In the title molecule, C$_{22}$H$_{19}$NO$_2$S, 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 (Chaudhary 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.

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
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 under-

way.

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$_2$O$_3$·2H$_2$O/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). Analysis 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$_2$S

$M_r = 361.44$

Orthorhombic, $Pbc2_1$

$\alpha = 6.287$ (5) Å

$\beta = 13.248$ (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

$\theta = 2.4$–32.5°

$\mu = 0.19$ mm$^{-1}$

$T = 293$ (2) K

Block, pale yellow

0.35 x 0.2 x 0.2 mm

Data collection

DIPlabo 32001 diffractometer

ω scans

5948 measured reflections

5640 independent reflections

4167 reflections with $I > 2\sigma(I)$

$R_{int} = 0.016$

$\Delta\rho_{max} = 0.19$ e Å$^{-3}$

Extinction correction: SHELXL97

Extinction coefficient: 0.011 (15)

Absolute structure: Flack (1983), with 2218 Friedel pairs

Flack parameter: 0.40 (8)

The H atoms were placed at idealized positions and allowed to ride on their parent C atoms, with C–H = 0.96 Å and $U_{eq}(H) = 1.2U_{eq}(C)$. The value of the Flack parameter (Flack, 1983) indicates an inversion twin. Pbc$_2_1$ is a unconventional setting of Pca$_2_1$. Since the transformation to the conventional setting did not yield a better solution, Pbc$_{21}$ was retained.

### Table 1

| 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 | 123.33 (17) |
| C16—N18—C19 | 120.82 (15) | N18—C19—C20 | 119.71 (16) |
| S14—C13—N18 | 105.11 (12) | N18—C19—C20 | 120.52 (16) |
| S14—C13—C10 | 114.27 (14) | O25—C22—C23 | 124.62 (19) |

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.

The H atoms were placed at idealized positions and allowed to ride on their parent C atoms, with C–H = 0.96 Å and $U_{eq}(H) = 1.2U_{eq}(C)$. The value of the Flack parameter (Flack, 1983) indicates an inversion twin. Pbc$_{21}$ is a unconventional setting of Pca$_{21}$. Since the transformation to the conventional setting did not yield a better solution, Pbc$_{21}$ was retained.
Data collection: *XPRESS* (MacScience, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZ0* (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*.

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References


