

H. S. Yathirajan,^a A. N. Mayekar,^a B. Narayana,^b Sarojini^c and Michael Bolte^{d*}^aDepartment of Studies in Chemistry, University of Mysore, Manasagangothri, Mysore 570 006, India, ^bDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, India, ^cDepartment of Chemistry, P. A. College of Engineering, Nadupadavu, Mangalagangothri 574 153, India, and ^dInstitut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Max-von-Laue-Strasse 7, 60438 Frankfurt/Main, GermanyCorrespondence e-mail:
bolte@chemie.uni-frankfurt.de

Key indicators

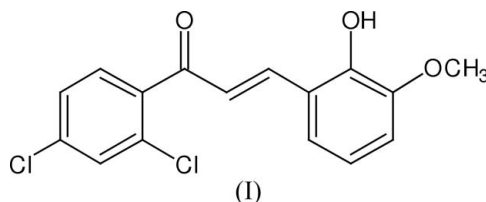
Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.029
 wR factor = 0.081
Data-to-parameter ratio = 16.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**(2E)-1-(2,4-Dichlorophenyl)-3-(2-hydroxy-3-methoxyphenyl)prop-2-en-1-one**

The geometric parameters of the title molecule, $\text{C}_{16}\text{H}_{12}\text{Cl}_2\text{O}_3$, are in the normal ranges. The central double bond is *trans* configured. In the crystal structure, molecules are linked into centrosymmetric $\text{O}-\text{H}\cdots\text{O}$ hydrogen-bonded dimers. In addition, there is a single $\pi-\pi$ stacking interaction between benzene rings of the dichlorophenyl groups.

Received 18 December 2006
Accepted 18 December 2006

Comment

The background to this study is set out in the preceding paper (Yathirajan *et al.*, 2006). In continuation of our work on chalcones, the present paper reports the crystal structure of a newly synthesized chalcone.



The molecular structure of (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Allen *et al.* 1987). The carbonyl group is twisted by -42.28 (17°) from the plane of the dichlorophenyl ring. The torsion angle between the carbonyl group and the C atoms of the $\text{C}=\text{C}$ double bond is -6.3 (2°). The torsion angle between the C atoms of the double bond and the adjacent aromatic ring ($\text{C}2=\text{C}3-\text{C}21-\text{C}26$) is -11.92 (19°). The two benzene rings are not coplanar [dihedral angle = 61.59 (4°)]. The crystal packing (Fig. 2) is characterized by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 1) linking the molecules into centrosymmetric dimers. In addition, a $\pi-\pi$ stacking interaction between two dichlorophenyl rings can be observed (centroid-centroid distance = 3.727 Å; symmetry operator to generate the second molecule: $-\frac{1}{2} + x, y, -\frac{1}{2} - z$).

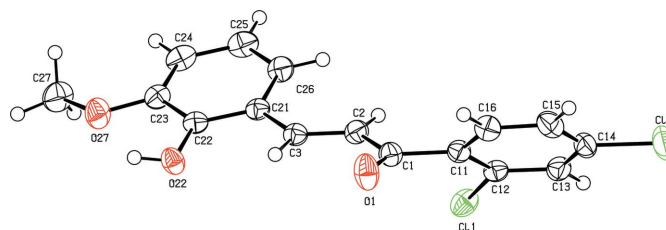


Figure 1
The molecular structure of (I) showing displacement ellipsoids drawn at the 50% probability level.

Experimental

To a stirred solution of 2,4-dichloroacetophenone (1.89 g, 0.01 mol) and 2-hydroxy-3-methoxybenzaldehyde (1.52 g, 0.01 mol) in methanol (25 ml), 40% KOH solution (5 ml) was added. The mixture was stirred overnight, quenched in ice, acidified and filtered. The solid that precipitated was filtered off and washed with water, dried and recrystallized from acetone–toluene (1:1) mixture (m.p. 385–387 K). Analysis for $C_{16}H_{12}Cl_2O_3$ found (calculated): C 59.38 (59.46), H 3.63 (3.74)%.

Crystal data

$C_{16}H_{12}Cl_2O_3$	$Z = 8$
$M_r = 323.16$	$D_x = 1.463 \text{ Mg m}^{-3}$
Orthorhombic, <i>Pbca</i>	Mo $K\alpha$ radiation
$a = 7.1477 (3) \text{ \AA}$	$\mu = 0.45 \text{ mm}^{-1}$
$b = 16.9604 (11) \text{ \AA}$	$T = 173 (2) \text{ K}$
$c = 24.2071 (13) \text{ \AA}$	Block, brown–yellow
$V = 2934.6 (3) \text{ \AA}^3$	$0.35 \times 0.34 \times 0.33 \text{ mm}$

Data collection

Stoe IPDS-II two-circle diffractometer	21745 measured reflections
ω scans	3277 independent reflections
Absorption correction: multi-scan (<i>MULABS</i> ; Spek, 2003; Blessing, 1995)	2843 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.859$, $T_{\max} = 0.866$	$R_{\text{int}} = 0.027$
	$\theta_{\text{max}} = 27.3^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.5024P]$
$R[F^2 > 2\sigma(F^2)] = 0.029$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.081$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
3277 reflections	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
196 parameters	Extinction correction: <i>SHELXL97</i>
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.0047 (5)

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O22-H22\cdots O1^i$	0.86 (2)	1.99 (2)	2.7898 (14)	155 (2)

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

H atoms were found in a difference map, but those bonded to C atoms were refined using a riding model with $C-H = 0.95 \text{ \AA}$ for C_{aromatic} and $C-H = 0.98 \text{ \AA}$ for C_{methyl} . $U_{\text{iso}}(\text{H})$ was set to $1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. The methyl group was allowed to rotate but not to tip. The hydroxyl H atom was freely refined.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve

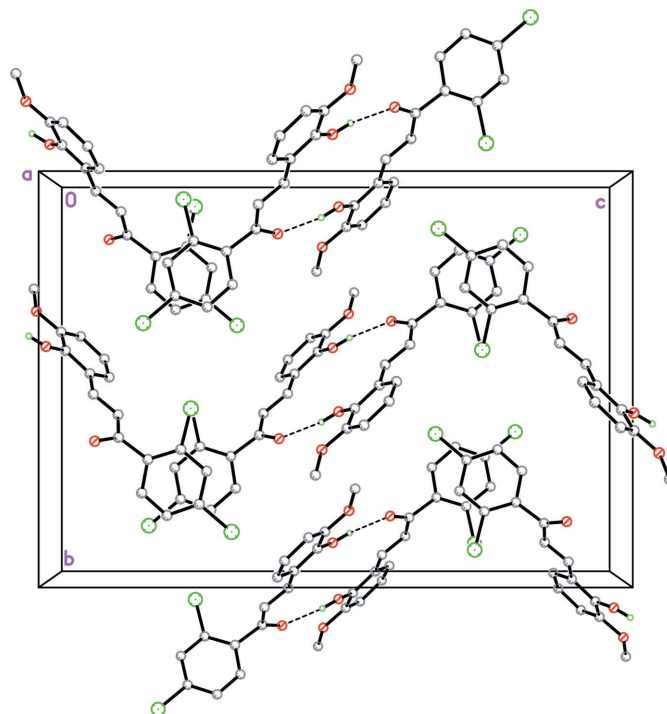


Figure 2

Partial packing diagram with H atoms not involved in hydrogen bonding omitted for clarity. Hydrogen bonds are shown as dashed lines.

structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

ANM thanks the University of Mysore for permission to carry out the research work. BKS thanks AICTE, Government of India, for financial assistance through the Career Award for Young Teachers Scheme.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.
- Yathirajan, H. S., Mayekar, A. N., Sarojini, B. K., Narayana, B. & Bolte, M. (2006). *Acta Cryst.* **E62**, o426–o427.