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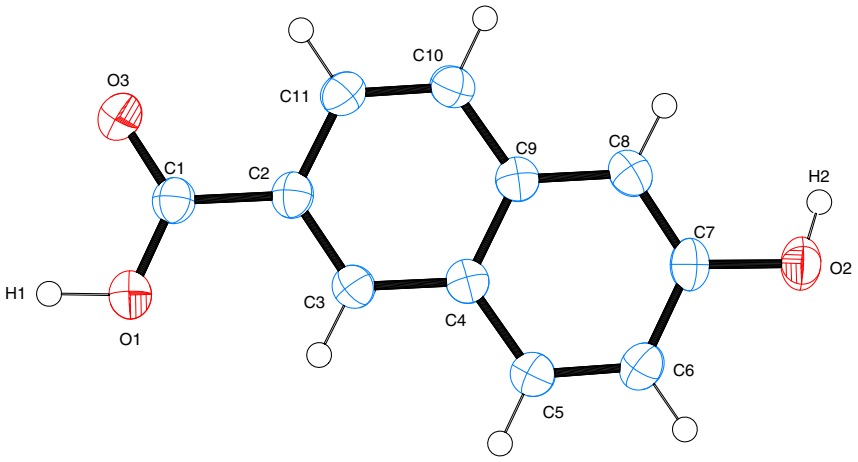
The crystal structure of a new polymorph of 6-hydroxy-2-naphthoic acid, $C_{11}H_8O_3$



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Abstract

$C_{11}H_8O_3$, monoclinic, $P2_1/c$ (no. 14), a = 8.6478(8) Å, b = 5.2261(5) Å, c = 18.7921(18) Å, β = 94.518(3)°, V = 846.66(14) Å^3, Z = 4, $R_{gt}(F)$ = 0.0419, $wR_{ref}(F^2)$ = 0.1181, T = 173 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.

1 Source of materials

6–Hydroxy-2-naphthoic acid was commercially sourced and was not purified further. An amount of 0.0635 g of 6-hydroxy-2-naphthoic acid (0.337 mmol) and 0.0670 g 4-aminoantipyrine (0.328 mmol) were dissolved in 5 mL of a 50/50 (vol/vol) AP-grade methanol/1,2-dichloroethane mixture. The mixture was stirred at 27 °C in a polytop vial. The solution was left to evaporate slowly at room temperature, with the cap being left only slightly open. Colourless plates formed after 3 days.

Table 1: Data collection and handling.

Crystal:	Colourless plate
Size:	0.28 × 0.12 × 0.05 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.11 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Venture Microfocus Photon III, ω
θ_{max}, completeness:	28.0°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8668, 2043, 0.050
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1311
$N(param)_{refined}$:	133
Programs:	Bruker [1], SHELX [2, 3], WinGX/ORTEP-3 [4, 5], PLATON [6]

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 O-bound H atoms involved in hydrogen bonding interactions were

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.81899 (18)	0.4941 (3)	0.55233 (9)	0.0315 (4)
C2	0.67520 (18)	0.4821 (3)	0.58940 (8)	0.0304 (4)
C3	0.55872 (19)	0.6568 (3)	0.57537 (8)	0.0314 (4)
H3	0.571947	0.790471	0.542146	0.038*
C4	0.41878 (18)	0.6413 (3)	0.60963 (8)	0.0296 (4)
C5	0.29684 (19)	0.8197 (3)	0.59664 (9)	0.0345 (4)
H5	0.306845	0.95312	0.562967	0.041*
C6	0.1651 (2)	0.8030 (3)	0.63177 (9)	0.0347 (4)
H6	0.084072	0.924134	0.622707	0.042*
C7	0.15007 (19)	0.6054 (3)	0.68145 (9)	0.0325 (4)
C8	0.26281 (19)	0.4266 (3)	0.69461 (9)	0.0327 (4)
H8	0.248988	0.292491	0.727655	0.039*
C9	0.40051 (18)	0.4403 (3)	0.65907 (8)	0.0294 (4)
C10	0.52289 (19)	0.2637 (3)	0.67250 (9)	0.0341 (4)
H10	0.511929	0.128701	0.705574	0.041*
C11	0.65640 (19)	0.2832 (3)	0.63894 (9)	0.0348 (4)
H11	0.73732	0.162391	0.648883	0.042*
O1	0.83436 (15)	0.6859 (2)	0.51021 (7)	0.0412 (3)
H1	0.933 (2)	0.680 (4)	0.4850 (10)	0.051*
O2	0.01563 (14)	0.6079 (3)	0.71664 (7)	0.0401 (3)
H2	0.010 (2)	0.470 (4)	0.7381 (11)	0.051*
O3	0.91920 (13)	0.3217 (2)	0.56176 (6)	0.0383 (3)

allowed to refine freely. Diagrams and publication material were generated using ORTEP-3 [4], WinGX [5] and PLATON [6].

3 Comment

6–Hydroxy-2-naphthoic acid is used as a co-monomer of meltable thermotropic co-polyestres of 4-hydrobenzoic acid [7], as well as being used as a polymer material in polymeric liquid crystals having high performance properties like thermal stability [8].

6–Hydroxy-2-naphthoic acid crystallizes in the monoclinic $P2_1/c$ space group and the asymmetric unit contains one molecule of 6-hydroxy-2-naphthoic acid.

One polymorph of 6-hydroxy-2-naphthoic acid has already been published (CCDC Identifier: JACDAV). In the packing of the previously published polymorph, the carboxylic acids hydrogen bond to each other *via* COOH···COOH homosynthons and the phenol OH hydrogen atoms bond to the *anti*-lone pair of the acid carbonyl of adjacent atoms [9]. However, in the polymorph described in this paper, the COOH group of each molecule is similarly hydrogen bonded to an adjacent molecule *via* the COOH···COOH homosynthon, but the hydroxyl group on each molecule is hydrogen bonded by its hydrogen donor atom to the oxygen acceptor atom of the hydroxyl group of an adjacent molecule. In the crystal packing of the new polymorph, the molecules arrange themselves in 2D sheets with a 2_1 screw axis parallel to the *b*-axis and a glide plane perpendicular to the *b*-axis.

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