

3-(2-Chlorobenzyl)isocoumarin

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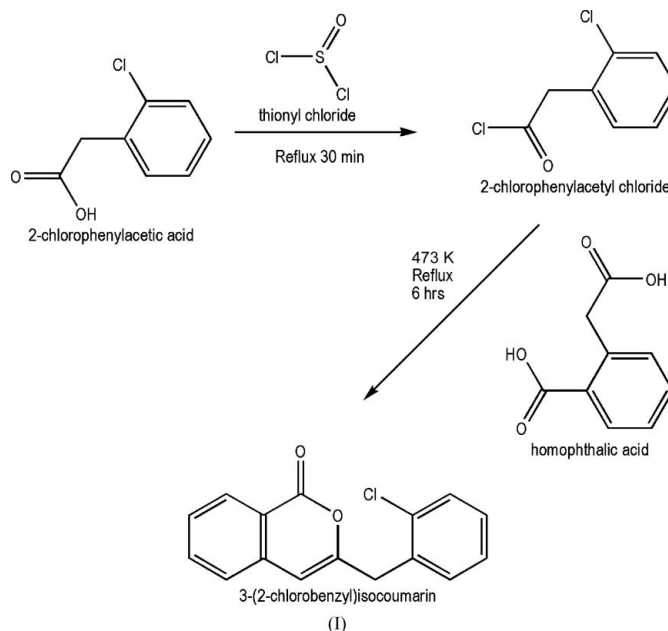
Key indicators

Single-crystal X-ray study
 $T = 273\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.032
 wR factor = 0.100
Data-to-parameter ratio = 18.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.In the title compound, $\text{C}_{16}\text{H}_{11}\text{ClO}_2$, an unnaturally occurring
isocoumarin, the benzene ring is nearly perpendicular to the
planar isocoumarin ring system. The crystal packing is
stabilized by intra- and intermolecular $\text{C}-\text{H}\cdots\text{Cl}$ and $\text{C}-$
 $\text{H}\cdots\text{O}$ hydrogen bonds.

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Comment

The isocoumarin nucleus is an abundant structural motif in
natural products (Barry, 1964). Many constituents of the
steadily growing class of known isocoumarins exhibit valuable
biological properties such as antifungal (Sturtz *et al.*, 2002),
antitumor or cytotoxic, anti-inflammatory, anti-allergic (Rossi
et al., 2003) and enzyme inhibitory activity (Powers *et al.*,
2002). Naturally occurring haloisocoumarins and their halo-
geno-3,4-dihydroisocoumarin derivatives are very rare;
however, a few examples of naturally occurring chlorine-
containing isocoumarins are known (Thomas & Jens, 1999). In
view of the importance of this class of compounds, the title
compound, (I), has been synthesized and its crystal structure is
reported here.In (I), all bond lengths and angles are within normal ranges
(Allen *et al.*, 1987). The isocoumarin ring system is planar, with
a maximum displacement from the mean plane of 0.0252 (12) \AA for C9. The benzene ring is nearly perpendicular
to the isocoumarin ring, the dihedral angle being 88.01 (4) $^\circ$. In
this conformation, an intramolecular $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen

bond is observed (Table 1). The crystal packing is stabilized by intermolecular C—H...O hydrogen-bond interactions (Table 1 and Fig. 2).

Experimental

A mixture of 2-chlorophenylacetic acid (10 g, 58 mmol) and thionyl chloride (5.1 ml, 69 mmol) was heated for 30 min in the presence of a few drops of DMF under reflux at 343 K to give 2-chlorophenylacetyl chloride. Completion of reaction was indicated by the disappearance of gas evolution. Removal of excess thionyl chloride was carried out under reduced pressure to afford 2-chlorophenylacetyl chloride. Homophthalic acid (2.5 g, 13 mmol) was then added and the solution was refluxed for 6 h at 473 K with stirring. The reaction mixture was extracted with ethyl acetate (3 × 100 ml); an aqueous solution of sodium carbonate (5%, 200 ml) was added to remove the unreacted homophthalic acid. The organic layer was separated, concentrated and chromatographed on silica gel using petroleum ether (313–353 K fractions) as eluent to afford the title compound (yield 73%; m.p. 370 K). Colourless single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate solution.

Crystal data

$C_{16}H_{11}ClO_2$	$V = 627.98 (6) \text{ \AA}^3$
$M_r = 270.70$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.432 \text{ Mg m}^{-3}$
$a = 7.2238 (4) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 8.1750 (4) \text{ \AA}$	$\mu = 0.30 \text{ mm}^{-1}$
$c = 10.7958 (6) \text{ \AA}$	$T = 273 (2) \text{ K}$
$\alpha = 83.978 (3)^\circ$	Block, colourless
$\beta = 82.089 (4)^\circ$	$0.25 \times 0.17 \times 0.13 \text{ mm}$
$\gamma = 89.509 (4)^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	3093 independent reflections
φ and ω scans	2390 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{int} = 0.019$
11570 measured reflections	$\theta_{max} = 28.3^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0507P)^2 + 0.0671P]$
$R[F^2 > 2\sigma(F^2)] = 0.033$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.100$	$(\Delta/\sigma)_{max} = 0.010$
$S = 1.08$	$\Delta\rho_{max} = 0.21 \text{ e \AA}^{-3}$
3093 reflections	$\Delta\rho_{min} = -0.15 \text{ e \AA}^{-3}$
172 parameters	
H-atom parameters constrained	

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C10-H10B\cdots Cl1$	0.97	2.59	3.0998 (14)	113
$Cl1-H1\cdots O2^i$	0.93	2.47	3.3333 (16)	155

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

All H atoms were placed in idealized positions and constrained to ride on their parent atoms, with $C-H = 0.93 \text{ \AA}$ and $U_{iso}(H) = 1.2U_{eq}(C)$.

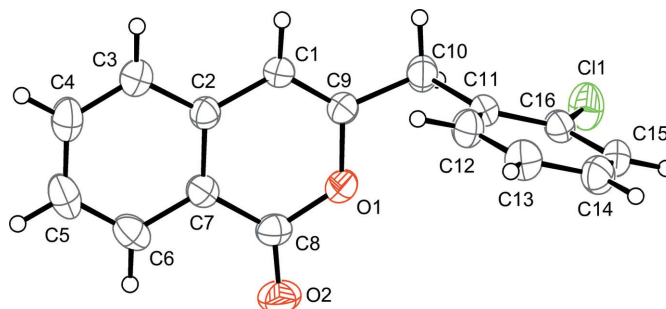


Figure 1

View of the title compound with displacement ellipsoids drawn at the 40% probability level.

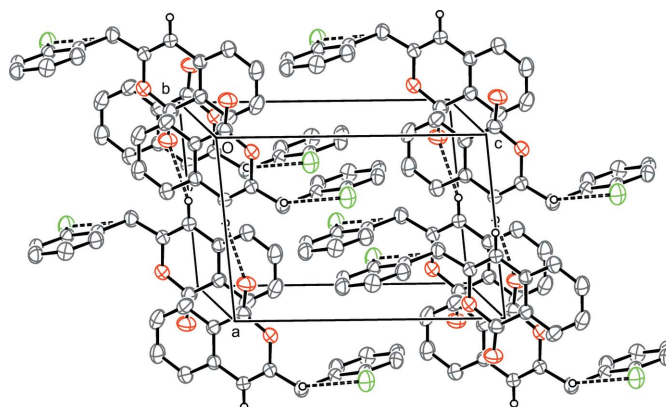


Figure 2

The packing of the title compound with displacement ellipsoids drawn at the 40% probability level. Only H atoms involved in hydrogen bonding (dashed lines) are shown.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1999); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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