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Methyl 3-dimethylamino-2-{2-[2-(dimethylamino)-1-(3-phenyl-1,2,4-oxadiazol-5-yl)-vinyloxy]phenyl}acrylate

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Key indicators

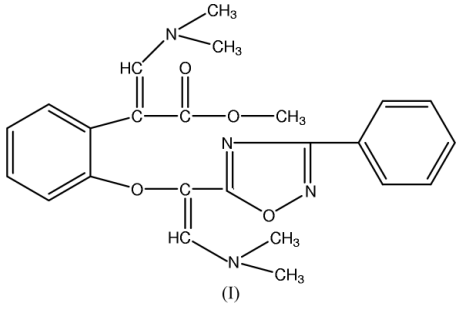
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.073
wR factor = 0.259
Data-to-parameter ratio = 15.6

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

The title compound, $C_{24}H_{26}N_4O_4$, was synthesized by the reaction of methyl {2-[(3-phenyl-1,2,4-oxadiazol-5-yl)-methoxy]phenyl}acetate and *N*,*N*-dimethylformamide dimethyl acetal and crystallizes in the triclinic space group $P\overline{1}$ with two independent molecules in the asymmetric unit. The crystal packing is stabilized mainly by van der Waals interactions.

Comment

1,2,4–Oxadiazoles represent an important class of five-membered heterocycles. Some derivatives of 1,2,4-oxadiazoles have intrinsic analgesic (Terashita *et al.*, 2002), antiinflammatory (Nicolaides *et al.*, 1998) and antipicornaviral (Romero, 2001) properties and are efficient as agonists (*e.g.* for angiotensin (Naka & Kubo, 1999) and adhesion (Juraszyk *et al.*, 1997) for different receptors.



(I)

We report here the crystal structure of the title compound, (I), synthesized from methyl {2-[(3-phenyl-1,2,4-oxadiazol-5-yl)methoxy]phenyl}acetate (Wang *et al.*, 2004) and *N*,*N*-dimethylformamide dimethyl acetal. The molecular structure of (I) is shown in Fig. 1 and bond lengths and angles are detailed in Table 1. The crystal packing is stabilized mainly by van der Waals interactions, though there are short intra- and intermolecular C—H···O contacts (Fig. 2 and Table 2).

Experimental

Methyl {2-[(3-phenyl-1,2,4-oxadiazol-5-yl)methoxy]phenyl}acetate (14 mmol) was dissolved in dimethylformamide (20 ml) and *N*,*N*-dimethylformamide dimethyl acetal (8 ml) was added in one portion. The resulting mixture was refluxed for 6 h, then concentrated under reduced pressure to afford crude compound (I). Pure compound (I) was obtained by crystallizing from ethyl acetate (15 ml) and petroleum ether (b.p. 445–447 K) (7.5 ml). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. Spectroscopic analysis, ^{1}H NMR ($CDCl_3$, p.p.m.): 7.99–8.00 (*m*, 2H), 7.83 (*m*, 1H), 7.43–7.55 (*m*, 3H), 7.35 (*m*, 1H), 7.17–7.21 (*m*, 2H), 6.95–6.97 (*m*, 1H), 6.90–6.91 (*m*, 1H), 3.57 (*s*, 3H), 3.02 (*s*, 6H), 2.86 (*s*, 6H).

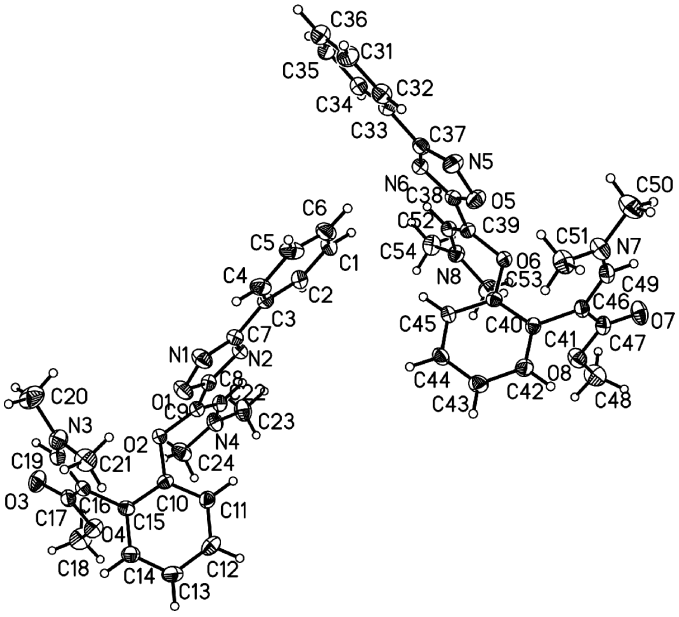


Figure 1
A view of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 25% probability level.

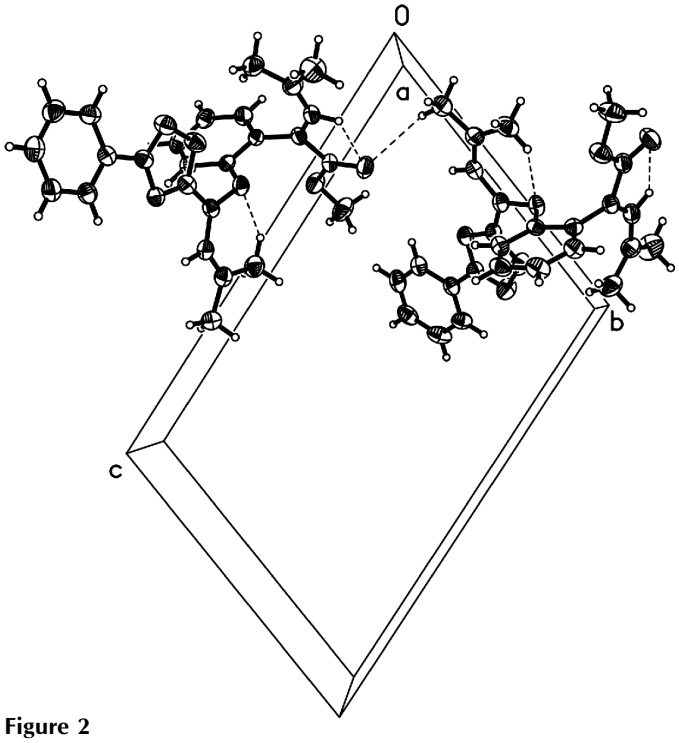


Figure 2
Short C—H···O contacts (dashed lines) in the crystal structure of (I).

Crystal data

$C_{24}H_{26}N_4O_4$
M_r = 434.49
Triclinic, $P\overline{1}$
a = 11.220 (2) Å
b = 12.260 (3) Å
c = 17.762 (4) Å
α = 71.06 (3)°
β = 86.35 (3)°
γ = 85.19 (3)°
V = 2301.1 (8) Å^3
Z = 4
D_x = 1.254 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–13°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.50 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.961, T_{max} = 0.974
9501 measured reflections
9014 independent reflections
4761 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 26.0°
$h = 0 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -21 \rightarrow 21$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.259
S = 1.12
9014 reflections
578 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 1.9P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.019
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0092 (15)

Table 1
Selected geometric parameters (Å, °).

O1—C8	1.371 (5)	N4—C23	1.453 (6)
O1—N1	1.424 (5)	N5—C37	1.304 (5)
O2—C9	1.378 (5)	N6—C38	1.303 (5)
O2—C10	1.397 (5)	N6—C37	1.365 (5)
O3—C17	1.220 (5)	N7—C49	1.338 (6)
O4—C17	1.351 (5)	N7—C51	1.437 (6)
O4—C18	1.433 (6)	N7—C50	1.441 (6)
O5—C38	1.354 (5)	N8—C52	1.337 (5)
O5—N5	1.422 (5)	N8—C53	1.448 (6)
O6—C40	1.386 (5)	N8—C54	1.462 (6)
O6—C39	1.389 (5)	C8—C9	1.414 (6)
O7—C47	1.215 (5)	C9—C22	1.371 (6)
O8—C47	1.351 (5)	C15—C16	1.482 (6)
O8—C48	1.443 (6)	C16—C19	1.351 (6)
N1—C7	1.295 (6)	C16—C17	1.472 (6)
N2—C8	1.303 (5)	C33—C37	1.470 (6)
N2—C7	1.367 (5)	C38—C39	1.437 (6)
N3—C19	1.328 (6)	C39—C52	1.345 (6)
N3—C21	1.445 (7)	C41—C46	1.499 (6)
N3—C20	1.462 (7)	C46—C49	1.353 (6)
N4—C22	1.336 (6)	C46—C47	1.454 (6)
N4—C24	1.432 (6)		
C8—O1—N1	105.9 (3)	C22—C9—C8	119.4 (4)
C9—O2—C10	118.4 (3)	C15—C10—O2	115.1 (3)
C17—O4—C18	115.7 (4)	C10—C15—C16	120.5 (4)
C38—O5—N5	106.1 (3)	C19—C16—C17	114.1 (4)
C40—O6—C39	117.6 (3)	C19—C16—C15	126.9 (4)
C47—O8—C48	116.3 (4)	O3—C17—O4	121.3 (4)
C7—N1—O1	103.2 (4)	O3—C17—C16	125.7 (4)
C8—N2—C7	103.3 (4)	N3—C19—C16	130.9 (4)
C19—N3—C21	124.1 (4)	N4—C22—C9	130.8 (4)
C19—N3—C20	120.3 (5)	C34—C33—C37	120.8 (4)
C21—N3—C20	115.6 (5)	C32—C33—C37	120.4 (4)
C22—N4—C24	124.6 (4)	N5—C37—N6	115.6 (4)
C22—N4—C23	119.5 (4)	N5—C37—C33	121.0 (4)
C24—N4—C23	115.9 (4)	N6—C38—O5	112.8 (4)
C37—N5—O5	102.8 (3)	N6—C38—C39	130.1 (4)
C38—N6—C37	102.8 (4)	C52—C39—C38	119.8 (4)
C49—N7—C51	124.0 (4)	O6—C39—C38	115.6 (4)
C49—N7—C50	120.0 (4)	C45—C40—O6	122.9 (4)
C51—N7—C50	116.0 (5)	O6—C40—C41	116.0 (3)
C52—N8—C53	125.3 (4)	C42—C41—C46	124.0 (4)
C52—N8—C54	119.5 (4)	C40—C41—C46	118.8 (4)
C53—N8—C54	115.2 (4)	C49—C46—C47	114.8 (4)
C4—C3—C7	120.6 (4)	C49—C46—C41	125.5 (4)
N1—C7—N2	115.7 (4)	O7—C47—O8	120.7 (4)
N1—C7—C3	121.6 (4)	O8—C47—C46	112.3 (4)
N2—C8—O1	112.0 (4)	N7—C49—C46	132.3 (4)
N2—C8—C9	131.6 (4)	C39—C52—N8	130.2 (4)
C22—C9—O2	123.2 (4)		

Table 2
Short C—H···O contacts (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C19—H19*A*···O3	0.93	2.33	2.747 (6)	107
C23—H23*A*···O7^{i}	0.96	2.51	3.280 (6)	137
C24—H24*A*···O2	0.96	2.14	2.918 (7)	137
C49—H49*A*···O7	0.93	2.34	2.763 (6)	107
C53—H53*A*···O6	0.96	2.16	2.929 (6)	136

Symmetry code: (i) $1-x, -y, 1-z$.

All H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined using a riding model, with U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 Software; data reduction: *XCAD*4 (Harms, 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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