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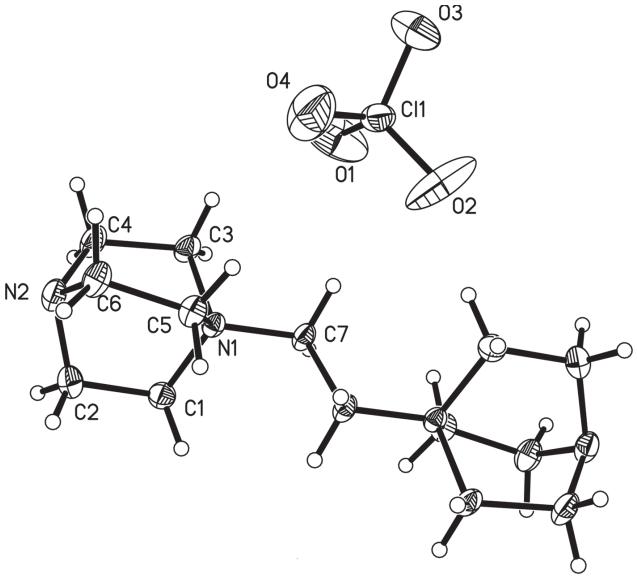
Crystal structure of the salt 1,1′-(ethane-1,2-diyl)bis(1,4-diazabicyclo[2.2.2]octan-1-ium) diperchlorate, $C_{14}H_{28}N_4(ClO_4)_2$



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Abstract

$C_{14}H_{28}N_4O_8Cl_2$, monoclinic, $P2_1/n$ $a = 8.7538(18)$ Å, $b = 11.393(2)$ Å, $c = 9.942(4)$ Å, $\beta = 97.50(3)°$, $V = 983.1(5)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0706$, $wR_{ref}(F^2) = 0.2036$, $T = 293(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

1,1′-(ethane-1,2-diyl)bis(1,4-diazabicyclo[2.2.2]octan-1-ium)dibromide was synthesised according to the method of Abbiss et al. [3]. 1,1′-(ethane-1,2-diyl)bis(1,4-diazabicyclo[2.2.2]octan-1-ium) dibromide (4.118 g, 10 mmol) and $NaClO_4 \cdot H_2O$ (2.809 g, 20 mmol) were mixed and stirred in aqueous solution (30 mL). Crystals were obtained by slow evaporation at room temperature with a yield of 86%. $C_{14}H_{28}C_{l2}N_4O_8$ (451.3): Cacld. (%): C, 37.26; H, 6.25; N, 12.41; O, 28.36. Found: C, 36.41; H, 6.94; N, 13.11; O, 28.59. **IR** (KBr disc, v, cm^{-1}): 459, 625, 914, 1052, 1454, 1466, 2245, 2870, 2951, 3310.

Table 1: Data collection and handling.

Crystal:	colorless block
Size:	0.20 × 0.20 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.38 mm^{-1}
Diffractometer, scan mode:	Rigaku RAPIDII, ω-scans
$2\theta_{max}$, completeness:	27.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6531, 2254, 0.083
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1757
$N(param)_{refined}$:	127
Programs:	Rigaku programs [1], SHELX [2]

Experimental details

Hydrogen atoms were placed in calculated positions and refined as a riding mode, with C–H = 0.96 Å (methylene) with $U_{iso}(H) = 1.2\,U_{eq}(C)$.

Discussion

Organic-inorganic crystalline materials can be exploited for many purposes such as nonlinear optical materials, dielectric, piezoelectric, pyroelectric, ferroelectric, semiconductors and other physical properties [4–7]. Salts, co-crystals, polymorphs, solvates and hydrates are of general interest. Furthermore, there is a research interest in organic salts because of their brilliant properties [8–11]. The basic structural unit of the compound comprises two perchlorate anions and one 1,1′-(ethane-1,2-diyl)bis(1,4-diazabicyclo[2.2.2]octan-1-ium) cation. The perchlorate anions adopt tetrahedral geometry with Cl—O bond length of 1.390(4)–1.442(3) Å and bond angles from 105.9(2)° to 113.3(3)°. The diazabicyclo[2.2.2]octane moiety (*cf.* the figure)

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
N1	0.6575(2)	0.90612(16)	0.6044(2)	0.0251(5)
N2	0.7986(3)	0.7339(2)	0.7379(2)	0.0383(6)
C1	0.5962(3)	0.8879(2)	0.7392(3)	0.0343(6)
H1A	0.4847	0.8827	0.7249	0.041*
H1B	0.6253	0.9535	0.7994	0.041*
C2	0.6655(4)	0.7735(3)	0.8023(3)	0.0429(7)
H2A	0.6975	0.7856	0.8984	0.052*
H2B	0.5872	0.7128	0.7928	0.052*
C3	0.8296(3)	0.9301(2)	0.6356(3)	0.0347(6)
H3A	0.8465	1.0068	0.6772	0.042*
H3B	0.8763	0.9293	0.5523	0.042*
C4	0.9037(3)	0.8343(3)	0.7331(3)	0.0420(7)
H4A	0.9989	0.8078	0.7030	0.050*
H4B	0.9283	0.8671	0.8234	0.050*
C5	0.6372(3)	0.7927(2)	0.5237(3)	0.0323(6)
H5A	0.6641	0.8050	0.4332	0.039*
H5B	0.5306	0.7674	0.5158	0.039*
C6	0.7426(4)	0.6983(2)	0.5977(3)	0.0424(7)
H6A	0.6862	0.6251	0.5989	0.051*
H6B	0.8300	0.6850	0.5487	0.051*
C7	0.5853(3)	1.0103(2)	0.5277(3)	0.0301(6)
H7A	0.6422	1.0276	0.4527	0.036*
H7B	0.5930	1.0780	0.5873	0.036*
Cl1	0.77899(8)	0.90725(7)	0.17026(8)	0.0442(3)
O1	0.8337(5)	0.9569(4)	0.2973(3)	0.1199(16)
O2	0.6277(4)	0.9380(4)	0.1235(6)	0.1373(18)
O3	0.8819(4)	0.9364(3)	0.0773(3)	0.0834(10)
O4	0.7820(4)	0.7810(3)	0.1805(5)	0.1056(13)

in the cation has a little torsion with the torsion angle of N—C—C—N ranging from 15.0(3)° to 15.9(3)°, which loses its typical D_{3h} symmetry. Two kinds of weak hydrogen bonds, C—H···N and C—H···O, form a three dimensional network. The cation chains are parallel to the (101) plane and extend along *b* axis. The perchlorate anions also have two orientations which fill in the inner cation layers connecting the chains via C—H···O (dH···A = 2.53–2.57 Å) becoming 3D hydrogen bond network.

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