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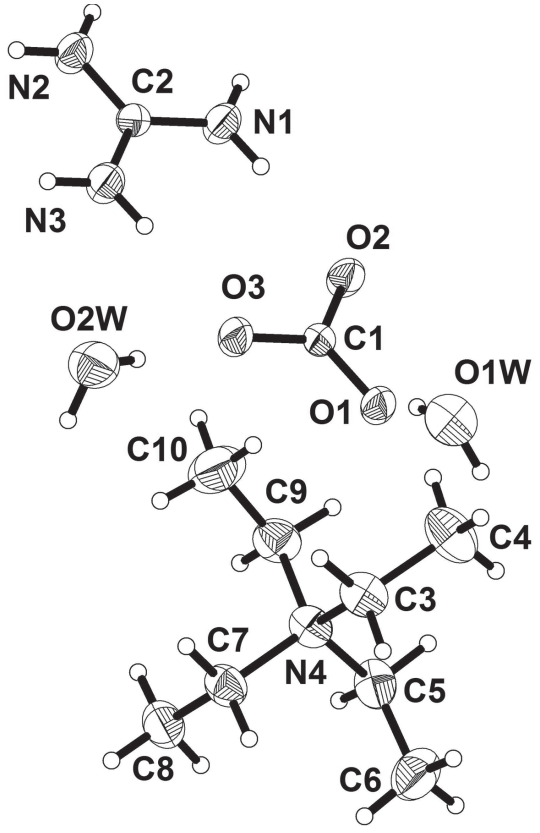
Crystal structure of guanidinium tetraethylammonium carbonate dihydrate, $C_{10}H_{30}N_4O_5$



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Abstract

$C_{10}H_{30}N_4O_5$, orthorhombic, $Pna2_1$, $a = 18.1934(5)$ Å, $b = 7.25480(10)$ Å, $c = 11.8168(2)$ Å, $V = 1559.69(5)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0358$, $wR_{ref}(F^2) = 0.0978$, $T = 296$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.27 × 0.18 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	CCD area detector, ϕ and ω
θ_{max}:	28.4°
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5319, 3177, 0.018
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2624
$N(param)_{refined}$:	186
Programs:	SHELX [5], Bruker programs [6]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	−0.02838(11)	−0.7083(2)	−0.44106(18)	0.0307(4)
O1	−0.03627(9)	−0.86223(19)	−0.49343(14)	0.0418(4)
O1W	−0.17061(9)	−0.9882(3)	−0.57830(19)	0.0619(5)
H1WA	−0.1347(13)	−0.922(4)	−0.553(3)	0.093*
N1	−0.01436(11)	−0.3684(2)	−0.22345(15)	0.0419(4)
H1A	−0.0217	−0.4699	−0.2593	0.050*
H1B	−0.0017	−0.3706	−0.1533	0.050*
C2	−0.02255(11)	−0.2092(3)	−0.27606(19)	0.0313(4)
O2	−0.01008(10)	−0.7079(2)	−0.33643(14)	0.0440(4)
O2W	−0.39442(11)	−0.8224(3)	−1.16101(19)	0.0644(5)
H2WA	−0.4142(19)	−0.896(4)	−1.114(2)	0.097*
H2WB	−0.4194(19)	−0.823(4)	−1.2226(19)	0.097*
N2	−0.01251(11)	−0.0529(2)	−0.22059(15)	0.0416(4)
H2A	−0.0186	0.0508	−0.2546	0.050*
H2B	0.0001	−0.0549	−0.1505	0.050*
C3	−0.27918(11)	−0.9603(3)	−0.9266(2)	0.0487(5)
H3A	−0.3101	−0.8518	−0.9323	0.058*
H3B	−0.2891	−1.0369	−0.9921	0.058*
O3	−0.03917(10)	−0.5567(2)	−0.49364(14)	0.0468(4)
N3	−0.04196(11)	−0.2071(2)	−0.38360(17)	0.0401(4)
H3C	−0.0492	−0.3091	−0.4190	0.048*
H3D	−0.0474	−0.1039	−0.4183	0.048*
C4	−0.30034(15)	−1.0653(5)	−0.8219(3)	0.0706(8)
H4A	−0.3514	−1.0983	−0.8259	0.106*
H4B	−0.2711	−1.1750	−0.8164	0.106*
H4C	−0.2921	−0.9896	−0.7564	0.106*
N4	−0.19935(8)	−0.8998(2)	−0.93074(16)	0.0377(4)
C5	−0.14850(12)	−1.0634(3)	−0.9226(2)	0.0501(6)
H5A	−0.0983	−1.0186	−0.9226	0.060*
H5B	−0.1569	−1.1237	−0.8505	0.060*
C6	−0.15599(17)	−1.2048(4)	−1.0147(3)	0.0708(9)

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
H6A	−0.1215	−1.3028	−1.0020	0.106*
H6B	−0.2050	−1.2537	−1.0143	0.106*
H6C	−0.1463	−1.1483	−1.0866	0.106*
C7	−0.18861(14)	−0.7970(3)	−1.0415(2)	0.0467(6)
H7A	−0.2017	−0.8788	−1.1033	0.056*
H7B	−0.2224	−0.6936	−1.0434	0.056*
C8	−0.11182(15)	−0.7256(4)	−1.0617(3)	0.0568(7)
H8A	−0.1100	−0.6629	−1.1332	0.085*
H8B	−0.0987	−0.6415	−1.0023	0.085*
H8C	−0.0779	−0.8270	−1.0624	0.085*
C9	−0.18157(15)	−0.7747(4)	−0.8315(2)	0.0535(6)
H9A	−0.1846	−0.8465	−0.7625	0.064*
H9B	−0.1312	−0.7327	−0.8392	0.064*
C10	−0.2307(2)	−0.6081(4)	−0.8195(3)	0.0802(9)
H10A	−0.2155	−0.5373	−0.7550	0.120*
H10B	−0.2271	−0.5336	−0.8864	0.120*
H10C	−0.2806	−0.6475	−0.8095	0.120*
H1WB	−0.1495(15)	−1.083(3)	−0.607(3)	0.072(10)*

and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

4,4′-Oxybis(benzoic acid) (A. R.), guanidine hydrochloride (A. R.) and tetraethylammonium hydroxide (25% aqueous solution) were dissolved in small amount of water/ethanol (v/v, 1:2) to yield a clean solution. After vigorous stirring for an hour, the solution was set aside to generate colorless block crystals of the title compound after about 2 weeks. 4,4′-Oxybis(benzoic acid) was not present in the final structure. It can be concluded that carbonate should be from carbon dioxide during the stirring.

Comment

Guanidinium and carbonate ions both possess perfect triangular planar geometry with the ability to form the related hydrogen bonds, but the difference in the former is hydrogen bond donor and the latter is the acceptor. In the corresponding crystal structures, these ions are usually present as the ancillary molecules to help the formation of the structures involving tetraalkylammonium and some aromatic carboxylic acids [1–4].

In the asymmetric unit of the title compound, there are one guanidinium cation, one tetraethylammonium cation, one carbonate anion and two water molecules. With the help of N—H···O and O—H···O hydrogen bonds guanidinium cations and water molecules are connected with carbonate anions to yield the hydrogen-bonded layers along the (100) plane. The interlayer distance is about $a/2 = 9.1$ Å. The tetraethylammonium is located between the layers to form the stable crystal structure.

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References

1. Mak, T. C.; Xue, F.: Supramolecular rosette ribbon constructed from guanidinium and hydrogen carbonate ions in the crystal engineering of hydrogen-bonded networks. J. Am. Chem. Soc. **122** (2000) 9860–9861.
2. Han, J.; Yau, C. W.; Chan, C. W.; Mak, T. C.: Anionic host layers constructed with guanidinium-hydrogen carbonate dimer 2:1 rosette-ribbons and bridging carboxylate connectors. Crys. Growth Des. **12** (2012) 4457–4465.
3. Han, J.; Yau, C. W.; Lam, C. K.; Mak, T. C.: Designed supramolecular assembly of hydrogen-bonded anionic rosette layers. J. Am. Chem. Soc. **130** (2008) 10315–10326.
4. Lam, C. K.; Xue, F.; Zhang, J. P.; Chen, X. M.; Mak, T. C.: Hydrogen-bonded anionic rosette networks assembled with guanidinium and C_3-symmetric oxoanion building blocks. J. Am. Chem. Soc. **127** (2005) 11536–11537.
5. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
6. Bruker: SAINT-Plus, SHELXTL and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA, 2004.