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{3-[2-Methoxy-5-(1-methyl-1-phenylethyl)phenyl]-2,5-diphenyl-1,2,3,4-tetrahydro-4-isoxazol-4-yl}-(2-thienyl)methanone

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.038
wR factor = 0.104
Data-to-parameter ratio = 7.5

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

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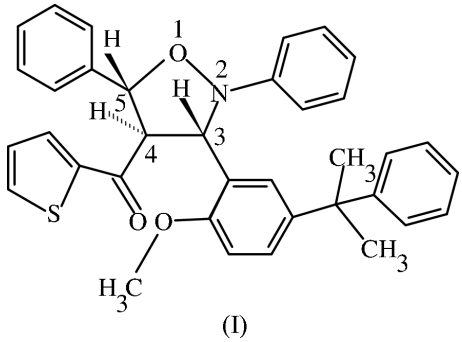
In the crystal structure of the title compound, $C_{36}H_{33}NO_3S$, the five-membered isoxazolidine ring has an envelope conformation, with the O atom as the flap. The three substituents on this ring have all-*trans* stereochemistry.

Comment

The chemistry of isoxazolidines has been extensively reviewed by Lang & Lin (1984). Isoxazolidines obtained from biphenyl nitrones and activated olefins possess some interesting biological activities (Kumar *et al.*, 2003). A number of carbohydrate derivatives of porphyrins, which have been synthesized and tested for photodynamic therapy (PDT) of cancer (Pandey, 2000) readily undergo 1,3-dipolar cycloaddition reactions with sugar nitrones to yield active isoxazolidines (Silva *et al.*, 2002). Sulfur heterocycles have also been shown to exhibit some biological activities (Haxtable, 1986; Colwell *et al.*, 1974; Mizzoni & Eisman, 1958). Recently, we have synthesized a set of substituted isoxazolidines *via* 1,3-dipolar cycloaddition reactions, and their structural features have been investigated with the combined use of NMR and X-ray studies (Sridharan *et al.*, 2004*a*). In continuation of this work, a series of isoxazolidines with sulfur heterocycles have been synthesized (Sridharan *et al.*, 2004*b*). These may exhibit enhanced biological activity due to the presence of two heterocyclic rings.



(I)

Here we report the crystal structure of one of these isoxazolidines, (I). As observed in related systems, the title compound has all-*trans* substitution at atoms C14, C6, C7 (Fig. 1). The five-membered isoxazolidine ring has an envelope conformation, with the O atom as the flap. O2 is displaced by 0.537 (3) Å from the mean plane of the other four atoms. If we denote rings S1–C1/C4, C8/C13, C15/C20, C25/C30 and C31/C36 by *R*1, *R*2, *R*3, *R*4 and *R*5, respectively, the dihedral angles *R*1/*R*2, *R*3/*R*4, *R*3/*R*5 and *R*4/*R*5 are 61.8 (2), 80.5 (2), 73.8 (1) and 38.6 (2)°, respectively. The bond lengths and angles are unexceptional.

Experimental

A mixture of 1.73 g (0.005 mol) of α-(5-(1-methyl-1-phenylethyl)-2-methoxyphenyl-*N*-phenyl nitrone and 1.07 g (0.005 mol) of *E*-3-phenyl-1-(2-thienyl)-2-propen-1-one was refluxed in 50 ml of toluene for 12 h. After completion of the reaction, the solvent was evaporated under reduced pressure and pure (I) was separated through a silica column, using petroleum ether–ethyl acetate as eluent. It was recrystallized from a petroleum ether–ethyl acetate mixture.

Crystal data

$C_{36}H_{33}NO_3S$
M_r = 559.69
Orthorhombic, $Pna2_1$
a = 19.712 (3) Å
b = 9.823 (3) Å
c = 15.805 (2) Å
V = 3060.3 (11) Å^3
Z = 4
D_x = 1.215 Mg m^{-3}
D_m = 1.211 Mg m^{-3}
Density measured by flotation using a mixture of carbon tetrachloride and xylene
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9.7–13.7°
μ = 0.14 mm^{-1}
T = 293 (2) K
Block, colorless
0.30 × 0.20 × 0.15 mm

Data collection

Nonius MACH3 four-circle diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.966, T_{max} = 0.980
3141 measured reflections
2786 independent reflections
1545 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.0°
$h = 0 \rightarrow 23$
$k = -1 \rightarrow 11$
$l = 0 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.104
S = 1.02
2786 reflections
370 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0534P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Absolute structure: Flack (1983), 634 Friedel pairs
Flack parameter = −0.01 (15)

Table 1
Selected torsion angles (°).

C4—C5—C6—C7	125.2 (4)	N1—C14—C15—C16	−12.1 (6)
C6—C7—C13—C8	−89.9 (5)	C32—C31—N1—C14	−141.1 (4)

The H atoms were placed in geometrically calculated positions and included in the refinement in the riding-model approximation, with C—H in the range 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq} of the carrier atom.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, (1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97.

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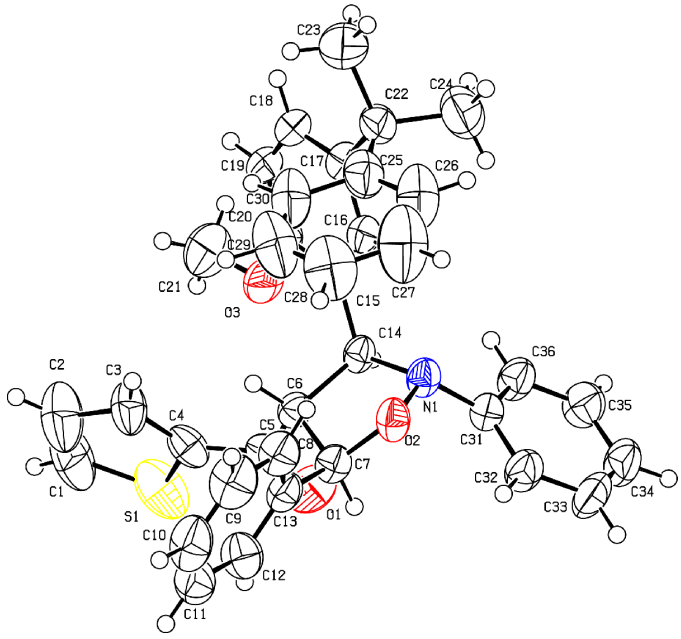

Figure 1
The molecular structure of the title compound, with the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level (Johnson, 1976).

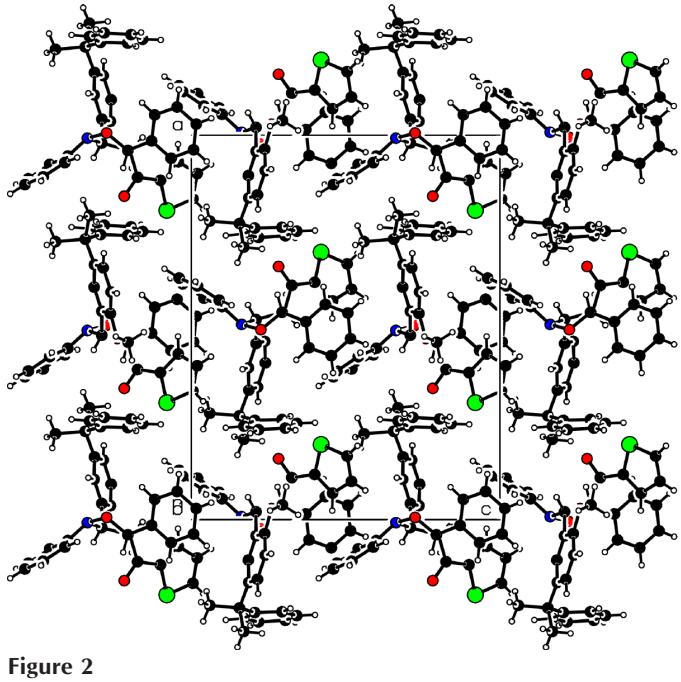

Figure 2
Packing diagram, viewed down the *b* axis.

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