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# A new heterocylic ring system in (4*SR*,6*SR*)-6-(2-phenylethyl)-4-phenyl-1,4,3-oxathiazin-2(6*H*)-one

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**Key indicators**

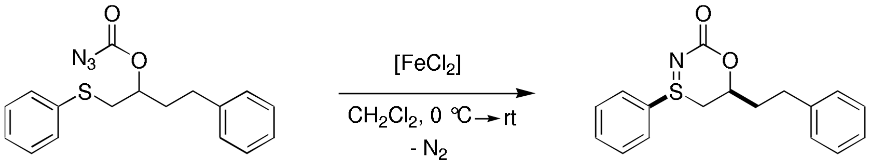
Single-crystal X-ray study
*T* = 213 K
Mean *σ*(C–C) = 0.003 Å
*R* factor = 0.038
*wR* factor = 0.105
Data-to-parameter ratio = 14.5

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

The title compound, $C_{17}H_{17}NO_2S$, is formed by reaction of 2-azidocarbonyloxy-4-phenyl-1-thiophenylbutane with iron(II) chloride as catalyst. This is the first example of the 1,4,3-oxathiazin-2(6*H*)-one ring system.

## Comment

During our studies on iron(II)-catalyzed nitrogen transfer, we developed an intramolecular variant generating cyclic systems (Bach *et al.*, 2000*a*,*b*; Bach & Körber, 1998, 1999, 2000). By treating a thio-substituted alkoxycarbonylazide with catalytic amounts of iron(II) chloride in dichloromethane, the title compound, (I), was precipitated as a mixture of diastereoisomers (55:45). After recrystallization, it could be obtained in diastereoisomerically pure form.



(I)

The interesting 1,4,3-oxathiazin-2(6*H*)-one ring system constitutes a new class of heterocycle. The ring atoms N3, C2 with O2, O1 and C6 are approximately coplanar [maximum deviation from the best plane 0.047 (1) Å]; S4 is 0.559 (1) Å and C5 is 1.030 (2) Å above this plane. The phenyl ring at S4 shows a remarkable rotation towards the N5 atom [torsion angle N3—S4—C7—C12 −24.4 (2)°]. The corresponding 1,4,3-oxathiazin-2(6*H*)-one 4-oxides have been prepared earlier and an X-ray structure analysis was reported (Hwang *et al.*, 1987). Heterocyclic sulfoximines show a wide variety of physiological activities (Kennewell & Taylor, 1980).

## Experimental

Compound (I) was prepared by reaction of 2-azidocarbonyloxy-4-phenyl-1-thiophenylbutane with iron(II) chloride in dichloromethane according to the scheme above. The product was purified by chromatography and then dissolved in dichloromethane/*tert*-butyl methyl ether, from which crystals suitable for X-ray analysis were obtained.

*Crystal data*

$C_{17}H_{17}NO_2S$
$M_r$ = 299.38
Orthorhombic, *Pbca*
*a* = 9.0561 (4) Å
*b* = 11.5330 (5) Å
*c* = 28.0118 (13) Å
*V* = 2925.7 (2) $Å^3$
*Z* = 8
$D_x$ = 1.359 Mg $m^{-3}$

Cu *Kα* radiation
Cell parameters from 25 reflections
*θ* = 18.2–19.9°
*μ* = 1.99 $mm^{-1}$
*T* = 213 (2) K
Square plate, colourless
0.25 × 0.22 × 0.06 mm

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*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega$ scans
Absorption correction: numerical *via* indexed faces (*SHELXTL*; Sheldrick, 1996)
$T_{\min} = 0.636$, $T_{\max} = 0.886$
5578 measured reflections
2779 independent reflections
2222 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.046$
$\theta_{\max} = 70.3°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 14$
$l = -34 \rightarrow 34$
2 standard reflections
frequency: 120 min
intensity decay: 1.9%

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.106$
$S = 1.05$
2779 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.7475P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.26$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.49$ e Å$^{-3}$
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00049 (9)

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

| | | | |
|---|---|---|---|
| O1—C2 | 1.375 (2) | S4—C5 | 1.8002 (19) |
| O1—C6 | 1.446 (2) | C5—C6 | 1.516 (2) |
| C2—O2 | 1.217 (2) | C6—C13 | 1.522 (3) |
| C2—N3 | 1.357 (3) | C13—C14 | 1.532 (3) |
| N3—S4 | 1.6176 (17) | C14—C15 | 1.511 (3) |
| S4—C7 | 1.792 (2) | | |
| C2—O1—C6 | 120.37 (15) | C6—C5—S4 | 104.89 (12) |
| O2—C2—N3 | 123.06 (18) | O1—C6—C5 | 109.15 (14) |
| O2—C2—O1 | 115.86 (18) | O1—C6—C13 | 106.54 (15) |
| N3—C2—O1 | 120.94 (17) | C5—C6—C13 | 111.25 (15) |
| C2—N3—S4 | 122.82 (14) | C8—C7—S4 | 116.91 (15) |
| N3—S4—C7 | 105.65 (9) | C12—C7—S4 | 120.81 (16) |
| N3—S4—C5 | 105.40 (9) | C6—C13—C14 | 113.86 (16) |
| C7—S4—C5 | 100.24 (9) | C15—C14—C13 | 115.52 (17) |
| C6—O1—C2—O2 | −172.27 (17) | C2—O1—C6—C13 | −173.30 (16) |
| C6—O1—C2—N3 | 3.6 (3) | S4—C5—C6—O1 | 65.97 (16) |
| O2—C2—N3—S4 | −159.80 (16) | S4—C5—C6—C13 | −176.75 (13) |
| O1—C2—N3—S4 | 24.6 (3) | N3—S4—C7—C8 | 154.71 (15) |
| C2—N3—S4—C7 | 101.39 (18) | C5—S4—C7—C8 | −95.97 (16) |
| C2—N3—S4—C5 | −4.2 (2) | O1—C6—C13—C14 | −64.6 (2) |
| N3—S4—C5—C6 | −38.78 (14) | C5—C6—C13—C14 | 176.58 (16) |
| C7—S4—C5—C6 | −148.29 (13) | C6—C13—C14—C15 | −67.0 (2) |
| C2—O1—C6—C5 | −53.1 (2) | C13—C14—C15—C16 | −41.3 (3) |

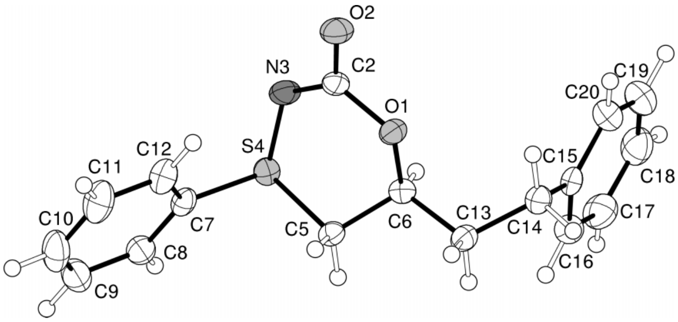

**Figure 1**
*DIAMOND* (Brandenburg, 1999) drawing of a molecule of (I) with the atomic numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1993); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXL*97.

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