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Crystal structure of N^1,N^3 -bis(2-hydroxyethyl)- N^1,N^1,N^3,N^3 -tetramethylpropane-1,3-diaminium dibromide, $C_{11}H_{28}Br_2N_2O_2$

<https://doi.org/10.1515/ncrs-2019-0258>

Received April 8, 2019; accepted June 5, 2019; available online June 19, 2019

Abstract

$C_{11}H_{28}Br_2N_2O_2$, triclinic, $P\bar{1}$ (no. 2), $a = 12.0519(11)$ Å, $b = 12.6670(12)$ Å, $c = 12.9161(12)$ Å, $\alpha = 117.088(3)^\circ$, $\beta = 92.459(1)^\circ$, $\gamma = 108.199(2)^\circ$, $V = 1627.2(3)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0645$, $wR_{ref}(F^2) = 0.1632$, $T = 298(2)$ K.

CCDC no.: 1585931

The molecular 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

2-(Dimethylamino)ethanol (1.78 g, 20 mmol) and 1,3-dibromopropane (2.02 g, 10 mmol) were mixed in 5 mL N,N -dimethylformamide. The mixture was stirred for 5 h at 333 K (monitored by TLC). After added into 10 mL diethyl ether, the mixture was kept at 253 K about 4 hours, and then filtrated. The residue was recrystallized from methanol/diethyl ether (1:2, v/v) to afford the white crystals. Yield: 85%.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.38 \times 0.30 \times 0.18$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	4.98 mm^{-1}
Diffractometer, scan mode:	APEX2 CCD, φ and ω
$\theta_{\max}$, completeness:	25.0° , 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8270, 5610, 0.070
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3016
$N(\text{param})_{\text{refined}}$:	315
Programs:	Bruker [1], SHELX [2, 3]

Experimental details

All H atoms were placed in idealized positions and treated as riding on their parent atoms, with $d(\text{C}-\text{H}) = 0.97$ Å (methylene) and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$; $d(\text{C}-\text{H}) = 0.96$ Å (methyl) and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$; $d(\text{O}-\text{H}) = 0.82$ Å (OH) and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

Comment

Due to the antibacterial activities, quaternary ammonium salts (QASs) were used for disinfectants, antiseptics and a variety of clinical purposes such as preoperative disinfection of unbroken skin, application to mucous membranes, and disinfection of noncritical surfaces [4, 5]. For example, alkyl-QAS, dialkyl-QAS, QAS-iodine and QAS-chitosan have received increasing attention due to their vital antibacterial activities [6–8]. Most of these disinfectants are mono-QASs [9–12]. However, recent studies have reported that the increased tolerance and decreased sensitivity of bacteria to limit the use of mono-QAS products. Therefore, the development of double-QASs with improving antibacterial activities has important research value. In this paper, 2-(dimethylamino)ethanol has been selected as activity donors to construct novel double-QAS because 2-(dimethylamino)ethanol contains a flexible hydroxyl group. Based on the literature reports, the double-QAS was used as a bridging organic ligand to construct coordination polymers [13, 14]. Herein, it is expected that the double-QAS with two flexible hydroxyl groups can pass through cell membrane into the cell to passivate enzyme and damage bacteria [15–17].

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	0.48959(8)	0.06821(8)	0.27297(7)	0.0426(3)
Br2	0.06635(8)	0.20681(9)	0.89507(8)	0.0472(3)
Br3	0.07569(8)	0.75058(9)	0.47256(9)	0.0520(3)
Br4	0.50123(8)	0.43624(9)	0.22863(8)	0.0573(3)
N1	0.2501(5)	0.6074(6)	−0.0131(5)	0.0332(15)
N2	0.2540(5)	0.9828(5)	−0.0677(5)	0.0254(14)
N3	0.2467(5)	0.5090(6)	0.4722(5)	0.0310(15)
N4	0.2520(5)	0.1357(6)	0.5234(5)	0.0315(15)
O1	0.2425(7)	0.6961(7)	0.2677(6)	0.086(2)
H1	0.212515	0.716668	0.326161	0.129*
O2	0.1087(5)	1.0903(5)	0.1933(5)	0.0496(15)
H2	0.065188	1.023750	0.189623	0.074*
O3	0.0454(5)	0.3455(6)	0.1748(5)	0.069(2)
H3	0.050742	0.310565	0.105022	0.083*
O4	0.4305(6)	0.1563(7)	0.7328(6)	0.072(2)
H4	0.448606	0.100862	0.737026	0.086*
C1	0.3175(7)	0.7283(7)	−0.0128(7)	0.0316(18)
H1A	0.371757	0.712182	−0.066274	0.038*
H1B	0.365653	0.791086	0.066749	0.038*
C2	0.2388(7)	0.7856(7)	−0.0501(7)	0.0338(19)
H2A	0.192300	0.725735	−0.130811	0.041*
H2B	0.183863	0.802772	0.002523	0.041*
C3	0.3202(6)	0.9094(6)	−0.0428(6)	0.0278(17)
H3A	0.371737	0.964290	0.036249	0.033*
H3B	0.370931	0.889442	−0.099465	0.033*
C4	0.1570(8)	0.6190(8)	0.0611(7)	0.047(2)
H4A	0.122831	0.539506	0.062383	0.056*
H4B	0.093247	0.627845	0.021109	0.056*
C5	0.1982(9)	0.7296(10)	0.1911(8)	0.060(3)
H5A	0.259842	0.803115	0.195228	0.072*
H5B	0.131092	0.753823	0.216510	0.072*
C6	0.1883(8)	0.5022(8)	−0.1383(7)	0.050(2)
H6A	0.123027	0.517789	−0.165879	0.076*
H6B	0.158202	0.421793	−0.139664	0.076*
H6C	0.244167	0.499840	−0.189348	0.076*
C7	0.3418(8)	0.5709(9)	0.0331(8)	0.057(3)
H7A	0.391659	0.548984	−0.023029	0.085*
H7B	0.301355	0.498943	0.043097	0.085*
H7C	0.390363	0.641837	0.108297	0.085*
C8	0.1601(6)	1.0055(7)	0.0042(6)	0.0301(18)
H8A	0.095880	0.924000	−0.021768	0.036*
H8B	0.126796	1.057915	−0.012654	0.036*
C9	0.2053(7)	1.0700(8)	0.1380(6)	0.040(2)
H9A	0.233750	1.016283	0.157672	0.048*
H9B	0.271004	1.151004	0.165916	0.048*
C10	0.3464(7)	1.1089(7)	−0.0415(7)	0.041(2)
H10A	0.406178	1.093894	−0.087094	0.062*
H10B	0.383440	1.158228	0.041911	0.062*
H10C	0.308528	1.154820	−0.062614	0.062*
C11	0.1930(7)	0.9118(7)	−0.1969(6)	0.040(2)
H11A	0.130091	0.833460	−0.214508	0.060*
H11B	0.250148	0.893110	−0.245049	0.060*
H11C	0.159864	0.963490	−0.213751	0.060*
C12	0.3085(7)	0.4193(7)	0.4687(6)	0.0298(18)
H12A	0.332704	0.385227	0.393206	0.036*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H12B	0.380470	0.468356	0.531512	0.036*
C13	0.2327(7)	0.3103(7)	0.4830(7)	0.0339(19)
H13A	0.167692	0.251121	0.413593	0.041*
H13B	0.198889	0.341390	0.552715	0.041*
C14	0.3133(7)	0.2439(7)	0.4972(6)	0.0307(18)
H14A	0.382217	0.306938	0.561611	0.037*
H14B	0.342235	0.209288	0.424703	0.037*
C15	0.1355(6)	0.4437(7)	0.3733(6)	0.0335(19)
H15A	0.079695	0.374032	0.379987	0.040*
H15B	0.097648	0.504480	0.385680	0.040*
C16	0.1568(7)	0.3915(8)	0.2492(7)	0.045(2)
H16A	0.214869	0.458073	0.240349	0.054*
H16B	0.186113	0.323136	0.230610	0.054*
C17	0.3388(7)	0.6167(7)	0.4647(7)	0.039(2)
H17A	0.301486	0.670681	0.457780	0.059*
H17B	0.371863	0.581865	0.396095	0.059*
H17C	0.401602	0.665963	0.535291	0.059*
C18	0.2116(8)	0.5683(8)	0.5889(7)	0.043(2)
H18A	0.278356	0.601479	0.652816	0.065*
H18B	0.145803	0.504930	0.593061	0.065*
H18C	0.188085	0.636568	0.595681	0.065*
C19	0.3410(7)	0.0738(8)	0.5258(7)	0.039(2)
H19A	0.299527	−0.002604	0.530387	0.047*
H19B	0.366595	0.046869	0.450845	0.047*
C20	0.4509(9)	0.1550(10)	0.6257(9)	0.064(3)
H20A	0.514197	0.122951	0.601798	0.076*
H20B	0.478007	0.241863	0.639431	0.076*
C21	0.2107(8)	0.1804(8)	0.6375(7)	0.047(2)
H21A	0.144001	0.204244	0.629255	0.071*
H21B	0.274789	0.252933	0.700125	0.071*
H21C	0.186576	0.112832	0.656636	0.071*
C22	0.1476(8)	0.0372(8)	0.4229(7)	0.051(2)
H22A	0.109704	−0.032272	0.437193	0.076*
H22B	0.174596	0.005863	0.349740	0.076*
H22C	0.091235	0.074705	0.417068	0.076*

There are two QAS molecules in the asymmetric unit of the title crystal structure (*cf.* the figure). Bond lengths and angles are all in the expected ranges. Single-crystal structure analysis reveals that configurations of two QAS molecules are different. In a molecule, two ethoxy groups adopt the *cis*-configuration compared with central 1,3-propane diamine group. The QAS molecule looks like an ‘U’ type. In another molecule, two ethoxy groups adopt the *trans*-configuration and it looks like an ‘S’ type. This flexibility of the double-QAS is easier to pass through cell membrane into the cell [18, 19].

The asymmetric unit (two dications and four bromide anions) of the title crystal structure is shown in the figure. Bond lengths and angles are in the expected ranges [14].

Acknowledgements: This work was supported by the Foundation of Yantai city (No. 2018XSCC048).

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