Effect of methylene versus ethylene linkers on structural properties of tert-butyl and mesityl bis(imidazolium) bromide salts

Emily S. Thompson, Elisa M. Olivas, Adrian Torres, Briana C. Arreaga, Hector L. Alarcon, Deandrea Dolberry, Jacob P. Brannon and S. Chantal E. Stieber*

Department of Chemistry & Biochemistry, California State Polytechnic University, Pomona, 3801 W. Temple Ave., Pomona, CA 91768, USA. *Correspondence e-mail: sestieber@cpp.edu

The crystal structures of ligand precursor bis(imidazolium) salts 1,1'-methylenebis(3-tert-butylimidazolium) dibromide monohydrate, C_{15}H_{26}N_{4}^{+}/C_{2}Br^{-}/C_{0}/C_{1}H_{2}O or \[\text{tBuNHC}_2\text{Me}\]\[\text{Br}\]_2\text{H}_2\text{O}, 1,1'- (ethane-1,2-diyl)bis(3-tert-butylimidazolium) dibromide dihydrate, C_{16}H_{28}N_{4}^{+}/C_{2}Br^{-}/C_{0}/C_{1}2\text{H}_2\text{O} or \[\text{tBuNHC}_2\text{Et}\]\[\text{Br}\]_2\text{H}_2\text{O}, and 1,1'-methylenebis[3-(2,4,6-trimethylphenyl)imidazolium] dibromide dihydrate, C_{25}H_{30}N_{4}^{2+}/C_{1}2\text{Br}^{-}/C_{0}/C_{1}2\text{H}_2\text{O} or \[\text{MesNHC}_2\text{Me}\]\[\text{Br}\]_2\text{H}_2\text{O}, and 1,1'- (ethane-1,2-diyl)bis[3-(2,4,6-trimethylphenyl)imidazolium] dibromide tetrahydrate, C_{26}H_{32}N_{4}^{2+}/C_{1}2\text{Br}^{-}/C_{0}/C_{1}4\text{H}_2\text{O} or \[\text{MesNHC}_2\text{Et}\]\[\text{Br}\]_2\text{H}_2\text{O}, are reported. At 293 K, \[\text{tBuNHC}_2\text{Me}\]\[\text{Br}\]_2\text{H}_2\text{O} crystallizes in the \(P_{21/c}\) space group, while \[\text{tBuNHC}_2\text{Et}\]\[\text{Br}\]_2\text{H}_2\text{O} crystallizes in the \(P_{21/n}\) space group at 100 K. At 112 K, \[\text{MesNHC}_2\text{Me}\]\[\text{Br}\]_2\text{H}_2\text{O} crystallizes in the orthorhombic space group \(Pccn\) while \[\text{MesNHC}_2\text{Et}\]\[\text{Br}\]_2\text{H}_2\text{O} crystallizes in the \(P_{21/c}\) space group at 100 K. Bond distances and angles within the imidazolium rings are generally comparable among the four structures. All four bis(imidazolium) salts co-crystallize with one to four molecules of water.

1. Chemical context

Bis(imidazolium) salts are common precursors for the synthesis of bidentate N-heterocyclic carbene (NHC_2) ligands, which can be used to stabilize a variety of metal complexes and catalysts. Bis(imidazolium) salts, \([\text{NHC}_2R^{1}]_2X\) are relatively modular in that modifications can be relatively easily made to exterior groups attached to each NHC (R), the moiety linking the two NHC groups (R^1), and the counter-ion (X). One general synthetic approach for synthesizing bis(imidazolium) salts is where two equivalents of an alkyl or aryl imidazole are combined with one equivalent of an organic dihalide reagent and refluxed to afford the final product (Gardiner et al., 1999). A simplified procedure for a variety of ligand salts using pressure tubes resulting in yields that were generally over 80% was also reported (Scherg et al., 2006). Some reports have gone even further to minimize solvent in the synthesis of these ligand precursors, including a solvent-free synthesis (Cao et al., 2011, 2012). This implies that the exterior R groups can easily be modified by changing the alkyl or aryl group on the starting imidazole. The linking group R^1 and counter-ion X can be modified by changing the organic dihalide reagent. In this fashion, a library of bis(imidazolium) salts can be relatively easily synthesized from alkyl or aryl imidazoles, and some are also commercially available. Some of the most widely reported bis(imidazolium) salts are those with tert-butyl (tBu) and mesityl (Mes) exterior R groups.
and methylene (Me) or ethylene (Et) linking \( R^1 \) groups. \( [\text{MesNHC}_2\text{Et}]\text{Br}_2 \) was even reported to be a stand-alone catalyst for the conversion of arylaldehydes to carboxylic acids in combination with water and \( K_2\text{CO}_3 \) in DMSO (Yang et al., 2013). Methylene linkers are quite commonly used for complexing to metals, and although examples with ethylene linkers are fewer, comparative studies report that changing the linker affects catalysis. For example, shorter methylene linkers \( (R^1) \) were reported to be more effective for hydrosilylation reactions with \( \text{RhI} \) complexes than ethylene linkers (Riederer et al., 2010).

The bidentate NHC ligand system is highly versatile for stabilizing a range of metals, some of which result in catalytically active systems. For example, \( [\text{tBuNHC}_2\text{Me}]\text{Br}_2 \) and \( [\text{MesNHC}_2\text{Et}]\text{Br}_2 \) were used as precursors for synthesis of rhodium complexes (Leung et al., 2006). \( [\text{MesNHC}_2\text{Et}]\text{Cl}_2 \) was used for synthesis of aluminum, gallium, and indium complexes (Baker et al., 2002). \( [\text{MesNHC}_2\text{Et}]\text{Br}_2 \) was reported for synthesizing rhenium complexes (Hock et al., 2014; Hiltner et al., 2010), palladium complexes via \( \text{Pd(OAc)}_2 \)-assisted deprotometalation (Wierenga et al., 2019), palladium complexes via silver transmetallation (Sluijter et al., 2013), and for palladium catalysts for Suzuki and Heck coupling reactions (Lee et al., 2004). Other reported palladium complexes were active for dehalogenation of aryl halides (Viciu et al., 2001).

Examples with first row transition metals are fewer, with nickel being the most commonly reported. Nickel carbonato complexes were synthesized with \( [\text{MesNHC}_2\text{Me}]\text{Cl}_2 \) and \( [\text{MesNHC}_2\text{Et}]\text{Cl}_2 \) ligand precursors (Guo et al., 2013). Iron complexes for use in aryl Grignard-alkyl halide cross-coupling reactions were synthesized using various bis(imidazolium) salts including \( [\text{tBuNHC}_2\text{Me}]\text{Cl}_2 \), \( [\text{RhNHC}_2\text{Me}]\text{Br}_2 \), \( [\text{MesNHC}_2\text{Me}]\text{Cl}_2 \), \( [\text{MesNHC}_2\text{Me}]\text{Br}_2 \), and \( [\text{MesNHC}_2\text{Et}]\text{Br}_2 \) (Meyer et al., 2011).

When used for stabilizing bimetallic systems, \( [\text{tBuNHC}_2\text{Et}]\text{Cl}_2 \) and \( [\text{MesNHC}_2\text{Et}]\text{Cl}_2 \) have been used as precursors for dipalladium complexes for Heck reactions (Li et al., 2013; Yang et al., 2012; Cao et al., 2010), while \( [\text{tBuNHC}_2\text{Et}]\text{Br}_2 \) was a precursor for dimetallic Rh complexes (Wells et al., 2008) and mixed-metal Rh/Pd (Zamora et al., 2009) and Ir/Rh (Frey et al., 2006). Similarly, \( [\text{MesNHC}_2\text{Me}]\text{Br}_2 \) and \( [\text{MeNH-}\text{C}_2\text{Et}]\text{Br}_2 \) were used to synthesize bimetallic gold catalysts for cross-coupling and hydroamination reactions (Baron et al., 2018).

While bis(imidazolium) salts are common ligand precursors, few have been structurally characterized (Rheingold, 2019). This work presents structural characterization and a comparison of supramolecular features for methylene- versus ethylene-linked bis(imidazolium) salts with tert-butyl and mesityl ancillary groups.

2. Structural commentary

All four bis(imidazolium) salts were recrystallized from hot methanol and each compound co-crystallizes with one or more molecules of water. Fig. 1 depicts \( [\text{tBuNHC}_2\text{Me}]\text{Br}_2 \cdot \text{H}_2\text{O} \) while Fig. 2 depicts \( [\text{tBuNHC}_2\text{Et}]\text{Br}_2 \cdot 2\text{H}_2\text{O} \).

Bond distances in the imidazolium rings of \( [\text{tBuNHC}_2\text{Me}]\text{Br}_2 \) and \( [\text{tBuNHC}_2\text{Et}]\text{Br}_2 \) are mostly the same within experimental error, with backbone C2–C3 distances of 1.348 (4) and 1.349 (3) Å, respectively. The N–C distances are also mostly comparable with \( [\text{tBuNHC}_2\text{Me}]\text{Br}_2 \) having an N1–C2 and an N2–C3 distance of 1.389 (3) Å and N1–C1 and N2–C1 distances both being 1.337 (3) Å, while \( [\text{tBuNHC}_2\text{Et}]\text{Br}_2 \) has an N1–C2 distance of 1.388 (3) Å, an N2–C3 distance of 1.388 (3) Å, an
N1—C1 distance of 1.327 (3) Å and an N2—C1 distance of 1.331 (3) Å. For the linker, the N2—C7 distance is 1.463 (3) Å for [tBuNHC2Me][Br]2·2H2O and 1.468 (3) Å for [tBuNHNC2Et][Br]2·2H2O.

Bond angles in the imidazolium rings are also quite similar in [tBuNHC2Me][Br]2·2H2O and [tBuNHNC2Et][Br]2·2H2O. For [tBuNHC2Me][Br]2·2H2O, bond angles include C1—N1—C2 at 108.2 (2)°, N1—C2—C3 at 107.6 (2)°, C2—C3—N2 at 106.9 (2)°, C3—N2—C1 at 108.6 (2)°, and N2—C1—N1 at 108.7 (2)°. For [tBuNHNC2Et][Br]2·2H2O, bond angles include C1—N1—C2 at 108.21 (19)°, N1—C2—C3 at 107.3 (2)°, C2—C3—N2 at 106.9 (2)°, C3—N2—C1 at 108.54 (19)°, and N2—C1—N1 at 109.02 (19)°.

Fig. 3 depicts [MesNHC2Me][Br]2·2H2O while Fig. 4 depicts [MesNHNC2Et][Br]2·4H2O. Notably, [MesNHNC2Et][Br]2·4H2O is the only compound of the four for which the asymmetric unit contains only half of the molecule.

Bond distances in the imidazolium rings of [MesNHC2Me][Br]2·2H2O and [MesNHNC2Et][Br]2·4H2O are mostly the same within experimental error, with backbone C2—C3 distances of 1.344 (3) Å and 1.3506 (19) Å, respectively. N—C distances are also mostly the same with [MesNHC2Me][Br]2·2H2O having an N1—C2 distance of 1.387 (3) Å, an N2—C3 distance of 1.380 (3) Å, an N1—C1 distance of 1.326 (3) Å, and an N2—C1 distance of 1.341 (3) Å. Similarly, [MesNHNC2Et][Br]2·4H2O has an N1—C2 distance of 1.3872 (16) Å, an N2—C3 distance of 1.3841 (16) Å, an N1—C1 distance of 1.3322 (16) Å and an N2—C1 distance of 1.3314 (16) Å. For the linker, the N2—C7 distance is 1.457 (3) Å for [MesNHC2Me][Br]2·2H2O and 1.4653 (16) Å for [MesNHNC2Et][Br]2·4H2O.

Figure 3
View of [MesNHC2Me][Br]2·2H2O with 50% probability ellipsoids.

Figure 4
View of [MesNHNC2Et][Br]2·4H2O with 50% probability ellipsoids.

3. Supramolecular features

The supramolecular structure of [tBuNHC2Me][Br]2·2H2O is stabilized by hydrogen bonding (Fig. 5, Table 1). Distances between centroids of neighboring imidazoles are greater than 5 Å, suggesting no π-stacking interactions (Janiak, 2000). Hydrogen bonding between one bromide atom and one water molecule is found with Br1···H1D having a distance of 2.575 (4) Å. One tert-butyl group has positional disorder.

The supramolecular structure of [tBuNHNC2Et][Br]2·2H2O is stabilized by extensive hydrogen bonding (Fig. 6, Table 1). Distances between centroids of neighboring imidazoles are greater than 5 Å, suggesting no π-stacking interactions (Janiak, 2000). Several hydrogen-bonding interactions are found between bromide ions and water molecules, including Br2···H1B (2.439 Å) and Br1···H1A (2.398 Å).

The supramolecular structure of [MesNHC2Me][Br]2·2H2O is also stabilized by hydrogen bonding (Fig. 7, Table 1). No π-stacking interactions were found as distances between...
centroids of aromatic rings of neighboring molecules are greater than 5 Å (Janiak, 2000). Several hydrogen-bonding interactions are observed between bromide ions and water molecules as well as neighboring water molecules, including \( \text{Br}_1 \cdot \cdot \cdot \text{H}_2 \text{A} \) at 2.413 Å, \( \text{Br}_2 \cdot \cdot \cdot \text{H}_1 \text{A} \) at 2.463 Å, and \( \text{O}_1 \cdot \cdot \cdot \text{H}_2 \text{B} \) at 2.125 Å.

The supramolecular structure of \( \text{[MesNHC}_2\text{Et][Br]}_2\cdot4\text{H}_2\text{O} \) is also stabilized by hydrogen bonding (Fig. 8, Table 1). No \( \pi \)-stacking is observed between mesityl groups, similar to \( \text{[MesNHC}_2\text{Me][Br]}_2\cdot2\text{H}_2\text{O} \) as the distance between centroids of the mesityl groups of neighboring fragments is greater than 4.5 Å (Janiak, 2000). Hydrogen-bonding interactions include \( \text{O}_1 \cdot \cdot \cdot \text{H}_2 \text{B} \) at 1.994 (2) Å, \( \text{O}_2 \cdot \cdot \cdot \text{H}_1 \text{E} \) at 2.001 (3) Å, and \( \text{Br}_1 \cdot \cdot \cdot \text{H}_1 \text{D} \) at 2.585 (2) Å.

4. Database survey
A survey of the Cambridge Structural Database (Web accessed March 24, 2022; Groom et al., 2016) and SciFinder (SciFinder, 2022) yielded no exact matches for the unit cells of \( \text{[MesNHC}_2\text{Me][Br]}_2\cdot2\text{H}_2\text{O} \), \( \text{[MesNHC}_2\text{Et][Br]}_2\cdot2\text{H}_2\text{O} \), or \( \text{[MesNHC}_2\text{Et][Br]}_2\cdot4\text{H}_2\text{O} \). A deposited dataset for \( \text{[MesNHC}_2\text{Me][Br]}_2\cdot2\text{H}_2\text{O} \) was found (Rheingold, 2019) with a slightly higher \( R \) of 3.94% and data collection at a higher temperature of 150 K, as compared to \( R \) of 3.18% and temperature of 112 K in the current report. As discussed in the introduction, the syntheses of all of the reported structures are reported based on the SciFinder search; however, no additional structural data were found.

5. Synthesis and crystallization

General considerations. All reagents were purchased from commercial suppliers and used without further purification.

### Table 1

| Compound                        | Atoms                   | Distance   |
|---------------------------------|-------------------------|------------|
| \( \text{[MesNHC}_2\text{Me][Br]}_2\cdot2\text{H}_2\text{O} \) | \( \text{Br} \cdot \cdot \cdot \text{H}_1 \text{D} \) | 2.575 (4)  |
| \( \text{[MesNHC}_2\text{Et}[\text{Br]}_2\cdot2\text{H}_2\text{O} \) | \( \text{Br} \cdot \cdot \cdot \text{H}_1 \text{A} \) | 2.398      |
| \( \text{[MesNHC}_2\text{Me}[\text{Br]}_2\cdot2\text{H}_2\text{O} \) | \( \text{Br} \cdot \cdot \cdot \text{H}_1 \text{A} \) | 2.463      |
| \( \text{[MesNHC}_2\text{Et}[\text{Br]}_2\cdot4\text{H}_2\text{O} \) | \( \text{O} \cdot \cdot \cdot \text{H}_2 \text{B} \) | 2.125      |
| \( \text{[MesNHC}_2\text{Me}[\text{Br]}_2\cdot2\text{H}_2\text{O} \) | \( \text{O} \cdot \cdot \cdot \text{H}_2 \text{B} \) | 1.994 (2)  |
| \( \text{[MesNHC}_2\text{Et}[\text{Br]}_2\cdot4\text{H}_2\text{O} \) | \( \text{O} \cdot \cdot \cdot \text{H}_1 \text{E} \) | 2.001 (3)  |
| \( \text{[MesNHC}_2\text{Me}[\text{Br]}_2\cdot2\text{H}_2\text{O} \) | \( \text{Br} \cdot \cdot \cdot \text{H}_1 \text{D} \) | 2.585 (2)  |

Figure 6
View of four molecules of \( \text{[MesNHC}_2\text{Et}[\text{Br]}_2\cdot2\text{H}_2\text{O} \) with 50% probability ellipsoids, highlighting intermolecular distances.

Figure 7
View of eight molecules of \( \text{[MesNHC}_2\text{Me}[\text{Br]}_2\cdot2\text{H}_2\text{O} \) with 50% probability ellipsoids, highlighting intermolecular distances.
NMR data were collected on a Varian 400 MHz spectrometer and referenced to residual CHCl₃.

**Synthesis of 1-tert-butyl-1H-imidazole, ([dub]Im).** The procedure was adapted from a literature procedure (Liu et al., 2003). A round-bottom flask was charged with 10.0 mL (95 mmol, 1 eq.) of tert-butylamine, 11.0 mL of 40% glyoxal (95 mmol, 1 eq.), approximately 100 mL of methanol, and approximately 25 mL of deionized water and a stir bar, then heated to 343 K under reflux. 7.81 mL of 37% formaldehyde (95 mmol, 1 eq.) were added, followed by 3.70 mL of ammonium hydroxide (95 mmol, 1 eq.) added dropwise over 5 minutes while stirring. The solution was refluxed at 343 K for 5 h, resulting in a light red–orange solution. Excess solvent was removed in vacuo, and the resulting product was diluted with approximately 150 mL of dichloromethane and washed twice with 50 mL of deionized H₂O until the aqueous layers ran clear. The product was vacuum distilled at ~373 K, yielding a clear liquid, which was weighed in a tared vial, resulting in 7.95 g (34% yield) of [dub]Im, and characterized by ¹H NMR spectroscopy in CDCl₃.

**Synthesis of 1-(2,4,6-trimethylphenyl)-1H-imidazole, ([Mes]Im).** The procedure was adapted from a literature procedure (Liu et al., 2003; Gardiner et al., 1999). A 250 mL three-neck round-bottom flask was charged with 15.000 g (110.9 mmol, 1 eq.) of 2,4,6-trimethylaniline, 16.090 g (110.9 mmol, 1 eq.) of 40% glyoxal, and 300 mL of methanol. 1H NMR data were consistent with those previously reported (Scherg et al., 2006).

**Synthesis of 1,1'-di(tert-butyl)-3,3'-ethylene-diimidazolium dibromide, ([dub]NHC₂Et)[Br]₂.** A 250 mL round-bottomed flask was charged with 2.017 g (16.2 mmol, 2.5 eq.) of [dub]Im, 0.562 mL (6.45 mmol, 1 eq.) of dibromoethane, a stir bar, and ~20 mL of toluene. The mixture was refluxed at 423 K and stirred for 46 h, at which point the solution was a rusty brown color. The flask was then placed in an ice bath, and the resulting precipitate was collected via vacuum filtration and washed twice with ~5 mL of cold toluene. The resulting solids were dried and weighed, yielding 1.727 g (61% yield) of [dub]NHC₂Et[Br]₂ and single crystals suitable for X-ray diffraction were obtained via recrystallization from hot methanol. ¹H NMR data were consistent with those previously reported (Scherg et al., 2006).

**Synthesis of 1,1'-di(mesityl)-3,3'-methylene-diimidazolium dibromide, ([Mes]NHC₂Me)[Br]₂.** The procedure was adapted from a literature procedure (Gardiner et al., 1999). 5.00 g (26.8 mmol, 2.5 eq.) of MesIm we added to a 50 mL round-bottomed flask with a stir bar and ~20 mL of toluene. The mixture was refluxed at 423 K and stirred for 19 h, resulting in a cloudy yellow solution. The product was obtained in 17% yield (1.10 g) as tan crystals identified as [MesNHC₂Me][Br]₂ suitable for X-ray diffraction and characterized by ¹H NMR.

**Synthesis of 1,1'-di(merosityl)-3,3'-ethylene-diimidazolium dibromide, ([Mes]NHC₂Et)[Br]₂.** A 250 mL three-neck round-bottom flask was charged with 4.438 g (23.8 mmol, 2.5 eq.) of MesIm, 0.824 mL (9.52 mmol, 1 eq.) of 1,2-dibromoethane, and ~20 mL of toluene. The reaction mixture was heated to 423 K and refluxed for 19 h, resulting in a cloudy yellow solution. The solution was cooled in an ice bath, and the resulting precipitate was collected and recrystallized from ~25 mL of hot methanol, resulting in 2.962 g (55% yield) of tan crystals which were analyzed via ¹H NMR spectroscopy and identified as [MesNHC₂Et][Br]₂.

6. Refinement
Crystal data, data collection and structure refinement details are summarized in Table 2. Most hydrogen atoms were placed in calculated positions using the AFIX commands of SHELXL and included as riding contributions with distances of 0.95 Å for C—H, 0.99 Å for CH₂ and 0.98 Å for CH₃. Methyl H atoms were allowed to rotate but not to tip to best fit the experimental electron density. Uiso values of riding H atoms were set to 1.2 times Ueq(C) for CH₂ and CH₃, and 1.5 times Ueq(C) for CH₃ and H₂O. For [Mes]NHC₂Me][Br]₂, the SADI command of SHELX was used to model disorder in one of the tert-butyl moieties for N4—OAA and N4—C12.
## Table 2

Experimental details.

| Crystal data | [1,8-NHC2Me][Br]2·2H2O | [1,8-NHC2Me][Br]2·H2O | [1,8-NHC2Et][Br]2·H2O | [1,8-NHC2Et][Br]2·4H2O |
|--------------|-------------------------|------------------------|------------------------|------------------------|
| Chemical formula | C$_2$H$_4$N$_2$$^{2-}$·2Br$^-·$2H$_2$O | C$_2$H$_4$N$_2$$^{2-}$·2Br$^-·$2H$_2$O | C$_2$H$_4$N$_2$$^{2-}$·2Br$^-·$2H$_2$O | C$_2$H$_4$N$_2$$^{2-}$·2Br$^-·$4H$_2$O |
| $M_1$ | 582.38 | 440.23 | 472.27 | 632.42 |
| Crystal system, space group | Orthorhombic, Pccn | Monoclinic, $P2_1/c$ | Monoclinic, $P2_1/c$ | Monoclinic, $P2_1/c$ |
| Temperature (K) | 112 | 293 | 100 | 100 |
| $\alpha$, $\beta$, $\gamma$ ($^\circ$) | 90, 90, 90 | 90, 101.35 (3), 90 | 90, 112.766 (1), 90 | 90, 108.379 (1), 90 |
| $V$ ($\text{Å}^3$) | 5464.6 (2) | 1995 (2) | 2110.9 (12) | 1437.78 (6) |
| Z | 8 | 4 | 4 | 2 |
| Radiation type | Mo Kα | Mo Kα | Mo Kα | Mo Kα |
| No. of measured, independent and observed reflections | 39085, 5954, 5530 | 33565, 4402, 3780 | 31474, 4664, 4168 | 28199, 3165, 3028 |
| $R_{	ext{min}}$, $R_{	ext{max}}$ | 0.386, 0.748 | 0.544, 0.747 | 0.496, 0.748 | 0.544, 0.750 |
| No. of reflections | 39085, 5954, 5530 | 33565, 4402, 3780 | 31474, 4664, 4168 | 28199, 3165, 3028 |
| $R_{	ext{wp}}$, $R_{	ext{e}}$ | 0.035, 0.043 | 0.043, 0.059 | 0.059 | 0.025 |
| No. of parameters | 338 | 259 | 253 | 194 |
| No. of restraints | 0 | 3 | 0 | 0 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta \rho_{\text{max}}$, $\Delta \rho_{\text{min}}$ (e·Å$^{-3}$) | 0.42, −0.54 | 1.49, −1.10 | 0.60, −0.61 | 0.33, −0.29 |

Data collection

| Diffractometer | Bruker Venture D8 Kappa | Bruker APEXII CCD | Bruker Venture D8 Kappa | Bruker Venture D8 Kappa |
|----------------|-------------------------|-------------------|-------------------------|-------------------------|
| Absorption correction | Multi-scan (SADABS; Bruker, 2016) | Multi-scan (SADABS; Bruker, 2016) | Multi-scan (SADABS; Bruker, 2016) | Multi-scan (SADABS; Bruker, 2016) |
| $T_{	ext{min}}$, $T_{	ext{max}}$ | 0.032, 0.066, 1.19 | 0.035, 0.068, 1.08 | 0.031, 0.066, 1.11 | 0.018, 0.044, 1.10 |
| No. of reflections | 5954 | 4402 | 4664 | 3165 |
| No. of parameters | 338 | 259 | 253 | 194 |
| No. of restraints | 0 | 3 | 0 | 0 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta \rho_{\text{max}}$, $\Delta \rho_{\text{min}}$ (e·Å$^{-3}$) | 0.42, −0.54 | 1.49, −1.10 | 0.60, −0.61 | 0.33, −0.29 |

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C04AA—C00N and C14—C12, and C1AA—C0AA and C13—C12 to restrain distances within a sigma of 0.02 Å. The population parameters for the disordered tert-butyl groups are 0.54019 for C12—C14, and 0.45981 for C00N, C0AA, and C1AA. The highest peak and deepest hole are both near a heavy atom Br1 with a distance of 0.88 Å from the highest peak of 1.49 e Å$^{-3}$ and a distance of 0.73 Å from the deepest hole of −1.10 e Å$^{-3}$.

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**Effect of methylene versus ethylene linkers on structural properties of tert-butyl and mesityl bis(imidazolium) bromide salts**

Emily S. Thompson, Elisa M. Olivas, Adrian Torres, Briana C. Arreaga, Hector L. Alarcon, Deandrea Dolberry, Jacob P. Brannon and S. Chantal E. Stieber

Computing details

For all structures, data collection: APEX3 (Bruker, 2016); cell refinement: SAINT (Bruker, 2016); data reduction: SAINT (Bruker, 2016). Program(s) used to solve structure: SHELXT (Sheldrick, 2015a) for sces01006_0m, est01043_0m, est01041d_0ma; olex2.solve (Bourhis et al., 2015) for at01019_0ma. For all structures, program(s) used to refine structure: SHELXL (Sheldrick, 2015b); molecular graphics: OLEX2 (Dolomanov et al., 2009); software used to prepare material for publication: OLEX2 (Dolomanov et al., 2009).

1,1′-Methylenebis(3-tert-butylimidazolium) dibromide monohydrate (sces01006_0m)

**Crystal data**

\[\text{C}_{15}\text{H}_{26}\text{N}_4^+\cdot 2\text{Br}^-\cdot \text{H}_2\text{O}\]  
\[M_r = 440.23\]

Monoclinic, \(\text{P}_{2_1}/c\)

\(a = 7.211\) (5) Å  
\(b = 18.311\) (17) Å  
\(c = 15.409\) (5) Å  
\(\beta = 101.35\) (3)°  
\(V = 1995\) (2) Å³  
\(Z = 4\)

**Data collection**

Bruker APEXII CCD  
\(\varphi\) and \(\omega\) scans  
Absorption correction: multi-scan  
(SADABS; Bruker, 2016)

\(T_{\text{min}} = 0.544, T_{\text{max}} = 0.747\)

33565 measured reflections

4402 independent reflections  
3780 reflections with \(I > 2\sigma(I)\)

\(R_{\text{int}} = 0.043\)

\(\theta_{\text{max}} = 27.1°, \theta_{\text{min}} = 2.7°\)

\(h = -9\rightarrow 8\)

\(k = -23\rightarrow 23\)

\(l = -19\rightarrow 19\)

**Refinement**

Refinement on \(F^2\)

Least-squares matrix: full  
\(R(F^2 > 2\sigma(F^2)) = 0.035\)

\(wR(F^2) = 0.068\)

\(S = 1.08\)

4402 reflections  
259 parameters  
3 restraints

Primary atom site location: dual  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement

\(w = 1/[\sigma(F^2) + (0.0006P)^2 + 4.1194P]\)  
where \(P = (F^2 + 2F_O^2)/3\)
(Δ/σ)_{max} = 0.001
Δρ_{max} = 1.49 \text{ e Å}^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

| Atom | x    | y    | z    | U_{iso} or U_{eq} | Occ. (<1) |
|------|------|------|------|------------------|-----------|
| Br2  | 0.83773 (4) | 0.86331 (2) | 0.55378 (2) | 0.03323 (9) | 0.54 (3) |
| Br1  | 1.05299 (4) | 0.61882 (2) | 0.35706 (2) | 0.03727 (10) | 0.54 (3) |
| N3   | 0.4514 (3) | 0.71177 (11) | 0.18236 (13) | 0.0175 (4) | 0.54 (3) |
| N1   | 0.5823 (3) | 0.48054 (11) | 0.16964 (13) | 0.0162 (4) | 0.54 (3) |
| O1   | 0.8362 (4) | 0.78047 (16) | 0.35775 (17) | 0.0419 (6) | 0.54 (3) |
| N2   | 0.4742 (3) | 0.58205 (11) | 0.21324 (13) | 0.0170 (4) | 0.54 (3) |
| N4   | 0.5288 (3) | 0.80162 (12) | 0.10500 (14) | 0.0225 (5) | 0.54 (3) |
| C1   | 0.6311 (4) | 0.54357 (13) | 0.21172 (16) | 0.0181 (5) | 0.54 (3) |
| H5   | 0.753852 | 0.558228 | 0.235943 | 0.022* | 0.54 (3) |
| C5   | 0.2905 (4) | 0.73014 (14) | 0.12095 (18) | 0.022 (5) | 0.54 (3) |
| C4   | 0.5935 (4) | 0.75572 (14) | 0.17061 (16) | 0.0211 (5) | 0.54 (3) |
| H9   | 0.716674 | 0.754245 | 0.203041 | 0.025* | 0.54 (3) |
| C7   | 0.4681 (4) | 0.65531 (14) | 0.25048 (16) | 0.0212 (5) | 0.54 (3) |
| H8A  | 0.361201 | 0.658686 | 0.279890 | 0.025* | 0.54 (3) |
| H8B  | 0.582372 | 0.663573 | 0.294434 | 0.025* | 0.54 (3) |
| C8   | 0.7152 (4) | 0.42139 (14) | 0.15085 (18) | 0.0207 (5) | 0.54 (3) |
| C6   | 0.3396 (4) | 0.78602 (15) | 0.07277 (19) | 0.0251 (6) | 0.54 (3) |
| C2   | 0.3869 (4) | 0.47957 (15) | 0.14242 (18) | 0.0216 (5) | 0.54 (3) |
| C3   | 0.3195 (4) | 0.54263 (15) | 0.16910 (19) | 0.0239 (6) | 0.54 (3) |
| C13  | 0.5981 (4) | 0.86039 (16) | −0.02694 (18) | 0.0290 (6) | 0.54 (3) |
| H15A | 0.613085 | 0.812884 | −0.050991 | 0.043* | 0.54 (3) |
| H15B | 0.674562 | 0.895007 | −0.050884 | 0.043* | 0.54 (3) |
| H15C | 0.467761 | 0.874789 | −0.042171 | 0.043* | 0.54 (3) |
| H15D | 0.641328 | 0.812747 | −0.039773 | 0.043* | 0.46 (3) |
| H15E | 0.674290 | 0.896891 | −0.047847 | 0.043* | 0.46 (3) |
| H15F | 0.468475 | 0.866370 | −0.055952 | 0.043* | 0.46 (3) |
| C12  | 0.6629 (17) | 0.8579 (5) | 0.0781 (8) | 0.014 (2) | 0.54 (3) |
| C11  | 0.6634 (5) | 0.35011 (16) | 0.1920 (2) | 0.0402 (8) | 0.54 (3) |
| H3A  | 0.676730 | 0.356294 | 0.254815 | 0.060* | 0.54 (3) |
| H3B  | 0.746068 | 0.311841 | 0.180278 | 0.060* | 0.54 (3) |
| H3C  | 0.534897 | 0.337462 | 0.166825 | 0.060* | 0.54 (3) |
| C10  | 0.9155 (4) | 0.44460 (17) | 0.1903 (3) | 0.0408 (8) | 0.54 (3) |
| H1A  | 0.944282 | 0.489150 | 0.162880 | 0.061* | 0.54 (3) |
| H1B  | 1.002017 | 0.407121 | 0.180300 | 0.061* | 0.54 (3) |
| H1C  | 0.927348 | 0.452256 | 0.252799 | 0.061* | 0.54 (3) |
| C9   | 0.6908 (5) | 0.4157 (2) | 0.0510 (2) | 0.0399 (8) | 0.54 (3) |
| H4A  | 0.564299 | 0.400122 | 0.026305 | 0.060* | 0.54 (3) |
### Atomic displacement parameters (Å²)

|         | \(U_11\)       | \(U_22\)       | \(U_33\)       | \(U_{12}\)       | \(U_{13}\)       | \(U_{23}\)       |
|---------|-----------------|-----------------|-----------------|------------------|------------------|------------------|
| Br2     | 0.02663 (15)    | 0.03928 (17)    | 0.02985 (16)    | 0.01469 (13)     | −0.00401 (11)    | −0.00144 (13)    |
| Br1     | 0.02382 (15)    | 0.0520 (2)      | 0.03702 (18)    | −0.01154 (13)    | 0.00837 (12)     | −0.02086 (14)    |
| N3      | 0.0203 (11)     | 0.0185 (10)     | 0.0146 (10)     | −0.0049 (8)      | 0.0058 (8)       | −0.0011 (8)      |
| N1      | 0.0134 (10)     | 0.0203 (11)     | 0.0140 (10)     | −0.0024 (8)      | 0.0008 (8)       | 0.0046 (8)       |
| O1      | 0.0559 (17)     | 0.0417 (15)     | 0.0233 (13)     | −0.0041 (13)     | −0.0040 (11)     | 0.0034 (11)      |
| N2      | 0.0182 (11)     | 0.0207 (11)     | 0.0125 (10)     | −0.0039 (8)      | 0.0040 (8)       | 0.0026 (8)       |
| N4      | 0.0315 (13)     | 0.0209 (11)     | 0.0153 (11)     | −0.0117 (9)      | 0.0053 (9)       | −0.0034 (8)      |
| C1      | 0.0149 (12)     | 0.0223 (13)     | 0.0156 (12)     | −0.0039 (10)     | −0.0009 (9)      | 0.0041 (10)      |
| C5      | 0.0210 (14)     | 0.0194 (13)     | 0.0241 (14)     | −0.0026 (10)     | 0.0002 (11)      | −0.0025 (10)     |
| C4      | 0.0243 (14)     | 0.0283 (14)     | 0.0106 (12)     | −0.0092 (11)     | 0.0035 (10)      | −0.0022 (10)     |
| C7      | 0.0285 (14)     | 0.0233 (13)     | 0.0135 (12)     | −0.0036 (11)     | 0.0083 (10)      | 0.0014 (10)      |
| C8      | 0.0176 (13)     | 0.0180 (12)     | 0.0271 (14)     | −0.0015 (10)     | 0.0060 (11)      | 0.0024 (10)      |
| C6      | 0.0304 (15)     | 0.0196 (13)     | 0.0227 (14)     | −0.0033 (11)     | −0.0010 (12)     | −0.0013 (11)     |
| C2      | 0.0126 (12)     | 0.0246 (14)     | 0.0266 (14)     | −0.0068 (10)     | 0.0016 (10)      | 0.0013 (11)      |
| C3      | 0.0136 (13)     | 0.0256 (14)     | 0.0328 (16)     | −0.0051 (11)     | 0.0053 (11)      | 0.0022 (11)      |
| C13     | 0.0353 (16)     | 0.0344 (16)     | 0.0195 (14)     | −0.0012 (13)     | 0.0109 (12)      | 0.0030 (12)      |
| C12     | 0.023 (5)       | 0.011 (3)       | 0.008 (4)       | −0.003 (3)       | 0.002 (4)        | 0.000 (3)        |
| C11     | 0.0429 (19)     | 0.0235 (15)     | 0.060 (2)       | 0.0033 (13)      | 0.0254 (17)      | 0.0115 (14)      |
| C10     | 0.0166 (15)     | 0.0285 (16)     | 0.072 (2)       | 0.0027 (12)      | −0.0039 (15)     | −0.0055 (16)     |
Geometric parameters (Å, °)

|     |   |     |     |     |     |     |
|-----|---|-----|-----|-----|-----|-----|
|     |   |     |     |     |     |     |
| C9  | 0.0406 (19) | 0.050 (2) | 0.0316 (17) | 0.0052 (15) | 0.0133 (14) | −0.0072 (15) |
| C15 | 0.014 (4) | 0.028 (5) | 0.031 (3) | −0.009 (3) | 0.003 (3) | 0.000 (3) |
| C14 | 0.032 (5) | 0.020 (3) | 0.028 (4) | −0.006 (3) | 0.014 (3) | −0.002 (3) |
| C14A | 0.066 (9) | 0.011 (3) | 0.030 (5) | −0.003 (4) | 0.026 (6) | 0.000 (3) |
| C12A | 0.025 (6) | 0.022 (5) | 0.014 (4) | −0.005 (4) | −0.004 (4) | 0.008 (3) |
| C15A | 0.028 (6) | 0.047 (8) | 0.032 (5) | −0.018 (5) | −0.010 (5) | 0.022 (5) |

Supporting information
| Bond                  | Angle (°)   | Bond                  | Angle (°)   |
|-----------------------|-------------|-----------------------|-------------|
| C1—N2—C7              | 125.5 (2)   | C8—C11—H3A           | 109.5       |
| C1—N2—C3              | 108.6 (2)   | C8—C11—H3B           | 109.5       |
| C3—N2—C7              | 125.8 (2)   | C8—C11—H3C           | 109.5       |
| C4—N4—C6              | 108.4 (2)   | H3A—C11—H3B          | 109.5       |
| C4—N4—C12             | 119.2 (6)   | H3A—C11—H3C          | 109.5       |
| C4—N4—C12A            | 133.6 (7)   | H3B—C11—H3C          | 109.5       |
| C6—N4—C12             | 132.4 (6)   | C8—C10—H1A           | 109.5       |
| C6—N4—C12A            | 117.7 (7)   | C8—C10—H1B           | 109.5       |
| N1—C1—N2              | 108.7 (2)   | C8—C10—H1C           | 109.5       |
| N1—C1—H5              | 125.6       | H1A—C10—H1B          | 109.5       |
| N2—C1—H5              | 125.6       | H1A—C10—H1C          | 109.5       |
| N3—C5—H10             | 123 (2)     | H1B—C10—H1C          | 109.5       |
| C6—C5—N3              | 106.5 (2)   | C8—C9—H4A            | 109.5       |
| C6—C5—H10             | 131 (2)     | C8—C9—H4B            | 109.5       |
| N3—C4—H9              | 125.7       | C8—C9—H4C            | 109.5       |
| N4—C4—N3              | 108.6 (2)   | H4A—C9—H4B           | 109.5       |
| N4—C4—H9              | 125.7       | H4A—C9—H4C           | 109.5       |
| N3—C7—N2              | 111.8 (2)   | H4B—C9—H4C           | 109.5       |
| N3—C7—H8A             | 109.3       | C12—C15—H13A         | 109.5       |
| N3—C7—H8B             | 109.3       | C12—C15—H13B         | 109.5       |
| N2—C7—H8A             | 109.3       | C12—C15—H13C         | 109.5       |
| N2—C7—H8B             | 109.3       | H13A—C15—H13B        | 109.5       |
| H8A—C7—H8B            | 107.9       | H13A—C15—H13C        | 109.5       |
| N1—C8—C11             | 108.4 (2)   | H13B—C15—H13C        | 109.5       |
| N1—C8—C10             | 108.2 (2)   | C12—C14—H14A         | 109.5       |
| N1—C8—C9              | 107.0 (2)   | C12—C14—H14B         | 109.5       |
| C10—C8—C11            | 111.4 (3)   | C12—C14—H14C         | 109.5       |
| C10—C8—C9             | 109.7 (3)   | H14A—C14—H14B        | 109.5       |
| C9—C8—C11             | 111.9 (3)   | H14A—C14—H14C        | 109.5       |
| N4—C6—H11             | 122 (2)     | H14B—C14—H14C        | 109.5       |
| C5—C6—N4              | 107.7 (2)   | H00A—C14A—H00B       | 109.5       |
| C5—C6—H11             | 131 (2)     | H00A—C14A—H00C       | 109.5       |
| N1—C2—H6              | 118.1 (18)  | H00B—C14A—H00C       | 109.5       |
| C3—C2—N1              | 107.6 (2)   | C12A—C14A—H00A       | 109.5       |
| C3—C2—H6              | 134.3 (18)  | C12A—C14A—H00B       | 109.5       |
| N2—C3—H7              | 120 (2)     | C12A—C14A—H00C       | 109.5       |
| C2—C3—N2              | 106.9 (2)   | N4—C12A—C14A         | 111.9 (9)   |
| C2—C3—H7              | 133 (2)     | C13—C12A—N4          | 110.5 (9)   |
| H15A—C13—H15B         | 109.5       | C13—C12A—C14A        | 113.1 (8)   |
| H15A—C13—H15C         | 109.5       | C13—C12A—C15A        | 107.5 (10)  |
| H15B—C13—H15C         | 109.5       | C15A—C12A—N4         | 101.8 (9)   |
| H15D—C13—H15E         | 109.5       | C15A—C12A—C14A       | 111.4 (10)  |
| H15D—C13—H15F         | 109.5       | C12A—C15A—H1AA       | 109.5       |
| H15E—C13—H15F         | 109.5       | C12A—C15A—H1AB       | 109.5       |
| C12—C13—H15A          | 109.5       | H1AA—C15A—H1AC       | 109.5       |
| C12—C13—H15B          | 109.5       | H1AA—C15A—H1AC       | 109.5       |
| C12—C13—H15C          | 109.5       | H1AB—C15A—H1AC       | 109.5       |
| C12A—C13—H15D         | 109.5       | H1AB—C15A—H1AC       | 109.5       |
C12A—C13—H15E 109.5

N3—C5—C6—N4 −0.3 (3) C7—N2—C1—N1 −177.6 (2)
N1—C2—C3—C11 122.5 (3) C8—N1—C1—N2 178.4 (2)
C1—N1—C8—C10 1.6 (3) C8—N1—C2—C3 −179.9 (2)
C1—N1—C8—C9 −116.6 (3) C6—N4—C4—N3 −0.8 (3)
C1—N1—C2—C3 −0.3 (3) C6—N4—C12—C13 −36.8 (9)
C7—N2—C3—C2 177.5 (2) C7—N2—C3—C2 177.5 (2)
C1—N1—C8—C10 1.6 (3) C8—N1—C1—N2 178.4 (2)
C1—N1—C8—C9 −116.6 (3) C6—N4—C4—N3 −0.8 (3)
C1—N1—C2—C3 −0.3 (3) C6—N4—C12—C13 −36.8 (9)
C7—N2—C3—C2 177.5 (2) C7—N2—C3—C2 177.5 (2)

Crystal data

C16H28N42+·2Br−·2H2O  

Mr = 472.27

Monoclinic, P2₁/n  
a = 17.1577 (6) Å  
b = 7.3180 (2) Å  
c = 18.2712 (6) Å  
β = 112.786 (1)°  

V = 2115.09 (12) Å³  
Z = 4

Dx = 1.483 Mg m⁻³

Mo Kα radiation, λ = 0.71073 Å
Cell parameters from 9074 reflections

θ(max) = 27.1°, θ(min) = 2.6°
μ = 3.85 mm⁻¹
T = 100 K
 Prism, clear colourless
0.2 × 0.1 × 0.05 mm

Data collection

Bruker Venture D8 Kappa  

diffractometer  
4664 independent reflections
φ and ω scans  
4168 reflections with I > 2σ(I)
Absorption correction: multi-scan  
(SADABS; Bruker, 2016)

Refinement

Refinement on F²  
4664 reflections
Least-squares matrix: full  
253 parameters
R(F²) = 0.031  
0 restraints

1,1'-(Ethane-1,2-diyl)bis(3-tert-butylimidazolium) dibromide dihydrate (est01043_0m)
Primary atom site location: dual  
Hydrogen site location: mixed  
H atoms treated by a mixture of independent and constrained refinement

\[ w = 1/[\sigma^2(F_o^2) + (0.013P)^2 + 2.745P] \]
where \( P = (F_o^2 + 2F_c^2)/3 \)

\( (\Delta/\sigma)_{\text{max}} = 0.001 \)
\( \Delta\rho_{\text{max}} = 0.60 \text{ e Å}^{-3} \)
\( \Delta\rho_{\text{min}} = -0.61 \text{ e Å}^{-3} \)

**Special details**

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)**

|       | x      | y      | z      | Uiso*/Ueq |
|-------|--------|--------|--------|-----------|
| Br2   | 0.42021(2) | 1.06846(3) | 0.17436(2) | 0.01794(7) |
| Br1   | 0.64204(2) | 1.22356(3) | 0.46410(2) | 0.01716(7) |
| O2    | 0.60635(11) | 0.9669(3) | 0.30798(12) | 0.0253(4) |
| H2C   | 0.612198 | 1.048204 | 0.344458 | 0.038* |
| H2D   | 0.556728 | 0.988924 | 0.271398 | 0.038* |
| O1    | 0.84726(12) | 1.2201(3) | 0.50226(12) | 0.0273(4) |
| H1C   | 0.874519 | 1.260282 | 0.550269 | 0.041* |
| H1D   | 0.793229 | 1.222132 | 0.491369 | 0.041* |
| N1    | 0.63968(11) | 0.5405(3) | 0.63652(11) | 0.0097(4) |
| N2    | 0.70265(12) | 0.7072(3) | 0.57816(11) | 0.0111(4) |
| N3    | 0.79306(12) | 0.7383(3) | 0.42286(12) | 0.0113(4) |
| N4    | 0.86030(11) | 0.8912(2) | 0.36416(11) | 0.0103(4) |
| C1    | 0.70549(14) | 0.5497(3) | 0.61555(13) | 0.0100(4) |
| C9    | 0.61678(14) | 0.3834(3) | 0.67751(14) | 0.0119(5) |
| C7    | 0.76212(14) | 0.7620(3) | 0.54214(14) | 0.0123(5) |
| H1A   | 0.818644 | 0.709890 | 0.572987 | 0.015* |
| H1B   | 0.767285 | 0.896775 | 0.543092 | 0.015* |
| C4    | 0.79441(14) | 0.8939(3) | 0.38544(13) | 0.0108(4) |
| C3    | 0.63152(15) | 0.8025(3) | 0.57441(15) | 0.0152(5) |
| C10   | 0.68416(16) | 0.2364(3) | 0.69468(15) | 0.0165(5) |
| H9A   | 0.688285 | 0.198067 | 0.644913 | 0.025* |
| H9B   | 0.668775 | 0.131113 | 0.719524 | 0.025* |
| H9C   | 0.738750 | 0.285198 | 0.730682 | 0.025* |
| C2    | 0.59238(15) | 0.6988(3) | 0.61096(15) | 0.0154(5) |
| C8    | 0.73079(14) | 0.6942(3) | 0.45708(14) | 0.0133(5) |
| H2A   | 0.721968 | 0.560336 | 0.455808 | 0.016* |
| H2B   | 0.676038 | 0.752701 | 0.425349 | 0.016* |
| C11   | 0.61411(16) | 0.4549(3) | 0.75498(15) | 0.0176(5) |
| H7A   | 0.670172 | 0.500229 | 0.789205 | 0.026* |
| H7B   | 0.597527 | 0.355834 | 0.782054 | 0.026* |
| H7C   | 0.572921 | 0.554573 | 0.743410 | 0.026* |
| C5    | 0.86096(15) | 0.6322(3) | 0.42626(15) | 0.0155(5) |
| C12   | 0.53083(15) | 0.3119(4) | 0.62096(16) | 0.0196(5) |
| H8A   | 0.488167 | 0.407825 | 0.611530 | 0.029* |

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**sup-7**
### Supporting Information

**Atomic displacement parameters (Å²)**

|   | U^11 | U^22 | U^33 | U^12 | U^13 | U^23 |
|---|------|------|------|------|------|------|
| Br2 | 0.01224 (11) | 0.01866 (13) | 0.02260 (14) | -0.00149 (9) | 0.00642 (10) | 0.00053 (10) |
| Br1 | 0.02064 (13) | 0.01617 (12) | 0.01448 (13) | -0.00222 (9) | 0.00658 (10) | 0.00156 (9) |
| O2 | 0.01999 (9) | 0.0299 (11) | 0.0227 (11) | 0.0067 (8) | 0.0046 (8) | -0.0070 (8) |
| O1 | 0.0261 (10) | 0.0346 (11) | 0.0234 (11) | 0.0008 (9) | 0.0120 (8) | -0.0035 (9) |
| N1 | 0.0115 (9) | 0.0117 (9) | 0.0082 (9) | -0.0001 (7) | 0.0063 (7) | 0.0017 (7) |
| N2 | 0.0120 (9) | 0.0141 (9) | 0.0088 (10) | -0.0008 (7) | 0.0059 (8) | 0.0006 (8) |
| N3 | 0.0135 (9) | 0.0113 (9) | 0.0118 (10) | -0.0022 (7) | 0.0078 (8) | 0.0015 (7) |
| N4 | 0.0129 (9) | 0.0103 (9) | 0.0098 (10) | -0.0010 (7) | 0.0068 (8) | 0.0018 (7) |
| C1 | 0.0114 (10) | 0.0116 (11) | 0.0084 (11) | 0.0009 (9) | 0.0054 (9) | 0.0002 (8) |
| C9 | 0.0148 (11) | 0.0132 (11) | 0.0103 (11) | -0.0025 (9) | 0.0078 (9) | 0.0027 (9) |
| C7 | 0.0146 (11) | 0.0155 (11) | 0.0094 (11) | -0.0034 (9) | 0.0075 (9) | 0.0031 (9) |
| C4 | 0.0125 (10) | 0.0127 (11) | 0.0087 (11) | -0.0010 (9) | 0.0056 (9) | 0.0005 (9) |
| C3 | 0.0170 (12) | 0.0142 (12) | 0.0157 (13) | 0.0041 (9) | 0.0079 (10) | 0.0038 (10) |
| C10 | 0.0214 (12) | 0.0145 (11) | 0.0168 (13) | 0.0004 (9) | 0.0110 (10) | 0.0029 (10) |
| C2 | 0.0150 (11) | 0.0157 (12) | 0.0182 (13) | 0.0048 (9) | 0.0094 (10) | 0.0032 (10) |
| C8 | 0.0135 (11) | 0.0177 (12) | 0.0121 (12) | -0.0047 (9) | 0.0085 (9) | 0.0008 (9) |
| C11 | 0.0228 (12) | 0.0200 (12) | 0.0166 (13) | 0.0004 (10) | 0.0149 (10) | 0.0027 (10) |
| C5 | 0.0192 (12) | 0.0108 (11) | 0.0182 (13) | 0.0020 (9) | 0.0092 (10) | 0.0024 (9) |
| C12 | 0.0157 (12) | 0.0218 (13) | 0.0207 (14) | -0.0074 (10) | 0.0065 (10) | 0.0012 (10) |
| C16 | 0.0217 (12) | 0.0160 (12) | 0.0179 (13) | 0.0024 (10) | 0.0127 (10) | 0.0053 (10) |
| C13 | 0.0143 (11) | 0.0132 (11) | 0.0133 (12) | -0.0032 (9) | 0.0085 (9) | 0.0034 (9) |
| C15 | 0.0240 (13) | 0.0212 (13) | 0.0166 (13) | -0.0010 (10) | 0.0144 (11) | 0.0019 (10) |

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|  | 0.0173 (12) | 0.0140 (11) | 0.0200 (13) | 0.0027 (9) | 0.0120 (10) | 0.0014 (10) |
|---|---|---|---|---|---|---|
| C6 | 0.0164 (12) | 0.0215 (13) | 0.0199 (13) | −0.0055 (10) | 0.0063 (10) | 0.0031 (10) |

**Geometric parameters (Å, °)**

| Bond | Length (Å) | Angle (°) |
|---|---|---|
| O2—H2C | 0.8695 | C10—H9B 0.9800 |
| O2—H2D | 0.8698 | C10—H9C 0.9800 |
| O1—H1C | 0.8699 | C2—H4 0.94 (3) |
| O1—H1D | 0.8697 | C8—H2A 0.9900 |
| N1—C1 | 1.327 (3) | C8—H2B 0.9900 |
| N1—C9 | 1.505 (3) | C11—H7A 0.9800 |
| N1—C2 | 1.388 (3) | C11—H7B 0.9800 |
| N2—C1 | 1.331 (3) | C11—H7C 0.9800 |
| N2—C7 | 1.468 (3) | C5—C6 1.352 (3) |
| N2—C3 | 1.384 (3) | C5—H10 0.91 (3) |
| N3—C4 | 1.333 (3) | C12—H8A 0.9800 |
| N3—C8 | 1.468 (3) | C12—H8B 0.9800 |
| N3—C5 | 1.381 (3) | C12—H8C 0.9800 |
| N4—C4 | 1.330 (3) | C16—H16A 0.9800 |
| N4—C13 | 1.503 (3) | C16—H16B 0.9800 |
| N4—C6 | 1.384 (3) | C16—H16C 0.9800 |
| C1—H5 | 0.91 (2) | C16—C13 1.520 (3) |
| C9—C10 | 1.520 (3) | C13—C15 1.522 (3) |
| C9—C11 | 1.526 (3) | C13—C14 1.531 (3) |
| C9—C12 | 1.528 (3) | C15—H15A 0.9800 |
| C7—H1A | 0.9900 | C15—H15B 0.9800 |
| C7—H1B | 0.9900 | C15—H15C 0.9800 |
| C7—C8 | 1.518 (3) | C6—H11 0.93 (3) |
| C4—H12 | 0.91 (2) | C14—H14A 0.9800 |
| C3—C2 | 1.349 (3) | C14—H14B 0.9800 |
| C3—H3 | 0.90 (3) | C14—H14C 0.9800 |
| C10—H9A | 0.9800 |

**Bond Angles**

| Bond | Angle (°) |
|---|---|
| O2—H2C—H2D | 104.5 |
| H1C—O1—H1D | 109.5 |
| C1—N1—C9 | 126.71 (19) |
| C1—N1—C2 | 108.21 (19) |
| C2—N1—C9 | 125.02 (18) |
| C1—N2—C7 | 124.95 (19) |
| C1—N2—C3 | 108.54 (19) |
| C3—N2—C7 | 126.4 (2) |
| C4—N3—C8 | 124.51 (19) |
| C4—N3—C5 | 108.75 (19) |
| C5—N3—C8 | 126.7 (2) |
| C4—N4—C13 | 125.94 (19) |
| C4—N4—C6 | 108.16 (19) |
| C6—N4—C13 | 125.85 (19) |
| N1—C1—N2 | 109.02 (19) |
| Bond          | Distance (Å) | Bond          | Distance (Å) |
|---------------|--------------|---------------|--------------|
| N1—C1—H5     | 126.1 (16)   | C9—C12—H8C   | 109.5        |
| N2—C1—H5     | 124.9 (16)   | H8A—C12—H8B  | 109.5        |
| N1—C9—C10    | 108.60 (18)  | H8A—C12—H8C  | 109.5        |
| N1—C9—C11    | 107.87 (18)  | H8B—C12—H8C  | 109.5        |
| N1—C9—C12    | 107.13 (18)  | H16A—C16—H16B| 109.5        |
| C10—C9—C11   | 110.2 (2)    | H16A—C16—H16C| 109.5        |
| C10—C9—C12   | 110.8 (2)    | H16B—C16—H16C| 109.5        |
| C11—C9—C12   | 112.13 (19)  | C13—C16—H16A | 109.5        |
| N2—C7—H1A    | 109.7        | C13—C16—H16B | 109.5        |
| N2—C7—H1B    | 109.7        | C13—C16—H16C | 109.5        |
| N2—C7—C8     | 109.74 (18)  | N4—C13—C16   | 108.52 (18)  |
| H1A—C7—H1B   | 108.2        | N4—C13—C15   | 108.11 (19)  |
| C8—C7—H1A    | 109.7        | N4—C13—C14   | 107.00 (19)  |
| C8—C7—H1B    | 109.7        | C16—C13—C15  | 110.4 (2)    |
| N3—C4—H12    | 124.6 (15)   | C16—C13—C14  | 110.6 (2)    |
| N4—C4—N3     | 108.8 (2)    | C15—C13—C14  | 112.0 (2)    |
| N4—C4—H12    | 126.5 (15)   | C13—C15—H15A | 109.5        |
| N2—C3—H3     | 120.5 (17)   | C13—C15—H15B | 109.5        |
| C2—C3—N2     | 106.9 (2)    | C13—C15—H15C | 109.5        |
| C2—C3—H3     | 132.5 (17)   | H15A—C15—H15B| 109.5        |
| C9—C10—H9A   | 109.5        | H15A—C15—H15C| 109.5        |
| C9—C10—H9B   | 109.5        | H15B—C15—H15C| 109.5        |
| C9—C10—H9C   | 109.5        | N4—C6—H11    | 121.8 (17)   |
| H9A—C10—H9B  | 109.5        | C5—C6—N4     | 107.6 (2)    |
| H9A—C10—H9C  | 109.5        | C5—C6—H11    | 130.7 (17)   |
| H9B—C10—H9C  | 109.5        | C13—C14—H14A | 109.5        |
| N1—C2—H4     | 120.4 (18)   | C13—C14—H14B | 109.5        |
| C3—C2—N1     | 107.3 (2)    | C13—C14—H14C | 109.5        |
| C3—C2—H4     | 132.3 (17)   | H14A—C14—H14B| 109.5        |
| N3—C8—C7     | 109.68 (18)  | H14A—C14—H14C| 109.5        |
| N3—C8—H2A    | 109.7        | H14B—C14—H14C| 109.5        |
| N2—C7—C8—N3  | −176.23 (18) | C4—N4—C6—C5  | 0.0 (3)      |
| N2—C3—C2—N1  | −0.2 (3)     | C3—N2—C1—N1  | −0.5 (3)     |
| N3—C5—C6—N4  | 0.2 (3)      | C3—N2—C7—C8  | −86.8 (3)    |
| C1—N1—C9—C10 | 2.3 (3)      | C2—N1—C1—N2  | 0.4 (3)      |
| C1—N1—C9—C11 | 121.7 (2)    | C2—N1—C9—C10 | 179.3 (2)    |
| C1—N1—C9—C12 | −117.4 (2)   | C2—N1—C9—C11 | −61.3 (3)    |
| C1—N1—C2—C3  | −0.1 (3)     | C2—N1—C9—C12 | 59.6 (3)     |
| C1—N2—C7—C8  | 88.3 (3)     | C8—N3—C4—N4  | 178.2 (2)    |
| C1—N2—C3—C2  | 0.5 (3)      | C8—N3—C5—C6  | −178.1 (2)   |
| C9—N1—C1—N2  | 177.7 (2)    | C5—N3—C4—N4  | 0.4 (3)      |
| C9—N1—C2—C3  | −177.5 (2)   | C5—N3—C8—C7  | 88.4 (3)     |
| C7—N2—C1—N1  | −176.37 (19) | C13—N4—C4—N3 | −177.8 (2)   |
| C7—N2—C3—C2  | 176.3 (2)    | C13—N4—C6—C5 | 177.6 (2)    |
| C4—N3—C8—C7  | −88.9 (3)    | C6—N4—C4—N3  | −0.3 (3)     |
| C4—N3—C5—C6  | −0.4 (3)     | C6—N4—C13—C16| 176.4 (2)    |
| C4—N4—C13—C16| −6.5 (3)     | C6—N4—C13—C15| 56.6 (3)     |
C4—N4—C13—C15 $-126.3$ (2)  
C4—N4—C13—C14 $112.8$ (2)  
C6—N4—C13—C14 $-64.3$ (3)  

1,1′-Methylenebis[3-(2,4,6-trimethylphenyl)imidazolium] dibromide dihydrate (at01019_0ma)

Crystal data

$C_{25}H_{30}N_{42}^+·2Br^-·2H_2O$

$M_r = 582.38$

Orthorhombic, $Pccn$

$a = 21.5695$ (6) Å

$b = 28.3385$ (6) Å

$c = 8.9401$ (2) Å

$V = 5464.6$ (2) Å$^3$

$Z = 8$

$F(000) = 2384$

$D_x = 1.416$ Mg m$^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9959 reflections

$\theta = 2.6$–39.4°

$\mu = 2.99$ mm$^{-1}$

$T = 112$ K

Prism, clear colourless

$0.4 \times 0.3 \times 0.25$ mm

Data collection

Bruker Venture Kappa D diffractometer

$\varphi$ and $\omega$ scans

Absorption correction: multi-scan

(SADABS; Bruker, 2016)

$\theta_{\text{min}} = 0.386$, $\theta_{\text{max}} = 0.748$

39085 measured reflections

5530 independent reflections

5530 reflections with $I > 2\sigma(I)$

$R_{int} = 0.035$

$\theta_{\text{max}} = 27.1°$, $\theta_{\text{min}} = 2.6°$

$T_{\text{min}} = -24 \rightarrow 27$

$k = -31 \rightarrow 36$

$l = -11 \rightarrow 11$

Refinement

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.066$

$S = 1.19$

5954 reflections

338 parameters

0 restraints

Primary atom site location: iterative

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0149P)^2 + 6.9269P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.002$

$\Delta \rho_{\text{max}} = 0.42$ e Å$^{-3}$

$\Delta \rho_{\text{min}} = -0.54$ e Å$^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)

| x         | y         | z         | $U_{eq}$ / $U_{eq}$ | Occ. (<1) |
|-----------|-----------|-----------|---------------------|-----------|
| Br1       | 0.32147 (2)| 0.59206 (2)| 0.51936 (2)         | 0.01792   |
| Br2       | 0.32341 (2)| 0.42996 (2)| 0.77256 (2)         | 0.01874   |
| N2        | 0.14868 (8)| 0.40218 (6)| 0.76624 (19)        | 0.0134    |
| N3        | 0.17753 (8)| 0.54615 (6)| 0.5774 (2)          | 0.0167    |
| N1        | 0.17956 (8)| 0.47256 (6)| 0.7130 (2)          | 0.0158    |
| N4        | 0.14048 (8)| 0.61532 (6)| 0.53485 (19)        | 0.0151    |
| C4        | 0.17963 (9)| 0.42757 (7)| 0.6667 (2)          | 0.0165    |
| H4        | 0.198554 | 0.415943 | 0.578146 | 0.020*    |

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| Atom | X      | Y      | Z      | U11   | U22   | U33   | U12   | U13   | U23   |
|------|--------|--------|--------|-------|-------|-------|-------|-------|-------|
| C16  | 0.1731 9 | 0.5900 6 | 0.6302 (2) | 0.0154 (4) |
| H16  | 0.1905 8 | 0.601214 | 0.721117 | 0.018* |
| C17  | 0.12153 (9) | 0.66386 (7) | 0.5581 (2) | 0.0146 (4) |
| C5   | 0.13900 (9) | 0.35172 (7) | 0.7556 (2) | 0.0141 (4) |
| O2A  | 0.4650 (10) | 0.5643 (7) | 0.601 (3) | 0.037 (3) | 0.47 (9) |
| H2AA | 0.482702 | 0.563311 | 0.513609 | 0.055* | 0.47 (9) |
| H2AB | 0.427586 | 0.574062 | 0.582766 | 0.055* | 0.47 (9) |
| C12  | 0.22444 (10) | 0.34048 (8) | 0.9479 (2) | 0.0201 (4) |
| H12A | 0.256440 | 0.357156 | 0.890780 | 0.030* |
| H12B | 0.243324 | 0.314106 | 1.002226 | 0.030* |
| C14  | 0.14765 (13) | 0.54398 (8) | 0.4406 (3) | 0.0275 (5) |
| C18  | 0.07474 (9) | 0.67224 (7) | 0.6624 (2) | 0.0150 (4) |
| C22  | 0.15021 (10) | 0.69938 (7) | 0.4754 (2) | 0.0179 (4) |
| C6   | 0.17572 (9) | 0.32189 (7) | 0.8424 (2) | 0.0153 (4) |
| C24  | 0.04581 (10) | 0.63259 (7) | 0.7507 (2) | 0.0190 (4) |
| H24A | 0.037277 | 0.605939 | 0.683916 | 0.028* |
| H24B | 0.006967 | 0.643518 | 0.795745 | 0.028* |
| C3   | 0.12848 (12) | 0.43164 (8) | 0.8801 (3) | 0.0244 (5) |
| C10  | 0.09279 (10) | 0.33558 (7) | 0.6589 (2) | 0.0163 (4) |
| C15  | 0.12434 (13) | 0.58713 (8) | 0.4143 (3) | 0.0272 (5) |
| C1   | 0.21637 (10) | 0.50965 (7) | 0.6432 (3) | 0.0231 (5) |
| H1A  | 0.243891 | 0.524043 | 0.719194 | 0.028* |
| H1B  | 0.242843 | 0.495638 | 0.564401 | 0.028* |
| C19  | 0.05623 (10) | 0.71864 (7) | 0.6822 (2) | 0.0174 (4) |
| H19  | 0.024420 | 0.725449 | 0.752452 | 0.021* |
| C11  | 0.05222 (11) | 0.36931 (8) | 0.5741 (3) | 0.0242 (5) |
| H11A | 0.036347 | 0.393487 | 0.642452 | 0.036* |
| H11B | 0.017393 | 0.352060 | 0.529724 | 0.036* |
| H11C | 0.076445 | 0.384406 | 0.494617 | 0.036* |
| O1A  | 0.47220 (10) | 0.47135 (8) | 0.6838 (4) | 0.0715 (9) |
| H1AA | 0.487824 | 0.450597 | 0.622561 | 0.107* |
| H1AB | 0.436751 | 0.459386 | 0.710574 | 0.107* |
| C7   | 0.16563 (9) | 0.27361 (7) | 0.8269 (2) | 0.0175 (4) |
| H7   | 0.189642 | 0.252345 | 0.885177 | 0.021* |
| C2   | 0.14808 (12) | 0.47542 (8) | 0.8472 (3) | 0.0245 (5) |
| C20  | 0.08292 (10) | 0.75565 (7) | 0.6021 (2) | 0.0182 (4) |
| C23  | 0.20108 (11) | 0.68858 (8) | 0.3644 (3) | 0.0263 (5) |
| H23A | 0.236552 | 0.674719 | 0.416963 | 0.040* |
| H23B | 0.214017 | 0.717788 | 0.314740 | 0.040* |
| H23C | 0.185662 | 0.666212 | 0.289523 | 0.040* |
| C8   | 0.12146 (10) | 0.25546 (7) | 0.7288 (2) | 0.0184 (4) |
| C9   | 0.08556 (10) | 0.28686 (7) | 0.6465 (2) | 0.0179 (4) |
| H9   | 0.055181 | 0.274754 | 0.579845 | 0.021* |
| C13  | 0.11192 (11) | 0.20288 (7) | 0.7166 (3) | 0.0243 (5) |
| H13A | 0.152068 | 0.186833 | 0.723425 | 0.036* |
| H13B | 0.092477 | 0.195470 | 0.620360 | 0.036* |
Atomic displacement parameters (Å²)

|      | U¹¹  | U²²  | U³³  | U¹²  | U¹³  | U²³  |
|------|------|------|------|------|------|------|
| Br1  | 0.01793 (11) | 0.01900 (10) | 0.01682 (11) | −0.00262 (8) | 0.00203 (8) | −0.00139 (8) |
| Br2  | 0.02215 (11) | 0.01532 (10) | 0.01876 (11) | −0.00184 (8) | 0.00044 (8) | −0.00160 (8) |
| N2   | 0.0173 (8) | 0.0108 (8) | 0.0120 (8) | −0.0006 (6) | 0.0010 (6) | −0.0006 (6) |
| N3   | 0.0177 (9) | 0.0101 (8) | 0.0222 (9) | 0.0009 (6) | 0.0016 (7) | 0.0035 (7) |
| N1   | 0.0145 (8) | 0.0116 (8) | 0.0213 (9) | 0.0005 (6) | 0.0008 (7) | 0.0025 (7) |
| N4   | 0.0207 (9) | 0.0114 (8) | 0.0133 (8) | 0.0006 (6) | −0.0008 (7) | 0.0006 (7) |
| C4   | 0.0157 (10) | 0.0147 (9) | 0.0190 (10) | 0.0021 (8) | 0.0023 (8) | 0.0000 (8) |
| C16  | 0.0150 (10) | 0.0125 (9) | 0.0186 (10) | −0.0017 (7) | −0.0002 (8) | 0.0012 (8) |
| C17  | 0.0179 (10) | 0.0113 (9) | 0.0147 (10) | 0.0014 (7) | −0.0043 (8) | −0.0008 (8) |
| C5   | 0.0186 (10) | 0.0101 (9) | 0.0135 (10) | −0.0020 (7) | 0.0038 (8) | 0.0000 (7) |
| O2A  | 0.026 (5) | 0.040 (4) | 0.045 (6) | 0.008 (4) | 0.005 (4) | 0.005 (5) |
| C12  | 0.0208 (11) | 0.0196 (10) | 0.0200 (11) | −0.0019 (8) | −0.0023 (9) | 0.0016 (9) |
| C14  | 0.0443 (15) | 0.0135 (10) | 0.0248 (12) | 0.0035 (10) | −0.0058 (11) | −0.0070 (9) |
| C18  | 0.0174 (10) | 0.0128 (9) | 0.0148 (10) | −0.0008 (7) | −0.0047 (8) | 0.0020 (8) |
| C22  | 0.0198 (11) | 0.0149 (10) | 0.0192 (10) | −0.0008 (8) | −0.0009 (8) | 0.0011 (8) |
| C6   | 0.0135 (10) | 0.0168 (9) | 0.0157 (10) | −0.0010 (7) | 0.0034 (8) | 0.0016 (8) |
| C24  | 0.0206 (11) | 0.0157 (10) | 0.0205 (11) | 0.0016 (8) | 0.0014 (9) | 0.0045 (8) |
| C3   | 0.0376 (14) | 0.0183 (11) | 0.0174 (11) | 0.0000 (9) | 0.0088 (10) | −0.0005 (9) |
| C10  | 0.0177 (10) | 0.0163 (10) | 0.0150 (10) | −0.0026 (8) | 0.0008 (8) | 0.0027 (8) |
| C15  | 0.0446 (15) | 0.0182 (11) | 0.0188 (11) | 0.0055 (10) | −0.0107 (11) | −0.0043 (9) |
| C1   | 0.0170 (11) | 0.0122 (10) | 0.0402 (14) | 0.0011 (8) | 0.0022 (10) | 0.0103 (9) |
| C19  | 0.0182 (10) | 0.0151 (10) | 0.0188 (10) | 0.0023 (8) | −0.0018 (8) | −0.0007 (8) |
| C11  | 0.0257 (12) | 0.0202 (11) | 0.0266 (12) | −0.0037 (9) | −0.0083 (10) | 0.0042 (9) |
| O1A  | 0.0296 (12) | 0.0545 (14) | 0.130 (3) | 0.0055 (10) | 0.0180 (14) | 0.0468 (16) |
| C7   | 0.0168 (10) | 0.0147 (9) | 0.0208 (10) | 0.0018 (8) | 0.0026 (8) | 0.0041 (8) |
| C2   | 0.0364 (14) | 0.0154 (10) | 0.0216 (12) | 0.0015 (9) | 0.0050 (10) | −0.0040 (9) |
| C20  | 0.0212 (11) | 0.0107 (9) | 0.0227 (11) | 0.0007 (8) | −0.0062 (9) | −0.0007 (8) |
| C23  | 0.0283 (13) | 0.0216 (11) | 0.0291 (13) | 0.0004 (9) | 0.0091 (10) | 0.0042 (10) |
| C8   | 0.0202 (10) | 0.0147 (10) | 0.0201 (10) | −0.0028 (8) | 0.0083 (8) | −0.0002 (8) |
| C9   | 0.0203 (11) | 0.0170 (10) | 0.0164 (10) | −0.0057 (8) | 0.0002 (8) | −0.0013 (8) |
sup-14

C13 0.0267 (12) 0.0148 (10) 0.0313 (13) −0.0033 (8) 0.0054 (10) −0.0026 (9)  
C21 0.0225 (11) 0.0125 (9) 0.0242 (11) −0.0027 (8) −0.0021 (9) 0.0036 (9)  
C25 0.0283 (12) 0.0120 (10) 0.0366 (14) 0.0028 (9) 0.0002 (10) −0.0008 (10)  
O2 0.023 (4) 0.045 (5) 0.060 (13) 0.013 (3) 0.006 (5) 0.026 (7)  

**Geometric parameters (Å, °)**

| Bond (Å) | Bond Angle (°) |
|----------|----------------|
| N2—C4    | 1.325 (3)      |
| N2—C5    | 1.448 (2)      |
| N2—C3    | 1.387 (3)      |
| N3—C16   | 1.334 (3)      |
| N3—C14   | 1.384 (3)      |
| N3—C1    | 1.455 (3)      |
| N1—C4    | 1.340 (3)      |
| N1—C1    | 1.458 (3)      |
| N1—C2    | 1.380 (3)      |
| N4—C16   | 1.317 (3)      |
| N4—C17   | 1.450 (2)      |
| N4—C15   | 1.386 (3)      |
| C4—H4    | 0.9500         |
| C16—H16  | 0.9500         |
| C17—C18  | 1.394 (3)      |
| C5—C6    | 1.394 (3)      |
| C5—C10   | 1.396 (3)      |
| O2A—H2AA | 0.8694         |
| O2A—H2AB | 0.8679         |
| C12—H12A | 0.9800         |
| C12—H12B | 0.9800         |
| C12—H12C | 0.9800         |
| C12—C6   | 1.507 (3)      |
| C14—C15  | 1.343 (3)      |
| C14—H14  | 0.87 (3)       |
| C18—C24  | 1.508 (3)      |
| C18—C19  | 1.386 (3)      |
| C22—C23  | 1.511 (3)      |
| C22—C21  | 1.396 (3)      |
| C6—C7    | 1.392 (3)      |
| C4—N2—C5 | 124.39 (17)    |
| C4—N2—C3 | 108.93 (17)    |
| N3—N2—C5 | 126.68 (17)    |
| C16—N3—C14 | 108.72 (18)   |
| C16—N3—C1 | 124.13 (19)    |
| C14—N3—C1 | 126.41 (19)    |

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C4—N1—C1 123.56 (19) N1—C1—H1B 109.3
C4—N1—C2 108.94 (18) H1A—C1—H1B 107.9
C2—N1—C1 126.71 (19) C18—C19—H19 119.0
C16—N4—C17 124.98 (17) C18—C19—H20 122.0 (2)
C16—N4—C15 108.91 (17) C20—C19—H19 119.0
C15—N4—C17 126.00 (18) C10—C11—H11A 109.5
N2—C4—N1 107.98 (18) C10—C11—H11B 109.5
N1—C4—H4 126.0 H1A—C11—H11B 109.5
N3—C16—H16 125.8 H11A—C11—H11C 109.5
N4—C16—N3 108.43 (19) H11B—C11—H11C 109.5
N4—C16—H16 125.8 H1AA—O1A—H1AB 104.5
C18—C17—N4 117.50 (17) C6—C7—H7 118.9
C22—C17—N4 118.93 (18) C6—C7—H7 118.9
C22—C17—C18 123.56 (18) C6—C7—C8 122.13 (19)
C6—C5—N2 118.71 (18) C8—C7—C8 122.13 (19)
C6—C5—C10 123.44 (18) C8—C7—H7 118.9
C10—C5—N2 117.85 (18) N1—C2—H2 119.2 (19)
H2AA—O2A—H2AB 104.6 C9—C2—H2 133.8 (19)
H12A—C12—H12B 109.5 C3—C2—N1 106.9 (2)
H12A—C12—H12C 109.5 C3—C2—H2 121.0 (18)
H12B—C12—H12C 109.5 C3—C2—H2 132.2 (19)
C6—C12—H12A 109.5 C3—C2—H2 116.6 (2)
C6—C12—H12B 109.5 C3—C2—H2 116.6 (2)
C6—C12—H12C 109.5 C3—C2—H2 116.6 (2)
N3—C14—H14 121.0 (18) C7—C8—C13 120.2 (2)
C15—C14—N3 106.8 (2) C9—C8—C13 118.53 (19)
C15—C14—H14 132.2 (19) C9—C8—C7 118.53 (19)
C17—C18—C24 121.48 (18) C9—C8—C7 121.2 (2)
C19—C18—C17 117.11 (19) C9—C8—C13 119.0
C19—C18—C24 121.41 (19) C10—C9—H9 119.0
C17—C22—C23 121.64 (19) C8—C9—C10 122.0 (2)
C17—C22—C21 116.6 (2) C8—C9—H9 119.0
C21—C22—C23 121.72 (19) C8—C9—H9 119.0
C5—C6—C12 122.16 (18) C8—C9—H9 119.0
C7—C6—C5 116.87 (19) C8—C9—H9 119.0
C7—C6—C12 120.96 (19) C8—C9—H9 119.0
C18—C24—H24A 109.5 H1A—C13—H13A 109.5
C18—C24—H24B 109.5 H13A—C13—H13B 109.5
C18—C24—H24C 109.5 H13B—C13—H13C 109.5
H24A—C24—H24B 109.5 C22—C21—H21 118.9
H24A—C24—H24C 109.5 C20—C21—C22 122.1 (2)
H24B—C24—H24C 109.5 C20—C21—H21 118.9
N2—C3—H3 120.2 (17) C20—C25—H25A 109.5
C2—C3—N2 107.2 (2) C20—C25—H25B 109.5
C2—C3—H3 132.4 (17) C20—C25—H25C 109.5
C5—C10—C11 121.33 (18) H25A—C25—H25B 109.5
C9—C10—C5 116.98 (19) H25B—C25—H25C 109.5
1,1′-(Ethane-1,2-diyl)bis[3-(2,4,6-trimethylphenyl)imidazolium] dibromide tetrahydrate (est01041d_0ma)

Crystal data

C_{26}H_{32}N_{42}^{+}·2Br^{-}·4H_{2}O

$M_r = 632.42$

Monoclinic, $P2_1/c$

$a = 12.4230$ (3) Å

$b = 13.1447$ (3) Å

$c = 9.2780$ (2) Å

$\beta = 108.379$ (1)°

$V = 1437.78$ (6) Å$^3$

$Z = 2$

$\mu = 2.86$ mm$^{-1}$

$T = 100$ K

Prism, clear colourless

0.15 × 0.15 × 0.05 mm

$\theta = 2.3$–44.6°

9565 reflections

$F(000) = 652$

$D_x = 1.461$ Mg m$^{-3}$

$\lambda$ = 0.71073 Å

$\lambda$ = Mo Kα radiation

Cell parameters from 9565 reflections

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Data collection
Bruker Venture D8 Kappa diffractometer
φ and ω scans
Absorption correction: multi-scans (SADABS; Bruker, 2016)
T min = 0.544, T max = 0.750
28199 measured reflections
3165 independent reflections
3028 reflections with I > 2σ(I)
R int = 0.025
θ max = 27.1°, θ min = 2.8°
h = −15→15
k = −16→16
l = −11→11

Refinement
Refinement on F 2
Least-squares matrix: full
R[F 2 > 2σ(F 2)] = 0.018
wR(F 2) = 0.044
S = 1.10
3165 reflections
194 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement

Special details
Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å 2 )

| x       | y       | z       | U iso*/U eq |
|---------|---------|---------|------------|
| Br01    | −0.19328 (2) | 0.01828 (2) | 0.22033 (2) | 0.01558 (5) |
| O1      | −0.07000 (10) | 0.23627 (9)  | 0.37843 (13) | 0.0268 (2) |
| H1D     | −0.1073 (18)  | 0.1876 (18)  | 0.340 (2)    | 0.036 (5)* |
| H1E     | −0.0360 (18)  | 0.2528 (17)  | 0.326 (3)    | 0.039 (6)* |
| N1      | 0.28176 (8)   | 0.55577 (8)  | 0.81318 (11) | 0.0117 (2) |
| N2      | 0.12491 (9)   | 0.48768 (8)  | 0.67428 (12) | 0.0123 (2) |
| O2      | 0.04964 (10)  | 0.29104 (10) | 0.18398 (14) | 0.0276 (2) |
| H2A     | 0.0823 (18)   | 0.3420 (18)  | 0.200 (2)    | 0.036 (6)* |
| H2B     | 0.016 (2)     | 0.2902 (19)  | 0.096 (3)    | 0.051 (7)* |
| C12     | 0.71245 (12)  | 0.75891 (11) | 0.97675 (18) | 0.0250 (3) |
| H1A     | 0.736386      | 0.766617     | 0.886291     | 0.037* |
| H1B     | 0.768542      | 0.718094     | 1.052694     | 0.037* |
| H1C     | 0.706272      | 0.826152     | 1.019087     | 0.037* |
| C6      | 0.59853 (11)  | 0.70628 (10) | 0.93381 (15) | 0.0166 (3) |
| C10     | 0.51141 (11)  | 0.74146 (10) | 0.98445 (14) | 0.0166 (3) |
| H3      | 0.523920      | 0.800361     | 1.046905     | 0.020* |
| C8      | 0.40605 (11)  | 0.69326 (10) | 0.94673 (14) | 0.0148 (2) |
| C9      | 0.39084 (10)  | 0.60639 (9)  | 0.85679 (13) | 0.0117 (2) |
| C1      | 0.21894 (10)  | 0.53490 (9)  | 0.67103 (14) | 0.0126 (2) |
| H6      | 0.2362 (13)   | 0.5503 (13)  | 0.5832 (19)  | 0.015 (4)* |
| C7      | 0.03385 (10)  | 0.45390 (10) | 0.53959 (14) | 0.0132 (2) |
| H7A     | −0.016689     | 0.406014     | 0.569818     | 0.016* |
### Atomic displacement parameters (Å²)

|     | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{12}$  | $U^{13}$  | $U^{23}$  |
|-----|-----------|-----------|-----------|-----------|-----------|-----------|
| Br01| 0.01802 (7) | 0.01898 (8) | 0.01231 (7) | −0.00240 (5) | 0.00847 (5) | −0.00121 (5) |
| O1  | 0.0320 (6) | 0.0288 (6) | 0.0207 (5) | −0.0124 (5) | 0.0100 (5) | −0.0066 (5) |
| N1  | 0.0124 (5) | 0.0137 (5) | 0.0093 (5) | 0.0009 (4)  | 0.0037 (4) | 0.0012 (4)  |
| N2  | 0.0122 (5) | 0.0141 (5) | 0.0108 (5) | −0.0002 (4) | 0.0039 (4) | 0.0020 (4)  |
| O2  | 0.0332 (6) | 0.0284 (6) | 0.0201 (6) | −0.0086 (5) | 0.0068 (5) | 0.0010 (5)  |
| C12 | 0.0165 (6) | 0.0195 (7) | 0.0338 (8) | −0.0033 (5) | 0.0007 (6) | −0.0009 (6) |
| C6  | 0.0159 (6) | 0.0141 (6) | 0.0158 (6) | −0.0007 (5) | −0.0005 (5) | 0.0031 (5)  |
| C10 | 0.0206 (6) | 0.0125 (6) | 0.0133 (6) | 0.0006 (5)  | 0.0003 (5) | −0.0027 (5) |
| C8  | 0.0168 (6) | 0.0150 (6) | 0.0112 (5) | 0.0045 (5)  | 0.0024 (4) | 0.0008 (5)  |
| C9  | 0.0116 (5) | 0.0134 (6) | 0.0087 (5) | −0.0002 (4) | 0.0012 (4) | 0.0018 (4)  |
| C1  | 0.0141 (6) | 0.0132 (6) | 0.0109 (6) | −0.0006 (4) | 0.0046 (5) | 0.0007 (4)  |
| C7  | 0.0125 (5) | 0.0136 (6) | 0.0120 (5) | −0.0024 (5) | 0.0021 (4) | −0.0005 (5) |
| C4  | 0.0153 (6) | 0.0127 (6) | 0.0094 (5) | 0.0015 (5)  | 0.0027 (4) | 0.0008 (5)  |
| C5  | 0.0136 (5) | 0.0161 (6) | 0.0140 (6) | 0.0018 (5)  | 0.0036 (4) | 0.0020 (5)  |
| C13 | 0.0178 (6) | 0.0179 (7) | 0.0200 (6) | −0.0004 (5) | 0.0070 (5) | −0.0070 (5) |
| C11 | 0.0222 (7) | 0.0238 (7) | 0.0219 (7) | 0.0057 (5)  | 0.0076 (5) | −0.0074 (6) |
| C3  | 0.0155 (6) | 0.0228 (7) | 0.0127 (6) | 0.0005 (5)  | 0.0064 (5) | 0.0050 (5)  |
| C2  | 0.0164 (6) | 0.0224 (7) | 0.0109 (6) | 0.0025 (5)  | 0.0059 (5) | 0.0036 (5)  |

### Geometric parameters (Å, °)

| Bond            | Distance (Å) | Angle (°) |
|-----------------|--------------|-----------|
| O1—H1D         | 0.80 (2)     | C8—C11    | 1.5126 (17) |
| O1—H1E         | 0.77 (2)     | C9—C4     | 1.4042 (17) |
| N1—C9          | 1.4481 (15)  | C1—H6     | 0.928 (17)  |
| N1—C1          | 1.3322 (16)  | C7—C7i    | 1.525 (2)   |
| N1—C2          | 1.3872 (16)  | C7—H7A    | 0.9900      |
| N2—C1          | 1.3314 (16)  | C7—H7B    | 0.9900      |
| N2—C7          | 1.4653 (16)  | C4—C5     | 1.3909 (17) |

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N2—C3 1.3841 (16)  C4—C13 1.5095 (17)
O2—H2A 0.77 (2)  C5—H9 0.9500
O2—H2B 0.79 (3)  C13—H10A 0.9800
C12—H1A 0.9800  C13—H10B 0.9800
C12—H1B 0.9800  C13—H10C 0.9800
C12—H1C 0.9800  C11—H11A 0.9800
C12—C6 1.5114 (18)  C11—H11B 0.9800
C6—C10 1.3880 (19)  C11—H11C 0.9800
C6—C5 1.3915 (18)  C3—H12 0.928 (18)
C10—H3 0.9500  C3—C2 1.3506 (19)
C10—C8 1.3959 (18)  C2—H13 0.932 (19)
C8—C9 1.3915 (18)

H1D—O1—H1E 107 (2)  N2—C7—H7A 109.8
C1—N1—C9 125.15 (10)  N2—C7—H7B 109.8
C1—N1—C2 108.51 (11)  N2—C7—C7i 109.8
C2—N1—C9 126.34 (10)  C7i—C7—H7A 109.8
C1—N2—C7 124.67 (11)  C7i—C7—H7B 109.8
C1—N2—C3 108.89 (11)  H7A—C7—H7B 108.3
C3—N2—C7 126.41 (11)  C9—C4—C13 123.34 (11)
H2A—O2—H2B 106 (2)  C5—C4—C9 117.41 (11)
H1A—C12—H1B 109.5  C4—C5—C6 122.21 (12)
H1A—C12—H1C 109.5  C4—C5—H9 118.9
C6—C12—H1C 109.5  C4—C13—H10C 109.5
C10—C6—C12 121.57 (12)  C4—C13—C10 109.5
C10—C6—C5 118.20 (12)  C5—C13—H10B 109.5
C5—C6—C12 120.23 (12)  C1—C2—C3 129.0 (11)
C6—C10—H3 118.9  C8—C11—H11C 109.5
C6—C10—C8 122.24 (12)  C8—C11—H11B 109.5
C8—C10—H3 118.9  C8—C11—H11A 109.5
C10—C8—C11 119.19 (12)  H11A—C11—H11B 109.5
C9—C8—C10 117.53 (12)  H11A—C11—H11C 109.5
C9—C8—C11 123.28 (12)  H11B—C11—H11C 109.5
C8—C9—N1 118.87 (11)  N2—C3—H12 120.6 (11)
C8—C9—C4 122.39 (11)  C2—C3—N2 106.87 (11)
C4—C9—N1 118.73 (11)  C2—C3—H12 132.6 (11)
N1—C1—H6 126.8 (10)  N1—C2—H13 123.8 (11)
N2—C1—N1 108.54 (11)  C3—C2—N1 107.19 (11)
N2—C1—H6 124.6 (10)  C3—C2—H13 129.0 (11)
N2—C7—C7i 109.28 (13)
N1—C9—C4—C5 177.64 (10)  C1—N1—C9—C4 −53.80 (17)
N1—C9—C4—C13 −2.91 (18)  C1—N1—C2—C3 0.17 (15)
N2—C3—C2—N1 0.44 (15)  C1—N2—C7—C7i −73.94 (17)
C12—C6—C10—C8 −179.51 (12)  C1—N2—C3—C2 −0.90 (15)
| Bond/Angle/Distance | Value (°/Å) |
|--------------------|-------------|
| C12—C6—C5—C4      | 178.27 (12) |
| C6—C10—C8—C9      | 0.85 (19)   |
| C6—C10—C8—C11     | −179.00 (12)|
| C10—C6—C5—C4      | −0.78 (19)  |
| C10—C8—C9—N1      | −178.79 (11)|
| C10—C8—C9—C4      | 0.00 (18)   |
| C8—C9—C4—C5       | −1.16 (18)  |
| C8—C9—C4—C13      | 178.30 (12) |
| C9—N1—C1—N2       | 179.64 (11) |
| C9—N1—C2—C3       | 179.79 (12) |
| C9—C4—C5—C6       | 1.56 (18)   |
| C1—N1—C9—C8       | 125.04 (13) |
| C7—N2—C1—N1       | 179.04 (11) |
| C7—N2—C3—C2       | −178.88 (12)|
| C5—C6—C10—C8      | −0.48 (19)  |
| C13—C4—C5—C6      | −177.92 (12)|
| C11—C8—C9—N1      | 1.04 (18)   |
| C11—C8—C9—C4      | 179.84 (12) |
| C3—N2—C1—N2       | 125.04 (13) |
| C3—N2—C7—C7i      | 103.73 (16) |
| C2—N1—C9—C8       | −54.53 (17) |
| C2—N1—C9—C4       | 126.64 (13) |
| C2—N1—C1—N2       | −0.73 (14)  |

Symmetry code: (i) −x, −y+1, −z+1.