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7-Methyl-9-(4-methylbenzylidene)-1-(4-methylphenyl)-3,4-diphenyl-2-oxa-3,7-diazaspiro[4.5]decan-10-one

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Key indicators

Single-crystal X-ray study
T = 105 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.113
Data-to-parameter ratio = 19.6

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

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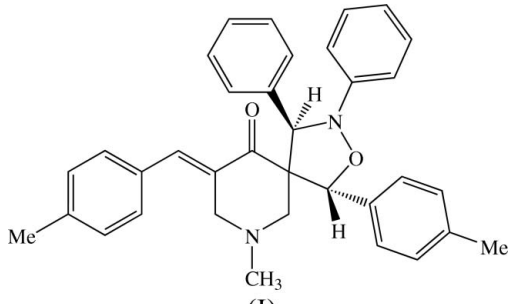
In the title compound, $C_{35}H_{34}N_2O_2$, the isoxazolidine ring adopts a twisted conformation and the piperidine ring is in a half-chair conformation. Weak C—H···π interactions are observed in the crystal structure.

Comment

The 1,3-dipolar cycloaddition of nitrones with olefinic dipolarophiles proceeds through a concerted mechanism yielding highly substituted isoxazolidines (Gothelf & Jorgensen, 1998). Isoxazolidines are potential precursors for biologically important compounds such as amino sugars (Gothelf & Jorgensen, 1998), alkaloids (Goti *et al.*, 1997), β-lactams (Ali *et al.*, 1988) and amino acids (Annuziata *et al.*, 1987), and exhibit antibacterial and antifungal activities (Kumar *et al.*, 2003). Highly substituted spiro-isoxazolidines result from the 1,3-dipolar cycloaddition of exocyclic olefins with nitrones and these spiro-isoxazolidines have also been transformed into complex heterocycles (Colombi *et al.*, 1978). Heterocycles with piperidine sub-structures display important biological activities, such as anticancer (Dimmock *et al.*, 2001) and cytotoxic (El-Subbagh *et al.*, 2000), besides being useful as synthons in the construction of alkaloid natural products (Lee *et al.*, 2001). The above importance of isoxazolidine and piperidine sub-structures led us to investigate the cycloaddition of 1-methyl-3,5-bis[(*E*)-(4-methylphenyl)methylidene]tetrahydro-4(1*H*)-pyridinone with *C*,*N*-diphenylnitrone. The present work reports the X-ray crystallographic study of one such substituted isoxazolidine, the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. The isoxazolidine ring adopts a twisted conformation with a pseudo-twofold axis passing through atom C4 and the midpoint of the C1—O2 bond. The piperidine ring adopts a half-chair conformation with atoms N7 and C6 deviating by −0.452 (2) and 0.346 (2) Å, respectively, from the least-squares plane defined by C5/C8/C9/C10. The dihedral angle

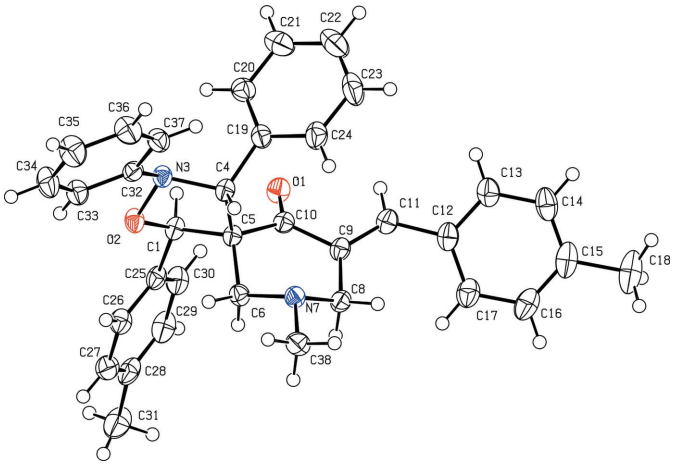


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering scheme.

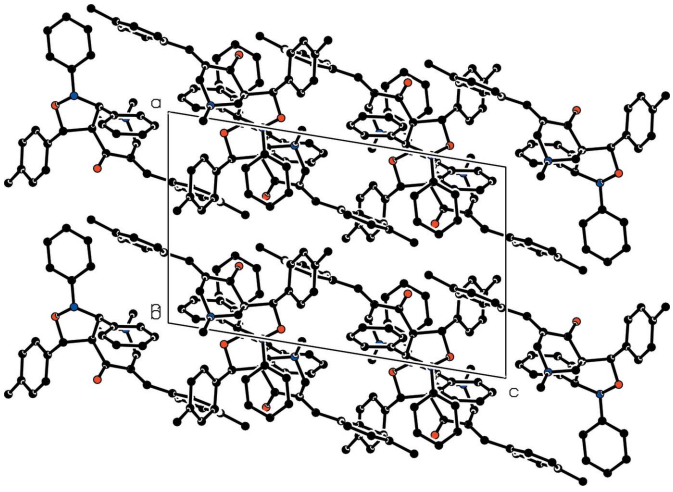


Figure 2
Packing diagram of (I). H atoms have been omitted for clarity.

between the C19–C24 and C32–C37 phenyl rings is 79.12 (6)°. The orientation of the (4-methylphenyl)methylidene substituent with respect to the attached piperidine ring may be influenced by the intramolecular C11—H11···O1 interaction (Table 1). The dihedral angle between the C5/C8/C9/C10 and C12–C17 planes is 48.77 (7)°.

In the crystal structure (Fig. 2), two weak C—H···π interactions, *viz*. C26—H26···*Cg*1^{i} and C38—H38*B*···*Cg*2^{ii} (*Cg*1 and *Cg*2 are the centroids of the rings C19–C24 and C12–C17, respectively; symmetry codes are given in Table 1) are observed. There are no π–π interactions or intermolecular C—H···O interactions.

Experimental

A mixture of 1-methyl-3,5-bis[(*E*)-(4-methylphenyl)methylidene]-tetrahydro-4(1*H*)-pyridinone (1 mol) and nitrone (4 mol) in toluene (15 mol) was refluxed for 10 h. The product was then purified by column chromatography employing petroleum ether–ethyl acetate as eluant and subsequently recrystallized from ethanol (yield 39%, m.p. 447–448 K).

Crystal data

$C_{35}H_{34}N_2O_2$
M_r = 514.64
Monoclinic, $P2_1/c$
a = 11.131 (2) Å
b = 14.206 (3) Å
c = 18.016 (4) Å
β = 99.32 (3)°
V = 2811.2 (10) Å^3
Z = 4
D_x = 1.216 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 105 (2) K
Block, colourless
0.28 × 0.14 × 0.12 mm

Data collection

Bruker SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.987, T_{max} = 0.991
41834 measured reflections
6967 independent reflections
5288 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 28.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.113
S = 1.02
6967 reflections
355 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 1.0249P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11···O1	0.93	2.40	2.7712 (17)	104
C26—H26···*Cg*1^{i}	0.93	2.97	3.7137 (17)	138
C38—H38*B*···*Cg*2^{ii}	0.96	2.95	3.7646 (18)	144

Symmetry codes: (i) $-x, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x, -y+2, -z$.

H atoms were placed in calculated positions (C—H = 0.93–0.98 Å), and allowed to ride on their carrier atoms, with U_{iso}(H) = 1.2U_{eq}(C) for CH_2 and CH groups, and 1.5U_{eq}(C) for CH_3 groups.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); 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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