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The crystal structure of 1-dodecylpyridin-1-ium bromide monohydrate, $C_{17}H_{32}BrNO$

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Abstract

$C_{17}H_{32}BrNO$, triclinic, $P\bar{1}$ (no. 2), $a = 5.344(3)$ Å, $b = 7.871(5)$ Å, $c = 23.614(15)$ Å, $\alpha = 95.990(12)^\circ$, $\beta = 95.747(12)^\circ$, $\gamma = 98.386(12)^\circ$, $V = 970.5(11)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0550$, $wR_{ref}(F^2) = 0.1527$, $T = 293(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was synthesized by heating 1-bromododecane in pyridine at reflux for 3 h. Excess pyridine was removed under reduced pressure and the resulting solid dissolved in a minimum of $CHCl_3$. Pouring this liquid slowly into stirring ethyl acetate resulted in the formation of a white solid, which was subsequently filtered and recrystallized from methanol. The solid was

Table 1: Data collection and handling.

Crystal:	Colourless needle
Size:	$0.25 \times 0.10 \times 0.10$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	2.12 mm^{-1}
Diffractometer, scan mode:	Bruker,
$\theta_{\max}$, completeness:	25.0° , 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4817, 3382, 0.031
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1876
$N(\text{param})_{\text{refined}}$:	182
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

dissolved in water and left to slowly evaporate, affording colourless needles of the title compound.

Experimental details

The structure was solved by Direct Methods with SHELX [2]. All H-atoms were positioned geometrically and refined using a riding model with $d(C-H) = 0.93$ Å, $U_{\text{iso}} = 1.2U_{\text{eq}}$ (C) for aromatic, 0.97 Å, $U_{\text{iso}} = 1.2U_{\text{eq}}$ (C) for CH_2 , 0.96 Å, $U_{\text{iso}} = 1.5U_{\text{eq}}$ (C) for CH_3 hydrogen atoms and $d(O-H) = 0.85$ Å, $U_{\text{iso}} = 1.2U_{\text{eq}}$ (O) for H_2O . Restraints were applied to the displacement parameters of C2, C3, C6, C7, C8, C9, C16, C17 to approximate typical behaviour.

Comment

In recent years, all kinds of ionic liquids, especially imidazolium and pyridinium ionic liquids have attracted extensive attention and research interest, because these

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Br1	0.46294 (11)	0.82802 (8)	0.12404 (3)	0.0721 (3)
C1	0.3469 (11)	0.3466 (7)	0.0733 (2)	0.0618 (15)
H1	0.5061	0.4157	0.0781	0.074*
C2	0.1999 (12)	0.3378 (8)	0.0239 (2)	0.0715 (17)
H2	0.2503	0.4032	−0.0048	0.086*
C3	−0.0312 (12)	0.2276 (8)	0.0166 (3)	0.0756 (17)
H3	−0.1357	0.2129	−0.0180	0.091*
C4	−0.1043 (11)	0.1404 (8)	0.0613 (3)	0.0706 (16)
H4	−0.2623	0.0701	0.0574	0.085*
C5	0.0521 (11)	0.1560 (7)	0.1107 (2)	0.0579 (14)
H5	0.0047	0.0946	0.1405	0.070*
C6	0.4453 (11)	0.2851 (8)	0.1721 (2)	0.0719 (17)
H6A	0.4472	0.1734	0.1859	0.086*
H6B	0.6178	0.3296	0.1656	0.086*
C7	0.3634 (11)	0.4049 (7)	0.21697 (19)	0.0633 (15)
H7A	0.1918	0.3605	0.2241	0.076*
H7B	0.3619	0.5172	0.2036	0.076*
C8	0.5425 (11)	0.4241 (8)	0.2723 (2)	0.0740 (17)
H8A	0.5423	0.3110	0.2851	0.089*
H8B	0.7139	0.4655	0.2644	0.089*
C9	0.4765 (12)	0.5454 (7)	0.3204 (2)	0.0686 (16)
H9A	0.3040	0.5064	0.3281	0.082*
H9B	0.4827	0.6601	0.3085	0.082*
C10	0.6566 (12)	0.5549 (9)	0.3746 (2)	0.0807 (19)
H10A	0.8295	0.5897	0.3663	0.097*
H10B	0.6460	0.4406	0.3870	0.097*
C11	0.5988 (12)	0.6840 (8)	0.4244 (2)	0.0759 (18)
H11A	0.6103	0.7986	0.4122	0.091*
H11B	0.4258	0.6496	0.4327	0.091*
C12	0.7776 (12)	0.6916 (8)	0.4777 (2)	0.0774 (18)
H12A	0.9503	0.7262	0.4692	0.093*
H12B	0.7667	0.5766	0.4896	0.093*
C13	0.7221 (11)	0.8207 (8)	0.5288 (2)	0.0752 (18)
H13A	0.7338	0.9359	0.5171	0.090*
H13B	0.5496	0.7863	0.5374	0.090*
C14	0.9011 (12)	0.8258 (8)	0.5812 (2)	0.0774 (18)
H14A	1.0735	0.8593	0.5724	0.093*
H14B	0.8888	0.7105	0.5928	0.093*
C15	0.8496 (12)	0.9539 (9)	0.6323 (2)	0.0819 (19)
H15A	0.8593	1.0685	0.6203	0.098*
H15B	0.6774	0.9193	0.6411	0.098*
C16	1.0293 (14)	0.9634 (9)	0.6856 (2)	0.091 (2)
H16A	1.2019	0.9964	0.6769	0.109*
H16B	1.0175	0.8494	0.6983	0.109*
C17	0.9764 (16)	1.0943 (12)	0.7354 (3)	0.129 (3)
H17A	0.9759	1.2058	0.7225	0.193*
H17B	1.1069	1.1026	0.7671	0.193*
H17C	0.8136	1.0551	0.7475	0.193*
N1	0.2765 (8)	0.2612 (5)	0.11630 (17)	0.0530 (11)
O1W	0.9057 (7)	0.5792 (5)	0.12690 (19)	0.0942 (14)
H1WA	0.7905	0.6431	0.1261	0.113*
H1WB	1.0483	0.6423	0.1261	0.113*

long-chain amphiphilic salts not only are surface-active, but also can form a new class of liquid crystals materials in suitable solvents, which combine the characteristics of ionic liquids and thermotropic liquid crystals [5]. They can be used as ionic conducting material, organic reaction solvent, functional nanomaterial template and ordered membrane [6–9]. The characteristics of their crystal structures are very important for the application of these materials and many crystal structures have been reported. However, there is no report of the crystal structure of the 1-dodecylpyridinium bromide monohydrate.

The asymmetric unit of the title compound (see the Figure) comprises the desired alkyl pyridinium cation with a bromide counter anion and a water molecule. The hydrophobic C12 alkyl chain has a *trans*-planar arrangement. The C–C bond lengths found in the hydrocarbon chain are between 1.479(7) Å–1.575(7) Å and the C–C–C bond angles are between 110.7(5)° and 114.6(5)°. These values are close to those found in 1-dodecylpyridinium chloride monohydrate [10] and 1-tetradecylpyridinium bromide monohydrate [11]. The dihedral angle formed between the alkyl chain and the pyridinium ring is 78.29(20)°, slightly less than the 79.16° seen in 1-dodecylpyridinium chloride monohydrate [10], and different to the 52.73(7)° seen in 1-tetradecylpyridinium bromide monohydrate [11]. The torsion angles in the hydrocarbon chain show that the largest deviation from the *trans*-conformation is 179.8(5)° for N–C6–C7–C8 slightly greater than the 179.0(4)° seen in 1-dodecylpyridinium chloride monohydrate [10] and 177.00(18)° seen in 1-tetradecylpyridinium bromide monohydrate [11]. The compound has a lipid bilayered structure and water molecules participate in the formation of the structure which is consistent with similar structures. The bromide ion accepts a weak hydrogen bond [O1W...Br1 = 3.284(4) Å and O1W–H1WA...Br1 = 179.6°] from the water molecule and symmetry links it to another water molecule [O1...Br1i = 3.328(4) Å and <(O1W–H1WB...Br1i) = 179.6 (i = 1 + X, +Y, +Z)], forming an infinite O–H...Br hydrogen bonded chain in the (100) direction. In addition, there are four non-classical hydrogen bonds between C–H donors and the water molecule or bromide anion. Two of these interactions involve the methylene group attached directly to the pyridinium nitrogen whilst two involve pyridinium C–H groups which is different from 1-tetradecylpyridinium bromide monohydrate.

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