Chao-Jun Du*, Zong-Dong Wang, Da-Feng Zhu, Yuan-Liang Zhu and Yan Zeng

Crystal structure of 3-iodophthalic acid, $C_8H_5IO_4$

Table 1: Data collection and handling.

Crystal:	Colourless needle
Size:	0.35 × 0.20 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	3.78 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} :	8356, 1724, 0.039
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1565
$N(\text{param})_{\text{refined}}$:	120
Programs:	Bruker [1], SHELX [2], Olex2 [3, 4]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.4001(6)	0.2386(3)	0.2537(2)	0.0105(5)
C2	0.2755(6)	0.2963(4)	0.3436(2)	0.0119(5)
C3	0.2739(6)	0.4767(4)	0.3720(2)	0.0157(6)
H3	0.1848(6)	0.5140(4)	0.4317(2)	0.0188(7)*
C4	0.4023(7)	0.6025(4)	0.3132(2)	0.0186(6)
H4	0.4029(7)	0.7262(4)	0.3334(2)	0.0223(7)*
C5	0.5291(6)	0.5487(4)	0.2253(2)	0.0162(6)
H5	0.6188(6)	0.6354(4)	0.1856(2)	0.0195(7)*
C6	0.5249(6)	0.3672(4)	0.1954(2)	0.0131(5)
C7	0.4006(6)	0.0442(4)	0.2180(2)	0.0113(5)
C8	0.1524(6)	0.1640(4)	0.4105(2)	0.0122(5)
I1	0.69137(4)	0.29139(3)	0.054076(16)	0.01835(7)
O1	0.1312(4)	−0.0804(2)	0.16793(16)	0.0154(4)
H1	−0.0148(9)	−0.0349(15)	0.174(2)	0.0231(6)*
O2	0.6351(4)	−0.0001(3)	0.22628(17)	0.0165(4)
O3	−0.0210(5)	0.2187(3)	0.47517(17)	0.0181(4)
H3a	−0.073(7)	0.142(2)	0.514(2)	0.0271(6)*
O4	0.2127(4)	0.0194(2)	0.40536(16)	0.0162(4)

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Abstract

$C_8H_5IO_4$, triclinic, $P\bar{1}$ (no. 2), $a = 4.5570(3)$ Å, $b = 7.7720(5)$ Å, $c = 12.7660(8)$ Å, $\alpha = 99.906(3)^\circ$, $\beta = 97.718(3)^\circ$, $\gamma = 105.651(3)^\circ$, $V = 421.02(5)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0191$, $wR_{\text{ref}}(F^2) = 0.0433$, $T = 150(2)$ K.

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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

All agents are commercial resource and used without further purification. The title compound was crystallized as follows:

***Corresponding author: Chao-Jun Du**, School of Biochemical and Chemical Engineering, Nanyang Institute of Technology, Nanyang, Henan Province 473000, P.R. China, e-mail: hyperchem@126.com. <https://orcid.org/0000-0002-4767-7057>

Zong-Dong Wang: Nanyang Radiation Environment Supervision and Management Station, Nanyang, Henan Province 473000, P.R. China

Da-Feng Zhu: Nanyang Ke Mei Environmental Testing Co. Ltd, Nanyang, Henan Province 473004, P.R. China

Yuan-Liang Zhu and Yan Zeng: School of Biochemical and Chemical Engineering, Nanyang Institute of Technology, Nanyang, Henan Province 473000, P.R. China

solid 3-iodophthalic acid (2.92 g, 0.01 mol) was added in 10 mL THF solution in a beaker with stirring for 15 min, filtered and let evaporate. Colorless crystals were obtained after one day.

Experimental details

The structure was solved by Direct Methods with the SHELXS program [2]. All H-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 = 0.840–845 Å, respectively. Unusual ellipsoids

may be the result of a non-optimal mounting strategy leading to problems during the data processing, but the atom-type assignments are unquestionable.

Comment

The crystal structures of 3-iodophthalic acid derivatives are rare and to the best of our knowledge, only three crystal structures of 3-iodophthalic acid analogues have been reported [5]. They are dimethyl 3,6-diiodo-4,5-dimethylphthalate, dimethyl 4,5-dibutyl-3,6-diiodophthalate and dimethyl 3,6-diiodo-4,5-dipropylphthalate. A crystal structure of 4-iodophthalic acid analogues, dimethyl 3-hydroxy-4-iodo-5-((6-methyl-4-oxohepta-2,5-dien-2-yl)oxy)phthalate, has also been reported elsewhere [6].

As shown in the figure, the title compound, 3-iodophthalic acid, with its molecule formula of C₈H₅IO₄, crystallizes in *P* $\bar{1}$. Two hydrogen bonds: the end-to-end model of O—H...O to generate one-dimensional chains and the head to head model of O—H...O to generate 8-membered ring, which are linked to construct strands along the [100] direction. A three-dimensional supramolecular structure will be constructed by weak I...O interactions (with a distance of 3.33 Å). All of the bond lengths and angles of 3-iodophthalic acid are comparable to its analogues [5–7].

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