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(*R*)-Methyl 2-(2-chlorophenyl)-2-(phenylsulfonyloxy)acetate

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Key indicators

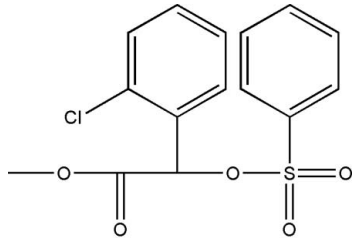
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.097
Data-to-parameter ratio = 13.7

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

The title compound, $C_{15}H_{13}ClO_5S$, has demonstrated itself to be particularly useful as an intermediate, notably for the synthesis of clopidrogel. The dihedral angle between the two aromatic rings is 21.0 (2)°.

Comment

The title compound, (I), was obtained in two stages from (*R*)-2-(2-chlorophenyl)-2-hydroxyacetic acid (Bousquet & Musolino, 2003). This is a practical method widely used in the synthesis of sulfonyloxyacetic esters. We report here its crystal structure.



(I)

The molecular structure of (I) is shown in Fig. 1. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). Rings *A* (C1–C6) and *B* (C10–C15) are, of course, planar and the dihedral angle between them is 21.0 (2)°.

Experimental

A 1000 ml reactor fitted with a double jacket and a valve in the bottom, a mechanical stirrer, a thermometer and a condenser was loaded with (*R*)-2-(2-chlorophenyl)-2-hydroxyacetic acid (120 g, 643 mmol), methanol (480 ml) and sulfuric acid (95%, 4.8 g). The solution was then heated under reflux for 2 h and the excess methanol was eliminated under reduced pressure. The oily residue was taken up in dichloromethane (650 ml) and an aqueous solution of potassium carbonate (10%, 240 g). After decanting, the chlorinated phase was washed with water (200 ml), and then concentrated under reduced pressure. (*R*)-Methyl 2-(2-chlorophenyl)-2-hydroxyacetate was obtained in the form of a colorless oil (yield 124.4 g, 94%). In a dry three-necked round-bottomed flask (100 ml), fitted with a magnetic stirrer, a condenser and a thermometer and operating under an atmosphere of nitrogen, were placed lithium perchlorate (3.81 g, 36 mmol), benzenesulfonyl chloride (30 mmol) and 1,2-dichloroethane (45 ml). Pyridine (36 mmol, 2.9 ml) was added to the solution. The inhomogeneous white reaction medium was stirred for 15 min, before adding (*R*)-methyl 2-(2-chlorophenyl)-2-hydroxyacetate (6 g) dissolved in 1,2-dichloroethane (15 ml). The resulting milky reaction mixture was stirred for 5 h and then poured over a stirred mixture of hydrochloric acid (1 *N*, 1210 ml) and dichloromethane (240 ml).

After decanting, the chlorinated phase was washed with water (120 ml) and then concentrated under reduced pressure. Purification on a silica column gave the product as colorless prisms (yield 90%; m.p. 342 K).

Crystal data

$C_{15}H_{13}ClO_5S$
M_r = 340.76
Monoclinic, $P2_1/c$
a = 7.4015 (16) Å
b = 22.945 (5) Å
c = 9.184 (2) Å
β = 96.145 (4)°
V = 1550.7 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 294 (2) K
0.18 × 0.14 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.931, T_{max} = 0.954
7855 measured reflections
2738 independent reflections
2103 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.098
S = 1.06
2738 reflections
200 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

H atoms were positioned geometrically, with C—H = 0.93 and 0.96 Å for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where x = 1.2 for aromatic and x = 1.5 for methyl H atoms.

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*.

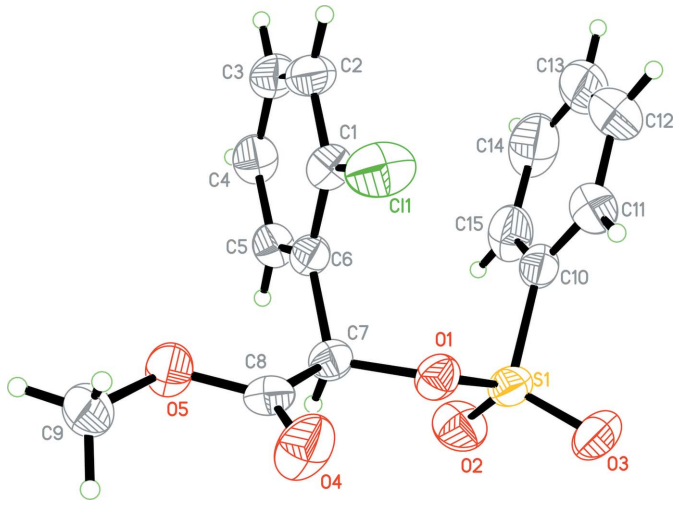


Figure 1
The molecular structure of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

References

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