Shu-Jing Li*, Ke Li and Yi-Wen Li

The crystal structure of 2-nitroisophthalic acid, $C_8H_5NO_6$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	$0.35 \times 0.10 \times 0.07$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.14 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	26.0° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4377, 857, 0.060
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 599
$N(\text{param})_{\text{refined}}$:	71
Programs:	Bruker [1], SHELX [2], Olex2 [3, 4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.5	0.7574(2)	0.25	0.0325(10)
H1	0.5	0.8234(2)	0.25	0.0391(11)*
C2	0.3545(5)	0.70918(16)	0.3198(2)	0.0246(6)
H2	0.2539(5)	0.74270(16)	0.3669(2)	0.0295(7)*
C3	0.3534(4)	0.61252(15)	0.3221(2)	0.0182(6)
C4	0.2000(4)	0.56172(15)	0.39952(19)	0.0162(5)
C5	0.5	0.5658(2)	0.25	0.0146(7)
N1	0.5	0.46333(17)	0.25	0.0165(6)
O1	0.2212(3)	0.47870(10)	0.42006(15)	0.0223(5)
O2	0.0421(3)	0.61606(11)	0.44293(15)	0.0231(5)
H2a	−0.042(3)	0.5844(4)	0.485(2)	0.0346(7)*
O3	0.3335(3)	0.42440(11)	0.18248(15)	0.0250(5)

<https://doi.org/10.1515/ncrs-2019-0704>

Received September 18, 2019; accepted November 8, 2019;
available online December 5, 2019

Abstract

$C_8H_5NO_6$, monoclinic, $C2/c$ (no. 15), $a = 5.4755(12)$ Å, $b = 14.377(3)$ Å, $c = 11.143(3)$ Å, $\beta = 99.096(9)^\circ$, $V = 866.2(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0455$, $wR_{\text{ref}}(F^2) = 0.1341$, $T = 150(2)$ K.

CCDC no.: 1964554

The molecular structure is shown in the figure (only the asymmetric unit is labelled). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All of starting materials were purchased from commercial sources and used as received. The title compound was synthesized in THF solution: solid 2-nitroisophthalic acid (2.11 g,

0.01 mol) was dissolved in 10 mL THF solution in a beaker equipped with a stirrer and stirred at room temperature for 5 min. Then the solution was filtered and let evaporate slowly at room temperature in air. Colorless single crystals were obtained after 12 hours, yield: 93.4% (based on 2-nitroisophthalic acid).

Experimental details

The structure was solved by direct methods with the SHELXS program. All H-atoms from C atoms and O atoms were positioned with idealized geometry and refined isotropically ($U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$) using a riding model with C—H = 0.95 Å and O—H = 0.84 Å, respectively.

*Corresponding author: Shu-Jing Li, Department of Chemistry and Chemical Engineering, Zhoukou Normal University, Zhoukou, Henan Province 466001, P.R. China, e-mail: zksylsj@163.com.
<https://orcid.org/0000-0002-5384-3402>

Ke Li and Yi-Wen Li: Department of Chemistry and Chemical Engineering, Zhoukou Normal University, Zhoukou, Henan Province 466001, P.R. China

Comment

Some 4- and 5-nitroisophthalic acids or their analogues have been reported [5–8]. However, no structural result have been reported for 2-nitroisophthalic acid or 2-nitroisophthalate. To the best of our knowledge, only one crystal structure of an analogue of 2-nitroisophthalic acid, bis(2,2,2-trinitroethyl) 2, 4,6-trinitroisophthalate, has been reported elsewhere [9].

As shown in the figure, there is a two-fold axis along the direction of the line of H1—C1—C5—N1. All of the atoms except the two O atoms from the nitro group are co-planar. The plane defined by the three atoms of the nitro group is almost perpendicular to the plane of benzene ring ($\sim 96^\circ$). There are hydrogen bonds: O—H $\cdots$ O(carboxyl). The carboxyl groups are linked to generate one-dimensional chains by the strong O—H $\cdots$ O(carboxyl)-type of hydrogen bond. All bond lengths and angles of the 2-nitroisophthalic acid are comparable to that reported in the literature [9].

References

1. Bruker. SAINT v8.37A. Bruker AXS Inc, Madison, WI, USA (2015).
2. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112–122.
3. 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.* **A71** (2015) 59–75.
4. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.
5. Liu, Y.-L.; Zou, P.; Xie, M.-H.; Luo, S.-N.; He, Y.-J.: Ethyl 3-carboxy-5-nitrobenzoate. *Acta Crystallogr.* **E65** (2009) o1174.
6. Luo, Y.-H.; Pan, M.-L.: 4-Nitroisophthalic acid. *Acta Crystallogr.* **E68** (2012) o206.
7. Saleh, M. I.; Tham, W.-T.; Adnan, R.; Kusriani, E.; Ng, S.-L.; Fun, H.-K.: A second orthorhombic polymorph of 5-nitroisophthalic acid monohydrate. *Acta Crystallogr.* **E62** (2006) o4837–o4839.
8. Domenicano, A.; Schultz, G.; Hargittai, I.; Colapietro, M.; Portalone, G.; George, P.; Bock, C. W.: Molecular structure of nitrobenzene in the planar and orthogonal conformations. *Struct. Chem.* **1** (1990) 107–122.
9. Xu, Z.; Yang, H.; Cheng, G.: Studies on the synthesis and properties of polynitro compounds based on esteryl backbones. *New J. Chem.* **40** (2016) 9936–9944.