Crystal structure of aqua-bis(*N,N,N*-trimethylammonioacetato)sodium bromide, $[\text{Na}(\text{H}_2\text{O})\{(\text{CH}_3)_3\text{NCH}_2\text{COO}\}_2]\text{Br}$

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Abstract

$\text{C}_{10}\text{H}_{24}\text{BrN}_2\text{NaO}_5$, monoclinic, $C121$ (no. 5), $a = 9.108(2)$ Å, $b = 6.531(1)$ Å, $c = 13.306(2)$ Å, $\beta = 105.11(2)^\circ$, $V = 764.2$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.032$, $wR_{\text{ref}}(F^2) = 0.092$, $T = 293$ K.

Source of material

Single crystals of the title compound have been grown from a saturated water solution of sodium bromide and betaine, by a controlled solvent evaporation method. A small single crystal of good quality, selected from a larger sample, was used for X-ray data collection.

Experimental details

All hydrogen atoms involved in C–H bonds were refined as riding using appropriate AFIX instructions with SHELXL-97 [1] defaults. The hydrogen atom of the water molecule was located on a Fourier difference synthesis and refined with an isotropic displacement parameter constrained to that of the parent atom using SHELXL-97 defaults [1]. The final structure was examined with PLATON [2] showing that there are no solvent-accessible voids.

Discussion

Interesting results on a new family of hydrates having the general formula $[(\text{CH}_3)_3\text{NCH}_2\text{COO}]_n\text{XY} \cdot m\text{H}_2\text{O}$, ($n = 1, 2$; $X = \text{K}, \text{Rb}$; $Y = \text{Br}, \text{I}$; $m = 2$) were recently reported in detail [3–7]. These compounds present weak polar properties related to the degrees of freedom of the water molecule, each compound revealing its own peculiarities [8–11]. The compounds mentioned above exhibit a phase transition of order-disorder type below room temperature, closely associated with the reorientation of the water molecules [9–11]. The title compound is a new member of this family with $n = 2$, $m = 1$, $X = \text{Na}$, $Y = \text{Br}$.

The asymmetric unit of the title structure contains one betaine molecule, one sodium and one bromide ion, as well as the oxygen atom of the water molecule. The latter three atoms occupy special positions. The betaine molecule is in its zwitterionic neutral form. The atoms O1, O2, C1, C2, N1 and C4 of the betaine molecule lie on the same plane within 0.044(7) Å. Moreover, the molecule deviates only slightly from a perfect C_s symmetry as indicated by the torsion angles $\text{C1}–\text{C2}–\text{N1}–\text{C5}$ ($-65.5(7)^\circ$) and $\text{C1}–\text{C2}–\text{N1}–\text{C3}$ ($58.1(6)^\circ$). All bond angles and distances are within the typical ranges for the betaine molecule. The sodium cation is coordinated by five oxygen atoms belonging to four betaine and one water molecule (figure, top). A number of sodium compounds in which the cation exhibits coordination five has been reported [12–14]. In this structure, the coordination polyhedron of the cation can be described as a distorted triangular bipyramid in which two betaine O2 atoms and one water O3 atom are on the equatorial plane, at nearly 120° ; the oxygen atoms lying at the polar vertices are betaine O1 atoms. There is one strong hydrogen bond between the water molecule and the O1 oxygen of the betaine molecule, the associated bond length and angle being 2.847(5) Å and $167(9)^\circ$, respectively.

The crystal structure can be described as a stacking of three types of alternating layers parallel to the ab plane (figure, bottom). One of these layers consists of interpenetrating centered rectangular networks of sodium ions and water molecules. The neighboring layer is formed by betaine molecules that coordinate to the Na^+ of the previous layer. The third one contains the bromide anions ensuring the charge neutrality of the whole structure.

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Table 1. Data collection and handling.

Crystal:	transparent colorless square prism, size 0.01 × 0.10 × 0.17 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	27.34 cm ⁻¹
Diffractometer, scan mode:	Enraf-Nonius CAD4, $\omega/2\theta$ (profile fit)
$2\theta_{\max}$:	54.92°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	1031, 952
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 898
$N(\text{param})_{\text{refined}}$:	95
Programs:	SHELX-97 [1], PLATON [2], ORTEP-II [15]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(2A)	4c	0.0007	0.6975	0.3114	0.037
H(2B)	4c	0.0630	0.8192	0.2306	0.037
H(3A)	4c	0.0845	0.2042	0.2362	0.067
H(3B)	4c	0.0390	0.3229	0.3260	0.067
H(3C)	4c	0.2089	0.3208	0.3205	0.067
H(4A)	4c	-0.1009	0.6220	0.1001	0.063
H(4B)	4c	-0.1537	0.4900	0.1826	0.063
H(4C)	4c	-0.0882	0.3827	0.0982	0.063
H(5A)	4c	0.1837	0.6612	0.1194	0.053
H(5B)	4c	0.1604	0.4275	0.0932	0.053
H(5C)	4c	0.2877	0.4985	0.1907	0.053
H(3)	4c	0.080(7)	0.53(2)	0.521(5)	0.067

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	2a	0	0.0000*	0	0.0510(4)	0.0269(3)	0.0431(4)	0	0.0105(2)	0
Na(1)	2b	0	0.9613(4)	½	0.035(1)	0.028(2)	0.029(1)	0	-0.0014(9)	0
O(1)	4c	0.2141(4)	0.9112(7)	0.4127(3)	0.039(2)	0.040(2)	0.051(2)	-0.006(2)	0.006(2)	-0.010(2)
O(2)	4c	0.3398(4)	0.6433(7)	0.3714(3)	0.030(2)	0.045(2)	0.040(2)	0.003(2)	0.005(1)	0.008(2)
C(1)	4c	0.2268(5)	0.7542(8)	0.3621(3)	0.032(2)	0.029(2)	0.028(2)	-0.004(2)	0.008(2)	0.005(2)
C(2)	4c	0.0837(7)	0.704(1)	0.2783(6)	0.030(2)	0.029(3)	0.032(3)	0.000(2)	0.005(2)	-0.005(2)
N(1)	4c	0.0765(3)	0.513(1)	0.2139(2)	0.029(1)	0.023(2)	0.031(1)	0.001(2)	0.009(1)	0.003(3)
C(3)	4c	0.105(1)	0.323(1)	0.2801(7)	0.065(5)	0.023(3)	0.049(4)	-0.002(3)	0.019(4)	0.007(3)
C(4)	4c	-0.0808(5)	0.501(2)	0.1422(4)	0.033(2)	0.044(2)	0.045(2)	-0.005(4)	0.003(2)	-0.008(4)
C(5)	4c	0.1869(5)	0.526(1)	0.1484(3)	0.042(2)	0.032(4)	0.037(2)	-0.001(2)	0.020(2)	-0.001(2)
O(3)	2b	0	0.6019(9)	½	0.050(3)	0.026(3)	0.058(3)	0	0.015(3)	0

* fixed due to the floating origin restraint

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