Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(Biphenyl-4-yl)-3-(4-methoxyphenyl)-1,3-thiazolidin-4-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.110
Data-to-parameter ratio = 23.8

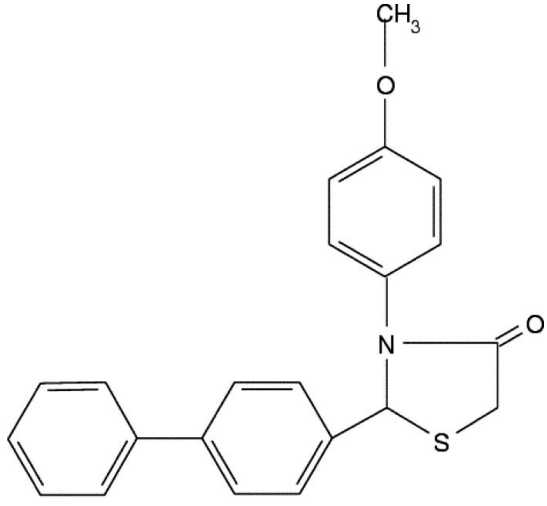
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 6 June 2005
Accepted 22 June 2005
Online 30 June 2005

In the title molecule, $C_{22}H_{19}NO_2S$, the thiazolidinone ring exhibits a flattened envelope conformation. The methoxyphenyl and biphenyl substituents are in pseudo-equatorial and pseudo-axial orientations, respectively, with respect to the thiazolidinone ring.

Comment

The thiazolidin-4-one ring system exists in a number of biologically active compounds which exhibit anticonvulsant (Ragab *et al.*, 1997), hypnotic (Chaudhary *et al.*, 1975), anti-inflammatory (Vigorita *et al.*, 2001), antiproteolytic (Chaudhari *et al.*, 1976) and antituberculous (Babaoglu *et al.*, 2003) properties. The usual conformations of the thiazolidin-4-one ring are envelope or half-chair (Diurno *et al.*, 1992). The structural and conformational features of thiazolidin-4-one derivatives are essential in the study of their structure–activity relationships. As part of our continuing research in the synthesis of nitrogen-containing biologically active heterocyclic compounds (Ravikumar *et al.*, 2003; Basappa *et al.*, 2003), the title compound, (I) (Fig. 1), has been synthesized and we present its crystal structure here.



(I)

The thiazolidinone ring in (I) exhibits a flattened envelope conformation, where atom S14 is displaced by 0.3918 (8) Å from the mean plane of atoms C15/C16/N18/C13. This conformation may be caused by the different steric hindrance of the substituents attached to atoms N18 and C13. These substituents, *viz*. methoxyphenyl and biphenyl, respectively, show pseudo-equatorial and pseudo-axial orientations, respectively, with respect to the thiazolidinone ring. Most of

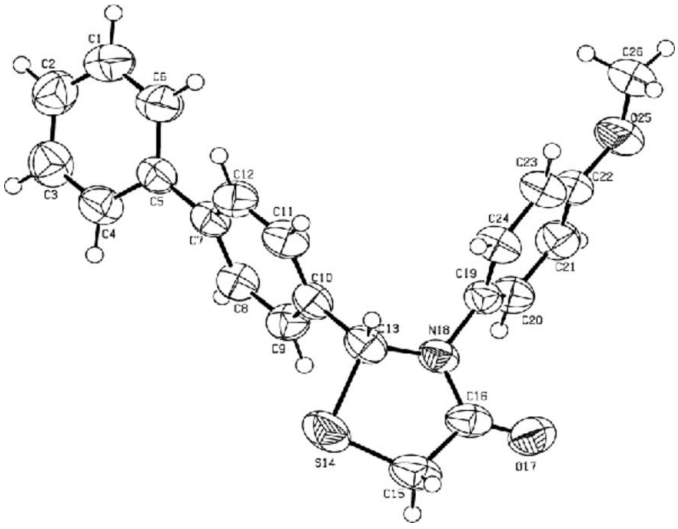


Figure 1
View of (I), with 50% probability displacement ellipsoids.

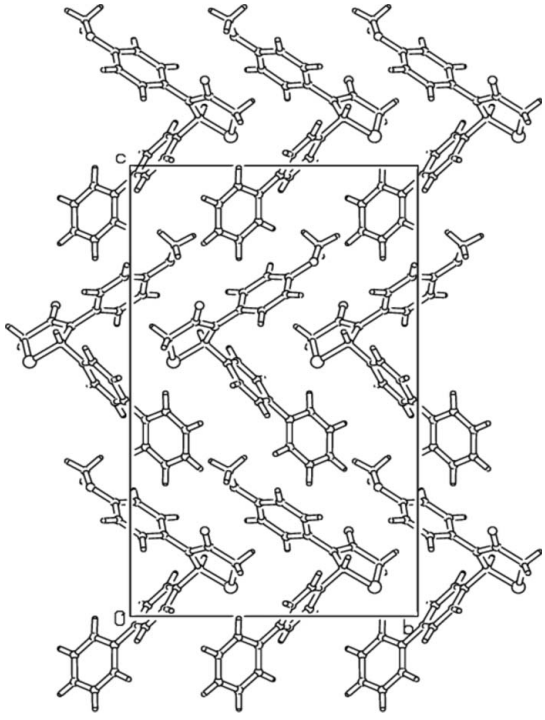


Figure 2
The crystal packing in (I), viewed down the *a* axis.

the bond lengths and angles (Table 1) have normal values. The crystal packing (Fig. 2) is stabilized by van der Waals forces.

A detailed study of the biological activity of (I) is underway.

Experimental

4-Methoxyaniline (5 g, 1 mol), 4-biphenylcarboxaldehyde (7.39 g, 1.0 mol) and anhydrous γ-ferrite (12.96 g, 2 mol) were refluxed with constant stirring in dry benzene for 30 min, after which thioglycolic acid (2.82 ml, 1 mol) was added to the reaction mixture. Reflux and stirring were continued for another 3 h. The reaction was monitored by thin-layer chromatography (R_F = 0.56). After completion of the reaction, a red–brown amorphous solid, $Fe_2O_3{\cdot}2H_2O/FeO(OH)$, was removed by filtration. The filtrate was concentrated to dryness under reduced pressure. The product was confirmed by spectroscopic characterization (yield 78°, m.p. 415–417 K). Anaylsis calculated: C 73.10, H 5.29, N 3.87, S 8.87%; found: C 73.17, H 5.22, N 3.89, S 8.86%. 1 g of (I) was taken up in 15 ml of methanol. Charcoal (1 g) was added and the solution was heated for 2 to 3 min. The hot solution was filtered through a Whatmann 42 filter paper. The solution was kept in a slightly opened conical flask. Crystals were obtained after a few days.

Crystal data

$C_{22}H_{19}NO_2S$
M_r = 361.44
Orthorhombic, $Pbc2_1$
a = 6.287 (5) Å
b = 13.248 (9) Å
c = 22.250 (9) Å
V = 1853.2 (2) Å^3
Z = 4
D_x = 1.295 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5948 reflections
θ = 2.4–32.5°
μ = 0.19 mm^{-1}
T = 293 (2) K
Block, pale yellow
0.35 × 0.2 × 0.2 mm

Data collection

DIPLabo 32001 diffractometer
ω scans
5948 measured reflections
5640 independent reflections
4167 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 32.5°
$h = -9 \rightarrow 9$
$k = -19 \rightarrow 19$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.110
S = 1.09
5640 reflections
237 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0375P)^2 + 0.2616P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0111 (13)
Absolute structure: Flack (1983), with 2218 Friedel pairs
Flack parameter: 0.40 (8)

Table 1
Selected geometric parameters (Å, °).

S14—C13	1.8261 (19)	O25—C26	1.413 (4)
S14—C15	1.778 (3)	N18—C13	1.465 (2)
O17—C16	1.222 (2)	N18—C16	1.355 (2)
O25—C22	1.367 (3)	N18—C19	1.442 (2)
C13—S14—C15	92.75 (10)	S14—C15—C16	108.15 (15)
C22—O25—C26	118.21 (19)	O17—C16—N18	124.48 (17)
C13—N18—C16	117.21 (15)	O17—C16—C15	123.18 (18)
C13—N18—C19	119.77 (14)	N18—C16—C15	112.33 (17)
C16—N18—C19	120.82 (15)	N18—C19—C20	119.71 (16)
S14—C13—N18	105.11 (12)	N18—C19—C24	120.52 (16)
S14—C13—C10	109.94 (14)	O25—C22—C21	115.40 (18)
N18—C13—C10	114.27 (14)	O25—C22—C23	124.62 (19)

The H atoms were placed at idealized positions and allowed to ride at the parent C atoms, with C—H = 0.96 Å and U_{iso}(H) = 1.2U_{eq}(C). The value of the Flack parameter (Flack, 1983) indicates an inversion twin. $Pbc2_1$ is a unconventional setting of $Pca2_1$. Since the transformation to the conventional setting did not yield a better solution, Pbc21 was retained.

Data collection: *XPRESS* (MacScience, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

We thank the DST, Government of India, for financial assistance under project No. SP/I2/FOO/93. MM thanks the CSIR, Government of India, for the award of a Senior Research Fellowship.

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