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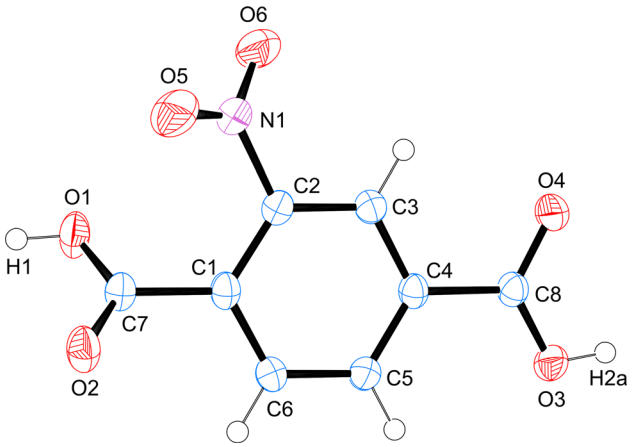
The crystal structure of nitroterephthalic acid, $C_8H_5NO_6$



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Abstract

$C_8H_5NO_6$, monoclinic, $C2/c$ (no.15), a = 12.8112(9) Å, b = 11.7614(8) Å, c = 11.1954(8) Å, β = 100.726(3)°, V = 1657.4(2) Å^3, Z = 8, $R_{gt}(F)$ = 0.042, $wR_{ref}(F^2)$ = 0.1247, T = 173 K.

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The crystal 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.

1 Source of materials

Nitroterephthalic acid was commercially sourced and was not purified further. An amount of 0.1004 g of nitroterephthalic acid (0.476 mmol) was dissolved in 3 ml of AP-grade methanol, the mixture was stirred and then heated at 50 °C in a polytop vial. The solution was left to evaporate slowly at room temperature, with the cap being left only slightly open. Colourless needles formed after seven days.

Table 1: Data collection and handling.

Crystal:	Needle, colourless
Size:	0.27 × 0.09 × 0.06 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.15 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Venture Photon, ω-scans
θ_{max}, completeness:	28°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	25064, 2014, 0.075
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1657
$N(param)_{refined}$:	136
Programs:	Bruker programs [1], SHELX [2, 3], WINGX [4], PLATON [5]

2 Experimental details

C-bound hydrogen atoms were located in the difference map then positioned geometrically and were allowed to ride on their respective parent atoms with thermal displacement parameters 1.2 times of the parent C atom. The coordinates and isotropic displacement parameters of the N-bound and O-bound H atoms involved in hydrogen bonding interactions were allowed to refine freely. Diagrams and publication material were generated using ORTEP [6], WINGX [4] and PLATON [5].

3 Discussion

Nitroterephthalic acid is a derivative of terephthalic acid and is synthesized by the nitration of terephthalic acid [7]. It is used as a modifier in polyethylene terephthalate [8] as well as in the modification in polyethylene glycol [9]. Nitroterephthalic acid is also used in the preparation of many metal organic frameworks [10, 11].

Nitroterephthalic acid crystallizes in the monoclinic space group $C2/c$ and the asymmetric unit contains one molecule of nitroterephthalic acid. The COOH group

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.34135 (11)	0.23098 (13)	0.73605 (12)	0.0237 (3)
C2	0.36042 (11)	0.32704 (13)	0.67086 (13)	0.0246 (3)
C3	0.39191 (11)	0.32075 (13)	0.56015 (13)	0.0249 (3)
H3	0.4032	0.3877	0.5169	0.03*
C4	0.40678 (11)	0.21368 (13)	0.51308 (12)	0.0229 (3)
C5	0.38817 (12)	0.11643 (13)	0.57525 (13)	0.0262 (3)
H5	0.3973	0.0437	0.5417	0.031*
C6	0.35597 (12)	0.12516 (13)	0.68707 (13)	0.0270 (3)
H6	0.3439	0.0582	0.73	0.032*
C7	0.30179 (11)	0.23826 (13)	0.85332 (13)	0.0248 (3)
C8	0.44830 (12)	0.20770 (13)	0.39755 (13)	0.0243 (3)
N1	0.35517 (11)	0.44047 (12)	0.72372 (13)	0.0330 (3)
O1	0.22013 (10)	0.30211 (11)	0.85094 (10)	0.0362 (3)
H1	0.2027	0.3019	0.9197	0.054*
O2	0.34512 (9)	0.18225 (11)	0.94139 (10)	0.0325 (3)
O3	0.46074 (9)	0.11143 (10)	0.35226 (9)	0.0299 (3)
H2A[a]	0.4844	0.1198	0.2877	0.045*
O4	0.46986 (11)	0.30093 (10)	0.35290 (10)	0.0392 (3)
H2B[a]	0.4926	0.289	0.2883	0.059*
O5	0.38634 (12)	0.45010 (12)	0.83345 (12)	0.0475 (4)
O6	0.32168 (14)	0.51810 (12)	0.65542 (13)	0.0522 (4)

[a]Occupancy: 0.5.

meta to the nitro position displays disorder with the hydrogen atom being found equally on either of the two oxygen atoms. In the diagram of the asymmetric unit, one of the two disordered positions has been omitted. All bond lengths in the structure are standard. Each nitroterephthalic acid molecule is hydrogen bonded to an adjacent molecule *via* a COOH···COOH homosynthon. The molecules arrange themselves in 1–D chains with alternating inversions and two-fold rotations. The centre of inversion and rotation axes are respectively found at the centre of the successive COOH···COOH homosynthons.

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References

1. Bruker. *Apex2 (version 2012.10–0), Saint (version 27B), Sadabs (version 2012/1)*; Bruker AXS Inc: Madison, Wisconsin, USA, 2012.
2. Sheldrick G. M. Shelxt – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Sheldrick G. M. Crystal structure refinement with Shelxl. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Farrugia L. J. *WinGX* suite for small-molecule single-crystal crystallography. *J. Appl. Crystallogr.* 1999, *32*, 837–838.
5. Spek A. L. Platon. *Acta Crystallogr. D* 2009, *65*, 148–155.
6. Farrugia L. J. WinGX and ORTEP for Windows: an update. *J. Appl. Crystallogr*. 2012, *45*, 849–854.
7. Ghaemy M., Mighani H. Synthesis and identification of dinitro– and diamino– terephthalic acid. *Chin. Chem. Lett.* 2009, *20*, 800–804.
8. Bardak F. Experimental and DFT analysis of structural and spectroscopic features of nitroterephthalic acid, and computational insights into its molecular interactions with hER–α *via* molecular docking. *J. Mol. Struct.* 2019, *1175*, 458–470.
9. Zhang H., Wang Z., Liu O. Development and validation of a GC—FID method for quantitative analysis of oleic acid and related fatty acids. *J. Pharmaceut. Anal.* 2015, *5*, 223–230.
10. Lee G., Yoo D. K., Ahmed I., Lee H. J., Jhung S. H. Metal–organic frameworks composed of nitro groups: preparation and applications in adsorption and catalysis. *Chem. Eng. J.* 2023, *451*, 138538.
11. Hasanova S. S., Yolchueva E. A., Mashadi A. Q., Muhammad S., Ashfaq M., Muhammed M. E., Munawar K. S., Tahir M. N., Al-Sehemi A. G., AlarfajiSynthesis S. S. Characterization, crystal structures, and supramolecular assembly of copper complexes derived from nitroterephthalic acid along with hirshfeld surface analysis and quantum chemical studies. *ACS Omega* 2023, *8*, 8530–8540.