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2-(3-Phenyl-1,2,4-oxadiazol-5-yl)-4*H*-1-benzopyran-3-ol

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.176
Data-to-parameter ratio = 12.9

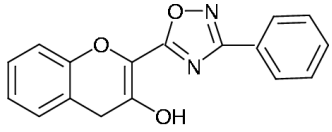
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{17}H_{12}N_2O_3$, was synthesized by the reaction of methyl {2-[(3-phenyl-1,2,4-oxadiazol-5-yl)-methoxy]phenyl}acetate and sodium hydride. The molecule adopts the enol form, stabilized by an intramolecular O—H···N hydrogen bond, which gives rise to a hydrogen-bonded six-membered pseudo-ring. All non-H atoms are coplanar within 0.13 Å.

Comment

1,2,4-Oxadiazole derivatives are of great interest because of their biological properties. Some derivatives of 1,2,4-oxadiazole have intrinsic analgesic (Terashita *et al.*, 2002), anti-inflammatory (Nicolaides *et al.*, 1998) and antipicornaviral (Romero, 2001) properties, and exhibit high efficacy as agonists [*e.g.* for muscarinic (Macor *et al.*, 1996) and adrenergic (Quagliato & Andrae, 2002)] and antagonists [*e.g.* for angiotensin (Naka & Kubo, 1999) and adhesion (Juraszyk *et al.*, 1997)] for different receptors.

The title compound, (I), was synthesized by the reaction of methyl {2-[(3-phenyl-1,2,4-oxadiazol-5-yl)methoxy]phenyl}-acetate (Wang *et al.*, 2004) and sodium hydride. The molecular structure of (I) is shown in Fig. 1. Selected bond lengths and angles are listed in Table 1.



(I)

The structural study of compound (I) confirmed that the molecule adopts the enol form, stabilized by a relatively strong intramolecular O2—H2*A*···N2 bond [O2—H2*A* = 0.87 (4) Å, H2*A*···N2 = 2.01 (4) Å, O2···N2 = 2.736 (3) Å and O2—H2*A*···N2 = 141 (3)°]. As a result, the C9—C17 bond has a length of 1.347 (3) Å, typical for an olefinic bond, and all atoms of the molecule, with the exception of H16*A* and H16*B*, are coplanar within 0.13 Å.

Experimental

Methyl {2-[(3-phenyl-1,2,4-oxadiazol-5-yl)methoxy]phenyl}acetate (20 mmol), synthesized according to the method of Wang *et al.* (2004), was dissolved in dimethylformamide (DMF, 20 ml) and added dropwise at 278 K to a suspension of sodium hydride (20 mmol) in DMF (10 ml). The mixture was stirred overnight at room temperature and then poured into cold dilute HCl to precipitate the title compound. Pure (I) was obtained by recrystallization from ethyl acetate. Crystals of (I) (m.p. 458–459 K), suitable for X-ray diffraction, were obtained by slow evaporation of an ethanol solution. ^{1}H

NMR ($CDCl_3$): δ 10.09 (*m*, 1H), 8.11–8.13 (*m*, 2H), 7.53–7.58 (*m*, 3H), 7.07–7.28 (*m*, 4H), 3.89 (*s*, 2H).

Crystal data

$C_{17}H_{12}N_2O_3$
M_r = 292.29
Monoclinic, $P2_1/n$
a = 5.675 (1) Å
b = 9.346 (2) Å
c = 25.440 (5) Å
β = 92.06 (3)°
V = 1348.4 (5) Å^3
Z = 4
D_x = 1.440 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–13°
μ = 0.10 mm^{-1}
T = 293 (2) K
Block, yellow
0.4 × 0.4 × 0.3 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.960, T_{max} = 0.970
2902 measured reflections
2625 independent reflections
1885 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 26.0°
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 11$
$l = -31 \rightarrow 31$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.176
S = 1.05
2625 reflections
203 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.56P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—N1	1.420 (3)	N2—C7	1.384 (3)
O1—C8	1.337 (3)	N2—C8	1.302 (3)
O2—C17	1.344 (3)	C5—C7	1.473 (3)
O2—H2*A*	0.87 (4)	C8—C9	1.437 (3)
O3—C9	1.385 (3)	C9—C17	1.347 (3)
O3—C10	1.378 (3)	C15—C16	1.509 (3)
N1—C7	1.298 (3)	C16—C17	1.484 (3)
C8—O1—N1	105.56 (17)	N2—C8—C9	126.3 (2)
C17—O2—H2*A*	112 (2)	C17—C9—O3	124.4 (2)
C10—O3—C9	117.56 (18)	C17—C9—C8	122.5 (2)
C7—N1—O1	104.01 (18)	O3—C10—C15	122.6 (2)
C8—N2—C7	102.33 (19)	C10—C15—C16	121.2 (2)
C4—C5—C7	120.8 (2)	C17—C16—C15	111.73 (19)
N1—C7—N2	114.2 (2)	O2—C17—C9	124.1 (2)
N1—C7—C5	122.6 (2)	O2—C17—C16	113.9 (2)
N2—C8—O1	113.9 (2)	C9—C17—C16	122.1 (2)

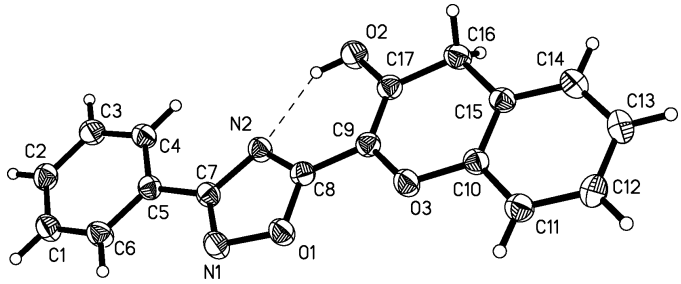


Figure 1
A view of the molecular structure of (I). The dashed line indicates the O—H···N hydrogen bond. Displacement ellipsoids are drawn at the 30% probability level.

All H atoms bonded to C atoms were placed geometrically at distances of 0.93–0.97 Å and included in the refinement in the riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(carrier atom). Hydroxy atom H2*A*, which participates in the intramolecular hydrogen bond, was located in a difference map and refined isotropically.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; 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: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXL*97.

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