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Key indicators

Single-crystal X-ray study

T = 273 K

Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$

R factor = 0.049

wR factor = 0.140

Data-to-parameter ratio = 14.1

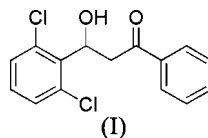
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

3-(2,6-Dichlorophenyl)-3-hydroxy-1-phenylpropan-1-one

In the crystal structure of the title compound, $\text{C}_{15}\text{H}_{12}\text{Cl}_2\text{O}_2$, the chloro-substituted benzene rings are not parallel; the other rings are nearly parallel and the molecules stack in layers parallel to these rings. There are two molecules in the asymmetric unit.

Comment

The structure of the title compound, (I), indicated by ^1H NMR, ^{13}C NMR and IR spectroscopy was confirmed by X-ray diffraction (Fig. 1). There is an active H-atom signal, which disappears after D_2O exchange in ^1H NMR and no $\text{C}=\text{C}$ signal in ^{13}C NMR. In the IR spectrum, a strong hydroxy signal exists. The bond lengths and angles (Table 1) are normal. There are two molecules in the asymmetric unit.



Experimental

The title compound, (I), $\text{C}_{15}\text{H}_{12}\text{Cl}_2\text{O}_2$, was obtained in high yield in the usual manner (Li *et al.*, 2004) from the aldol reaction of 2,6-dichlorobenzaldehyde and acetophenone. Under the same conditions chalcones were the principal products when using 2-chlorobenzaldehyde or 2,4-dichlorobenzaldehyde (House *et al.*, 1973; Mukaiyama *et al.*, 1974; Sakthivel *et al.*, 2001). In a two-necked flask fitted with a magnetic bar and a pressure-equalizing dropping funnel, 10% NaOH (25 ml), acetophenone (6 ml, 0.05 mol) and ethanol (10 ml) were charged. A mixture of 2,6-dichlorobenzaldehyde (8.75 g, 0.05 mol) and ethanol (20 ml) was added dropwise with stirring. After addition and stirring at room temperature for 4 h, the mixture was cooled in an ice-bath to precipitate the product completely. The crude product was isolated and washed to neutral pH. The analytically pure light-green product (14.16 g, yield 96%) was obtained after crystallization from ethanol (95%). Crystals suitable for X-ray analysis were obtained by slow evaporation of an absolute ethanol solution at 293 K (m.p. 374 K). IR (KBr, cm^{-1}): 3494 (s, OH), 3079 (s, H-Ar), 2956 (w, C-H₂), 1673 (s, O=C). ^1H NMR (300 MHz, CDCl_3): δ 7.14–8.00 (m, 8H, H-Ar), 6.14–6.20 (m, 1H, CH), 3.98–4.07 (m, 1H, OH), 3.33 (d, 2H, CH₂). ^{13}C NMR (300 MHz, CDCl_3): δ 198.3, 136.5, 136.3, 134.7, 133.6, 129.5, 129.3, 128.8, 128.3, 68.2, 43.6.

Crystal data

 $\text{C}_{15}\text{H}_{12}\text{Cl}_2\text{O}_2$ $M_r = 295.15$ Monoclinic, $P2_1/c$ $a = 16.2952$ (14) Å $b = 11.5678$ (10) Å $c = 14.6687$ (12) Å $\beta = 102.2300$ (10) $^\circ$ $V = 2702.3$ (4) Å^3

Z = 8

 $D_x = 1.451 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation

Cell parameters from 5943

reflections

 $\theta = 2.3\text{--}27.9^\circ$ $\mu = 0.47 \text{ mm}^{-1}$

T = 273 (2) K

Block, light green

0.54 × 0.44 × 0.34 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 16721 measured reflections
 6171 independent reflections

4449 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = -21 \rightarrow 20$
 $k = -15 \rightarrow 14$
 $l = -19 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.140$
 $S = 1.05$
 6171 reflections
 437 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0651P)^2 + 1.271P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.46 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.41 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0100 (10)

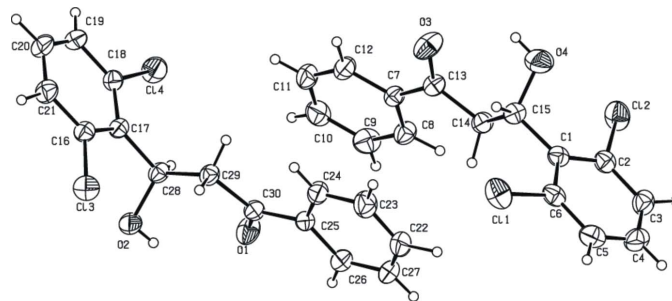


Figure 1 The structure of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level.

Table 1

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

C12—C2	1.740 (2)	C14—C15	1.493 (3)
C11—C6	1.746 (3)	C14—C13	1.500 (3)
C1—C15	1.511 (3)	C13—O3	1.220 (3)
C7—C13	1.481 (3)	C15—O4	1.412 (3)
C6—C1—C15	121.3 (2)	O3—C13—C7	120.2 (2)
C3—C2—C12	116.4 (2)	O3—C13—C14	120.5 (2)
C1—C2—C12	120.68 (19)	C7—C13—C14	119.4 (2)
C1—C6—C11	120.22 (19)	O4—C15—C14	114.3 (2)
C15—C14—C13	114.7 (2)	O4—C15—C1	108.5 (2)
C6—C1—C2—C12	178.83 (16)	C13—C14—C15—O4	62.6 (3)
C12—C7—C13—O3	12.9 (4)	C2—C1—C15—O4	59.5 (3)
C8—C7—C13—O3	-166.0 (3)	C6—C1—C15—O4	-122.3 (2)

Table 2

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O4—H4 \cdots O3	0.82	2.26	2.844 (3)	129
O2—H16 \cdots O1	0.77 (4)	2.41 (4)	2.990 (3)	134 (4)
O4—H4 \cdots Cl4 ⁱ	0.82	2.87	3.437 (2)	128
O2—H16 \cdots O3 ⁱⁱ	0.77 (4)	2.20 (4)	2.833 (3)	141 (4)

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

All H atoms were located in a difference map and refined freely, except atom H4, which was positioned geometrically and treated as riding [O—H = 0.82 \AA and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$].

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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