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Diethyl 3-[2-methoxy-5-(1-methyl-1-phenylethyl)phenyl]-2-phenyl-2,3,4,5-tetrahydroisoxazole-4,5-dicarboxylate

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Key indicators

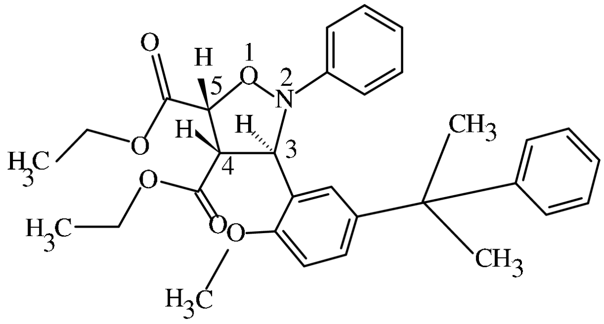
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.045
wR factor = 0.140
Data-to-parameter ratio = 13.8

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

The title compound, $C_{31}H_{35}NO_6$, has an envelope conformation for the central isoxazolidine ring, with one of the C atoms out of the plane of the other four atoms. The ester groups are *cis* to each other, while the C-aryl group is *trans* to the ester group in the isoxazolidine ring.

Comment

Recently, a set of new 2,3,4,5-tetrasubstituted isoxazolidines has been synthesized in our laboratory and their structural features have been investigated by NMR and X-ray analysis (Sridharan, Muthusubramanian *et al.*, 2004). Most of these isoxazolidines have all-*trans* stereochemistry around adjacent ring atoms. It has been planned to prepare analogous compounds with *cis* stereochemistry around atoms C8 and C15. With this view, 1,3-dipolar cycloaddition reactions of several α-(5-substituted-2-methoxyphenyl)-*N*-phenylnitrones with diethyl maleate have been investigated (Sridharan, Kalanidhi & Muthusubramanian, 2004). Diethyl maleate is a dipolarophile with considerable activity, though its reactivity has been limited when compared with its *trans* isomer (Padwa, 1984).



(I)

The title compound, (I), has been obtained as one of the products in the above process and its stereochemistry has been analyzed in this study. As expected, the substituents at C15 and C8 of the isoxazolidine ring are *cis* to each other, while those at C16 and C15 are *trans* to each other, confirming that the reaction is a concerted one. The torsion angles around C15—C8 and around C16—C15 clearly show that the H atoms attached at C15 and C8 are closer to each other than those attached at C16 and C15. The isoxazolidine ring adopts an envelope conformation, with atom C8 deviating by 0.2 (9) Å from the mean plane through the other four atoms. A mixture of α-(5-(1-methyl-1-phenylethyl)-2-methoxyphenyl-*N*-phenylnitrone (1.73 g, 0.005 mol) and diethyl maleate (0.86 g, 0.005 mol) was refluxed in toluene (50 ml) for 6 h. After

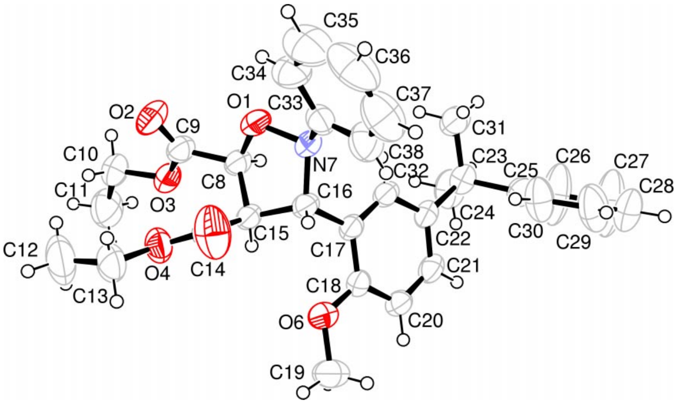


Figure 1
The molecular structure of the title compound with the atom-numbering scheme and 50% probability displacement ellipsoids (*ORTEP*II; Johnson, 1976).

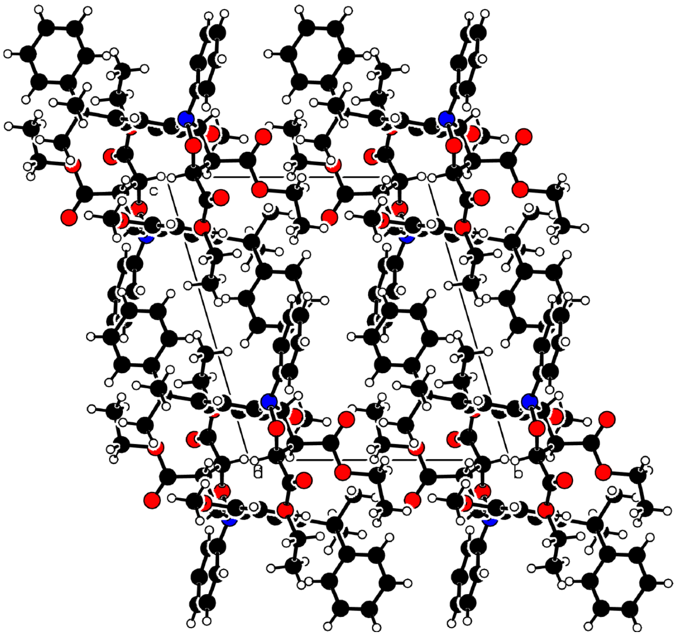


Figure 2
Packing diagram of the title molecule, viewed down the *b* axis.

completion of the reaction, the solvent was evaporated under reduced pressure and pure product (I) was obtained by recrystallization from a petroleum ether–ethyl acetate mixture.

Crystal data

$C_{31}H_{35}NO_6$
$M_r = 517.60$
Triclinic, $P\overline{1}$
$a = 10.8029$ (14) Å
$b = 10.9431$ (9) Å
$c = 12.376$ (2) Å
$\alpha = 105.92$ (1)°
$\beta = 93.838$ (12)°
$\gamma = 94.804$ (9)°
$V = 1395.8$ (3) Å^3
$Z = 2$
$D_x = 1.232$ Mg m^{-3}
$D_m = 1.230$ Mg m^{-3}
D_m measured by flotation in a mixture of carbon tetracholride and xylene
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 9.8$–$14.0°$
$\mu = 0.09$ 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)
$T_{\min} = 0.980$, $T_{\max} = 0.987$
5169 measured reflections
4725 independent reflections
2607 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.015$
$\theta_{\max} = 25.0°$
$h = -12 \rightarrow 10$
$k = -13 \rightarrow 12$
$l = -1 \rightarrow 14$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.140$
$S = 1.00$
4725 reflections
343 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.057P)^2 + 0.4626P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.15$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}

Table 1
Selected torsion angles (°).

C8—O1—N7—C33	−156.2 (2)	O5—C14—C15—C16	3.7 (4)
C8—O1—N7—C16	−28.4 (2)	O4—C14—C15—C16	−175.12 (19)
N7—O1—C8—C9	168.63 (19)	O5—C14—C15—C8	−109.0 (3)
N7—O1—C8—C15	43.6 (2)	O4—C14—C15—C8	72.2 (3)
O1—C8—C9—O2	−12.2 (4)	C9—C8—C15—C14	−40.4 (3)
C15—C8—C9—O2	103.7 (3)	O1—C8—C15—C16	−41.4 (2)
O1—C8—C9—O3	167.8 (2)	C9—C8—C15—C16	−159.6 (2)
C15—C8—C9—O3	−76.3 (3)	O1—N7—C16—C15	1.3 (2)

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

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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References

Enraf–Nonius (1994). *CAD*-4 *EXPRESS*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD*4. University of Marburg, Germany.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.*. **36**, 7–13.
Padwa, A. (1984). In *1,3-Dipolar Cycloaddition Chemistry*, Vols. 1–2. New York: Wiley-Interscience.
Sridharan, V., Muthusubramanian, S., Sivasubramanian, S. & Polborn, K. (2004). *Tetrahedron*, **60**, 8881–8892.
Sridharan, V., Kalanidhi, P. & Muthusubramanian, S. (2004). Unpublished results.

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