

1-(2-Hydroxy-3,5-dimethylphenyl)ethanone

Jian-Guo Chang,* Guo-Fang He,
Ji-Kun Li and Yan-Fei LiDepartment of Material Science and Chemical
Engineering, Taishan University, 271021 Taian,
Shandong, People's Republic of China

Correspondence e-mail: tsucjg@163.com

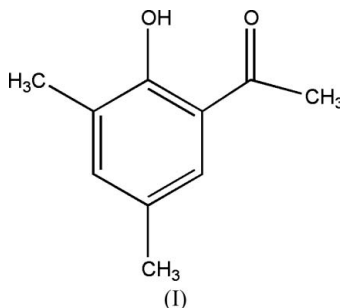
Key indicators

Single-crystal X-ray study
 $T = 273$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.065
 wR factor = 0.210
Data-to-parameter ratio = 13.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The structure of the title molecule, $\text{C}_{10}\text{H}_{12}\text{O}_2$, is stabilized by an intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond. There are two crystallographically independent molecules in the asymmetric unit.

Comment

The title compound, (I), is commonly used as a chelator to characterize metal ions in analytical chemistry (Jetley *et al.*, 1980; Patel & Patel, 1975). The structure of (I) was previously predicted on the basis of spectroscopic analysis and we report here the crystal structure of (I).



There are two crystallographically independent molecules in the asymmetric unit (Fig. 1). The conformations of the two molecules are almost the same, with no significant differences in bond lengths and angles. Each molecule exhibits an intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond between the hydroxy group and carbonyl O atom (Table 1).

Experimental

Acetyl chloride (3.92 g, 0.05 mol) was added dropwise to 3,5-dimethylphenol (6.11 g, 0.05 mol) and the mixture was reacted at room temperature for 1 h, then heated to 373 K for 2 h. After cooling to ambient temperature, the chloroform-diluted mixture was washed with sodium carbonate solution and water several times. The organic layer was dried with anhydrous sodium sulfate, filtered, and the solvent was removed. Further purification was carried out by vacuum distillation; 2,4-dimethylphenyl acetate was obtained (yield 83.6%). A mixture of 2,4-dimethylphenyl acetate (6.87 g, 0.042 mol) and anhydrous aluminium chloride (16.80 g, 0.126 mol) was then gradually heated to 373 K for 0.5 h; it was then heated to 423 K and kept at that temperature for 0.5 h. The reaction mixture was cooled to room temperature, hydrolysed, and extracted with 50 ml chloroform. The organic layer was washed with water, dried with anhydrous sodium sulfate and filtered. Chloroform was removed under reduced pressure. The residue was distilled under vacuum to obtain the title compound (yield 79.0%). The compound was recrystallized from ethyl acetate to obtain colourless single crystals suitable for X-ray diffraction.

Received 25 August 2006
Accepted 30 August 2006

Crystal data

C₁₀H₁₂O₂
 M_r = 164.20
 Monoclinic, P₂₁/c
 a = 14.46 (2) Å
 b = 8.076 (15) Å
 c = 16.03 (3) Å
 β = 109.05 (2)°
 V = 1769 (5) Å³

Z = 8
 D_x = 1.233 Mg m⁻³
 Mo Kα radiation
 μ = 0.09 mm⁻¹
 T = 273 (2) K
 Block, colourless
 0.55 × 0.37 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 T_{min} = 0.939, T_{max} = 0.979

6791 measured reflections
 3087 independent reflections
 1913 reflections with I > 2σ(I)
 R_{int} = 0.033
 θ_{max} = 25.0°

Refinement

Refinement on F²
 R[F² > 2σ(F²)] = 0.065
 wR(F²) = 0.210
 S = 1.00
 3087 reflections
 225 parameters
 H-atom parameters constrained

w = 1/[σ²(F_o²) + (0.1P)² + 0.55P]
 where P = (F_o² + 2F_c²)/3
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.23 e Å⁻³
 Δρ_{min} = -0.29 e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...O2	0.82	1.80	2.521 (6)	146
O3—H3...O4	0.82	1.81	2.527 (6)	146

All H atoms were positioned geometrically and treated as riding on their parent atoms, with methyl C—H = 0.96 Å, aromatic C—H = 0.93 Å, O—H = 0.82 Å, and U_{iso}(H) = 1.5U_{eq}(methyl C and O) and 1.2U_{eq}(aromatic C).

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve

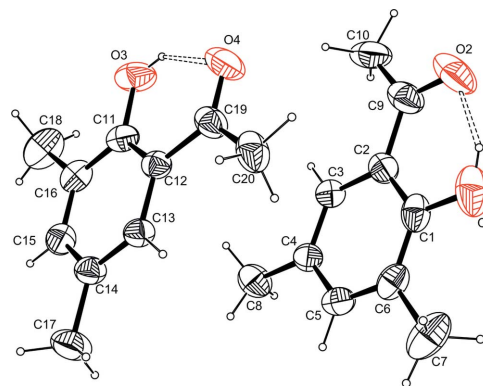


Figure 1

The asymmetric unit of (I). Displacement ellipsoids are drawn at the 30% probability level. H bonds are drawn as dashed lines.

structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: ORTEP III (Burnett & Johnson, 1996) and ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXTL (Sheldrick, 1997b).

This project was supported by the Postgraduate Foundation of Taishan University (No. Y05-2-09).

References

Burnett, M. N. & Johnson, C. K. (1996). ORTEP III. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
 Farrugia, L. J. (1997). J. Appl. Cryst. 30, 565.
 Jetley, U. K., Singh, Jai & Rastogi, S. N. (1980). Acta Cienc. India Chem. 6, 174-175.
 Patel, C. B. & Patel, R. P. (1975). J. Indian Chem. Soc. 52, 312-314.
 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (1997a). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Sheldrick, G. M. (1997b). SHELXTL. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
 Siemens (1996). SMART and SAINT. Versions 4.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.