

Xu Bin, Jin-Xiu Ji, Zhi-Qiang Fen
and Jin-Tang Wang*Department of Applied Chemistry, College of
Science, Nanjing University of Technology,
Nanjing 210009, People's Republic of China

Correspondence e-mail: wjt@njut.edu.cn

Key indicators

Single-crystal X-ray study
 $T = 288$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.060
 wR factor = 0.170
Data-to-parameter ratio = 17.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

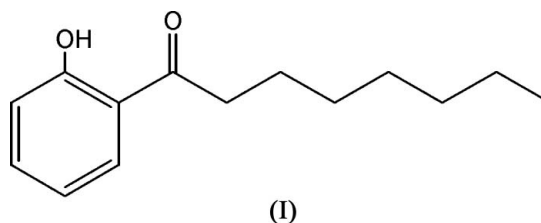
1-(2-Hydroxyphenyl)octan-1-one

The title compound, $\text{C}_{14}\text{H}_{20}\text{O}_2$, was synthesized by the reaction of phenol and octanoyl chloride. All non-H atoms are coplanar, with an r.m.s. deviation of 0.031 Å. In the crystal structure, molecules are linked into chains along the a axis by intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

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Comment

Hydroxyphenyl ketones are useful for preparing the corresponding normal alkylphenols by Clemmensen reduction (Read *et al.*, 1955) or the Huang–Minlon modification of the Wolff–Kishner reduction (Furniss *et al.*, 2004). Ketone derivatives are significant materials for the treatment of cardiovascular disease in relation to calcium-ATPase inhibition (Don *et al.*, 1994). We report here the crystal structure of the title compound, (I).



All non-H atoms are coplanar, with an r.m.s. deviation of 0.031 Å (Fig. 1). The planarity can be attributed to the intramolecular $\text{O}2-\text{H}2\text{A}\cdots\text{O}1$ hydrogen bond and delocalization of the electrons over the benzene ring and carbonyl group. Selected bond lengths and angles are given in Table 1. In the crystal structure, molecules are linked by intermolecular $\text{C}7-\text{H}7\text{B}\cdots\text{O}2$ hydrogen bonds (Table 2), forming chains along the a axis.

Experimental

Phenol (14 g, 0.15 mol) was dissolved in tetrachloroethane (30 ml) at 283 K and anhydrous aluminium chloride (20 g, 0.15 mol) was added at such a rate that the temperature did not rise above 288 K. The cooling bath was then removed and capryl chloride (16 g, 0.1 mol) was added. The resulting mixture was heated and stirred at 403–413 K for 6 h, and was hydrolysed by pouring on to ice. The product was steam-distilled to remove the solvent and excess phenol. The low melting-point solid was separated and washed free of mineral acids. It was further washed with a solution of sodium hydroxide (3 g) and ethyl alcohol (40 ml) in water (160 ml). The insoluble portion was separated by filtration and was crystallized from ligroin (333–363 K) and gave 8 g of light yellow flaky crystals. Recrystallization from ligroin (333–363 K) gave suitable single crystals for the X-ray diffraction study.

Crystal data

$C_{14}H_{20}O_2$
 $M_r = 220.30$
 Triclinic, $P\bar{1}$
 $a = 6.8560$ (14) Å
 $b = 8.4850$ (17) Å
 $c = 11.547$ (2) Å
 $\alpha = 95.20$ (3)°
 $\beta = 102.54$ (3)°
 $\gamma = 93.15$ (3)°
 $V = 651.1$ (2) Å³

$Z = 2$
 $D_x = 1.124$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 10\text{--}13^\circ$
 $\mu = 0.07$ mm⁻¹
 $T = 288$ (2) K
 Block, light yellow
 0.40 × 0.20 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.971$, $T_{\max} = 0.986$
 2781 measured reflections
 2555 independent reflections
 1229 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = 0 \rightarrow 8$
 $k = -10 \rightarrow 10$
 $l = -14 \rightarrow 13$
 3 standard reflections every 200 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.060$
 $wR(F^2) = 0.170$
 $S = 1.03$
 2555 reflections
 146 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.02P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.14$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.029 (6)

Table 1

Selected geometric parameters (Å, °).

| | | | |
|----------|-----------|------------|-----------|
| O1–C8 | 1.232 (3) | C8–C9 | 1.470 (3) |
| O2–C14 | 1.348 (3) | | |
| O1–C8–C9 | 120.8 (2) | O2–C14–C13 | 117.7 (2) |
| O1–C8–C7 | 119.0 (2) | O2–C14–C9 | 122.1 (2) |

Table 2

Hydrogen-bond geometry (Å, °).

| $D\text{--}H\cdots A$ | $D\text{--}H$ | $H\cdots A$ | $D\cdots A$ | $D\text{--}H\cdots A$ |
|---------------------------------|---------------|-------------|-------------|-----------------------|
| O2–H2A \cdots O1 | 0.82 | 1.82 | 2.542 (2) | 146 |
| C7–H7B \cdots O2 ⁱ | 0.97 | 2.59 | 3.377 (3) | 138 |

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

The H atoms were positioned geometrically, with O–H = 0.82 Å and C–H = 0.93 (aromatic), 0.96 (CH₃) or 0.97 Å (CH₂), and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C}, \text{O})$.

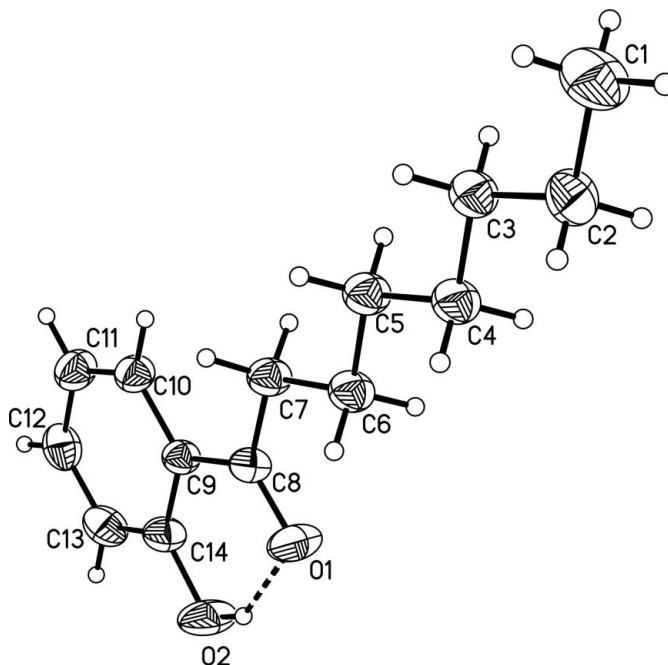


Figure 1

The structure of (I). Displacement ellipsoids are drawn at the 30% probability level. The dashed line indicates the O–H \cdots O hydrogen bond.

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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References

- Bruker (2000). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Don, R. B., Charles, D. C. & Li, Q. (1994). US Patent No WO9428886.
 Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
 Furniss, S. B., Hannaford, A. J., Smith, P. W. G. & Tatchell, A. R. (2004). *Vogel's Textbook of Practical Organic Chemistry*, 5th ed., pp. 827, 831. London: Addison-Wesley Longman Limited.
 Harms, K. & Wocadlo, S. (1996). *XCAD4*. University of Marburg, Germany.
 North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
 Read, R. R. & Wood, J. Jr (1955). *Organic Syntheses*, Collected Vol. 3, p. 444.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.