2786 measured reflections
2605 independent reflections
3 standard reflections
frequency: 30 min
intensity decay: 0.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.075$
$S = 1.122$
2605 reflections
143 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0534P)^2 + 0.2809P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.286$ e Å^{-3}
$\Delta\rho_{\min} = -1.252$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

Te—C1	2.020 (3)	Te—Cl1	2.551 (1)
Te—C2	2.146 (3)	C1—C3	1.196 (4)
Te—Cl2	2.486 (1)		
C1—Te—C2	97.66 (13)	C1—Te—Cl1	87.84 (10)
C1—Te—Cl2	89.94 (10)	C2—Te—Cl1	87.98 (9)
C2—Te—Cl2	86.59 (9)	Cl2—Te—Cl1	173.81 (3)

H atoms were refined with fixed geometry, each riding on a carrier atom, with an isotropic displacement parameter equal to 1.5 (for methyl-H atoms) or 1.2 (for the other H atoms) times that of the carrier atom. The highest peak and the deepest hole in the final difference Fourier map were at 0.95 and 0.94 Å from Te, respectively.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai, 1995). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GS1025). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 650–652

Methyl (2*S*)-2-[(5*R*)-3,5-diphenyl-4,5-dihydro-1,2,4-oxadiazol-4-yl]-3-phenylpropanoate

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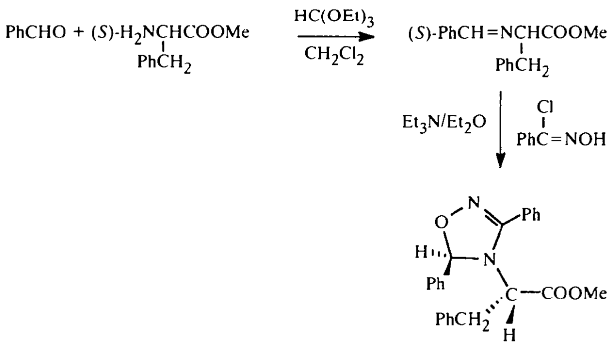
Abstract

In the title compound, $C_{24}H_{22}N_2O_3$, the 4,5-dihydro-1,2,4-oxadiazole ring system is in an envelope conformation with the C1, N1, C8 and N2 sets of atoms in a common plane with O1 0.110 (3) Å out of this plane. The chiral C atom in the five-membered ring, C1, is in the *R* configuration, which was identified by referring to the known configuration of the atom C15 in the (*S*)-phenylalanine moiety.

Comment

4,5-Dihydro-1,2,4-oxadiazole derivatives are important pharmaceutical agents with antiviral (Franco *et al.*, 1964), antifungal (Manrao & Kanta, 1995), and nematocidal and insecticidal activities (Sousa *et al.*, 1965). They have also been used as antimalarial drugs (Carrington, 1955) and potential chemoprophylactic agents of schistosomiasis (Hu *et al.*, 1997). Recently, it has been found that they are potential central nervous system depressants (Xu *et al.*, 1998), muscarinic agonists as insecticides and acaricides (Dick *et al.*, 1997), and muscarinic receptor 'super agonists' for treatment of Alzheimer's disease (Macor *et al.*, 1996).

An important method of synthesis of 4,5-dihydro-1,2,4-oxadiazole derivatives is the cycloaddition reaction of a Schiff base and nitrile oxide. Numerous 4,5-dihydro-1,2,4-oxadiazole derivatives can be synthesized from amino acid esters, aldehydes and ketones, and nitrile oxides as building blocks by the combinatorial chemistry method for bio-assay. The title compound, (I), as a model, has been synthesized from *N*-benzylidene-(*S*)-phenylalanine methyl ester, which was generated from benzaldehyde and methyl (*S*)-phenylalanine, and benzonitrile oxide. In order to identify the configuration of the chiral carbon atom C1 formed in the cycloaddition, the title compound was subjected to X-ray diffraction analysis.



(I)

The dihydro 1,2,4-oxadiazole ring adopts an envelope conformation with the C1, N1, C8 and N2 atoms in a plane with O1 0.110 (3) Å above the plane. The value of the torsion angle C1—N1—C8—N2 is −1.4 (3)°. The C8—N1 and C8—C9 bond lengths (Table 1) are both shorter than standard values (*cf.* Csp^2—Nsp^3 of 1.416 Å, and Csp^2—C*ar* of 1.485 Å; *International Tables for Crystallography*, Vol. C). This indicates that there is some delocalization of the N1 lone pair into these bonds, and implies the presence of a non-planar conjugated system between the C1, N2, C8 and N1 atoms of the oxadiazole ring and the C9 phenyl ring [dihedral angle 36.3 (3)°].

The configuration of the atom C1 was identified as *R* by referring to the known *S* configuration of the atom C15, which is the known configuration in the natural (*S*)-phenylalanine starting material.

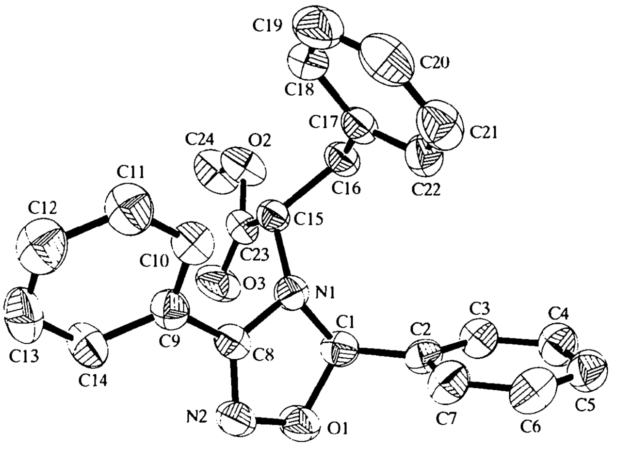


Fig. 1. Perspective view (Sheldrick, 1995) of (I) with the atom labelling. Displacement ellipsoids are shown at the 30% probability level.

Experimental

To a stirred solution of the *N*-benzylidene-(*S*)-phenylalanine methyl ester (1.07 g, 4 mmol) and the triethylamine (0.70 ml, 5 mmol) in anhydrous diethyl ether (25 ml), a solution of α-chlorobenzaldehyde oxime (0.62 g, 5 mmol) in anhydrous diethyl ether (5 ml) was slowly added dropwise. The reaction mixture was then stirred overnight at room temperature, washed twice with water and dried with anhydrous $MgSO_4$. The solvent was then evaporated at reduced pressure to obtain a brown oil. This material was recrystallized from diethyl ether to yield white crystals of the title compound (0.62g with yield 40%, m.p. 376–377 K). Elemental analysis for $C_{24}H_{22}N_2O_3$ (386.44): calculated C 74.58, H 5.74, N 7.25%; found C 74.62, H 5.81, N 7.00%. The colourless single crystal used for analysis was obtained by evaporation from a saturated diethyl ether solution of the compound.

Crystal data

$C_{24}H_{22}N_2O_3$
M_r = 386.44
Orthorhombic
$P2_12_12_1$
a = 14.671 (4) Å
b = 15.135 (2) Å
c = 9.328 (2) Å
V = 2071.2 (8) Å^3
Z = 4
D_x = 1.239 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 136 reflections
θ = 2.0–25.0°
μ = 0.082 mm^{-1}
T = 293 (2) K
Prism
0.3 × 0.2 × 0.2 mm
Colourless

Data collection

Rigaku R-AXIS II*C* IP diffractometer
Oscillation IP photos
Absorption correction: none
5627 measured reflections
3262 independent reflections
2720 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.039
$\theta_{\rm max}$ = 25.67°
$h = 0 \rightarrow 17$
$k = -18 \rightarrow 17$
$l = -11 \rightarrow 11$
3 standard reflections every 100 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.123$
$S = 1.096$
3262 reflections
263 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0499P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.112$ e Å^{-3}
$\Delta\rho_{\min} = -0.107$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997*a*)
Extinction coefficient: 0.024 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—N2	1.431 (3)	N1—C1	1.473 (3)
O1—C1	1.439 (4)	N2—C8	1.287 (3)
O2—C23	1.321 (4)	C1—C2	1.491 (4)
O2—C24	1.450 (4)	C8—C9	1.470 (4)
O3—C23	1.187 (3)	C15—C23	1.513 (4)
N1—C8	1.389 (4)	C15—C16	1.535 (4)
N1—C15	1.455 (3)	C16—C17	1.507 (4)
N2—O1—C1	109.71 (19)	N2—C8—N1	115.6 (2)
C23—O2—C24	117.3 (3)	N1—C15—C23	111.5 (2)
C8—N1—C1	105.2 (2)	N1—C15—C16	111.9 (2)
C8—N2—O1	105.8 (2)	C17—C16—C15	111.5 (2)
O1—C1—N1	103.1 (2)	O3—C23—O2	122.4 (3)
O1—C1—C2	110.8 (2)	O3—C23—C15	125.2 (3)
N1—C1—C2	114.0 (2)	O2—C23—C15	112.4 (2)

The data were collected using radiation from a rotating-anode generator operating at 50 kV and 90 mA by taking 23 oscillation photos in the range of 0–184° with exposing time of 10 min per frame. Crystal-to-detector distance was 78.1 mm. The structure was solved by direct methods. All the non-H atoms were refined anisotropically. All H atoms were included with the riding model.

Data collection: R-AXIS II*C* diffractometer software (Rigaku, 1995). Cell refinement: R-AXIS II*C* diffractometer software. Data reduction: R-AXIS II*C* data processing software. Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*b*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*). Molecular graphics: *SHELXTL* (Sheldrick, 1995). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1227). Services for accessing these data are described at the back of the journal.

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3,5-Di-*tert*-butyl-1,2-benzoquinone

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Abstract

The molecular structure of the title compound, $C_{14}H_{20}O_2$, (I), has been investigated using X-ray diffraction. When compared with the semiquinone (DTBSQ) molecule in a coordinated state, *e.g.* [Cu{NH(py)$_2$}-(DTBSQ)]$ClO_4\cdot0.5C_4H_8O$ [(II); Thompson & Calabrese (1985). *Inorg. Chem.* **24**, 3167–3171], the different C—O distances [1.214 (3) and 1.217 (3) Å in (I); 1.293 (7) and 1.304 (7) Å in (II)] reveal information about the electron distributions of the carbonyl group in different environments.

Comment

When we studied the auto-oxidation of 3,5-di-*tert*-butylcatechol (DTBC) by copper complexes (Chyn & Urbach, 1991), it was revealed that the intermediates have various possible structural states, and that the oxidated compound exists as a moiety of 3,5-di-*tert*-butyl-1,2-benzoquinone (DTBQ) in copper complexes. To investigate the coordinating character of the carbonyl groups, the crystal structure of DTBQ, (I), has been established. Comparison with the cation [Cu{NH(py)$_2$}-