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3-Phenylsulfonyl-3-(2-propenyl)chroman-4-one

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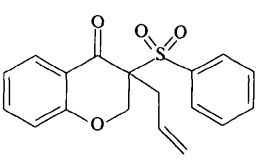
Abstract

In the title compound, $C_{18}H_{16}O_4S$, the pyran ring adopts a sofa conformation. The bond angles around the two planar C atoms in the ring deviate from their ideal values.

Comment

Isoflavonoids, which are built upon a 3-phenylchroman skeleton, represent an important and distinctive subclass of flavonoids. They are found in plants belonging to the subfamily Papilionoidae of the Leguminosae and are known to possess antifungal and antibacterial properties (Dewick, 1988). The title compound, (I), is a key intermediate in the synthesis of isoflavonones.



(I)

The bond angles around the C3 and C9 atoms deviate considerably from the ideal value of 120°. The shortening of the double bond between the C17 and C18 atoms may be due to the large thermal motion of the C18 atom. The dihedral angle between the two phenyl rings is 33.6 (1)°. The pyran ring adopts a sofa conformation, with C1 displaced by 0.532 Å from the mean plane formed by the other atoms in the ring (C2, C3, C8, C9 and O1), instead of the normal half-chair conformation (Alex, Srinivasan, Krishnasamy, Suresh, Iyer & Iyer, 1993). The crystal structure is stabilized by intermolecular van der Waals contacts.

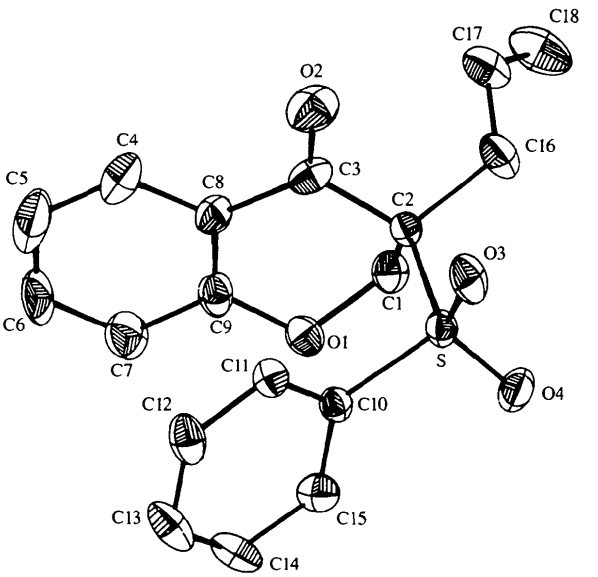


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the molecular structure and atom numbering of (I). The displacement ellipsoids are drawn at the 50% probability level.

Experimental

3-Phenylsulfonylchroman-4-one in DMF was added to sodium hydride also in DMF. To this solution was added an excess of 3-bromopropene and the mixture was kept in an ice bath and stirred for 2 h. The reaction mixture was purified by column chromatography to yield the propenylated sulfone as a yellow solid. The compound was characterized by IR, NMR and mass spectral studies (Santhosh, 1994).

Crystal data

$C_{18}H_{16}O_4S$
M_r = 328.37
Monoclinic
$P2_1/c$
a = 9.890 (3) Å
b = 14.122 (3) Å
c = 11.856 (3) Å
β = 98.04 (2)°
V = 1639.5 (7) Å^3
Z = 4
D_x = 1.330 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 8–14°
μ = 0.214 mm^{-1}
T = 293 (2) K
Cylindrical
0.40 × 0.25 × 0.22 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
2765 measured reflections
2606 independent reflections
2267 reflections with $I > 3\sigma(I)$
$R_{int} = 0.0179$
$\theta_{max} = 24.97°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 16$
$l = -14 \rightarrow 13$
2 standard reflections
every 100 reflections
frequency: 60 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0502$
$wR(F^2) = 0.1337$
$S = 1.088$
2606 reflections
272 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0642P)^2 + 1.1547P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.656$ e Å^{-3}
$\Delta\rho_{min} = -0.188$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S—C10	1.763 (3)	C2—C1	1.519 (4)
S—C2	1.839 (3)	C2—C3	1.518 (4)
O1—C9	1.381 (3)	C9—C8	1.373 (4)
O1—C1	1.408 (4)	C8—C3	1.495 (4)
O2—C3	1.189 (4)	C17—C18	1.282 (6)
C10—S—C2	108.93 (12)	O1—C9—C7	115.1 (3)
C9—O1—C1	114.0 (2)	O2—C3—C8	122.7 (3)
C1—C2—C3	110.7 (2)	O2—C3—C2	122.3 (3)
O1—C1—C2	113.7 (2)	C8—C3—C2	115.0 (2)
C8—C9—O1	124.1 (3)		
C10—S—C2—C16	170.2 (2)	O1—C9—C8—C3	−2.9 (4)
C9—O1—C1—C2	50.9 (3)	C9—C8—C3—C2	−1.4 (4)
C3—C2—C1—O1	−53.5 (4)	S—C2—C3—O2	83.4 (3)
C16—C2—C1—O1	−177.5 (3)	C1—C2—C3—C8	27.7 (3)
C1—O1—C9—C8	−22.3 (4)	C3—C2—C16—C17	−63.0 (4)

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976).

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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: VJ1050). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two Monoclinic Forms of Diclofenac Acid

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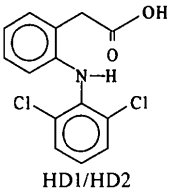
Abstract

Diclofenac acid, [2-(2,6-dichlorophenylamino)phenyl]-acetic acid, $C_{14}H_{11}Cl_2NO_2$, crystallizes in two polymorphic forms in the monoclinic system. In both forms, molecules are linked to each other through the carboxyl groups giving rise to centrosymmetric dimers. No interaction among different dimers has been found.

Comment

The crystal structure determination of diclofenac acid (HD) is part of our structural studies on non-steroidal anti-inflammatory agents (Castellari & Sabatino, 1994, 1996; Castellari & Ottani, 1995, 1996, 1997). In this paper we describe the X-ray crystal and molecular structures of two monoclinic forms of diclofenac acid, namely, HD1 (space group $P2_1/c$) and HD2 (space group $C2/c$). The crystal data of HD2 have been



HD1/HD2