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(3-Chlorophenyl)(2-hydroxy-5-methylphenyl)-methanone

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.151
Data-to-parameter ratio = 12.5

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

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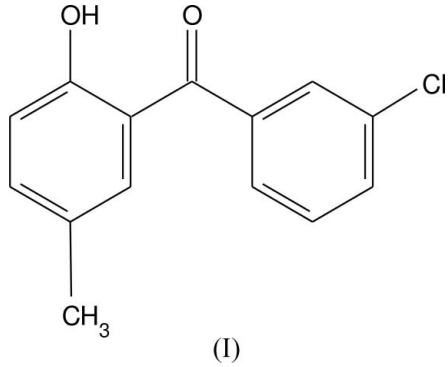
In the title compound, $C_{15}H_{14}ClO_2$, the dihedral angle between the two benzene rings is 57.37 (12)°.

Comment

The significance of benzophenone analogues in biological systems, as well as in chemotherapy, is now well established (Hsieh *et al.*, 2003; Revesz *et al.*, 2004). The chemistry of hydroxybenzophenones constitutes a central and important area of interest in synthetic organic, medicinal and pharmacological chemistry (Cuesta-Rubio *et al.*, 2002; Schlitzer *et al.*, 2002; Vidya *et al.*, 2003). Serving as attractive scaffolds for drug design and conferring drug-like characteristics on numerous structural motifs, halo-substituted hydroxybenzophenones are finding increasing applications in organic and medicinal chemistry (Khanum *et al.*, 2005). Based on the above observations, the title compound, (I), was synthesized and its crystal structure is reported here.



(I)

The molecule of (I) is non-planar (Fig. 1). The dihedral angle between the two benzene rings is 57.37 (12)°, a value much smaller than that of 75.2° observed for (2-chlorophenyl)(3,4-dimethoxyphenyl)methanone, (II) (Mahendra *et al.*, 2003). The bond lengths and angles have normal values and are comparable with those reported for (II). The crystal packing is stabilized by intramolecular O9—H9···O12 and intermolecular C6—H6···O12 hydrogen bonds (Table 2), which link the molecules into chains (Fig. 2). A detailed study of the biological activity of (I) is underway.

Experimental

A solution of anhydrous aluminium chloride (3.2 g, 0.02 mol) in dry nitrobenzene (25 ml) was added to 4-methylphenyl chlorobenzoate (5 g, 0.02 mol) dissolved in nitrobenzene (10 ml). The mixture was protected from moisture by a calcium chloride guard tube and refluxed with stirring for 30 min. At the end of this period, the solution was cooled and treated with acidic ice-cold water. Nitro-

benzene was removed by steam distillation. The residual solid was crushed into a powder, extracted with 10% sodium hydroxide (150 ml), and the basic aqueous solution was neutralized with 10% hydrochloric acid. The product was extracted into diethyl ether and the ether layer washed well with a saturated sodium chloride solution. Evaporation of the ether after drying over anhydrous sodium sulfate followed by recrystallization from methanol gave (I) in 85% yield (m.p. 344–346 K). IR (Nujol): 1673 (C═O), 3550–3640 cm^{-1} (OH); ^1H NMR ($CDCl_3$): 2.2 (*s*, 3H, CH_3), 7.0–7.65 (*m*, 7H, Ar—H), 12.15 (*bs*, 1H, OH); MS (EI) *m*/*z*: 246 (M^+, 88); Analysis calculated for $C_{14}H_{11}ClO_2$: C 68.15, H 4.46, Cl 14.40%; found: C 68.17, H 4.44, Cl 14.42%.

Crystal data

$C_{14}H_{11}ClO_2$	D_x = 1.375 Mg m^{-3}
M_r = 246.68	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 3643 reflections
a = 10.485 (9) Å	θ = 2.3–25.0°
b = 7.823 (4) Å	μ = 0.31 mm^{-1}
c = 16.297 (13) Å	T = 295 (2) K
β = 116.949 (2)°	Block, pale yellow
V = 1191.59 (15) $Å^3$	0.3 × 0.2 × 0.2 mm
Z = 4	

Data collection

MacScience DIPLabo 32001 diffractometer	1690 reflections with $I > 2\sigma(I)$
	R_{int} = 0.017
ω scans	θ_{max} = 25.0°
Absorption correction: none	$h = -12 \rightarrow 12$
3643 measured reflections	$k = -8 \rightarrow 7$
1945 independent reflections	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.089P)^2 + 0.3587P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.049	
$wR(F^2)$ = 0.151	
S = 1.09	$(\Delta/\sigma)_{max} < 0.001$
1945 reflections	$\Delta\rho_{max}$ = 0.56 e $Å^{-3}$
156 parameters	$\Delta\rho_{min}$ = −0.33 e $Å^{-3}$
H-atom parameters constrained	Extinction correction: *SHELXL97*
	Extinction coefficient: 0.069 (8)

Table 1
Selected geometric parameters (Å, °).

Cl1—C15	1.747 (3)	O12—C11	1.234 (3)
O9—C8	1.351 (3)		
O9—C8—C7	117.9 (2)	O12—C11—C10	121.2 (2)
O9—C8—C10	123.05 (19)	Cl1—C15—C16	119.5 (3)
O12—C11—C13	118.0 (2)	Cl1—C15—C14	118.72 (19)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O9—H9···O12	0.82	1.87	2.585 (3)	145
C6—H6···O12^{i}	0.93	2.54	3.408 (3)	156

Symmetry code: (i) $x, -y+\frac{3}{2}, +z-\frac{1}{2}$.

Difficulties with processing some strong reflections led to their omission from the data set, limiting the completeness of data used in this determination. H atoms were placed at idealized positions and allowed to ride on their parent atoms, with C—H distances of 0.96 Å and U_{iso}(H) values set equal to xU_{eq}(carrier atom), where x = 1.5 for methyl and hydroxyl H atoms and 1.2 for other H atoms. A rotating group refinement was used for the methyl groups.

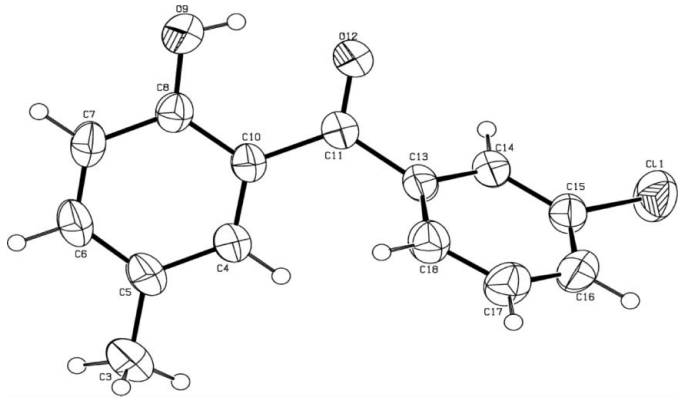


Figure 1
View of (I), shown with 50% probability displacement ellipsoids.

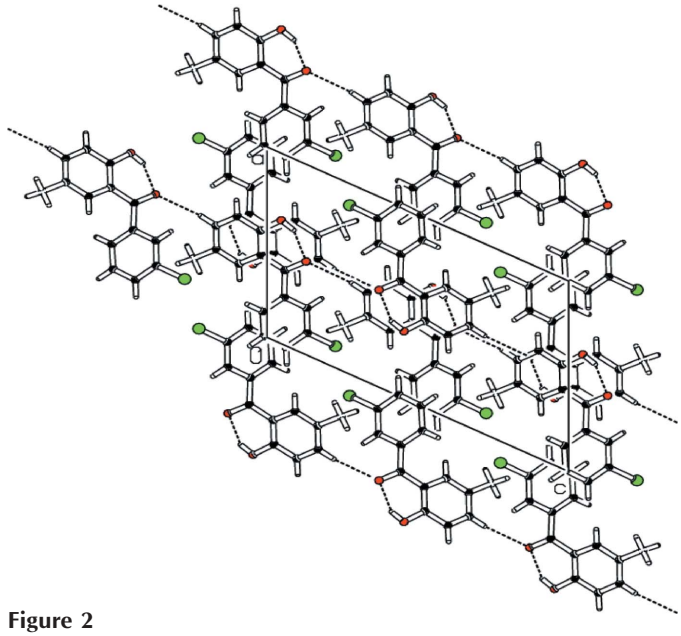


Figure 2
The crystal packing in (I), viewed down the *b* axis. Dashed lines indicate hydrogen bonds.

Data collection: *XPRESS* (MacScience, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski and Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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