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3-(2,4-Dichlorophenyl)-1-(2-furyl)-3-hydroxypropan-1-one

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The title compound, $C_{13}H_{10}Cl_2O_3$, was synthesized by the Reformatsky reaction of 2,4-dichlorobenzaldehyde and 2-(bromoacetyl)furan in an aqueous medium. Two intermolecular O—H···O hydrogen bonds are formed between the hydroxy and carbonyl groups of two molecules related by a center of symmetry.

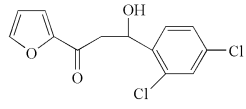
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.111
Data-to-parameter ratio = 13.7

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

Comment

We have recently investigated the Reformatsky reaction with heterocyclic compounds (Chung *et al.*, 2001) in aqueous media (Chan *et al.*, 1994; Li, 1996). A new compound, 3-(2,4-dichlorophenyl)-1-(2-furyl)-3-hydroxypropan-1-one, (I), was synthesized by the reaction of 2,4-dichlorobenzaldehyde and 2-(bromoacetyl)furan (Rho *et al.*, 1997) in an aqueous medium in the presence of zinc (Bieber *et al.*, 1997; Shen *et al.*, 1997). An X-ray crystal structure determination of (I) was carried out to elucidate the structure and the results are presented here.



(I)

The molecular structure is illustrated in Fig. 1. The dihedral angle between the benzene and furan rings is 28.8 (2)°. The angle C10—C9—C8 is 117.6 (2)°, indicating that C9 is sp^2 hybridized. Atoms C6, C7, C8 and C9 are almost coplanar [C6—C7—C8—C9 = −176.37 (19)°]. Two molecules related by a center of symmetry are linked by two intermolecular O—H···O hydrogen bonds (Table 2), resulting in the formation of a dimer. The crystal packing of the title compound is stabilized by O—H···O and weak C—H···O interactions (Fig. 2).

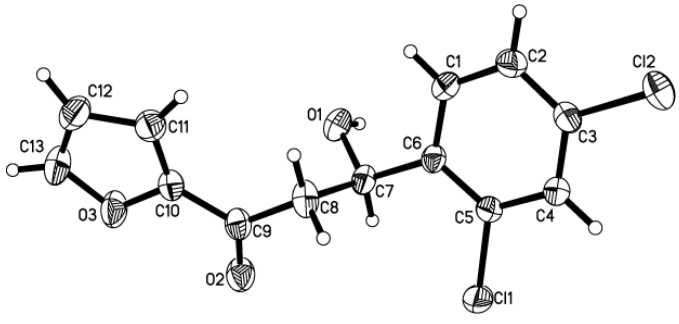


Figure 1
View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Experimental

To a saturated solution of calcium chloride (12 ml), in a round-bottomed flask, was added ammonium chloride (1.5 g). To this stirred mixture zinc powder (12 mmol), a trace amount of iodine, cetyl trimethylammonium bromide (0.005 g) and THF (1 ml) were added. A mixture of 2,4-dichlorobenzaldehyde (3 mmol) and 2-(bromoacetyl)furan (4.5 mmol) was immediately added. The reaction mixture was stirred at room temperature for 6 h and then quenched with 2 *M* HCl. The mixture was extracted with diethyl ether and dried over magnesium sulfate. After removal of the solvent under reduced pressure, the residue was purified by flash chromatography (ethyl acetate–petroleum ether). A colorless powder was obtained (yield 69%) and single crystals suitable for crystallographic analysis were obtained by slow evaporation of an ethyl acetate–petroleum ether solution (m.p. 340–341 K). Spectroscopic analysis: IR (KBr, ν cm^{-1}): 3443, 1651; ^{1}H NMR ($CDCl_3$, δ): 7.64–7.22 (*m*, 5H), 6.55 (*m*, 1H), 5.57 (*d*, 1H), 3.77 (*b*, 1H), 3.38 (*d*, 1H), 2.98 (*dd*, 1H). Analysis required for $C_{13}H_{10}Cl_2O_3$: C 54.74, H 3.51%; found: C 54.75, H 3.52%.

Crystal data

$C_{13}H_{10}Cl_2O_3$
M_r = 285.11
Triclinic, $P\overline{1}$
a = 7.933 (2) Å
b = 8.737 (3) Å
c = 9.907 (3) Å
α = 79.464 (4)°
β = 72.596 (4)°
γ = 79.918 (5)°
V = 638.9 (3) Å^3
Z = 2
D_x = 1.482 Mg m^{-3}
Mo Kα radiation
Cell parameters from 989 reflections
θ = 2.7–26.3°
μ = 0.50 mm^{-1}
T = 293 (2) K
Plate, colorless
0.22 × 0.20 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.882, T_{max} = 0.932
3322 measured reflections
2246 independent reflections
1808 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 25.0°
$h = -8 \rightarrow 9$
$k = -10 \rightarrow 9$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.111
S = 1.07
2246 reflections
164 parameters
H-atoms parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.1932P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C5	1.742 (2)	O1—C7	1.418 (3)
Cl2—C3	1.743 (2)	O2—C9	1.224 (3)
C13—O3—C10	106.5 (2)	O1—C7—C6	111.93 (18)
C2—C3—Cl2	119.98 (17)	O2—C9—C10	121.3 (2)
C6—C5—Cl1	120.29 (16)	O3—C10—C9	117.1 (2)
C5—C6—C7—O1	−149.0 (2)	C7—C8—C9—O2	58.6 (3)
O1—C7—C8—C9	62.0 (3)	O2—C9—C10—C11	178.9 (3)

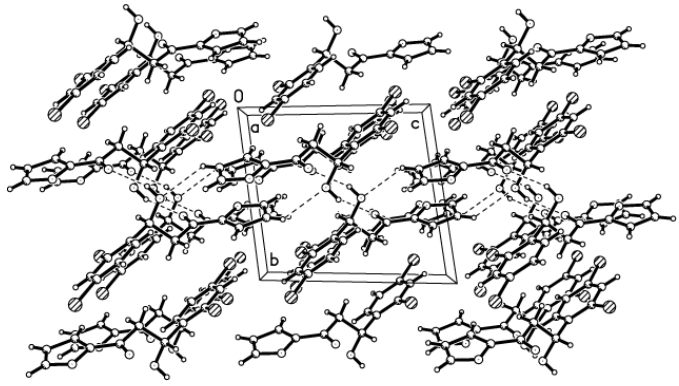


Figure 2
The crystal structure of (I), viewed along the *a* axis. Dashed lines indicate O—H···O and C—H···O interactions.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	2.09	2.839 (3)	152
C7—H7···Cl1	0.98	2.64	3.088 (2)	108
C12—H12···O1^{ii}	0.93	2.57	3.293 (3)	135

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $1-x, 1-y, -z$.

All H atoms were located in a difference Fourier map and were refined as riding (O—H = 0.82 Å and C—H = 0.93–0.98 Å), with U_{iso}(H) = 1.2 U_{eq}(carrier atom).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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