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A new triclinic polymorph of 4-*n*-hexyloxybenzoic acid

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.076
wR factor = 0.202
Data-to-parameter ratio = 16.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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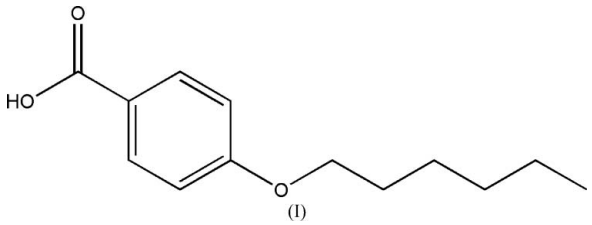
The title new triclinic polymorph of 4-*n*-hexyloxybenzoic acid, $C_{13}H_{18}O_3$, crystallizes with six independent molecules in the asymmetric unit. Four of these have the hydroxyl group arranged *cis* to the methylene group attached to the ether O atom, whereas in two molecules the carbonyl group is *cis* to the methylene group at the ether O atom. All molecules form hydrogen-bonded dimers through the carboxylic acid functionalities. The cell parameters for what appears to be the title polymorph had been originally inaccurately determined as monoclinic [a = 14.5 Å, b = 33.0 Å, c = 8.0 Å, α = 90.0°, β = 93.0° and γ = 90.0°; Bryan (1960). *J. Chem. Soc.* pp. 2517–2519]; no space group was given and no atomic coordinates were available. The other polymorph known for the title compound is a Z' = 1 phase crystallizing in $P2_1/c$ [Bryan *et al.* (1980). *Mol. Cryst. Liq. Cryst.* **62**, 311–326].

Comment

Metallomesogens, liquid crystalline compounds containing transition metals, are of great interest, because of their electric, magnetic and chromatic properties. Recently, ferrocene has been widely studied in this field (Deschenaux *et al.*, 1997). Liquid crystals are observed in a wide range of areas, in physical and biological sciences, ranging from obvious technological applications (Nierengarten *et al.*, 1999), such as twisted-nematic and ferroelectric display devices, to the widespread use of surfactants in the clearing industry. Ferrocene-based dendrimers display remarkable electrochemical properties and have been used for the construction of batteries (González *et al.*, 2002) and sensors (Valério *et al.*, 1997). The title compound, (I), is a precursor for the synthesis of liquid crystals having such potential applications.



(I)

The title compound crystallizes with six independent molecules in the asymmetric unit (Fig. 1). Four of them have the hydroxyl group *cis* to the methylene group attached to the ether O atom, and the two other molecules have the carbonyl group *cis* to the methylene group at the ether O atom. All molecules form hydrogen-bonded dimers in the crystal structure, through the carboxylic acid functionalities; two molecules form a centrosymmetric dimer with a symmetry-related

Figure 1
Structures of molecules 1–6 in (I). Displacement ellipsoids are drawn at the 50% probability level.

molecule, whereas the remainder are hydrogen bonded to another independent molecule in the asymmetric unit (Table 1, Fig. 2). The *n*-hexyl chains are fully extended in all the molecules, and geometric parameters are found to be in the usual ranges.

The cell parameters for (I) had been reported (Bryan, 1960), although with limited precision. No information was given about space group nor any atomic coordinates. In addition, two of the cell angles were reported to be 90°, suggesting monoclinic symmetry, whereas the actual symmetry is triclinic. Interestingly, the crystal structure of a monoclinic polymorph of (I) was subsequently characterized by the same author and co-workers (Bryan *et al.*, 1980) in space group $P2_1/c$.

Experimental

A solution of 0.5 *M* KOH in ethanol/water (95:5; 150 ml) was used to dissolve 4-*n*-hexyloxybenzoic acid (3 g, 12 mmol). The mixture was refluxed for 12 h, and then allowed to cool to room temperature. The mixture was acidified with 10% HCl and checked with litmus paper until acid pH. Compound (I) was then precipitated with water. The solid was filtered off and recrystallized from ethanol. Yield 82%, m.p. 365 K.

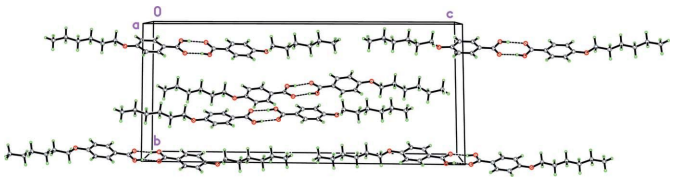


Figure 2
Packing diagram of (I), with a view along [100]. Hydrogen bonds are shown as dashed lines.

Crystal data

$C_{13}H_{18}O_3$
$M_r = 222.27$
Triclinic, $P\overline{1}$
$a = 7.9679$ (10) Å
$b = 14.4604$ (18) Å
$c = 33.389$ (4) Å
$\alpha = 88.809$ (10)°
$\beta = 85.795$ (9)°
$\gamma = 78.015$ (9)°
$V = 3753.0$ (8) Å^3
$Z = 12$
$D_x = 1.180$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 173$ (2) K
Block, colourless
0.35 × 0.33 × 0.32 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: none
31668 measured reflections
13939 independent reflections
10358 reflections with $I > 2\sigma(I)$
$R_{int} = 0.067$
$\theta_{max} = 25.7°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.076$
$wR(F^2) = 0.202$
$S = 1.12$
13939 reflections
872 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 7.8018P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0013 (2)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3^{i}	0.84	1.81	2.638 (4)	171
O2A—H2A···O3A^{ii}	0.84	1.81	2.644 (4)	173
O2B—H2B···O3C	0.84	1.83	2.663 (4)	174
O2C—H2C···O3B	0.84	1.84	2.679 (4)	175
O2D—H2D···O3E	0.84	1.81	2.647 (3)	172
O2E—H2E···O3D	0.84	1.84	2.675 (3)	174

Symmetry codes: (i) $-x+1, -y+2, -z+2$; (ii) $-x+1, -y+1, -z+1$.

H atoms were found in a difference map but were refined using a riding model, with constrained bond lengths: O—H = 0.84 Å, aromatic C—H = 0.95 Å, methylene C—H = 0.99 Å and methyl C—H = 0.98 Å. Isotropic displacement parameters for H atoms were fixed to U_{iso}(H) = 1.5U_{eq}(carrier C) for methyl groups and U_{iso}(H) = 1.2U_{eq}(carrier atom) for other H atoms. The hydroxyl groups were allowed to rotate but not to tip.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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A new triclinic polymorph of 4-*n*-hexyloxybenzoic acid. Corrigendum

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In the paper by Nazir, Zareen, Bolte, Butt & Siddiqi [*Acta Cryst.* (2007), E**63**, o12–o14], the name of the second author is given incorrectly. The correct author list is shown opposite.