Zhi Jian Hao*

Crystal structure of 1,4-bis(2-azidoethyl) piperazine-1,4-dium dichloride trihydrate, $C_8H_{18}N_8Cl_2 \cdot 3 H_2O$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.14 × 0.13 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.40 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	28.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	8680, 2128, 0.027
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1779
$N(param)_{refined}$:	96
Programs:	SHELX [1], Bruker [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U_{iso}^*/U_{eq}
C1	0.35227(9)	0.1350(2)	0.34714(12)	0.0365(3)
H1A	0.3392	0.0480	0.2928	0.044*
H1B	0.3695	0.0690	0.4098	0.044*
C2	0.41815(8)	0.2611(2)	0.33916(10)	0.0331(3)
H2A	0.4660	0.1903	0.3426	0.040*
H2B	0.4008	0.3206	0.2746	0.040*
C3	0.47365(8)	0.32390(18)	0.52172(10)	0.0292(3)
H3A	0.5201	0.2490	0.5243	0.035*
H3B	0.4332	0.2473	0.5360	0.035*
C4	0.50136(8)	0.5281(2)	0.40020(10)	0.0299(3)
H4A	0.4791	0.5830	0.3348	0.036*
H4B	0.5487	0.4586	0.3999	0.036*
Cl1	0.36825(2)	0.91868(6)	0.59134(3)	0.04183(14)
N1	0.16290(13)	0.1045(3)	0.3450(2)	0.0966(9)
N2	0.22194(9)	0.1649(2)	0.34533(14)	0.0577(4)
N3	0.28145(9)	0.2480(2)	0.34214(14)	0.0567(4)
N4	0.43998(6)	0.40341(15)	0.41950(8)	0.0250(2)
H4	0.3986	0.4699	0.4194	0.078*
O1	0.5000	0.8497(2)	0.2500	0.0686(6)
H1	0.5367	0.9130	0.2903	0.078*
O2	0.31915(7)	0.64256(18)	0.41511(10)	0.0521(3)
H2C	0.3262	0.7209	0.4613	0.078*
H2D	0.2706	0.6090	0.4058	0.078*

<https://doi.org/10.1515/ncrs-2019-0263>

Received April 10, 2019; accepted June 5, 2019; available online June 22, 2019

Abstract

$C_8H_{18}N_8O_3$, monoclinic, $C2/c$ (no. 15), $a = 17.3812(11)$ Å, $b = 7.3688(5)$ Å, $c = 14.0744(9)$ Å, $\beta = 107.915^\circ$, $V = 1715.2(2)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0365$, $wR_{ref}(F^2) = 0.1012$, $T = 296(2)$ K.

CCDC no.: 1908674

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

Single crystal of 1,4-bis(2-azidoethyl)piperazine-1,4-dium dichloride trihydrate was obtained by slow evaporation from the 1,4-bis(2-azidoethyl) piperazine-1,4-dium dichloride aqueous solution at temperature of 10 °C.

Experimental details

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

*Corresponding author: Zhi Jian Hao, Shenyang Institute of Technology, Fu shun 113122, P.R. China, e-mail: 136791302070@163.com

Comment

Amine azide compounds have higher density, positive enthalpy of formation than hydrazines, and their detonation products

are also pollution-free. So they are ideal alternatives for hydrazine based fuels [3]. In recent years, more researchers have paid attention to the synthesis and performance of azide amine compounds, and synthesized a variety of amine azide compounds [4–6]. In 2018, Gao *et al.* [7] already synthesized an amine azide compound containing a piperazine ring with higher density and obtained the single crystal of 1,4-bis(2-azidoethyl)piperazine-1,4-diium dichloride.

We obtained the title compound by almost the same method but obtained 1,4-bis(2-azidoethyl)piperazine-1,4-diium dichloride trihydrate at a low temperature. Due to the presence of crystal water, a large number of hydrogen bonds exist, giving the compound a much better stability. Geometric parameters are similar to those already reported [7].

Acknowledgements: We acknowledge the funding support received from the Liaoning Province Science and Technology Plan Project.

References

1. Sheldrick, G. M.: Crystal structure refinement with SHELX. *Acta Crystallogr.* **C71** (2015) 3–8.
2. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2014).
3. Zhang, Z. B.: Properties and research situation of tri-azido amine compounds. *Chem. Propellants Polym. Mater.* **9** (2011) 45–48.
4. Ghanbari, S.; Vaferi, B.: Experimental and theoretical investigation of water removal from DMAZ liquid fuel by an adsorption process. *Acta Astronaut.* **112** (2015) 19–28.
5. Reddy, G.; Song, J.; Mecchi, M. S.; Johnson, M. S.: Genotoxicity assessment of two hypergolic energetic propellant compounds. *Mutat. Res. Genet. Toxicol. Environ. Mutagen.* **700** (2010) 26–31.
6. Greene, B.; McClure, M. B.; Johnson, H. T.: Destruction or decomposition of hypergolic chemicals in a liquid propellant testing laboratory. *Chem. Health Saf.* **11** (2004) 6–13.
7. Gao, F. L.; Wang, Y. L.; Chen, B.: Crystal structure of 1,4-bis(2-azidoethyl) piperazine-1,4-diium dichloride, $C_8H_{18}N_8Cl_2$. *Z. Kristallogr. NCS* **233** (2018) 949–950.