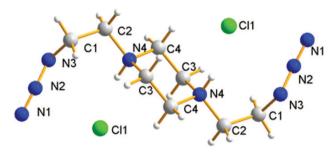
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Fu Lei Gao*, Ying Lei Wang, Bing Chen, Wei Xiao Liu and Ya Jing Liu

Crystal structure of 1,4-bis(2-azidoethyl) piperazine-1,4-diium dichloride, C₈H₁₈N₈Cl₂



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Abstract

 $C_8H_{18}N_8Cl_2$, monoclinic, $P2_1/n$ (no. 14), a = 6.5246(3) Å, b = 7.7090(4) Åc = 13.9243(6) Å $\beta = 101.240(3)^{\circ}$ $V = 686.93(6) \text{ Å}^3$, Z = 2, $R_{gt}(F) = 0.0320$, $wR_{ref}(F^2) = 0.0878$, T = 302(2) K.

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The crystal structure is shown in the figure. The non-hydrogen atoms of the asymmetric unit are labelled. 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-Azido-5-chloro-3-azo-pentane (50 mmol) was dissolved in distilled water (50 mL) and heated to the temperature of 40 °C. Perchloric acid (15 mmol) was added to the agueous solution dropwise slowly in 20 min, then the mixture was stirred for 5 h at 40 °C. After the removal of the solvent, the residue was poured into 10 mL cold water, the white solid was filter and washed with cold water. Crystals were obtained by slow evaporation from the aqueous solution at room temperature.

Ying Lei Wang, Bing Chen, Wei Xiao Liu and Ya Jing Liu: Xi'an Modern Chemistry Research Institute, Xi'an 710065, P.R. China

Table 1: Crystal collection and handling.

Crystal:	Block, colourless	
Size:	$0.130\times0.140\times0.150~\text{mm}$	
Wavelength:	Mo K_{α} radiation ($\lambda=0.71073$ Å)	
μ:	0.471 mm ⁻¹	
Diffractometer, scan mode:	Bruker APEX-II CCD, $oldsymbol{\phi}$ and	
	ω-scans	
$2 heta_{ exttt{max}}$, completeness:	28.4°, >99%	
$N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}, R_{\text{int}}$:	7168, 1725, 0.0288	
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{\rm obs} > 2\sigma(I_{\rm obs}), 1358$	
$N(param)_{refined}$:	87	
Programs:	Bruker programs [1], ShelX [2, 3]	

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

Atom	х	у	z	U _{iso} */U _{eq}
<u>C1</u>	0.9038(2)	0.20754(19)	0.67112(11)	0.0326(3)
H1A	0.8031	0.2265	0.7129	0.039
H1B	0.9586	0.0907	0.6823	0.039
C2	0.7997(2)	0.22910(18)	0.56512(11)	0.0310(3)
H2A	0.9052	0.2232	0.5249	0.037
H2B	0.7032	0.1338	0.5464	0.037
С3	0.4870(2)	0.40154(18)	0.58663(10)	0.0276(3)
НЗА	0.5227	0.3867	0.6571	0.033
H3B	0.3969	0.3062	0.5597	0.033
C4	0.3724(2)	0.57071(18)	0.56301(9)	0.0276(3)
H4A	0.2455	0.5692	0.5895	0.033
H4B	0.4592	0.6651	0.5940	0.033
Cl1	0.88140(5)	0.74563(4)	0.62576(2)	0.03553(14)
N1	1.1073(3)	0.4964(2)	0.84005(12)	0.0563(4)
N2	1.0842(2)	0.41570(16)	0.77156(10)	0.0371(3)
N3	1.0760(2)	0.33517(18)	0.69452(10)	0.0398(3)
N4	0.68245(17)	0.39803(14)	0.54507(8)	0.0241(3)
H4	0.761(2)	0.487(2)	0.5706(11)	0.031(4)

Experimental details

In the crystal structure, all hydrogen atoms were positioned geometrically and refined using a riding model.

Comment

Amine azides are low molecular weight organic amine compounds containing at least one tertiary nitrogen and one functional group that can be used as the fuel composition

^{*}Corresponding author: Fu Lei Gao, Xi'an Modern Chemistry Research Institute, Xi'an 710065, P.R. China, e-mail: gfl198510@163.com

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in hypergolic fuel propulsion systems or gelled propellants [4]. Such compounds are preferred to replace hydrazines due to the advantages of synthesized and evaluated as potentional hypergolic fuel compounds, such as dimethylaminoethylazide (DMAZ), pyrollidiylethylazide (PYAZ) and bis(ethylazide)methylamine (BAZ) [5–8]. DMAZ appears to be one of the better candidates for hydrazine compounds. But DMAZ systems can not meet higher performance standards set by monomethylhydrazine systems. In order to obtain a higher energy density fuel, an amine azide containing piperazine ring was synthesized [9]. As shown in the figure, the title structure contains one piperazine and two 2-azidoethanamine groups and has a higher energy density. Bonds lengths and angles of all moieties are in their normal ranges and excellently fit with those of related compounds [10].

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