

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

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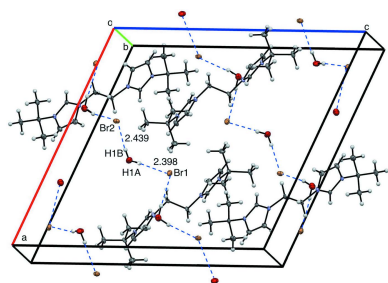
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The crystal structures of ligand precursor bis(imidazolium) salts 1,1'-methylenebis(3-*tert*-butylimidazolium) dibromide monohydrate, $C_{15}H_{26}N_4^{+} \cdot 2Br^{-} \cdot H_2O$ or $[^{tBu}NHC_2Me][Br]_2 \cdot H_2O$, 1,1'-(ethane-1,2-diyl)bis(3-*tert*-butylimidazolium) dibromide dihydrate, $C_{16}H_{28}N_4^{+} \cdot 2Br^{-} \cdot 2H_2O$ or $[^{tBu}NHC_2Et][Br]_2 \cdot 2H_2O$, 1,1'-methylenebis[3-(2,4,6-trimethylphenyl)imidazolium] dibromide dihydrate, $C_{25}H_{30}N_4^{2+} \cdot 2Br^{-} \cdot 2H_2O$ or $[^{Mes}NHC_2Me][Br]_2 \cdot 2H_2O$, and 1,1'-(ethane-1,2-diyl)bis[3-(2,4,6-trimethylphenyl)imidazolium] dibromide tetrahydrate, $C_{26}H_{32}N_4^{2+} \cdot 2Br^{-} \cdot 4H_2O$ or $[^{Mes}NHC_2Et][Br]_2 \cdot 4H_2O$, are reported. At 293 K, $[^{tBu}NHC_2Me][Br]_2 \cdot H_2O$ crystallizes in the $P2_1/c$ space group, while $[^{tBu}NHC_2Et][Br]_2 \cdot 2H_2O$ crystallizes in the $P2_1/n$ space group at 100 K. At 112 K, $[^{Mes}NHC_2Me][Br]_2 \cdot 2H_2O$ crystallizes in the orthorhombic space group $Pccn$ while $[^{Mes}NHC_2Et][Br]_2 \cdot 4H_2O$ crystallizes in the $P2_1/c$ 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 (NHC₂) ligands, which can be used to stabilize a variety of metal complexes and catalysts. Bis(imidazolium) salts, $[^R NHC_2 R^1][X]_2$ 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 (*t*Bu) and mesityl (*Mes*) exterior *R* groups



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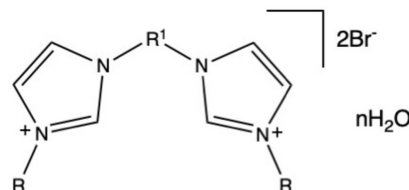
and methylene (Me) or ethylene (Et) linking R^1 groups. $[\text{MesNHC}_2\text{Et}][\text{Br}]_2$ was even reported to be a stand-alone catalyst for the conversion of arylaldehydes to carboxylic acids in combination with water and K_2CO_3 in DMSO (Yang *et al.*, 2013). Methylene linkers are quite commonly 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 Rh^I 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, $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Me}][\text{Br}]_2$ and $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Et}][\text{Br}]_2$ were used as precursors for synthesis of rhodium complexes (Leung *et al.*, 2006). $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Et}][\text{Cl}]_2$ 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* $\text{Pd}(\text{OAc})_2$ -assisted deprotometalation (Wierenga *et al.*, 2019), palladium complexes *via* silver transmetalation (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 carbonate 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 $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Me}][\text{Cl}]_2$, $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Me}][\text{Br}]_2$, $[\text{MesNHC}_2\text{Me}][\text{Cl}]_2$, $[\text{MesNHC}_2\text{Me}][\text{Br}]_2$, and $[\text{MesNHC}_2\text{Et}][\text{Br}]_2$ (Meyer *et al.*, 2011).

When used for stabilizing bimetallic systems, $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Et}][\text{Cl}]_2$ 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 $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Et}][\text{Br}]_2$ 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 $[\text{MesNHC}_2\text{Et}][\text{Br}]_2$ were used to synthesize bimetallic gold catalysts for cross-coupling and hydroamination reactions (Baron *et al.*, 2018).



$[\text{t}^{\text{Bu}}\text{NHC}_2\text{Me}][\text{Br}]_2$ $R = \text{tert-butyl}$; $R^1 = \text{CH}_2$, $n = 1$

$[\text{t}^{\text{Bu}}\text{NHC}_2\text{Et}][\text{Br}]_2$ $R = \text{tert-butyl}$; $R^1 = \text{CH}_2\text{CH}_2$, $n = 2$

$[\text{MesNHC}_2\text{Me}][\text{Br}]_2$ $R = 2,4,6\text{-trimethylphenyl}$; $R^1 = \text{CH}_2$, $n = 2$

$[\text{MesNHC}_2\text{Et}][\text{Br}]_2$ $R = 2,4,6\text{-trimethylphenyl}$; $R^1 = \text{CH}_2\text{CH}_2$, $n = 4$

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 $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Me}][\text{Br}]_2 \cdot \text{H}_2\text{O}$ while Fig. 2 depicts $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Et}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$.

Bond distances in the imidazolium rings of $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Me}][\text{Br}]_2 \cdot \text{H}_2\text{O}$ and $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Et}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ 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 $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Me}][\text{Br}]_2 \cdot \text{H}_2\text{O}$ 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 $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Et}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ has an N1—C2 distance of 1.388 (3) Å, an N2—C3 distance of 1.384 (3) Å, an

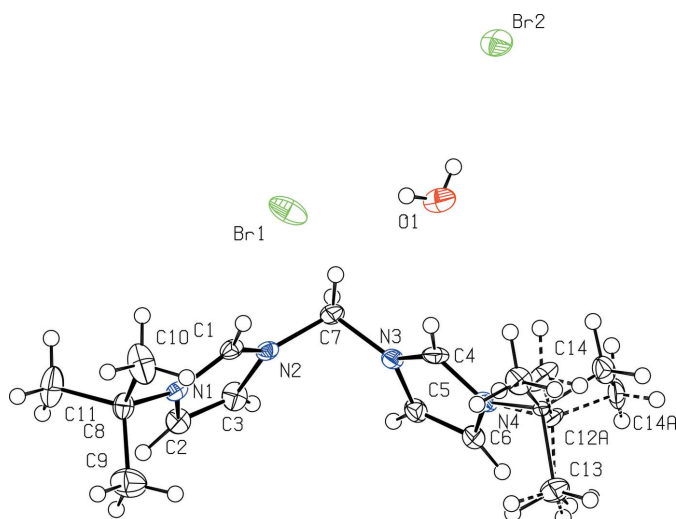


Figure 1
View of $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Me}][\text{Br}]_2 \cdot \text{H}_2\text{O}$ with 50% probability ellipsoids.

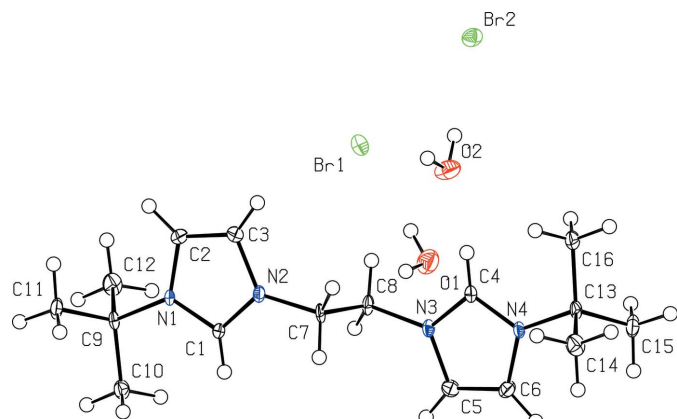


Figure 2
View of $[\text{t}^{\text{Bu}}\text{NHC}_2\text{Et}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ with 50% probability ellipsoids.

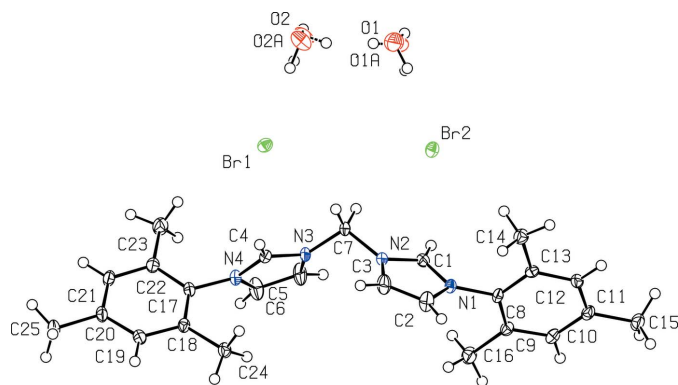


Figure 3
View of $[\text{MesNHC}_2\text{Me}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ with 50% probability ellipsoids.

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 $[\text{tBuNHC}_2\text{Me}][\text{Br}]_2 \cdot \text{H}_2\text{O}$ and 1.468 (3) Å for $[\text{tBuNHC}_2\text{Et}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$.

Bond angles in the imidazolium rings are also quite similar in $[\text{tBuNHC}_2\text{Me}][\text{Br}]_2 \cdot \text{H}_2\text{O}$ and $[\text{tBuNHC}_2\text{Et}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$. For $[\text{tBuNHC}_2\text{Me}][\text{Br}]_2 \cdot \text{H}_2\text{O}$, 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 $[\text{tBuNHC}_2\text{Et}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$, 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 $[\text{MesNHC}_2\text{Me}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ while Fig. 4 depicts $[\text{MesNHC}_2\text{Et}][\text{Br}]_2 \cdot 4\text{H}_2\text{O}$. Notably, $[\text{MesNHC}_2\text{Et}][\text{Br}]_2 \cdot 4\text{H}_2\text{O}$ 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 $[\text{MesNHC}_2\text{Me}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ and $[\text{MesNHC}_2\text{Et}][\text{Br}]_2 \cdot 4\text{H}_2\text{O}$ are mostly the same within experimental error, with backbone C2—C3 distances of 1.344 (3) and 1.3506 (19) Å, respectively. N—C distances are also mostly the same with $[\text{MesNHC}_2\text{Me}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ 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, $[\text{MesNHC}_2\text{Et}][\text{Br}]_2 \cdot 4\text{H}_2\text{O}$ 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 $[\text{MesNHC}_2\text{Me}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ and 1.4653 (16) Å for $[\text{MesNHC}_2\text{Et}][\text{Br}]_2 \cdot 4\text{H}_2\text{O}$.

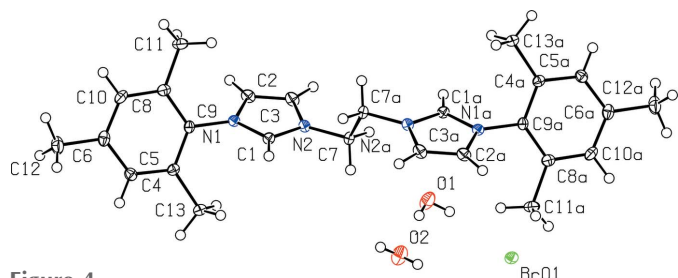


Figure 4
View of $[\text{MesNHC}_2\text{Et}][\text{Br}]_2 \cdot 4\text{H}_2\text{O}$ with 50% probability ellipsoids.

Bond angles in the imidazolium rings are also mostly the same for $[\text{MesNHC}_2\text{Me}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ and $[\text{MesNHC}_2\text{Et}][\text{Br}]_2 \cdot 4\text{H}_2\text{O}$. For $[\text{MesNHC}_2\text{Me}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$, bond angles include C1—N1—C2 at 108.92 (17)°, N1—C2—C3 at 107.20 (19)°, C2—C3—N2 at 106.95 (19)°, C3—N2—C1 at 108.96 (17)°, and N2—C1—N1 at 107.96 (18)°. For $[\text{MesNHC}_2\text{Et}][\text{Br}]_2 \cdot 4\text{H}_2\text{O}$, bond angles include C1—N1—C2 at 108.51 (11)°, N1—C2—C3 at 107.19 (11)°, C2—C3—N2 at 106.87 (11)°, C3—N2—C1 at 108.89 (11)°, and N2—C1—N1 at 108.54 (11)°. Overall, these data support that changing the linker R^1 group from methylene to ethylene does not significantly affect the imidazolium ring structures.

3. Supramolecular features

The supramolecular structure of $[\text{tBuNHC}_2\text{Me}][\text{Br}]_2 \cdot \text{H}_2\text{O}$ 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 $[\text{tBuNHC}_2\text{Et}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ 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 $[\text{MesNHC}_2\text{Me}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ is also stabilized by hydrogen bonding (Fig. 7, Table 1). No π -stacking interactions were found as distances between

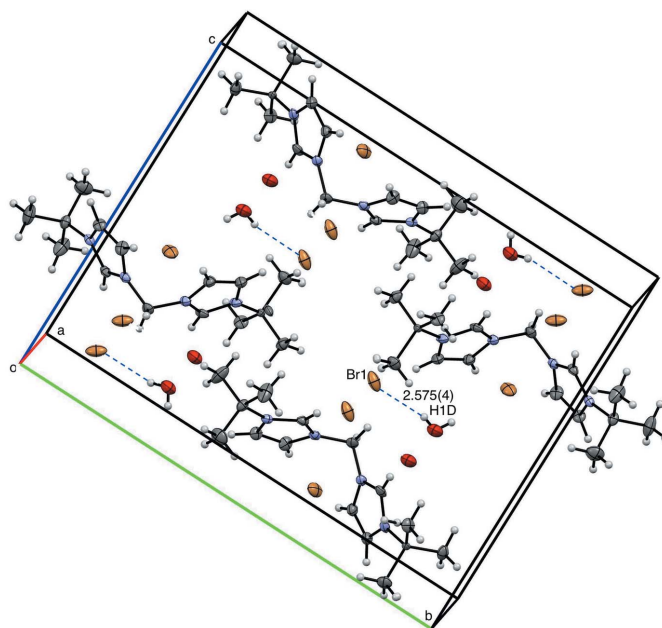


Figure 5
View of four molecules of $[\text{tBuNHC}_2\text{Me}][\text{Br}]_2 \cdot \text{H}_2\text{O}$ with 50% probability ellipsoids, highlighting intermolecular distances. Disordered *tert*-butyl groups are omitted for clarity.

Table 1

Intermolecular distances (Å) in the unit cells of $[^R\text{NHC}_2\text{R}_1][\text{X}]_2 \cdot n\text{H}_2\text{O}$.

Standard deviations for distances including some H atoms are omitted where H atoms were positionally fixed.

Compound	Atoms	Distance
$[^t\text{BuNHC}_2\text{Me}][\text{Br}]_2 \cdot \text{H}_2\text{O}$	Br1...H1D	2.575 (4)
$[^t\text{BuNHC}_2\text{Et}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$	Br1...H1A	2.398
	Br2...H1B	2.439
$[^{\text{Mes}}\text{NHC}_2\text{Me}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$	Br1...H2A	2.413
	Br2...H1A	2.463
	O1A...H2B	2.125
$[^{\text{Mes}}\text{NHC}_2\text{Et}][\text{Br}]_2 \cdot 4\text{H}_2\text{O}$	O1...H2B	1.994 (2)
	O2...H1E	2.001 (3)
	Br1...H1D	2.585 (2)

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 Br1...H2A at 2.413 Å, Br2...H1A at 2.463 Å, and O1A...H2B at 2.125 Å.

The supramolecular structure of $[^{\text{Mes}}\text{NHC}_2\text{Et}][\text{Br}]_2 \cdot 4\text{H}_2\text{O}$ is also stabilized by hydrogen bonding (Fig. 8, Table 1). No π -stacking is observed between mesityl groups, similar to $[^{\text{Mes}}\text{NHC}_2\text{Me}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ as the distance between centroids of the mesityl groups of neighboring fragments is greater than 4.5 Å (Janiak, 2000). Hydrogen-bonding interactions include O1...H2B at 1.994 (2) Å, O2...H1E at 2.001 (3) Å, and Br1...H1D 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 $[^t\text{BuNHC}_2\text{Me}][\text{Br}]_2 \cdot \text{H}_2\text{O}$, $[^t\text{BuNHC}_2\text{Et}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$, or $[^{\text{Mes}}\text{NHC}_2\text{Et}][\text{Br}]_2 \cdot 4\text{H}_2\text{O}$. A deposited dataset for $[^{\text{Mes}}\text{NHC}_2\text{Me}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ was found (Rheingold, 2019) with a slightly higher *R*1 of 3.94% and data collection at a higher

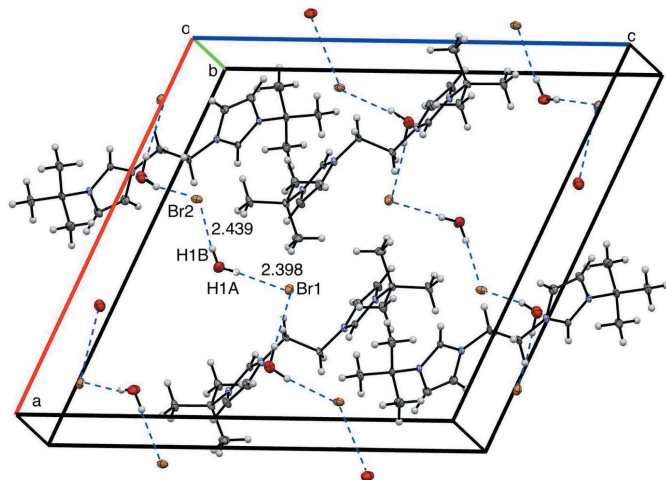


Figure 6
View of four molecules of $[^t\text{BuNHC}_2\text{Et}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ with 50% probability ellipsoids, highlighting intermolecular distances.

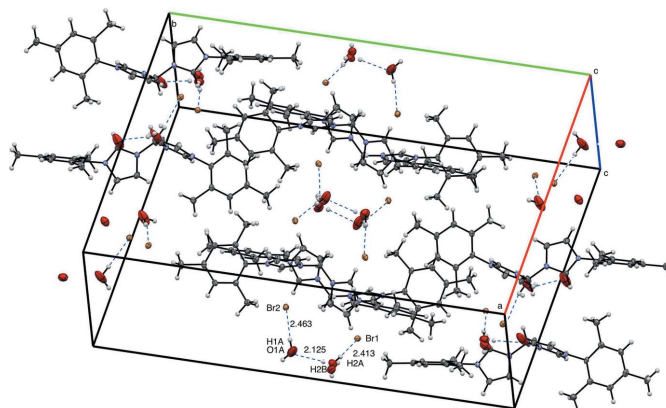


Figure 7
View of eight molecules of $[^{\text{Mes}}\text{NHC}_2\text{Me}][\text{Br}]_2 \cdot 2\text{H}_2\text{O}$ with 50% probability ellipsoids, highlighting intermolecular distances.

temperature of 150 K, as compared to *R*1 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. ^1H

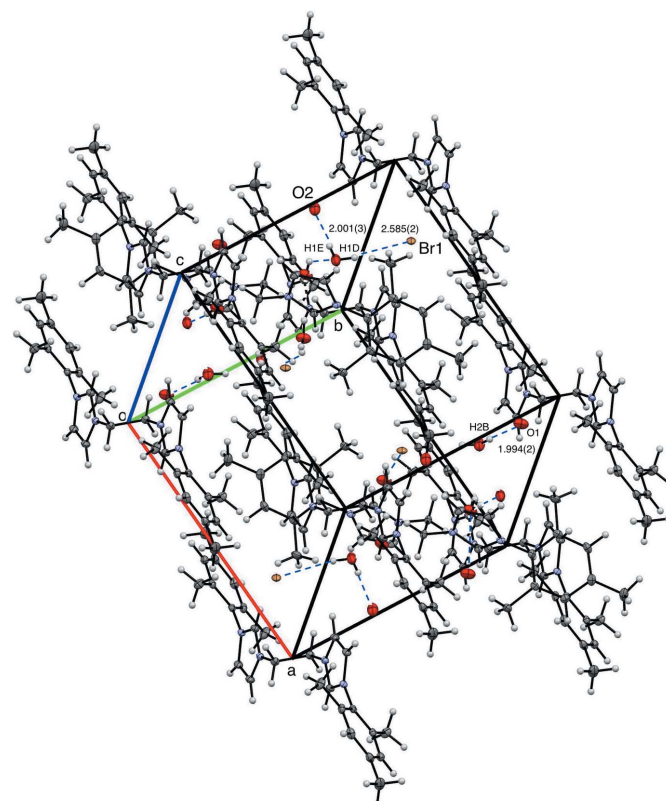


Figure 8
View of eight molecules of $[^{\text{Mes}}\text{NHC}_2\text{Et}][\text{Br}]_2 \cdot 4\text{H}_2\text{O}$ with 50% probability ellipsoids, highlighting intermolecular distances.

NMR data were collected on a Varian 400 MHz spectrometer and referenced to residual CHCl_3 .

Synthesis of 1-*tert*-butyl-1H-imidazole, (^{*t*Bu}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 H_2O 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 ^{*t*Bu}Im, and characterized by ^1H NMR spectroscopy in CDCl_3 .

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 ~ 75 mL of methanol and stirred for 24 h after which the solution turned orange with a yellow precipitate. 11.86 g (221.8 mmol, 2 eq.) of ammonium chloride, 18.00 g (221.8 mmol, 2 eq.) of 37% formaldehyde, and 300 mL of methanol were added, and the solution was refluxed for 24 h at 373 K, at which point the solution was deep brown. After being cooled to room temperature, 25.57 g (221.8 mmol, 2 eq.) of 85% phosphoric acid were added dropwise over ten minutes and the solution was refluxed for 16 h at 368 K. Excess solvent was removed *in vacuo* at 313 K, and the viscous brown residue was poured over ~ 300 g of ice and neutralized to pH 10 with a saturated solution of potassium hydroxide, resulting in a clear solution with a chunky brown precipitate. The product was taken into diethyl ether by washing the solution three times with ~ 100 mL of diethyl ether. The diethyl ether solution was washed thrice with ~ 100 mL of water, thrice with ~ 100 mL of brine, and dried overnight over sodium sulfate. Sodium sulfate solids were gravity filtered from the solution and the solvent was removed *in vacuo* resulting in a brown solid. The product was recrystallized from hot ethyl acetate, resulting in 9.49 g (46% yield) of tan crystals, which were characterized by ^1H NMR spectroscopy and identified as ^{Mes}Im.

Synthesis of 1,1'-di(*tert*-butyl)-3,3'-methylene-diimidazolium dibromide, [^{*t*Bu}NHC₂Me][Br]₂. 1.850 g (14.9 mmol, 2.5 eq.) of ^{*t*Bu}Im and 0.4194 mL (5.9 mmol, 1 eq.) of dibromomethane, a stir bar, and ~ 20 mL of toluene were stirred in a 50 mL round-bottomed flask. The solution was then heated to 423 K and refluxed for 46 h, resulting in the formation of a dark orange–brown solution. The solution was cooled in an ice bath, resulting in a fine white precipitate which was collected *via* vacuum filtration, washed twice with ~ 5 mL of cold

toluene, filtered and dried. 1.120 g (78.02% yield) of a fine white solid identified as [^{*t*Bu}NHC₂Me][Br]₂ were isolated. Crystals suitable for X-ray diffraction were obtained by recrystallization from hot methanol. The product was characterized by ^1H NMR spectroscopy. The ^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 [^{*t*Bu}NHC₂Et][Br]₂. A 250 mL round-bottomed flask was charged with 2.017 g (16.2 mmol, 2.5 eq.) of ^{*t*Bu}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 [^{*t*Bu}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 ^{Mes}Im we added to a 50 mL round-bottomed flask with a stir bar and ~ 20 mL of toluene. 0.754 mL (10.72 mmol, 1 eq.) of dibromomethane were added and the solution was refluxed at 423 K for 20 h. The solution was cooled in an ice bath, resulting in a white precipitate. The white solid was recrystallized from ~ 12 mL of hot methanol. The product was obtained in 17% yield (1.10 g) as tan crystals identified as [^{Mes}NHC₂Me][Br]₂ suitable for X-ray diffraction and characterized by ^1H NMR.

Synthesis of 1,1'-di(mesityl)-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 ^{Mes}Im, 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 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 [^{Mes}NHC₂Et][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 CH_2 and 0.98 Å for CH_3 . Methyl H atoms were allowed to rotate but not to tip to best fit the experimental electron density. U_{iso} values of riding H atoms were set to 1.2 times $U_{\text{eq}}(\text{C})$ for CH and CH_2 , and 1.5 times $U_{\text{eq}}(\text{C})$ for CH_3 and H_2O . For [^{*t*Bu}NHC₂Me][Br]₂, the SADI command of *SHELX* was used to model disorder in one of the *tert*-butyl moieties for N4–C0AA and N4–C12,

Table 2
Experimental details.

	[^{Mes} NHC ₂ Me][Br] ₂ ·2H ₂ O	[^t BuNHC ₂ Me][Br] ₂ ·H ₂ O	[^t BuNHC ₂ Et][Br] ₂ ·2H ₂ O	[^{Mes} NHC ₂ Et][Br] ₂ ·4H ₂ O
Crystal data				
Chemical formula	C ₂₅ H ₃₀ N ₄ ²⁺ ·2Br [−] ·2H ₂ O	C ₁₅ H ₂₆ N ₄ ⁺ ·2Br [−] ·H ₂ O	C ₁₆ H ₂₈ N ₄ ²⁺ ·2Br [−] ·2H ₂ O	C ₂₆ H ₃₂ N ₄ ²⁺ ·2Br [−] ·4H ₂ O
<i>M_r</i>	582.38	440.23	472.27	632.42
Crystal system, space group	Orthorhombic, <i>Pccn</i>	Monoclinic, <i>P2₁/c</i>	Monoclinic, <i>P2₁/n</i>	Monoclinic, <i>P2₁/c</i>
Temperature (K)	112	293	100	100
<i>a</i> , <i>b</i> , <i>c</i> (Å)	21.5695 (6), 28.3385 (6), 8.9401 (2)	7.211 (5), 18.311 (17), 15.409 (5)	17.1577 (6), 7.3180 (2), 18.2712 (6)	12.4230 (3), 13.1447 (3), 9.2780 (2)
α , β , γ (°)	90, 90, 90	90, 101.35 (3), 90	90, 112.786 (1), 90	90, 108.379 (1), 90
<i>V</i> (Å ³)	5464.6 (2)	1995 (2)	2115.09 (12)	1437.78 (6)
<i>Z</i>	8	4	4	2
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ^{−1})	2.99	4.07	3.85	2.86
Crystal size (mm)	0.4 × 0.3 × 0.25	0.3 × 0.15 × 0.1	0.2 × 0.1 × 0.05	0.15 × 0.15 × 0.05
Data collection				
Diffractometer	Bruker Venture D8 Kappa	Bruker APEXII CCD	Bruker Venture D8 Kappa	Bruker Venture D8 Kappa
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2016)	Multi-scan (<i>SADABS</i> ; Bruker, 2016)	Multi-scan (<i>SADABS</i> ; Bruker, 2016)	Multi-scan (<i>SADABS</i> ; Bruker, 2016)
<i>T_{min}</i> , <i>T_{max}</i>	0.386, 0.748	0.544, 0.747	0.496, 0.748	0.544, 0.750
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	39085, 5954, 5530	33565, 4402, 3780	31474, 4664, 4168	28199, 3165, 3028
<i>R_{int}</i> (sin θ/λ) _{max} (Å ^{−1})	0.035 0.641	0.043 0.641	0.059 0.641	0.025 0.641
Refinement				
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	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
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ^{−3})	0.42, −0.54	1.49, −1.10	0.60, −0.61	0.33, −0.29

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *olex2.solve* (Bourhis *et al.*, 2015), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b), and *OLEX2* (Dolomanov *et al.*, 2009).

C0AA—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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supporting information

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

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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 (scs01006_0m)

Crystal data

$C_{15}H_{26}N_4^+ \cdot 2Br^- \cdot H_2O$
 $M_r = 440.23$
 Monoclinic, $P2_1/c$
 $a = 7.211$ (5) Å
 $b = 18.311$ (17) Å
 $c = 15.409$ (5) Å
 $\beta = 101.35$ (3)°
 $V = 1995$ (2) Å³
 $Z = 4$

$F(000) = 896$
 $D_x = 1.466$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 9889 reflections
 $\theta = 2.6$ – 30.2 °
 $\mu = 4.07$ mm⁻¹
 $T = 293$ K
 Prism, clear colourless
 $0.3 \times 0.15 \times 0.1$ mm

Data collection

Bruker APEXII CCD
 diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2016)
 $T_{\min} = 0.544$, $T_{\max} = 0.747$
 33565 measured reflections

4402 independent reflections
 3780 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.043$
 $\theta_{\max} = 27.1$ °, $\theta_{\min} = 2.7$ °
 $h = -9 \rightarrow 8$
 $k = -23 \rightarrow 23$
 $l = -19 \rightarrow 19$

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
 $w = 1/[\sigma^2(F_o^2) + (0.0006P)^2 + 4.1194P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 1.49 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -1.10 \text{ e } \text{\AA}^{-3}$$

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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Br2	0.83773 (4)	0.86331 (2)	0.55378 (2)	0.03323 (9)	
Br1	1.05299 (4)	0.61882 (2)	0.35706 (2)	0.03727 (10)	
N3	0.4514 (3)	0.71177 (11)	0.18236 (13)	0.0175 (4)	
N1	0.5823 (3)	0.48054 (11)	0.16964 (13)	0.0162 (4)	
O1	0.8362 (4)	0.78047 (16)	0.35775 (17)	0.0419 (6)	
N2	0.4742 (3)	0.58205 (11)	0.21324 (13)	0.0170 (4)	
N4	0.5288 (3)	0.80162 (12)	0.10500 (14)	0.0225 (5)	
C1	0.6311 (4)	0.54357 (13)	0.21172 (16)	0.0181 (5)	
H5	0.753852	0.558228	0.235943	0.022*	
C5	0.2905 (4)	0.73014 (14)	0.12095 (18)	0.0221 (5)	
C4	0.5935 (4)	0.75572 (14)	0.17061 (16)	0.0211 (5)	
H9	0.716674	0.754245	0.203041	0.025*	
C7	0.4681 (4)	0.65531 (14)	0.25048 (16)	0.0212 (5)	
H8A	0.361201	0.658686	0.279890	0.025*	
H8B	0.582372	0.663573	0.294434	0.025*	
C8	0.7152 (4)	0.42139 (14)	0.15085 (18)	0.0207 (5)	
C6	0.3396 (4)	0.78602 (15)	0.07277 (19)	0.0251 (6)	
C2	0.3869 (4)	0.47957 (15)	0.14242 (18)	0.0216 (5)	
C3	0.3195 (4)	0.54263 (15)	0.16910 (19)	0.0239 (6)	
C13	0.5981 (4)	0.86039 (16)	−0.02694 (18)	0.0290 (6)	
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)	
H3A	0.676730	0.356294	0.254815	0.060*	
H3B	0.746068	0.311841	0.180278	0.060*	
H3C	0.534897	0.337462	0.166825	0.060*	
C10	0.9155 (4)	0.44460 (17)	0.1903 (3)	0.0408 (8)	
H1A	0.944282	0.489150	0.162880	0.061*	
H1B	1.002017	0.407121	0.180300	0.061*	
H1C	0.927348	0.452256	0.252799	0.061*	
C9	0.6908 (5)	0.4157 (2)	0.0510 (2)	0.0399 (8)	
H4A	0.564299	0.400122	0.026305	0.060*	

H4B	0.779299	0.380851	0.036360	0.060*	
H4C	0.713383	0.462547	0.027048	0.060*	
C15	0.8712 (15)	0.8362 (6)	0.1050 (8)	0.0245 (19)	0.54 (3)
H13A	0.906281	0.834192	0.168381	0.037*	0.54 (3)
H13B	0.948184	0.871611	0.082775	0.037*	0.54 (3)
H13C	0.889802	0.789067	0.080737	0.037*	0.54 (3)
C14	0.6197 (18)	0.9291 (5)	0.1206 (7)	0.025 (2)	0.54 (3)
H14A	0.487306	0.939908	0.103235	0.038*	0.54 (3)
H14B	0.692055	0.967891	0.101603	0.038*	0.54 (3)
H14C	0.652454	0.924641	0.183859	0.038*	0.54 (3)
H1D	0.905 (6)	0.746 (2)	0.365 (3)	0.061 (15)*	
C14A	0.530 (3)	0.9398 (5)	0.0928 (10)	0.034 (3)	0.46 (3)
H00A	0.398530	0.942603	0.064831	0.050*	0.46 (3)
H00B	0.596718	0.980230	0.074007	0.050*	0.46 (3)
H00C	0.540922	0.941501	0.155917	0.050*	0.46 (3)
C12A	0.614 (2)	0.8681 (6)	0.0668 (11)	0.021 (3)	0.46 (3)
C15A	0.823 (2)	0.8614 (11)	0.1096 (10)	0.038 (3)	0.46 (3)
H1AA	0.837624	0.866529	0.172622	0.057*	0.46 (3)
H1AB	0.893103	0.898947	0.087072	0.057*	0.46 (3)
H1AC	0.868755	0.814380	0.096076	0.057*	0.46 (3)
H1E	0.839 (5)	0.800 (2)	0.399 (3)	0.041 (12)*	
H6	0.331 (4)	0.4388 (16)	0.1108 (19)	0.023 (7)*	
H11	0.270 (5)	0.8115 (18)	0.027 (2)	0.035 (9)*	
H10	0.175 (5)	0.7062 (18)	0.118 (2)	0.037 (9)*	
H7	0.201 (5)	0.5612 (17)	0.164 (2)	0.034 (9)*	

Atomic displacement parameters ($\text{\AA}^2$)

	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)

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)

Geometric parameters (Å, °)

N3—C5	1.387 (3)	C13—H15D	0.9600
N3—C4	1.343 (3)	C13—H15E	0.9600
N3—C7	1.461 (3)	C13—H15F	0.9600
N1—C1	1.337 (3)	C13—C12	1.595 (13)
N1—C8	1.512 (3)	C13—C12A	1.434 (16)
N1—C2	1.389 (3)	C12—C15	1.530 (10)
O1—H1D	0.80 (4)	C12—C14	1.519 (10)
O1—H1E	0.73 (4)	C11—H3A	0.9600
N2—C1	1.337 (3)	C11—H3B	0.9600
N2—C7	1.463 (3)	C11—H3C	0.9600
N2—C3	1.389 (3)	C10—H1A	0.9600
N4—C4	1.327 (3)	C10—H1B	0.9600
N4—C6	1.387 (4)	C10—H1C	0.9600
N4—C12	1.525 (9)	C9—H4A	0.9600
N4—C12A	1.531 (11)	C9—H4B	0.9600
C1—H5	0.9300	C9—H4C	0.9600
C5—C6	1.352 (4)	C15—H13A	0.9600
C5—H10	0.94 (3)	C15—H13B	0.9600
C4—H9	0.9300	C15—H13C	0.9600
C7—H8A	0.9700	C14—H14A	0.9600
C7—H8B	0.9700	C14—H14B	0.9600
C8—C11	1.529 (4)	C14—H14C	0.9600
C8—C10	1.514 (4)	C14A—H00A	0.9600
C8—C9	1.518 (4)	C14A—H00B	0.9600
C6—H11	0.91 (3)	C14A—H00C	0.9600
C2—C3	1.348 (4)	C14A—C12A	1.531 (12)
C2—H6	0.94 (3)	C12A—C15A	1.527 (11)
C3—H7	0.91 (3)	C15A—H1AA	0.9600
C13—H15A	0.9600	C15A—H1AB	0.9600
C13—H15B	0.9600	C15A—H1AC	0.9600
C13—H15C	0.9600		
C5—N3—C7	127.0 (2)	C12A—C13—H15F	109.5
C4—N3—C5	108.7 (2)	N4—C12—C13	102.7 (7)
C4—N3—C7	124.3 (2)	N4—C12—C15	113.1 (8)
C1—N1—C8	126.5 (2)	C15—C12—C13	111.0 (8)
C1—N1—C2	108.2 (2)	C14—C12—N4	105.6 (7)
C2—N1—C8	125.2 (2)	C14—C12—C13	111.6 (6)
H1D—O1—H1E	112 (4)	C14—C12—C15	112.4 (7)

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	C12A—C15A—H1AC	109.5
C12—C13—H15B	109.5	H1AA—C15A—H1AB	109.5
C12—C13—H15C	109.5	H1AA—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—N2	−0.4 (3)	C7—N2—C3—C2	177.5 (2)
C1—N1—C8—C11	122.5 (3)	C8—N1—C1—N2	178.4 (2)
C1—N1—C8—C10	1.6 (3)	C8—N1—C2—C3	−177.9 (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)
C1—N2—C7—N3	97.2 (3)	C6—N4—C12—C15	−156.5 (6)
C1—N2—C3—C2	0.9 (3)	C6—N4—C12—C14	80.3 (8)
C5—N3—C4—N4	0.6 (3)	C6—N4—C12A—C13	−59.5 (11)
C5—N3—C7—N2	75.7 (3)	C6—N4—C12A—C14A	67.4 (12)
C4—N3—C5—C6	−0.2 (3)	C6—N4—C12A—C15A	−173.5 (9)
C4—N3—C7—N2	−105.5 (3)	C2—N1—C1—N2	0.8 (3)
C4—N4—C6—C5	0.7 (3)	C2—N1—C8—C11	−60.3 (3)
C4—N4—C12—C13	142.2 (5)	C2—N1—C8—C10	178.8 (3)
C4—N4—C12—C15	22.6 (10)	C2—N1—C8—C9	60.6 (3)
C4—N4—C12—C14	−100.7 (6)	C3—N2—C1—N1	−1.0 (3)
C4—N4—C12A—C13	127.1 (8)	C3—N2—C7—N3	−78.8 (3)
C4—N4—C12A—C14A	−105.9 (8)	C12—N4—C4—N3	179.9 (5)
C4—N4—C12A—C15A	13.1 (14)	C12—N4—C6—C5	179.8 (6)
C7—N3—C5—C6	178.7 (2)	C12A—N4—C4—N3	173.0 (8)
C7—N3—C4—N4	−178.3 (2)	C12A—N4—C6—C5	−174.3 (7)

1,1'-(Ethane-1,2-diyl)bis(3-*tert*-butylimidazolium) dibromide dihydrate (est01043_0m)

Crystal data

C₁₆H₂₈N₄²⁺·2Br[−]·2H₂O

M_r = 472.27

Monoclinic, *P*2₁/*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

F(000) = 968

D_x = 1.483 Mg m^{−3}

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 9074 reflections

θ = 2.4–38.4°

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

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2016)

T_{min} = 0.496, *T_{max}* = 0.748

31474 measured reflections

4664 independent reflections

4168 reflections with *I* > 2σ(*I*)

R_{int} = 0.059

θ_{max} = 27.1°, θ_{min} = 2.6°

h = −21→21

k = −9→9

l = −23→23

Refinement

Refinement on *F*²

Least-squares matrix: full

R [*F*² > 2σ(*F*²)] = 0.031

wR (*F*²) = 0.066

S = 1.11

4664 reflections

253 parameters

0 restraints

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 $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.60 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.61 \text{ e } \text{\AA}^{-3}$

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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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	0.668875	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*

H8B	0.515078	0.205199	0.644608	0.029*
H8C	0.534144	0.276943	0.570466	0.029*
C16	0.81928 (16)	1.1913 (3)	0.30107 (15)	0.0170 (5)
H16A	0.764854	1.140422	0.265770	0.025*
H16B	0.835344	1.291111	0.273954	0.025*
H16C	0.814374	1.238204	0.349352	0.025*
C13	0.88640 (14)	1.0428 (3)	0.32301 (14)	0.0126 (5)
C15	0.89362 (16)	0.9645 (3)	0.24873 (15)	0.0186 (5)
H15A	0.935772	0.866340	0.263706	0.028*
H15B	0.911020	1.061099	0.221125	0.028*
H15C	0.838699	0.915498	0.213542	0.028*
C6	0.90270 (15)	0.7277 (3)	0.38943 (15)	0.0157 (5)
C14	0.97108 (15)	1.1159 (4)	0.38186 (16)	0.0195 (5)
H14A	0.964657	1.157067	0.430264	0.029*
H14B	0.989184	1.218801	0.357887	0.029*
H14C	1.013621	1.018734	0.395112	0.029*
H5	0.7461 (15)	0.464 (3)	0.6249 (15)	0.006 (6)*
H4	0.5430 (18)	0.717 (4)	0.6210 (17)	0.023 (8)*
H12	0.7552 (15)	0.985 (3)	0.3754 (14)	0.003 (6)*
H11	0.9505 (17)	0.698 (4)	0.3796 (16)	0.018 (7)*
H3	0.6180 (17)	0.910 (4)	0.5485 (17)	0.018 (7)*
H10	0.8680 (18)	0.521 (4)	0.4505 (18)	0.028 (8)*

Atomic displacement parameters ($\text{\AA}^2$)

	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	0.0199 (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)

C6	0.0173 (12)	0.0140 (11)	0.0200 (13)	0.0027 (9)	0.0120 (10)	0.0014 (10)
C14	0.0164 (12)	0.0215 (13)	0.0199 (13)	−0.0055 (10)	0.0063 (10)	0.0031 (10)

Geometric parameters (Å, °)

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		
H2C—O2—H2D	104.5	N3—C8—H2B	109.7
H1C—O1—H1D	109.5	C7—C8—H2A	109.7
C1—N1—C9	126.71 (19)	C7—C8—H2B	109.7
C1—N1—C2	108.21 (19)	H2A—C8—H2B	108.2
C2—N1—C9	125.02 (18)	C9—C11—H7A	109.5
C1—N2—C7	124.95 (19)	C9—C11—H7B	109.5
C1—N2—C3	108.54 (19)	C9—C11—H7C	109.5
C3—N2—C7	126.4 (2)	H7A—C11—H7B	109.5
C4—N3—C8	124.51 (19)	H7A—C11—H7C	109.5
C4—N3—C5	108.75 (19)	H7B—C11—H7C	109.5
C5—N3—C8	126.7 (2)	N3—C5—H10	118.4 (19)
C4—N4—C13	125.94 (19)	C6—C5—N3	106.7 (2)
C4—N4—C6	108.16 (19)	C6—C5—H10	134.9 (19)
C6—N4—C13	125.85 (19)	C9—C12—H8A	109.5
N1—C1—N2	109.02 (19)	C9—C12—H8B	109.5

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)	C6—N4—C13—C14	−64.3 (3)
C4—N4—C13—C14	112.8 (2)		

1,1'-Methylenebis[3-(2,4,6-trimethylphenyl)imidazolium] dibromide dihydrate (at01019_0ma)*Crystal data*C₂₅H₃₀N₄²⁺·2Br[−]·2H₂O*M_r* = 582.38Orthorhombic, *Pccn**a* = 21.5695 (6) Å*b* = 28.3385 (6) Å*c* = 8.9401 (2) Å*V* = 5464.6 (2) Å³*Z* = 8*F*(000) = 2384*D_x* = 1.416 Mg m^{−3}Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 9959 reflections

θ = 2.6–39.4°

μ = 2.99 mm^{−1}*T* = 112 K

Prism, clear colourless

0.4 × 0.3 × 0.25 mm

Data collection

Bruker Venture Kappa D

diffractometer

φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2016)

*T*_{min} = 0.386, *T*_{max} = 0.748

39085 measured reflections

5954 independent reflections

5530 reflections with *I* > 2σ(*I*)*R*_{int} = 0.035θ_{max} = 27.1°, θ_{min} = 2.6°*h* = −24→27*k* = −31→36*l* = −11→11*Refinement*Refinement on *F*²

Least-squares matrix: full

R [*F*² > 2σ(*F*²)] = 0.032*wR*(*F*²) = 0.066*S* = 1.19

5954 reflections

338 parameters

0 restraints

Primary atom site location: iterative

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

w = 1/[σ²(*F*_o²) + (0.0149*P*)² + 6.9269*P*]where *P* = (*F*_o² + 2*F*_c²)/3(Δ/σ)_{max} = 0.002Δρ_{max} = 0.42 e Å^{−3}Δρ_{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 (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}	Occ. (<1)
Br1	0.32147 (2)	0.59206 (2)	0.51936 (2)	0.01792 (6)	
Br2	0.33241 (2)	0.42996 (2)	0.77256 (2)	0.01874 (6)	
N2	0.14868 (8)	0.40218 (6)	0.76624 (19)	0.0134 (3)	
N3	0.17753 (8)	0.54615 (6)	0.5774 (2)	0.0167 (4)	
N1	0.17956 (8)	0.47256 (6)	0.7130 (2)	0.0158 (4)	
N4	0.14048 (8)	0.61532 (6)	0.53485 (19)	0.0151 (3)	
C4	0.17963 (9)	0.42757 (7)	0.6667 (2)	0.0165 (4)	
H4	0.198554	0.415943	0.578146	0.020*	

C16	0.17313 (9)	0.59006 (7)	0.6302 (2)	0.0154 (4)	
H16	0.190588	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*	
H12C	0.205240	0.362270	1.019314	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*	
H24C	0.074401	0.622543	0.829693	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*	

H13C	0.085012	0.192160	0.798149	0.036*	
C21	0.12978 (10)	0.74543 (7)	0.5009 (2)	0.0197 (4)	
H21	0.148577	0.770574	0.447203	0.024*	
C25	0.06032 (11)	0.80563 (7)	0.6251 (3)	0.0256 (5)	
H25A	0.020408	0.809802	0.574412	0.038*	
H25B	0.090640	0.827784	0.583474	0.038*	
H25C	0.055261	0.811696	0.732324	0.038*	
H2	0.1460 (13)	0.5020 (10)	0.892 (3)	0.031*	
H15	0.1018 (13)	0.5989 (9)	0.332 (3)	0.031*	
H3	0.1080 (13)	0.4191 (10)	0.963 (3)	0.031*	
H14	0.1479 (13)	0.5182 (10)	0.388 (3)	0.031*	
O2	0.4729 (8)	0.5614 (8)	0.556 (4)	0.043 (5)	0.53 (9)
H2A	0.438 (4)	0.569 (4)	0.542 (11)	0.064*	0.53 (9)
H2B	0.465 (4)	0.542 (3)	0.606 (11)	0.064*	0.53 (9)

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
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)

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, °)

N2—C4	1.325 (3)	C3—C2	1.343 (3)
N2—C5	1.448 (2)	C3—H3	0.93 (3)
N2—C3	1.387 (3)	C10—C11	1.502 (3)
N3—C16	1.334 (3)	C10—C9	1.394 (3)
N3—C14	1.384 (3)	C15—H15	0.94 (3)
N3—C1	1.455 (3)	C1—H1A	0.9900
N1—C4	1.340 (3)	C1—H1B	0.9900
N1—C1	1.458 (3)	C19—H19	0.9500
N1—C2	1.380 (3)	C19—C20	1.394 (3)
N4—C16	1.317 (3)	C11—H11A	0.9800
N4—C17	1.450 (2)	C11—H11B	0.9800
N4—C15	1.386 (3)	C11—H11C	0.9800
C4—H4	0.9500	O1A—H1AA	0.8712
C16—H16	0.9500	O1A—H1AB	0.8701
C17—C18	1.394 (3)	C7—H7	0.9500
C17—C22	1.394 (3)	C7—C8	1.394 (3)
C5—C6	1.394 (3)	C2—H2	0.86 (3)
C5—C10	1.396 (3)	C20—C21	1.387 (3)
O2A—H2AA	0.8694	C20—C25	1.512 (3)
O2A—H2AB	0.8679	C23—H23A	0.9800
C12—H12A	0.9800	C23—H23B	0.9800
C12—H12B	0.9800	C23—H23C	0.9800
C12—H12C	0.9800	C8—C9	1.390 (3)
C12—C6	1.507 (3)	C8—C13	1.508 (3)
C14—C15	1.343 (3)	C9—H9	0.9500
C14—H14	0.87 (3)	C13—H13A	0.9800
C18—C24	1.508 (3)	C13—H13B	0.9800
C18—C19	1.386 (3)	C13—H13C	0.9800
C22—C23	1.511 (3)	C21—H21	0.9500
C22—C21	1.396 (3)	C25—H25A	0.9800
C6—C7	1.392 (3)	C25—H25B	0.9800
C24—H24A	0.9800	C25—H25C	0.9800
C24—H24B	0.9800	O2—H2A	0.79 (9)
C24—H24C	0.9800	O2—H2B	0.74 (11)
C4—N2—C5	124.39 (17)	C14—C15—N4	107.1 (2)
C4—N2—C3	108.93 (17)	C14—C15—H15	130.8 (17)
C3—N2—C5	126.68 (17)	N3—C1—N1	111.83 (17)
C16—N3—C14	108.72 (18)	N3—C1—H1A	109.3
C16—N3—C1	124.13 (19)	N3—C1—H1B	109.3
C14—N3—C1	126.41 (19)	N1—C1—H1A	109.3

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—C20	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
N2—C4—H4	126.0	C10—C11—H11C	109.5
N1—C4—H4	126.0	H11A—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—C8	122.13 (19)
C22—C17—C18	123.56 (18)	C8—C7—H7	118.9
C6—C5—N2	118.71 (18)	N1—C2—H2	119.2 (19)
C6—C5—C10	123.44 (18)	C3—C2—N1	106.9 (2)
C10—C5—N2	117.85 (18)	C3—C2—H2	133.8 (19)
H2AA—O2A—H2AB	104.6	C19—C20—C25	120.1 (2)
H12A—C12—H12B	109.5	C21—C20—C19	118.61 (19)
H12A—C12—H12C	109.5	C21—C20—C25	121.3 (2)
H12B—C12—H12C	109.5	C22—C23—H23A	109.5
C6—C12—H12A	109.5	C22—C23—H23B	109.5
C6—C12—H12B	109.5	C22—C23—H23C	109.5
C6—C12—H12C	109.5	H23A—C23—H23B	109.5
N3—C14—H14	121.0 (18)	H23A—C23—H23C	109.5
C15—C14—N3	106.8 (2)	H23B—C23—H23C	109.5
C15—C14—H14	132.2 (19)	C7—C8—C13	120.2 (2)
C17—C18—C24	121.48 (18)	C9—C8—C7	118.53 (19)
C19—C18—C17	117.11 (19)	C9—C8—C13	121.2 (2)
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—C13—H13A	109.5
C5—C6—C12	122.16 (18)	C8—C13—H13B	109.5
C7—C6—C5	116.87 (19)	C8—C13—H13C	109.5
C7—C6—C12	120.96 (19)	H13A—C13—H13B	109.5
C18—C24—H24A	109.5	H13A—C13—H13C	109.5
C18—C24—H24B	109.5	H13B—C13—H13C	109.5
C18—C24—H24C	109.5	C22—C21—H21	118.9
H24A—C24—H24B	109.5	C20—C21—C22	122.1 (2)
H24A—C24—H24C	109.5	C20—C21—H21	118.9
H24B—C24—H24C	109.5	C20—C25—H25A	109.5
N2—C3—H3	120.2 (17)	C20—C25—H25B	109.5
C2—C3—N2	107.2 (2)	C20—C25—H25C	109.5
C2—C3—H3	132.4 (17)	H25A—C25—H25B	109.5
C5—C10—C11	121.33 (18)	H25A—C25—H25C	109.5
C9—C10—C5	116.98 (19)	H25B—C25—H25C	109.5

C9—C10—C11	121.68 (19)	H2A—O2—H2B	94 (10)
N4—C15—H15	122.0 (17)		
N2—C5—C6—C12	0.8 (3)	C18—C17—C22—C23	179.6 (2)
N2—C5—C6—C7	−178.95 (18)	C18—C17—C22—C21	−0.1 (3)
N2—C5—C10—C11	−2.9 (3)	C18—C19—C20—C21	−0.7 (3)
N2—C5—C10—C9	177.94 (18)	C18—C19—C20—C25	178.7 (2)
N2—C3—C2—N1	0.5 (3)	C22—C17—C18—C24	−179.1 (2)
N3—C14—C15—N4	0.3 (3)	C22—C17—C18—C19	0.4 (3)
N4—C17—C18—C24	1.4 (3)	C6—C5—C10—C11	176.7 (2)
N4—C17—C18—C19	−178.99 (18)	C6—C5—C10—C9	−2.5 (3)
N4—C17—C22—C23	−1.0 (3)	C6—C7—C8—C9	−1.5 (3)
N4—C17—C22—C21	179.30 (18)	C6—C7—C8—C13	−179.6 (2)
C4—N2—C5—C6	101.6 (2)	C24—C18—C19—C20	179.5 (2)
C4—N2—C5—C10	−78.8 (3)	C3—N2—C4—N1	−0.4 (2)
C4—N2—C3—C2	−0.1 (3)	C3—N2—C5—C6	−77.5 (3)
C4—N1—C1—N3	116.6 (2)	C3—N2—C5—C10	102.1 (3)
C4—N1—C2—C3	−0.8 (3)	C10—C5—C6—C12	−178.8 (2)
C16—N3—C14—C15	−1.2 (3)	C10—C5—C6—C7	1.5 (3)
C16—N3—C1—N1	110.9 (2)	C15—N4—C16—N3	−1.4 (2)
C16—N4—C17—C18	−72.6 (3)	C15—N4—C17—C18	103.3 (3)
C16—N4—C17—C22	108.0 (2)	C15—N4—C17—C22	−76.2 (3)
C16—N4—C15—C14	0.7 (3)	C1—N3—C16—N4	172.30 (18)
C17—N4—C16—N3	175.03 (18)	C1—N3—C14—C15	−171.6 (2)
C17—N4—C15—C14	−175.7 (2)	C1—N1—C4—N2	171.14 (19)
C17—C18—C19—C20	0.0 (3)	C1—N1—C2—C3	−170.8 (2)
C17—C22—C21—C20	−0.6 (3)	C19—C20—C21—C22	1.0 (3)
C5—N2—C4—N1	−179.63 (18)	C11—C10—C9—C8	−177.7 (2)
C5—N2—C3—C2	179.1 (2)	C7—C8—C9—C10	0.4 (3)
C5—C6—C7—C8	0.6 (3)	C2—N1—C4—N2	0.7 (2)
C5—C10—C9—C8	1.5 (3)	C2—N1—C1—N3	−74.7 (3)
C12—C6—C7—C8	−179.16 (19)	C23—C22—C21—C20	179.7 (2)
C14—N3—C16—N4	1.6 (2)	C13—C8—C9—C10	178.5 (2)
C14—N3—C1—N1	−80.1 (3)	C25—C20—C21—C22	−178.4 (2)

1,1'-(Ethane-1,2-diyl)bis[3-(2,4,6-trimethylphenyl)imidazolium] dibromide tetrahydrate (est01041d_0ma)

Crystal data

$\text{C}_{26}\text{H}_{32}\text{N}_4^{2+} \cdot 2\text{Br}^- \cdot 4\text{H}_2\text{O}$

$M_r = 632.42$

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) Å³

$Z = 2$

$F(000) = 652$

$D_x = 1.461$ Mg m^{−3}

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

Cell parameters from 9565 reflections

$\theta = 2.3$ – 44.6°

$\mu = 2.86$ mm^{−1}

$T = 100$ K

Prism, clear colourless

$0.15 \times 0.15 \times 0.05$ mm

Data collection

Bruker Venture D8 Kappa
diffractometer

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2016)

$T_{\min} = 0.544$, $T_{\max} = 0.750$

28199 measured reflections

3165 independent reflections

3028 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 27.1^\circ$, $\theta_{\min} = 2.8^\circ$

$h = -15 \rightarrow 15$

$k = -16 \rightarrow 16$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.018$

$wR(F^2) = 0.044$

$S = 1.10$

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

$w = 1/[\sigma^2(F_o^2) + (0.0151P)^2 + 0.8539P]$

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

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.33 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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*

H7B	0.066579	0.418089	0.469255	0.016*
C4	0.47678 (10)	0.56704 (10)	0.80436 (13)	0.0128 (2)
C5	0.57920 (10)	0.61949 (10)	0.84337 (14)	0.0147 (2)
H9	0.637928	0.595239	0.807044	0.018*
C13	0.46334 (11)	0.47141 (10)	0.71025 (16)	0.0183 (3)
H10A	0.418593	0.486447	0.604922	0.027*
H10B	0.424390	0.419506	0.751255	0.027*
H10C	0.538291	0.446186	0.713460	0.027*
C11	0.31461 (12)	0.73712 (11)	1.00412 (16)	0.0225 (3)
H11A	0.240263	0.726481	0.927945	0.034*
H11B	0.327739	0.810168	1.022662	0.034*
H11C	0.316568	0.703141	1.098930	0.034*
C3	0.12649 (11)	0.47941 (11)	0.82365 (15)	0.0166 (3)
H12	0.0660 (15)	0.4502 (14)	0.8469 (19)	0.023 (4)*
C2	0.22470 (11)	0.52150 (11)	0.91051 (15)	0.0162 (3)
H13	0.2517 (15)	0.5293 (13)	1.016 (2)	0.022 (4)*

Atomic displacement parameters ($\text{\AA}^2$)

	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 ($\text{\AA}$, $^\circ$)

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—C7 ⁱ	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)

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)	C7 ⁱ —C7—H7A	109.8
C2—N1—C9	126.34 (10)	C7 ⁱ —C7—H7B	109.8
C1—N2—C7	124.67 (11)	H7A—C7—H7B	108.3
C1—N2—C3	108.89 (11)	C9—C4—C13	123.34 (11)
C3—N2—C7	126.41 (11)	C5—C4—C9	117.41 (11)
H2A—O2—H2B	106 (2)	C5—C4—C13	119.25 (11)
H1A—C12—H1B	109.5	C6—C5—H9	118.9
H1A—C12—H1C	109.5	C4—C5—C6	122.21 (12)
H1B—C12—H1C	109.5	C4—C5—H9	118.9
C6—C12—H1A	109.5	C4—C13—H10A	109.5
C6—C12—H1B	109.5	C4—C13—H10B	109.5
C6—C12—H1C	109.5	C4—C13—H10C	109.5
C10—C6—C12	121.57 (12)	H10A—C13—H10B	109.5
C10—C6—C5	118.20 (12)	H10A—C13—H10C	109.5
C5—C6—C12	120.23 (12)	H10B—C13—H10C	109.5
C6—C10—H3	118.9	C8—C11—H11A	109.5
C6—C10—C8	122.24 (12)	C8—C11—H11B	109.5
C8—C10—H3	118.9	C8—C11—H11C	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—C7 ⁱ	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—C7 ⁱ	−73.94 (17)
C12—C6—C10—C8	−179.51 (12)	C1—N2—C3—C2	−0.90 (15)

C12—C6—C5—C4	178.27 (12)	C7—N2—C1—N1	179.04 (11)
C6—C10—C8—C9	0.85 (19)	C7—N2—C3—C2	−178.88 (12)
C6—C10—C8—C11	−179.00 (12)	C5—C6—C10—C8	−0.48 (19)
C10—C6—C5—C4	−0.78 (19)	C13—C4—C5—C6	−177.92 (12)
C10—C8—C9—N1	−178.79 (11)	C11—C8—C9—N1	1.04 (18)
C10—C8—C9—C4	0.00 (18)	C11—C8—C9—C4	179.84 (12)
C8—C9—C4—C5	−1.16 (18)	C3—N2—C1—N1	1.01 (14)
C8—C9—C4—C13	178.30 (12)	C3—N2—C7—C7 ⁱ	103.73 (16)
C9—N1—C1—N2	179.64 (11)	C2—N1—C9—C8	−54.53 (17)
C9—N1—C2—C3	179.79 (12)	C2—N1—C9—C4	126.64 (13)
C9—C4—C5—C6	1.56 (18)	C2—N1—C1—N2	−0.73 (14)
C1—N1—C9—C8	125.04 (13)		

Symmetry code: (i) $-x, -y+1, -z+1$.