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The crystal structure of 2-amino-5-nitroanilinium iodide monohydrate, $C_6H_8IN_3O_2$

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.20 × 0.15 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	3.37 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	13473, 1936, 0.024
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1798
$N(\text{param})_{\text{refined}}$:	146
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Source of material

All of the reagents are commercially available and were used without further purification. 1.53 g 4-nitrobenzene-1,2-diamine (10 mmol) was added to a solution mixed by 9 mL THF and 1 mL hydroiodic acid (40%). After stirring for 10 min at room temperature, the solution was filtered and let evaporate automatically. Many yellow block crystals were obtained, yield 74.6% (based on 4-nitrobenzene-1,2-diamine).

Experimental details

The structure was solved by direct methods with the SHELXS-2018 program. All H-atoms from C atoms were positioned with idealized geometry and refined isotropically ($U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$) using a riding model with C–H = 0.95 Å. The H-atom from N and O were positioned using Q peaks and refined freely.

Comment

The crystal structures of 4-nitrobenzene-1,2-diamine has been reported [5, 6]. The mono-hydrochloric acid salt, and mono-hydrobromic acid salt were reported too [5]. And even the co-crystal structure of 4-nitrobenzene-1,2-diamine with 1,4,7,10,13,16-hexaoxacyclo-octadecane (18-crown-6) has yet been studied [7]. Moreover, the crystal structure of an analogue of the 4-nitrobenzene-1,2-diamine, 1,2-Diamino-4,5-dinitrobenzene, has been reported [8]. To the best of our

<https://doi.org/10.1515/ncrs-2021-0058>

Received February 8, 2021; accepted February 25, 2021;
published online March 19, 2021

Abstract

$C_6H_8IN_3O_2$, monoclinic, $P2_1/n$ (no. 14), $a = 7.0704(3)$ Å, $b = 15.7781(6)$ Å, $c = 9.1495(4)$ Å, $\beta = 112.114(1)^\circ$, $V = 945.61(7)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0187$, $wR_{\text{ref}}(F^2) = 0.0522$, $T = 150(2)$ K.

CCDC no.: 2065269

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.6528 (4)	0.10901 (15)	0.4044 (3)	0.0253 (5)
C2	0.5861 (4)	0.03329 (16)	0.3178 (3)	0.0264 (5)
C3	0.6662 (4)	−0.04302 (15)	0.3974 (3)	0.0265 (5)
H	0.625453	−0.095210	0.342738	0.032*
C4	0.8021 (4)	−0.04414 (15)	0.5520 (3)	0.0265 (5)
HA	0.853014	−0.096304	0.603720	0.032*
C5	0.8632 (3)	0.03254 (15)	0.6311 (3)	0.0243 (5)
C6	0.7899 (4)	0.10905 (15)	0.5583 (3)	0.0249 (5)
HB	0.833366	0.160878	0.613673	0.030*
I1	0.09198 (2)	0.28908 (2)	0.45256 (2)	0.02856 (8)
N1	0.5731 (4)	0.19135 (15)	0.3327 (3)	0.0286 (4)
N2	0.4455 (4)	0.03286 (17)	0.1677 (3)	0.0316 (5)
N3	1.0092 (3)	0.03308 (13)	0.7924 (2)	0.0275 (4)
O1	1.0708 (3)	0.10246 (12)	0.8557 (2)	0.0348 (4)
O2	1.0701 (3)	−0.03492 (12)	0.8601 (2)	0.0350 (4)
O1W	0.1563 (3)	0.20292 (13)	0.1247 (2)	0.0310 (4)
HC	0.631 (8)	0.208 (2)	0.257 (6)	0.074 (14)*
HD	0.453 (6)	0.188 (2)	0.284 (4)	0.047 (10)*
HE	0.410 (6)	−0.012 (3)	0.122 (5)	0.051 (10)*
HF	0.601 (6)	0.234 (3)	0.410 (5)	0.054 (10)*
HG	0.441 (6)	0.072 (3)	0.109 (5)	0.053 (11)*
HH	0.083 (6)	0.225 (2)	0.168 (5)	0.051 (11)*
HI	0.089 (6)	0.164 (3)	0.083 (5)	0.058 (12)*

knowledge, the crystal structure of the mono-hydriodic acid salt of 4-nitrobenzene-1,2-diamine has not been reported.

As shown in the figure the asymmetric unit of the title structure contains one substituted anilinium cation, one iodide counter anion and one water molecule. The non-hydrogen atoms of the cation are nearly co-planar. Only the N1 atom is protonated to counterbalance the negative charge of iodide anion. There are different types of hydrogen bonds, O–H...I and N–H...I, through which a two-dimensional structure is generated. All the bond lengths of the title compound are comparable with its analogues [5–9].

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: Key Science and Technology Projects in Henan Province (182102311076 and 182102210276), the State key Laboratory of Motor Vehicle Biofuel Technology (KFKT2021001), the Nanyang Institute of Technology Cross-Science Research Project (NITC2021007) and the Rare earth industry fund project of Institute of Green Process Manufacturing Innovation in Chinese Academy of Sciences (IAGM2020DB10).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. Bruker. SAINT v8.37A; Bruker AXS Inc: Madison, Wisconsin, USA, 2015.
2. Bourhis L. J., Dolomanov O. V., Gildea R. J., Howard J. A. K., Puschmann H. The anatomy of a comprehensive constrained, restrained refinement program for the modern computing environment—Olex2 dissected. *Acta Crystallogr.* 2015, *A71*, 59–75.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Sheldrick G. Using phases to determine the space group. *Acta Crystallogr.* 2018, *A74*, a353.
5. Geiger D. K., Parsons D. E. Hydrogen bonding in 4-nitrobenzene-1,2-diamine and two hydrohalide salts. *Acta Crystallogr.* 2014, *C70*, 681–688.
6. Shishkina S. V., Kononova I. S., Shishkina O. V., Boyko A. N. Influence of substituents on the acceptor properties of the amino groups in the diaminobenzene analogues. *CrystEngComm* 2017, *19*, 7162–7176.
7. Weber G. A 2:1 complex of 4-nitro-1,2-benzenediamine and 1,4,7,10,13,16-hexaoxacyclooctadecane (18-crown-6). *Acta Crystallogr.* 1982, *B38*, 629–632.
8. Siri O., Braunstein P. Tuning the synthesis of a dinitroaromatic towards a new trinitroaromatic stabilized energetic material. *New J. Chem.* 2005, *29*, 75–79.
9. Hosten E. C., Betz R. The crystal structure of 2,6-dimethyl-4-nitrophenol, C₈H₉NO₃. *Z. Kristallogr. NCS* 2021, *236*, 269–271.