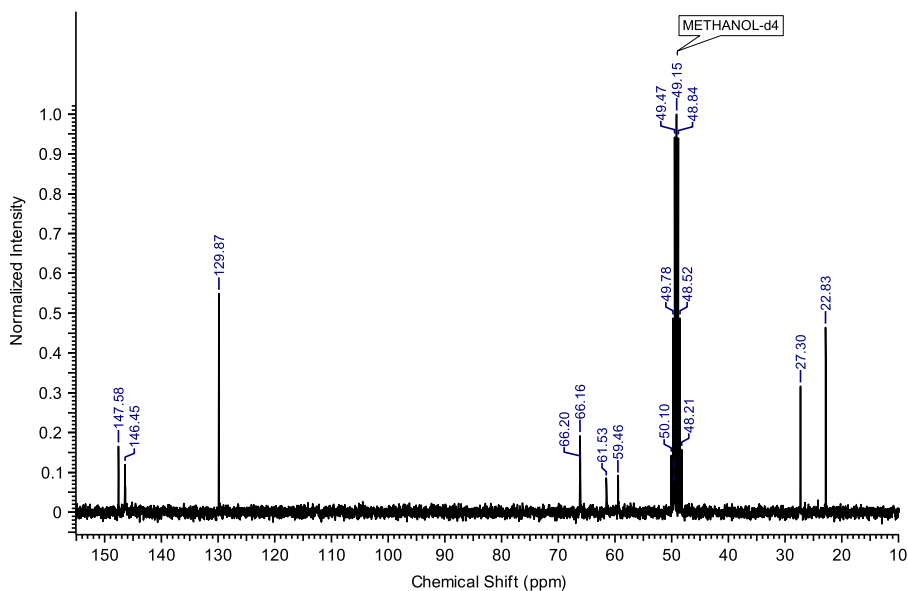


Fig. 20. ^{13}C NMR spectra of $\text{C}_3(\text{Pyr})(\text{MPyrr})\text{Br}_2$.

$\text{C}_8(\text{MPyrr})\text{Br}$: 1-methyl-1-octylpyrrolidinium bromide. $\text{C}_{13}\text{H}_{28}\text{BrN}$; from 20.44 g (240 mmol) of 1-methylpyrrolidine and 55.62 g (288 mmol) of 1-bromooctane, 62.61 g (224.99 mmol) of $\text{C}_8(\text{MPyrr})\text{Br}$ was obtained (yield: 94%); ^1H NMR (270 MHz, CDCl_3): δ 3.83–3.74 (m, 4H, $\text{N-CH}_2\text{-CH}_2\text{-CH}_2\text{-CH}_2\text{-N}$), 3.62–3.56 (m, 2H, $\text{N-CH}_2\text{-CH}_2\text{-C}_5\text{H}_{10}\text{-CH}_3$), 3.24 (s, 3H, N-CH_3), 2.28–2.20 (m, 4H, $\text{N-CH}_2\text{-CH}_2\text{-CH}_2\text{-CH}_2\text{-N}$), 1.77–1.66 (m, 2H, $\text{N-CH}_2\text{-CH}_2\text{-C}_5\text{H}_{10}\text{-CH}_3$), 1.36–1.14 (m, 10H, $\text{N-CH}_2\text{-CH}_2\text{-C}_5\text{H}_{10}\text{-CH}_3$), 0.81 (t, 3H, $\text{N-CH}_2\text{-CH}_2\text{-C}_5\text{H}_{10}\text{-CH}_3$). ^{13}C NMR (68 MHz, CDCl_3): δ 64.66 ($\text{N-CH}_2\text{-CH}_2\text{-CH}_2\text{-CH}_2\text{-N}$), 64.41 ($\text{N-CH}_2\text{-C}_6\text{H}_{12}\text{-CH}_3$), 48.89 (N-CH_3), 31.82 ($\text{N-C}_5\text{H}_{10}\text{-CH}_2\text{-CH}_2\text{-CH}_3$), 29.37 ($\text{N-C}_4\text{H}_8\text{-CH}_2\text{-C}_2\text{H}_4\text{-CH}_3$), 29.20

Table 2

Physical state, colour and melting point or thermal decomposition temperature of the synthesized ILs or molten salts.

Ionic liquid/Molten salt	Physical state at rt ^a	Colour	Melting point temperature (°C)	Thermal decomposition temperature ^b (°C)
C₈ (Mlm) Br	Liquid	Colourless	–	–
C₈ (Mlm) NTf₂	Liquid	Orange	–	–
C₈ (Mlm) SbF₆	Liquid	Light yellow	–	–
C₈ (Pyr) Br	Solid	White	46	–
C₈ (Pyr) NTf₂	Liquid	Light yellow	–	–
C₈ (Pyr) SbF₆	Solid	White	82	–
C₈ (MPyrr) Br	Solid	Light yellow	151	–
C₈ (MPyrr) NTf₂	Liquid	Light yellow	–	–
C₈ (MPyrr) SbF₆	Solid	White	46	–
C₂(Mlm)₂ Br₂	Solid	White	229	–
C₃(Mlm)₂ Br₂	Solid	White	132	–
C₄(Mlm)₂ Br₂	Liquid	Colourless	–	–
C₆(Mlm)₂ Br₂	Solid	White	156	–
C₈(Mlm)₂ Br₂	Solid	White	75	–
C₃(MPyrr)₂ Br₂	Liquid	Orange	–	–
C₄(MPyrr)₂ Br₂	Solid	Light brown	–	314
C₆(MPyrr)₂ Br₂	Solid	White	173	–
C₈(MPyrr)₂ Br₂	Solid	White	181	–
C₂(Pyr)₂ Br₂	Solid	White	–	300
C₃(Pyr)₂ Br₂	Solid	White	241	–
C₄(Pyr)₂ Br₂	Solid	White	–	262
C₆(Pyr)₂ Br₂	Solid	White	–	265
C₈(Pyr)₂ Br₂	Solid	White	193	–
C₁₂(Pyr)₂ Br₂	Solid	White	140	–
C₃(Pyr)(Mlm) Br₂	Solid	White	104	–
C₃(Pyr)(MPyrr) Br₂	Solid	Light brown	–	231

^a Room temperature.^b Decomposition temperature is reached before the melting of the ionic liquid or the molten salt.

(N-C₃H₆-CH₂-C₃H₆-CH₃), 26.61 (N-C₂H₄-CH₂-C₄H₈-CH₃), 24.31 (N-CH₂-CH₂-C₅H₁₀-CH₃), 22.74 (N-C₆H₁₂-CH₂-CH₃), 21.87 (N-CH₂-CH₂-CH₂-CH₂-N), 14.24 (N-C₇H₁₄-CH₃). MS m/z molecular ion: calcd. 198.2216; found 198.2227 (cation).

C₈(Pyr)Br: 1-octylpyridinium bromide. C₁₃H₂₂BrN; from 18.98 g (240 mmol) of pyridine and 55.62 g (288 mmol) of 1-bromooctane, 57.53 g (211.34 mmol) of C₈(Pyr)Br was obtained (yield: 88%); ¹H NMR (270 MHz, CDCl₃): δ 9.54 (d, 2H, N-CH-CH-CH-CH-N), 8.50 (t, 1H, N-CH-CH-CH-CH-CH-N), 8.13 (t, 2H, N-CH-CH-CH-CH-CH-CH-N), 4.93 (t, 2H, N-CH₂-CH₂-C₅H₁₀-CH₃), 2.02–1.95 (m, 2H, N-CH₂-CH₂-C₅H₁₀-CH₃), 1.33–1.09 (m, 10H, N-CH₂-CH₂-C₅H₁₀-CH₃), 0.77 (t, 3H, N-CH₂-CH₂-C₅H₁₀-CH₃). ¹³C NMR (68 MHz, CDCl₃): δ 145.59 (N-CH-CH-CH-CH-CH-N), 145.26 (N-CH-CH-CH-CH-CH-CH-N), 128.82 (N-CH-CH-CH-CH-CH-CH-CH-N), 62.36 (N-CH₂-C₆H₁₂-CH₃), 32.15 (N-C₅H₁₀-CH₂-CH₂-CH₃), 31.89 (N-CH₂-CH₂-C₅H₁₀-CH₃), 29.22 (N-C₃H₆-CH₂-CH₂-C₂H₄-CH₃), 26.28 (N-C₂H₄-CH₂-C₄H₈-CH₃), 22.78 (N-C₆H₁₂-CH₂-CH₃), 14.18 (N-C₇H₁₄-CH₃). MS m/z molecular ion: calcd. 192.1747; found 192.1753 (cation).

C₈(Mlm)NTf₂: 1-methyl-3-octylimidazolium bis (trifluoromethylsulfonyl) imide. C₁₄H₂₃F₆N₃O₄S₂; from 4.04 g (14.68 mmol) of C₈(Mlm)Br and 5.06 g (17.62 mmol) of LiNTf₂, 6.91 g (14.53 mmol) of C₈(Mlm)NTf₂ was obtained (yield: 99%); ¹H NMR (270 MHz, CD₄O): δ 8.87 (s, 1H, N-CH-N), 7.58 (d, 2H, N-CH-CH-N), 4.20 (t, 2H, N-CH₂-CH₂), 3.92 (s, 3H, N-CH₃), 1.94–1.83 (m, 2H, N-CH₂-CH₂-C₅H₁₀-CH₃), 1.37–1.28 (m, 10H, N-CH₂-CH₂-C₅H₁₀-CH₃), 0.90 (t, 3H, N-CH₂-CH₂-C₅H₁₀-CH₃). ¹³C NMR (68 MHz, CD₄O): δ 137.33 (N-CH-N), 125.05 (N-CH-CH-N), 123.75 (N-CH-CH-N), 50.98 (N-CH₂-CH₂), 36.54 (N-CH₃), 33.01 (N-CH₂-CH₂-C₅H₁₀-CH₃), 31.23 (N-C₅H₁₀-CH₂-CH₂-CH₃), 30.30 (N-C₄H₈-CH₂-C₂H₄-CH₃), 30.16 (N-C₃H₆-CH₂-C₃H₆-CH₃), 27.38 (N-C₂H₄-CH₂-C₄H₈-CH₃), 23.79 (N-C₆H₁₂-CH₂-CH₃), 14.52 (N-C₇H₁₄-CH₃). ¹⁹F NMR (200 MHz, CD₄O) δ –81.1 ppm. MS m/z molecular ion: calcd. 195.1856; found 195.1863 (cation), calcd. 279.9178; found 279.9180 (anion).

C₈(MPyrr)NTf₂: 1-methyl-1-octylpyrrolidinium bis (trifluoromethylsulfonyl) imide. C₁₅H₂₈F₆N₂O₄S₂; from 1.40 g (5.03 mmol) of C₈(MPyrr)Br and 1.73 g (6.04 mmol) of LiNTf₂, 2.34 g (4.89 mmol) of C₈(MPyrr)

NTf₂ was obtained (yield: 97%); ¹H NMR (270 MHz, CDCl₃): δ 3.57–3.45 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-N), 3.32–3.26 (m, 2H, N-CH₂-CH₂-C₅H₁₀-CH₃), 3.03 (s, 3H, N-CH₃), 2.31–2.20 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-N), 1.80–1.71 (m, 2H, N-CH₂-CH₂-C₅H₁₀-CH₃), 1.38–1.21 (m, 10H, N-CH₂-CH₂-C₅H₁₀-CH₃), 0.88 (t, 3H, N-CH₂-CH₂-C₅H₁₀-CH₃). ¹³C NMR (68 MHz, CDCl₃): δ 65.14 (N-CH₂-CH₂-CH₂-CH₂-N), 64.87 (N-CH₂-C₆H₁₂-CH₃), 48.69 (N-CH₃), 31.87 (N-C₅H₁₀-CH₂-CH₂-CH₃), 29.22 (N-C₃H₆-CH₂-CH₂-C₂H₄-CH₃), 24.14 (N-CH₂-CH₂-C₅H₁₀-CH₃), 22.85 (N-C₆H₁₂-CH₂-CH₃), 21.81 (N-CH₂-CH₂-CH₂-CH₂-N), 14.32 (N-C₇H₁₄-CH₃). ¹⁹F NMR (200 MHz, CDCl₃) δ –79.5 ppm. MS m/z molecular ion: calcd. 198.2216; found 198.2227 (cation), calcd. 279.9178; found 279.9179 (anion).

C₈(Pyr)NTf₂: 1-octylpyridinium bis (trifluoromethylsulfonyl) imide. C₁₅H₂₂F₆N₂O₄S₂; from 1.36 g (5 mmol) of C₈(Pyr)Br and 1.72 g (6 mmol) of LiNTf₂, 2.21 g (4.68 mmol) of C₈(Pyr)NTf₂ was obtained (yield: 94%); ¹H NMR (270 MHz, CDCl₃): δ 8.83 (d, 2H, N-CH-CH-CH-CH-CH-N), 8.48 (t, 1H, N-CH-CH-CH-CH-CH-N), 8.06 (t, 2H, N-CH-CH-CH-CH-CH-N), 4.59 (t, 2H, N-CH₂-CH₂-C₅H₁₀-CH₃), 2.05–1.94 (m, 2H, N-CH₂-CH₂-C₅H₁₀-CH₃), 1.40–1.19 (m, 10H, N-CH₂-CH₂-C₅H₁₀-CH₃), 0.86 (t, 3H, N-CH₂-CH₂-C₅H₁₀-CH₃). ¹³C NMR (68 MHz, CDCl₃): δ 145.75 (N-CH-CH-CH-CH-CH-N), 144.71 (N-CH-CH-CH-CH-CH-N), 128.99 (N-CH-CH-CH-CH-CH-N), 62.97 (N-CH₂-C₆H₁₂-CH₃), 31.87 (N-CH₂-CH₂-C₃H₆-CH₂-CH₂-CH₃), 29.17 (N-C₄H₈-CH₂-C₂H₄-CH₃), 29.08 (N-C₃H₆-CH₂-C₃H₆-CH₃), 26.23 (N-C₂H₄-CH₂-C₄H₈-CH₃), 22.83 (N-C₆H₁₂-CH₂-CH₃), 14.30 (N-C₇H₁₄-CH₃). ¹⁹F NMR (200 MHz, CDCl₃) δ –79.5 ppm. MS m/z molecular ion: calcd. 192.1747; found 192.1742 (cation), calcd. 279.9178; found 279.9188 (anion).

C₈(MIm)SbF₆: 1-methyl-3-octylimidazolium hexafluoroantimonate. C₁₂H₂₃F₆N₂Sb; from 1.35 g (4.92 mmol) of C₈(MIm)Br and 1.56 g (6.01 mmol) of NaSbF₆, 1.93 g (4.48 mmol) of C₈(MIm)SbF₆ was obtained (yield: 91%); ¹H NMR (270 MHz, DMSO-d₆): δ 9.09 (s, 1H, N-CH-N), 7.72 (d, 2H, N-CH-CH-N), 4.14 (t, 2H, N-CH₂-CH₂), 3.84 (s, 3H, N-CH₃), 1.82–1.73 (m, 2H, N-CH₂-CH₂-C₅H₁₀-CH₃), 1.32–1.18 (m, 10H, N-CH₂-CH₂-C₅H₁₀-CH₃), 0.86 (t, 3H, N-CH₂-CH₂-C₅H₁₀-CH₃). ¹³C NMR (68 MHz, DMSO-d₆): δ 136.47 (N-CH-N), 123.58 (N-CH-CH-N), 122.24 (N-CH-CH-N), 48.77 (N-CH₂-CH₂), 35.72 (N-CH₃), 31.15 (N-CH₂-CH₂-C₅H₁₀-CH₃), 29.35 (N-C₅H₁₀-CH₂-CH₂-CH₃), 28.45 (N-C₄H₈-CH₂-C₂H₄-CH₃), 28.32 (N-C₃H₆-CH₂-C₃H₆-CH₃), 25.47 (N-C₂H₄-CH₂-C₄H₈-CH₃), 22.04 (N-C₆H₁₂-CH₂-CH₃), 13.91 (N-C₇H₁₄-CH₃). ¹⁹F NMR (200 MHz, DMSO-d₆, at –40 °C) δ –121.4 ppm. MS m/z molecular ion: calcd. 195.1856; found 195.1865 (cation), calcd. 234.8948; found 234.8935 (anion).

C₈(MPyrr)SbF₆: 1-methyl-1-octylpyrrolidinium hexafluoroantimonate. C₁₃H₂₈F₆NSb; from 1.40 g (5.03 mmol) of C₈(MPyrr)Br and 1.56 g (6.03 mmol) of NaSbF₆, 2.05 g (4.72 mmol) of C₈(MPyrr)SbF₆ was obtained (yield: 94%); ¹H NMR (270 MHz, DMSO-d₆): δ 3.51–3.34 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-N), 3.31–3.23 (m, 2H, N-CH₂-CH₂-C₅H₁₀-CH₃), 2.97 (s, 3H, N-CH₃), 2.13–2.02 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-N), 1.74–1.63 (m, 2H, N-CH₂-CH₂-C₅H₁₀-CH₃), 1.35–1.23 (m, 10H, N-CH₂-CH₂-C₅H₁₀-CH₃), 0.87 (t, 3H, N-CH₂-CH₂-C₅H₁₀-CH₃). ¹³C NMR (68 MHz, DMSO-d₆): δ 63.38 (N-CH₂-CH₂-CH₂-CH₂-N), 63.09 (N-CH₂-C₆H₁₂-CH₃), 47.46 (N-CH₃), 31.14 (N-C₅H₁₀-CH₂-CH₂-CH₃), 28.43 (N-C₃H₆-CH₂-CH₂-C₂H₄-CH₃), 25.89 (N-CH₂-CH₂-C₅H₁₀-CH₃), 22.88 (N-C₆H₁₂-CH₂-CH₃), 22.01 (N-CH₂-CH₂-CH₂-CH₂-N), 21.05 (N-CH₂-CH₂-CH₂-CH₂-N), 13.91 (N-C₇H₁₄-CH₃). ¹⁹F NMR (200 MHz, DMSO-d₆, at –40 °C) δ –120.5 ppm. MS m/z molecular ion: calcd. 198.2216; found 198.2225 (cation), calcd. 234.8948; found 234.8939 (anion).

C₈(Pyr)SbF₆: 1-octylpyridinium hexafluoroantimonate. C₁₃H₂₂F₆NSb; from 1.37 g (5 mmol) of C₈(Pyr)Br and 1.56 g (6 mmol) of NaSbF₆, 2.00 g (4.67 mmol) of C₈(Pyr)SbF₆ was obtained (yield: 93%); ¹H NMR (270 MHz, DMSO-d₆): δ 9.08 (d, 2H, N-CH-CH-CH-CH-CH-N), 8.60 (t, 1H, N-CH-CH-CH-CH-CH-N), 8.16 (t, 2H, N-CH-CH-CH-CH-CH-N), 4.59 (t, 2H, N-CH₂-CH₂-C₅H₁₀-CH₃), 1.97–1.86 (m, 2H, N-CH₂-CH₂-C₅H₁₀-CH₃), 1.33–1.20 (m, 10H, N-CH₂-CH₂-C₅H₁₀-CH₃), 0.85 (t, 3H, N-CH₂-CH₂-C₅H₁₀-CH₃). ¹³C NMR (68 MHz, DMSO-d₆): δ 145.45 (N-CH-CH-CH-CH-CH-N), 144.72 (N-CH-CH-CH-CH-CH-N), 128.08 (N-CH-CH-CH-CH-CH-N), 60.80 (N-CH₂-C₆H₁₂-CH₃), 31.11 (N-C₅H₁₀-CH₂-CH₂-CH₃), 30.69 (N-CH₂-CH₂-C₅H₁₀-CH₃), 28.42 (N-C₄H₈-CH₂-C₂H₄-CH₃), 28.33 (N-C₃H₆-CH₂-C₃H₆-CH₃), 25.39 (N-C₂H₄-CH₂-C₄H₈-CH₃), 22.03 (N-C₆H₁₂-CH₂-CH₃), 13.92 (N-C₇H₁₄-CH₃). ¹⁹F NMR (200 MHz, DMSO-d₆, at –40 °C) δ –118.7 ppm. MS m/z molecular ion: calcd. 192.1747; found 192.1737 (cation), calcd. 234.8948; found 234.8943 (anion).

C₂(MIm)₂Br₂: 1,2-bis(3-methylimidazolium-1-yl)ethane dibromide. C₁₀H₁₆Br₂N₄; from 4.93 g (60 mmol) of 1-methylimidazole and 5.64 g (30 mmol) of 1,2-dibromoethane, 3.74 g (10.62 mmol) of C₂(MIm)₂Br₂ was obtained (yield: 35%); ¹H NMR (270 MHz, DMSO-d₆): δ 9.25 (s, 2H, N-CH-N), 7.75 (d, 4H, N-CH-CH-N), 4.76 (s, 4H, N-CH₂-CH₂-N), 3.86 (s, 6H, N-CH₃). ¹³C NMR (68 MHz, DMSO-d₆):

δ 137.16 (N-CH-N), 123.78 (N-CH-CH-N), 122.33 (N-CH-CH-N), 48.30 (N-CH₂-CH₂-N), 36.01 (N-CH₃). MS m/z molecular ion: calcd. 192.1371; found 192.1347 (cation²⁺).

C₃ (MIm)₂ Br₂: 1,3-bis(3-methylimidazolium-1-yl)propane dibromide. C₁₁H₁₈Br₂N₄; from 4.93 g (60 mmol) of 1-methylimidazole and 6.06 g (30 mmol) of 1,3-dibromopropane, 10.31 (28.16 mmol) g of C₃ (MIm)₂ Br₂ was obtained (yield: 94%); ¹H NMR (270 MHz, D₂O): δ 8.78 (s, 2H, N-CH-N), 7.47 (d, 4H, N-CH-CH-N), 4.30 (t, 4H, N-CH₂-CH₂-CH₂-N), 3.88 (s, 6H, N-CH₃), 2.56–2.45 (m, 2H, N-CH₂-CH₂-CH₂-N). ¹³C NMR (68 MHz, D₂O): δ 136.38 (N-CH-N), 124.16 (N-CH-CH-N), 122.36 (N-CH-CH-N), 46.49 (N-CH₂-CH₂-CH₂-N), 36.10 (N-CH₃), 29.95 (N-CH₂-CH₂-CH₂-N). MS m/z molecular ion: calcd. 103.0730; found 103.0727 (cation⁺).

C₄ (MIm)₂ Br₂: 1,4-bis(3-methylimidazolium-1-yl)butane dibromide. C₁₂H₂₀Br₂N₄; from 4.93 g (60 mmol) of 1-methylimidazole and 6.48 g (30 mmol) of 1,4-dibromobutane, 7.85 g (20.65 mmol) of C₄ (MIm)₂ Br₂ was obtained (yield: 69%); ¹H NMR (270 MHz, D₂O): δ 8.70 (s, 2H, N-CH-N), 7.42 (d, 4H, N-CH-CH-N), 4.21 (t, 4H, N-CH₂-C₂H₄-CH₂-N), 3.85 (s, 6H, N-CH₃), 1.90–1.84 (m, 4H, N-CH₂-C₂H₄-CH₂-N). ¹³C NMR (68 MHz, D₂O): δ 136.16 (N-CH-N), 123.92 (N-CH-CH-N), 122.28 (N-CH-CH-N), 48.92 (N-CH₂-C₂H₄-CH₂-N), 35.97 (N-CH₃), 26.41 (N-CH₂-C₂H₄-CH₂-N). MS m/z molecular ion: calcd. 110.0812; found 110.0805 (cation⁺).

C₆ (MIm)₂ Br₂: 1,6-bis(3-methylimidazolium-1-yl)hexane dibromide. C₁₄H₂₄Br₂N₄; from 4.93 g (60 mmol) of 1-methylimidazole and 7.32 g (30 mmol) of 1,6-dibromohexane, 11.26 g (27.59 mmol) of C₆ (MIm)₂ Br₂ was obtained (yield: 92%); ¹H NMR (270 MHz, DMSO-d₆): δ 9.31 (s, 2H, N-CH-N), 7.80 (d, 4H, N-CH-CH-N), 4.19 (t, 4H, N-CH₂-C₄H₈-CH₂-N), 3.87 (s, 6H, N-CH₃), 1.81–1.76 (m, 4H, N-CH₂-CH₂-C₂H₄-CH₂-N), 1.29–1.24 (m, 4H, N-C₂H₄-C₂H₄-C₂H₄-N). ¹³C NMR (68 MHz, DMSO-d₆): δ 136.50 (N-CH-N), 123.53 (N-CH-CH-N), 122.24 (N-CH-CH-N), 48.53 (N-CH₂-C₄H₈-CH₂-N), 35.77 (N-CH₃), 29.07 (N-CH₂-CH₂-C₂H₄-CH₂-N), 24.76 (N-C₂H₄-C₂H₄-C₂H₄-N). MS m/z molecular ion: calcd. 124.0997; found 124.0985 (cation⁺).

C₈ (MIm)₂ Br₂: 1,8-bis(3-methylimidazolium-1-yl)octane dibromide. C₁₆H₂₈Br₂N₄; from 4.93 g (60 mmol) of 1-methylimidazole and 8.16 g (30 mmol) of 1,8-dibromooctane, 12.90 g (29.57 mmol) of C₈ (MIm)₂ Br₂ was obtained (yield: 99%); ¹H NMR (270 MHz, D₂O): δ 8.66 (s, 2H, N-CH-N), 7.40 (d, 4H, N-CH-CH-N), 4.14 (t, 4H, N-CH₂-C₄H₈-CH₂-N), 3.84 (s, 6H, N-CH₃), 1.86–1.76 (m, 4H, N-CH₂-CH₂-C₄H₈-CH₂-N), 1.30–1.18 (m, 8H, N-C₂H₄-C₄H₈-C₂H₄-N). ¹³C NMR (68 MHz, D₂O): δ 135.97 (N-CH-N), 123.65 (N-CH-CH-N), 122.67 (N-CH-CH-N), 49.70 (N-CH₂-C₆H₁₂-CH₂-N), 35.85 (N-CH₃), 29.33 (N-CH₂-CH₂-C₄H₈-CH₂-N), 28.02 (N-C₂H₄-CH₂-C₄H₈-CH₂-N), 25.38 (N-C₃H₆-C₂H₄-C₃H₆-N). MS m/z molecular ion: calcd. 138.1151; found 138.1155 (cation⁺).

C₃ (MPyrr)₂ Br₂: 1,3-bis(1-methylpyrrolidinium-1-yl)propane dibromide. C₁₃H₂₈Br₂N₂; from 5.36 g (63 mmol) of 1-methylpyrrolidine and 6.06 g (30 mmol) of 1,3-dibromopropane, 10.28 g (27.62 mmol) of C₃ (MPyrr)₂ Br₂ was obtained (yield: 92%); ¹H NMR (270 MHz, D₂O): δ 3.65–3.41 (m, 12H, N-CH₂-CH₂), 3.10 (s, 6H, N-CH₃), 2.44–2.31 (m, 2H, N-CH₂-CH₂-CH₂-N), 2.28–2.17 (m, 8H, N-CH₂-CH₂-CH₂-N). ¹³C NMR (68 MHz, D₂O): δ 65.09 (N-CH₂-CH₂-CH₂-N), 60.46 (N-CH₂-CH₂-CH₂-N), 48.51 (N-CH₃), 21.52 (N-CH₂-CH₂-CH₂-N), 19.13 (N-CH₂-CH₂-CH₂-N). MS m/z molecular ion: calcd. 106.1123; found 106.1111 (cation⁺).

C₄ (MPyrr)₂ Br₂: 1,4-bis(1-methylpyrrolidinium-1-yl)butane dibromide. C₁₄H₃₀Br₂N₂; from 5.36 g (63 mmol) of 1-methylpyrrolidine and 6.48 g (30 mmol) of 1,4-dibromobutane, 8.04 g (20.82 mmol) of C₄ (MPyrr)₂ Br₂ was obtained (yield: 69%); ¹H NMR (270 MHz, D₂O): δ 3.54–3.37 (m, 12H, N-CH₂-CH₂), 3.02 (s, 6H, N-CH₃), 2.21–2.14 (m, 8H, N-CH₂-CH₂-CH₂-CH₂-N ring), 1.90–1.84 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-N alkyl linkage chain). ¹³C NMR (68 MHz, D₂O): δ 64.67 (N-CH₂-CH₂-CH₂-CH₂-N ring), 63.32 (N-CH₂-CH₂-CH₂-CH₂-N alkyl linkage chain), 48.29 (N-CH₃), 21.47 (N-CH₂-CH₂-CH₂-CH₂-N ring), 20.66 (N-CH₂-CH₂-CH₂-CH₂-N alkyl linkage chain). MS m/z molecular ion: calcd. 113.1201; found 113.1187 (cation⁺).

C₆ (MPyrr)₂ Br₂: 1,6-bis(1-methylpyrrolidinium-1-yl)hexane dibromide. C₁₆H₃₄Br₂N₂; from 5.36 g (63 mmol) of 1-methylpyrrolidine and 7.32 g (30 mmol) of 1,6-dibromohexane, 11.68 g (29.18 mmol) of C₆ (MPyrr)₂ Br₂ was obtained (yield: 94%); ¹H NMR (270 MHz, D₂O): δ 3.51–3.27 (m, 12H, N-CH₂-CH₂), 3.00 (s, 6H, N-CH₃), 2.22–2.11 (m, 8H, N-CH₂-CH₂-CH₂-CH₂-N), 1.85–1.73 (m, 4H, N-CH₂-CH₂-C₂H₄-CH₂-N), 1.43–1.37 (m, 4H, N-C₂H₄-C₂H₄-C₂H₄-N). ¹³C NMR (68 MHz, D₂O): δ 64.48 (N-CH₂-CH₂-CH₂-CH₂-N), 64.22 (N-CH₂-C₄H₆-CH₂-N), 48.27 (N-CH₃), 25.47 (N-C₂H₄-C₂H₄-C₂H₄-N),

23.18 (N-CH₂-CH₂-C₂H₄-CH₂-CH₂-N), 21.48 (N-CH₂-CH₂-CH₂-CH₂-N). MS m/z molecular ion: calcd. 127.1357; found 127.1315 (cation⁺).

C₈ (MPyrr)₂ Br₂: 1,8-bis(1-methylpyrrolidinium-1-yl)octane dibromide. C₁₈H₃₈Br₂N₂; from 5.36 g (63 mmol) of 1-methylpyrrolidine and 8.16 g (30 mmol) of 1,8-dibromooctane, 12.63 g (28.55 mmol) of C₈ (MPyrr)₂ Br₂ was obtained (yield: 95%); ¹H NMR (270 MHz, D₂O): δ 3.53–3.25 (m, 12H, N-CH₂-CH₂), 2.99 (s, 6H, N-CH₃), 2.23–2.10 (m, 8H, N-CH₂-CH₂-CH₂-CH₂-N), 1.81–1.70 (m, 4H, N-CH₂-CH₂-C₄H₈-CH₂-CH₂-N), 1.34 (s, 8H, N-C₂H₄-C₄H₈-C₂H₄-N). ¹³C NMR (68 MHz, D₂O): δ 64.46 (N-CH₂-CH₂-CH₂-CH₂-N), 64.42 (N-CH₂-C₆H₁₂-CH₂-N), 48.25 (N-CH₃), 28.10 (N-C₃H₆-C₂H₄-C₃H₆-N), 25.70 (N-C₂H₄-CH₂-C₂H₄-CH₂-C₂H₄-N), 23.31 (N-CH₂-CH₂-C₄H₈-CH₂-CH₂-N), 21.47 (N-CH₂-CH₂-CH₂-CH₂-N). MS m/z molecular ion: calcd. 141.1488; found 141.1491 (cation⁺).

C₂ (Pyr)₂ Br₂: 1,2-bis(pyridinium-1-yl)ethane dibromide. C₁₂H₁₄Br₂N₂; from 5.93 g (75 mmol) of pyridine and 5.64 g (30 mmol) of 1,2-dibromoethane, 5.96 g (17.22 mmol) of C₂ (Pyr)₂ Br₂ was obtained (yield: 57%); ¹H NMR (270 MHz, D₂O): δ 8.84 (d, 4H, N-CH-CH), 8.65 (t, 2H, N-CH-CH-CH-CH-CH-N), 8.11 (t, 4H, N-CH-CH-CH-CH-CH-CH-N), 5.34 (s, 4H, N-CH₂-CH₂-N). ¹³C NMR (68 MHz, D₂O): δ 147.74 (N-CH-CH-CH-CH-CH-N), 144.93 (N-CH-CH-CH-CH-CH-N), 129.39 (N-CH-CH-CH-CH-CH-N), 60.31 (N-CH₂-CH₂-N). MS m/z molecular ion: calcd. 186.1153; found 186.1165 (cation²⁺).

C₃ (Pyr)₂ Br₂: 1,3-bis(pyridinium-1-yl)propane dibromide. C₁₃H₁₆Br₂N₂; from 5.93 g (75 mmol) of pyridine and 6.06 g (30 mmol) of 1,3-dibromopropane, 10.42 g (28.94 mmol) of C₃ (Pyr)₂ Br₂ was obtained (yield: 96%); ¹H NMR (270 MHz, D₂O): δ 8.90 (d, 4H, N-CH-CH), 8.57 (t, 2H, N-CH-CH-CH-CH-CH-N), 8.09 (t, 4H, N-CH-CH-CH-CH-CH-CH-N), 4.80 (t, 4H, N-CH₂-CH₂-CH₂-N), 2.86–2.75 (m, 2H, N-CH₂-CH₂-CH₂-N). ¹³C NMR (68 MHz, D₂O): δ 146.54 (N-CH-CH-CH-CH-CH-CH-N), 144.62 (N-CH-CH-CH-CH-CH-CH-N), 128.85 (N-CH-CH-CH-CH-CH-CH-N), 58.30 (N-CH₂-CH₂-CH₂-N), 31.96 (N-CH₂-CH₂-CH₂-N). MS m/z molecular ion: calcd. 100.0654; found 100.0672 (cation⁺).

C₄ (Pyr)₂ Br₂: 1,4-bis(pyridinium-1-yl)butane dibromide. C₁₄H₁₈Br₂N₂; from 5.93 g (75 mmol) of pyridine and 6.48 g (30 mmol) of 1,4-dibromobutane, 10.22 g (27.32 mmol) of C₄ (Pyr)₂ Br₂ was obtained (yield: 91%); ¹H NMR (270 MHz, D₂O): δ 8.85 (d, 4H, N-CH-CH), 8.54 (t, 2H, N-CH-CH-CH-CH-CH-N), 8.07 (t, 4H, N-CH-CH-CH-CH-CH-CH-N), 4.72–4.61 (m, 4H, N-CH₂-C₂H₄-CH₂-N), 2.18–2.06 (m, 4H, N-CH₂-C₂H₄-CH₂-N). ¹³C NMR (68 MHz, D₂O): δ 146.15 (N-CH-CH-CH-CH-CH-CH-N), 144.45 (N-CH-CH-CH-CH-CH-CH-N), 128.65 (N-CH-CH-CH-CH-CH-CH-N), 61.03 (N-CH₂-C₂H₄-CH₂-N), 27.49 (N-CH₂-C₂H₄-CH₂-N). MS m/z molecular ion: calcd. 107.0732; found 107.0696 (cation⁺).

C₆ (Pyr)₂ Br₂: 1,6-bis(pyridinium-1-yl)hexane dibromide. C₁₆H₂₂Br₂N₂; from 5.93 g (75 mmol) of pyridine and 7.32 g (30 mmol) of 1,6-dibromohexane, 11.55 g (28.72 mmol) of C₆ (Pyr)₂ Br₂ was obtained (yield: 96%); ¹H NMR (270 MHz, D₂O): δ 8.83 (d, 4H, N-CH-CH), 8.53 (t, 2H, N-CH-CH-CH-CH-CH-N), 8.05 (t, 4H, N-CH-CH-CH-CH-CH-CH-N), 4.60 (t, 4H, N-CH₂-C₄H₈-CH₂-N), 2.06–1.94 (m, 4H, N-CH₂-CH₂-C₂H₄-CH₂-CH₂-N), 1.44–1.33 (m, 4H, N-C₂H₄-C₂H₄-C₂H₄-N). ¹³C NMR (68 MHz, D₂O): δ 145.79 (N-CH-CH-CH-CH-CH-CH-N), 144.39 (N-CH-CH-CH-CH-CH-CH-N), 128.45 (N-CH-CH-CH-CH-CH-CH-N), 61.92 (N-CH₂-C₄H₈-CH₂-N), 30.49 (N-CH₂-CH₂-C₂H₄-CH₂-CH₂-N), 24.99 (N-C₂H₄-C₂H₄-C₂H₄-N). MS m/z molecular ion: calcd. 121.0888; found 121.0873 (cation⁺).

C₈ (Pyr)₂ Br₂: 1,8-bis(pyridinium-1-yl)octane dibromide. C₁₈H₂₆Br₂N₂; from 5.93 g (75 mmol) of pyridine and 8.16 g (30 mmol) of 1,8-dibromooctane, 12.17 g (28.30 mmol) of C₈ (Pyr)₂ Br₂ was obtained (yield: 94%); ¹H NMR (270 MHz, D₂O): δ 8.82 (d, 4H, N-CH-CH), 8.52 (t, 2H, N-CH-CH-CH-CH-CH-N), 8.04 (t, 4H, N-CH-CH-CH-CH-CH-CH-N), 4.58 (t, 4H, N-CH₂-C₆H₁₂-CH₂-N), 2.04–1.93 (m, 4H, N-CH₂-CH₂-C₄H₈-CH₂-CH₂-N), 1.37–1.24 (m, 8H, N-CH₂-CH₂-C₄H₈-CH₂-CH₂-N). ¹³C NMR (68 MHz, D₂O): δ 145.69 (N-CH-CH-CH-CH-CH-CH-N), 144.37 (N-CH-CH-CH-CH-CH-CH-N), 128.39 (N-CH-CH-CH-CH-CH-CH-N), 62.12 (N-CH₂-C₆H₁₂-CH₂-N), 30.65 (N-CH₂-CH₂-C₄H₈-CH₂-CH₂-N), 28.00 (N-C₂H₄-CH₂-C₂H₄-CH₂-C₂H₄-N), 25.25 (N-C₃H₆-C₂H₄-C₃H₆-N). MS m/z molecular ion: calcd. 135.1044; found 135.1037 (cation⁺).

C₁₂ (Pyr)₂ Br₂: 1,12-bis(pyridinium-1-yl)dodecane dibromide. C₂₂H₃₄Br₂N₂; from 5.93 g (75 mmol) of pyridine and 9.84 g (30 mmol) of 1,12-dibromododecane, 13.86 g (28.51 mmol) of C₁₂ (Pyr)₂ Br₂ was obtained (yield: 95%); ¹H NMR (270 MHz, D₂O): δ 8.82 (d, 4H, N-CH-CH), 8.51 (t, 2H, N-CH-CH-CH-CH-CH-N), 8.04 (t, 4H, N-CH-CH-CH-CH-CH-CH-N), 4.58 (t, 4H, N-CH₂-C₁₀H₂₀-CH₂-N), 2.03–1.92 (m, 4H, N-CH₂-CH₂-C₈H₁₆-CH₂-CH₂-N), 1.30–1.19 (m, 16H, N-CH₂-CH₂-C₈H₁₆-CH₂-CH₂-N). ¹³C NMR (68 MHz, D₂O): δ 145.68 (N-CH-CH-CH-CH-CH-CH-N), 144.36 (N-CH-CH-CH-CH-CH-CH-N), 128.38 (N-CH-CH-CH-CH-CH-CH-N), 62.17 (N-CH₂-C₁₀H₂₀-CH₂-N), 30.69 (N-CH₂-CH₂-C₈H₁₆-CH₂-CH₂-N), 28.68 (N-C₂H₄-CH₂-C₆H₁₂-

$\text{CH}_2\text{-C}_2\text{H}_4\text{-N}$), 28.59 ($\text{N-C}_3\text{H}_6\text{-CH}_2\text{-C}_4\text{H}_8\text{-CH}_2\text{-C}_3\text{H}_6\text{-N}$), 28.23 ($\text{N-C}_4\text{H}_8\text{-CH}_2\text{-C}_2\text{H}_4\text{-CH}_2\text{-C}_4\text{H}_8\text{-N}$), 25.35 ($\text{N-C}_5\text{H}_{10}\text{-C}_2\text{H}_4\text{-C}_5\text{H}_{10}\text{-N}$). MS m/z molecular ion: calcd. 163.1357; found 163.1370 (cation⁺).

C₃ (Pyr) (Mlm) Br₂: 1-(pyridinium-1-yl)-3-(3-methylimidazolium-1-yl)propane dibromide C₁₂H₁₇Br₂N₃; from 4.42 g (15 mmol) of 1-(3-bromopropyl)pyridinium bromide and 1.48 g (18 mmol) of 1-methylimidazole, 5.1 g (14.04 mmol) of C₃ (Pyr) (Mlm) Br₂ was obtained (yield: 94%); ¹H NMR (270 MHz, methanol-d₄): δ 9.18 (d, 2H, N-CH-CH-CH-CH-CH-N), 9.14 (s, 1H, N-CH-N), 8.66 (t, 1H, N-CH-CH-CH-CH-CH-N), 8.18 (t, 2H, N-CH-CH-CH-CH-CH-N), 7.78 (t, 1H, N-CH-CH-N), 7.64 (t, 1H, N-CH-CH-N), 4.86 (t, 32H, N-CH₂-CH₂-CH₂-N), 4.50 (t, 2H, N-CH₂-CH₂-CH₂-N), 3.98 (s, 3H, N-CH₃), 2.77–2.66 (m, 2H, N-CH₂-CH₂-CH₂-N). ¹³C NMR (68 MHz, methanol-d₄): δ 147.48 (N-CH-CH-CH-CH-CH-N), 146.41 (N-CH-CH-CH-CH-CH-N), 138.48 (N-CH-N), 129.86 (N-CH-CH-CH-CH-CH-N), 125.40 (N-CH-CH-N), 123.89 (N-CH-CH-N), 59.68 (N-CH₂-CH₂-CH₂-N), 47.61 (N-CH₂-CH₂-CH₂-N), 36.95 (N-CH₃), 32.81 (N-CH₂-CH₂-CH₂-N). MS m/z molecular ion: calcd. 101.5708; found 101.5682 (cation⁺).

C₃ (Pyr) (MPyrr) Br₂: 1-(pyridinium-1-yl)-3-(1-methylpyrrolidinium-1-yl)propane dibromide C₁₃H₂₂Br₂N₂; from 4.42 g (15 mmol) of 1-(3-bromopropyl)pyridinium bromide and 1.66 g (19.5 mmol) of 1-methylpyrrolidine, 4.71 g (12.86 mmol) of C₃ (Pyr) (MPyrr) Br₂ was obtained (yield: 86%); ¹H NMR (270 MHz, methanol-d₄): δ 9.22 (d, 2H, N-CH-CH-CH-CH-CH-N), 8.67 (t, 1H, N-CH-CH-CH-CH-CH-N), 8.20 (t, 2H, N-CH-CH-CH-CH-CH-N), 4.85 (t, 2H, N_{aromatic}-CH₂-CH₂-CH₂-N), 3.71–3.63 (m, 6H, N_{aromatic}-CH₂-CH₂-CH₂-N y CH₂-N_{pyrrol}-CH₂), 3.17 (s, 3H, N-CH₃), 2.70–2.64 (m, 2H, N_{aromatic}-CH₂-CH₂-CH₂-N), 2.31–2.24 (m, 4H, N-CH₂-CH₂-CH₂-CH₂-N). ¹³C NMR (68 MHz, methanol-d₄): δ 147.58 (N-CH-CH-CH-CH-CH-N), 146.45 (N-CH-CH-CH-CH-CH-N), 129.87 (N-CH-CH-CH-CH-CH-N), 66.20y 66.16 (CH₂-N_{pyrrol}-CH₂), 61.53 (N_{aromatic}-CH₂-CH₂-CH₂-N), 59.46 (N_{aromatic}-CH₂-CH₂-CH₂-N), 49.60 (N-CH₃), 27.30 (N-CH₂-CH₂-CH₂-CH₂-N), 22.83 (N_{aromatic}-CH₂-CH₂-CH₂-N). MS m/z molecular ion: calcd. 103.0888; found 103.0862 (cation⁺).

Melting point temperature of the ILs or molten salts which are solid at room temperature was recorded on a Büchi Melting Point B-540 instrument. When the thermal decomposition of the IL or the molten salt was reached before its melting, thermal decomposition temperature was determined by Differential Scanning Calorimetry on a DSC 2920 (TA instruments) instrument.

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