

1-(4-Hydroxyphenyl)butan-1-one

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Key indicators

Single-crystal X-ray study

$T = 293$ K

Mean $\sigma(\text{C}-\text{C}) = 0.008$ Å

R factor = 0.099

wR factor = 0.168

Data-to-parameter ratio = 16.8

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

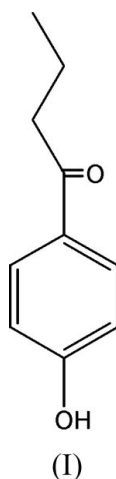
The title compound, $\text{C}_{10}\text{H}_{12}\text{O}_2$, was synthesized by the Fries rearrangement of phenyl butyrate. There are two independent molecules in the asymmetric unit, and all non-H atoms in each molecule are coplanar, with r.m.s. deviations of 0.050 and 0.024 Å. In the crystal structure, each molecule is linked to symmetry-equivalent molecules by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds to form chains along the a axis.

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Comment

Hydroxyphenyl ketones are effective materials for preparing the corresponding normal alkylphenols *via* Clemmensen reduction (Read & Wood, 1955) or the Huang–Minlon modification of Wolff–Kishner reduction (Furniss *et al.*, 2004). The title ketone, (I), is a useful intermediate for the sulfamate ester which is used for the treatment of arthritis and osteoporosis (Sek *et al.*, 1990). The crystal structure of 1-(2-hydroxyphenyl)octan-1-one has already been reported (Bin *et al.*, 2006). We report here the crystal structure of the title compound, (I).



There are two independent molecules in the asymmetric unit of (I) and they have essentially the same geometry. The structure of both molecules is shown in Fig. 1, and selected bond lengths and angles are given in Table 1. All non-H atoms in each molecule are coplanar, with r.m.s. deviations of 0.050 and 0.024 Å. The planarity can be attributed to the delocalization of the electrons over the benzene ring and carbonyl group.

In the crystal structure, each molecule is linked by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds to symmetry-equivalent molecules (Table 2), forming chains along the a axis (Fig. 2).

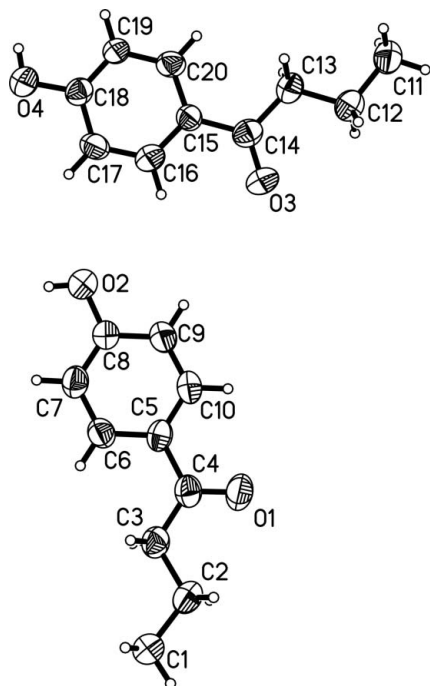


Figure 1
The asymmetric unit of (I). Displacement ellipsoids are drawn at the 30% probability level.

Experimental

Anhydrous aluminium chloride (310 g, 2.32 mol) and tetrachloroethane (300 ml) were placed in a 2 l four-necked round-bottomed flask, fitted with a reflux condenser, a mechanical stirrer and a 100 ml dropping funnel. Phenyl butyrate (339 g, 2.07 mol) was added slowly through the dropping funnel to the stirred suspension over a period of 1.5 h. The mixture was then heated to 313 K for about 2 h, heated and stirred at 373–393 K for 5 h, and then hydrolyzed by pouring on to ice. The product was steam-distilled to remove the solvent and excess phenol. The residue was separated and washed free of mineral acids. It was further washed with a solution of sodium hydroxide (19.2 g) and ethanol (162 ml) in water (972 ml). The oil layer was removed and the alkaline extract was acidified with hydrochloric acid. The resulting solid was crystallized from methanol by slow evaporation to give crystals of (I) suitable for X-ray diffraction.

Crystal data

$C_{10}H_{12}O_2$	$Z = 8$
$M_r = 164.20$	$D_x = 1.206 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 8.2650 (17) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$b = 30.986 (6) \text{ \AA}$	$T = 293 (2) \text{ K}$
$c = 7.9200 (16) \text{ \AA}$	Needle, colourless
$\beta = 116.94 (3)^\circ$	$0.40 \times 0.20 \times 0.10 \text{ mm}$
$V = 1808.2 (6) \text{ \AA}^3$	

Data collection

Enraf–Nonius CAD-4 diffractometer	3535 independent reflections
$\omega/2\theta$ scans	1507 reflections with $I > 2\sigma(I)$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$\theta_{\text{max}} = 26.0^\circ$
$T_{\text{min}} = 0.968$, $T_{\text{max}} = 0.992$	3 standard reflections
3535 measured reflections	every 200 reflections
	intensity decay: none

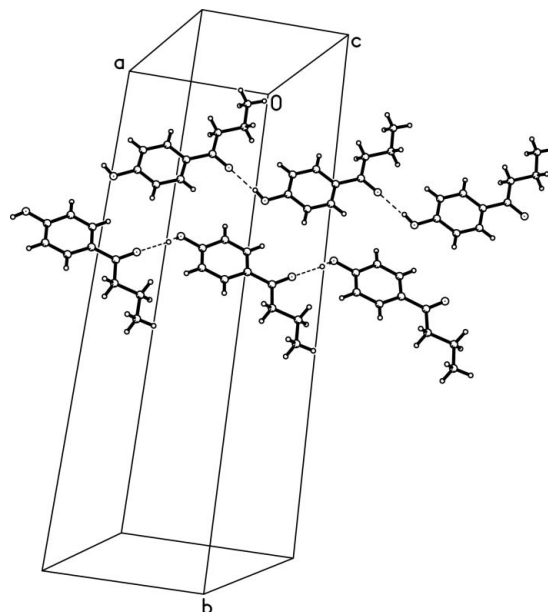


Figure 2
Part of the crystal packing of (I), showing O–H...O hydrogen-bonded (dashed lines) chains.

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.099$	$w = 1/[\sigma^2(F_o^2) + (0.01P)^2 + 2P]$
$wR(F^2) = 0.168$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{\text{max}} = 0.006$
3535 reflections	$\Delta\rho_{\text{max}} = 0.15 \text{ e \AA}^{-3}$
211 parameters	$\Delta\rho_{\text{min}} = -0.13 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1–C4	1.235 (5)	O3–C14	1.228 (5)
O2–C8	1.362 (6)	O4–C18	1.375 (5)
O1–C4–C3	121.5 (5)	O3–C14–C15	121.9 (5)
O1–C4–C5	118.4 (5)	O3–C14–C13	119.9 (5)
C7–C8–O2	122.9 (5)	C19–C18–O4	121.9 (5)
O2–C8–C9	116.6 (5)	O4–C18–C17	116.4 (5)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2–H2C...O1 ⁱ	0.82	1.93	2.747 (6)	172
O4–H4A...O3 ⁱ	0.82	1.92	2.737 (6)	172

Symmetry code: (i) $x + 1, y, z$.

H atoms were positioned geometrically, with O–H = 0.82 \AA , and C–H = 0.93 (aromatic), 0.96 (CH_3) or 0.97 \AA (CH_2), and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{eq}}(\text{C}, \text{O})$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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