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## Key indicators

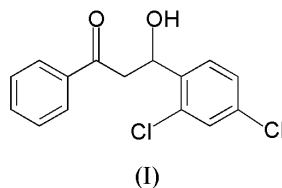
Single-crystal X-ray study  
T = 293 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$   
R factor = 0.042  
wR factor = 0.111  
Data-to-parameter ratio = 16.3For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.3-(2,4-Dichlorophenyl)-3-hydroxy-1-phenyl-  
propan-1-one

In the title compound,  $\text{C}_{15}\text{H}_{12}\text{Cl}_2\text{O}_2$ , the two benzene rings are approximately orthogonal. Two intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds are formed between the hydroxyl and carbonyl groups of two molecules related by a symmetry center.

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## Comment

A new compound, (I), was synthesized by an extension of the Reformatsky reaction (Bieber *et al.*, 1997) in aqueous media (Chan *et al.*, 1994; Shen *et al.*, 1997). A new aqueous system (Bieber *et al.*, 1998; Li, 1996) was designed in the reaction of a 2-bromoketone with an aromatic aldehyde and good results were obtained. It is the first time that the reaction has been carried out in aqueous media in the presence of zinc. An X-ray crystal structure determination of (I) was carried out in order to elucidate the structure and the results are presented here.



The molecule of (I) is shown in Fig. 1. The two benzene rings are approximately orthogonal, forming a dihedral angle of  $79.6(1)^\circ$ . Two molecules related by a centre of symmetry are linked by two intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds (Table 2), resulting in the formation of a dimer. In addition, some weaker hydrogen bonds also exist and the relevant data are listed in Table 2.  $\pi-\pi$  Stacking interactions (Janiak, 2000) are observed between two adjacent chlorine-substituted phenyl rings. The interplanar distance of the two rings is  $3.439(4) \text{ \AA}$  and the centroids of the two rings are offset laterally by  $1.510(4) \text{ \AA}$ . The distance is comparable to that in graphite (Wells, 1975).

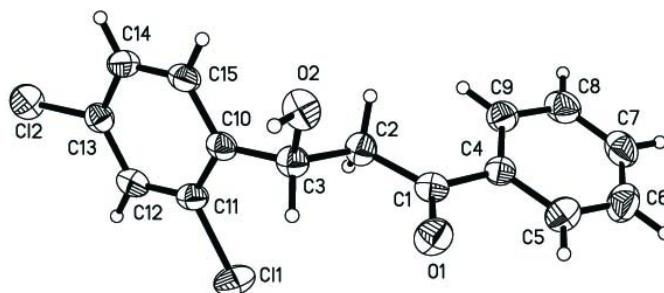


Figure 1

View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

## Experimental

The title compound was synthesized by adding a mixture of 2,4-dichlorobenzaldehyde (3 mmol) and 2-bromoacetophenone (4.5 mmol) to a mixture of a saturated solution of calcium chloride (12 ml), zinc (12 mmol), ammonium chloride (1.5 g), a trace amount of iodine, cetyl trimethylammonium bromide (0.005 g) and THF (1 ml). The mixture was stirred at room temperature for 2 h and extracted with ethyl ether, evaporated and separated by flash chromatography (ethyl acetate–petroleum ether). A colorless powder was obtained (yield 86%) and single crystals suitable for crystallographic analysis were obtained by slow evaporation of an ethyl acetate–petroleum ether solution (m.p. 365–367 K). Spectroscopic analysis, IR (KBr,  $\nu$   $\text{cm}^{-1}$ ): 3512, 1668;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ,  $\delta$ , p.p.m.): 7.96–7.24 (*m*, 8H), 5.60 (*d*, 1H), 3.52 (*d*, 1H), 3.09 (*dd*, 1H). Analysis required for  $\text{C}_{15}\text{H}_{12}\text{Cl}_2\text{O}_2$ : C 61.02, H 4.07%; found: C 61.02, H 4.04%.

### Crystal data

$\text{C}_{15}\text{H}_{12}\text{Cl}_2\text{O}_2$	$D_x = 1.430 \text{ Mg m}^{-3}$
$M_r = 295.15$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 893 reflections
$a = 9.967$ (3) Å	$\theta = 2.7$ – $25.3^\circ$
$b = 15.042$ (4) Å	$\mu = 0.47 \text{ mm}^{-1}$
$c = 10.271$ (3) Å	$T = 293$ (2) K
$\beta = 117.121$ (4) $^\circ$	Prism, colorless
$V = 1370.5$ (7) Å $^3$	$0.26 \times 0.20 \times 0.16 \text{ mm}$
$Z = 4$	

### Data collection

Bruker SMART CCD area-detector diffractometer	2815 independent reflections
$\varphi$ and $\omega$ scans	1935 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.030$
$T_{\text{min}} = 0.820$ , $T_{\text{max}} = 0.928$	$\theta_{\text{max}} = 26.4^\circ$
7777 measured reflections	$h = -10 \rightarrow 12$
	$k = -18 \rightarrow 18$
	$l = -11 \rightarrow 12$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 0.2758P]$
$R[F^2 > 2\sigma(F^2)] = 0.042$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.111$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.17 \text{ e \AA}^{-3}$
2815 reflections	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
173 parameters	
H atoms treated by a mixture of independent and constrained refinement	

**Table 1**

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

C1–C11	1.744 (2)	O1–C1	1.224 (3)
C12–C13	1.740 (2)	O2–C3	1.424 (3)
O2–C3–C2	105.82 (18)	C14–C13–C12	119.49 (18)
C12–C11–C11	117.69 (17)	O1–C1–C4–C9	–173.7 (2)
O1–C1–C2–C3	10.8 (3)	O2–C3–C10–C15	19.9 (3)
C1–C2–C3–O2	78.3 (2)		

**Table 2**

Hydrogen-bonding geometry (Å,  $^\circ$ ).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O2–H2 $\cdots$ O1 $^i$	0.82	2.12	2.915 (2)	162
C3–H3 $\cdots$ C11	0.98	2.74	3.076 (3)	101
C3–H3 $\cdots$ O1	0.98	2.46	2.792 (3)	99
C5–H5 $\cdots$ O1	0.93	2.45	2.763 (3)	99
C15–H15 $\cdots$ O2	0.93	2.46	2.800 (4)	102

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

All H atoms were located from a difference Fourier map and were refined as riding (O–H = 0.82 Å and C–H = 0.93–0.98 Å).

Data collection: SMART (Bruker, 1997); cell refinement: SMART; data reduction: SAINT (Bruker, 1997); program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); 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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## References

- Bieber, L. W., da Silva, M. F., da Costa, R. C. & Silva, L. O. S. (1998). *Tetrahedron Lett.* **39**, 3655–3658.
- Bieber, L. W., Malvestiti, I. & Storch, E. C. (1997). *J. Org. Chem.* **62**, 9061–9064.
- Bruker (1997). SMART, SAINT and SHELXTL. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chan, T. H., Li, C. J. & Lee, M. C. (1994). *Can. J. Chem.* **72**, 1181–1192.
- Janiak, C. (2000). *J. Chem. Soc. Dalton Trans.* pp. 3885–3896.
- Li, C. J. (1996). *Tetrahedron*, **52**, 5643–5668.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). SHELXL97. University of Göttingen, Germany.
- Shen, Z., Zhang, J. Q., Zou, H. X. & Yan, M. M. (1997). *Tetrahedron Lett.* **38**, 2733–2736.
- Wells, A. F. (1975). *Structural Inorganic Chemistry*, 4th ed., p. 734. Oxford University Press.