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Methyl 3-(dimethylamino)-2-(2-{2-(dimethylamino)-1-[3-(2-methylphenyl)-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) = 0.004 Å
R factor = 0.060
wR factor = 0.193
Data-to-parameter ratio = 15.7

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

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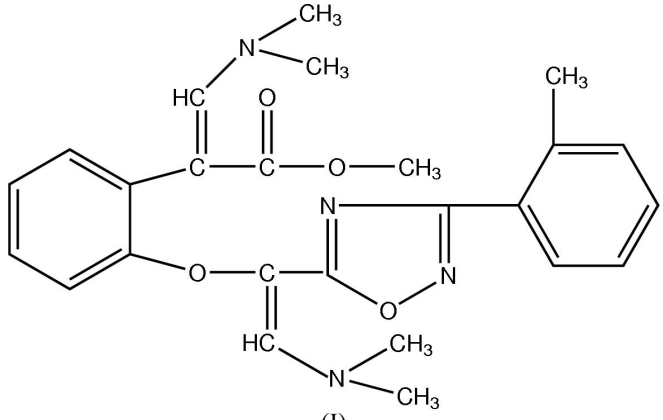
The title compound, $C_{25}H_{28}N_4O_4$, was synthesized by the reaction of methyl 2-{[3-(2-methylphenyl)-1,2,4-oxadiazol-5-yl]methoxy}phenyl acetate and *N*,*N*-dimethylformamide dimethyl acetal. In the crystal structure, there are intramolecular C—H···O and C—H···N hydrogen bonds and intermolecular C—H···π 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), anti-inflammatory (Nicolaides *et al.*, 1998) and antipicornaviral (Romero, 2001) properties and are efficient as agonists [*e.g.* forangiotensin (Naka *et al.*, 1999) and adhesion agents (Juraszyk *et al.*, 1997)] for different receptors. We report here the crystal structure of the title compound, (I).



(I)

The molecular structure of (I) is shown in Fig. 1, where the dashed lines indicate intramolecular C—H···O and C—H···N hydrogen bonds. Selected bond lengths and angles are given in Table 1. There are also C—H···π interactions in the crystal structure (Fig. 2). Full details of the hydrogen bonding are given in Table 2. The combination of both types of weak interactions generates a three-dimensional network.

Experimental

Methyl 2-{[3-(2-methylphenyl)-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 and then concentrated under reduced pressure to afford crude compound (I). Pure compound (I) was obtained by crystallization from ethyl acetate (15 ml) and petrolum ether (7.5 ml). Crystals of (I) suitable for X-ray

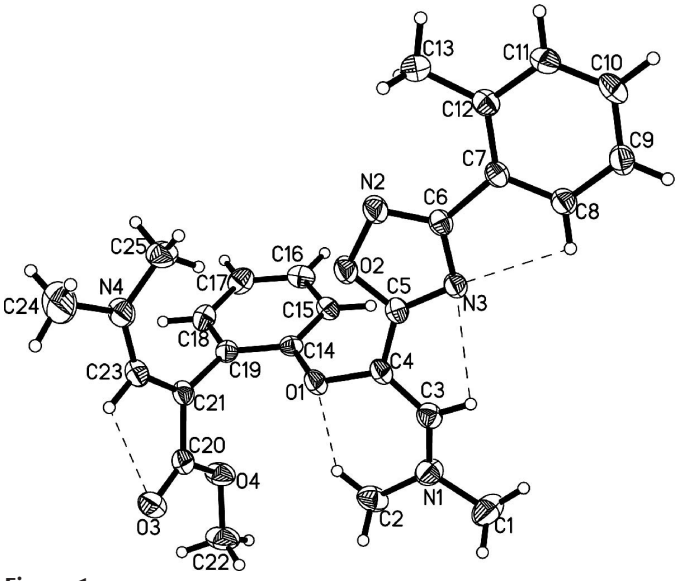


Figure 1
A view of the molecular structure of (I). Dashed lines indicate intramolecular C—H···O and C—H···N hydrogen bonds. Displacement ellipsoids are drawn at the 30% probability level.

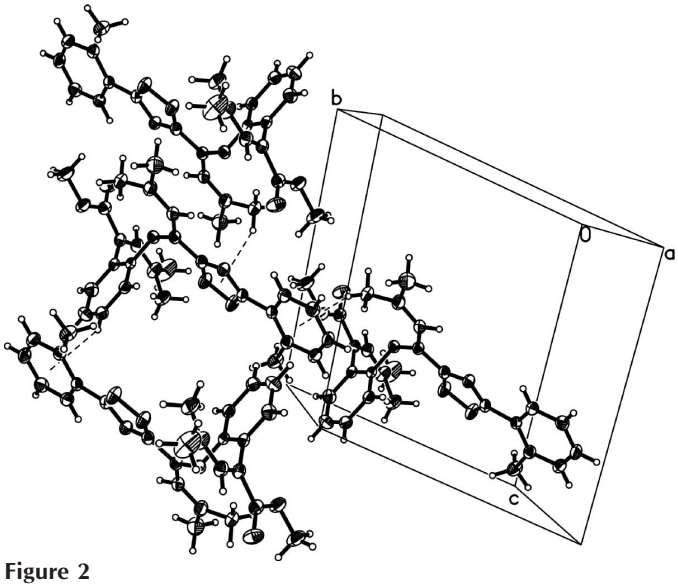


Figure 2
The C—H···π interactions in (I), shown as dashed lines.

diffraction were obtained by slow evaporation of an ethanol solution. Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 7.86–7.88 (*m*, 1H), 7.75 (*m*, 1H), 7.35–7.37 (*m*, 1H), 7.30–32 (*m*, 2H), 7.28–7.30 (*m*, 1H), 7.23–7.25 (*m*, 2H), 7.01 (*m*, 1H), 6.95–6.97 (*m*, 1H), 3.61 (*m*, 3H), 3.06 (*s*, 6H), 2.89 (*m*, 6H), 2.59 (*s*, 3H).

Crystal data

$C_{25}H_{28}N_4O_4$
M_r = 448.51
Triclinic, $P\overline{1}$
a = 10.250 (2) Å
b = 10.691 (2) Å
c = 12.263 (3) Å
α = 105.46 (3)°
β = 110.74 (3)°
γ = 92.30 (3)°
V = 1197.7 (6) Å^3
Z = 2
D_x = 1.244 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
0.4 × 0.3 × 0.3 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4945 measured reflections
4671 independent reflections
2950 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 26.0°
$h = 0 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.193
S = 1.05
4671 reflections
298 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.19P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C14	1.391 (3)	N3—C6	1.381 (3)
O1—C4	1.400 (3)	N4—C23	1.347 (4)
O2—C5	1.343 (3)	N4—C25	1.444 (4)
O2—N2	1.429 (3)	N4—C24	1.447 (4)
O3—C20	1.214 (3)	C3—C4	1.354 (4)
O4—C20	1.357 (3)	C4—C5	1.435 (4)
O4—C22	1.444 (3)	C6—C7	1.487 (3)
N1—C3	1.341 (3)	C12—C13	1.502 (4)
N1—C1	1.450 (4)	C19—C21	1.483 (4)
N1—C2	1.457 (4)	C20—C21	1.458 (4)
N2—C6	1.303 (3)	C21—C23	1.360 (4)
N3—C5	1.296 (3)		
C14—O1—C4	117.52 (18)	N2—C6—N3	114.6 (2)
C5—O2—N2	106.35 (19)	N2—C6—C7	124.5 (2)
C20—O4—C22	116.2 (2)	N3—C6—C7	120.9 (2)
C3—N1—C1	120.5 (3)	C8—C7—C6	117.0 (2)
C3—N1—C2	123.6 (3)	C12—C7—C6	123.0 (2)
C1—N1—C2	115.9 (3)	C11—C12—C13	118.3 (3)
C6—N2—O2	102.9 (2)	C7—C12—C13	124.5 (2)
C5—N3—C6	103.1 (2)	C15—C14—O1	122.8 (2)
C23—N4—C25	123.7 (2)	C19—C14—O1	115.6 (2)
C23—N4—C24	120.1 (3)	C18—C19—C21	120.8 (2)
C25—N4—C24	116.2 (3)	C14—C19—C21	122.3 (2)
N1—C3—C4	132.3 (3)	O3—C20—O4	121.2 (3)
C3—C4—O1	123.9 (2)	O3—C20—C21	127.3 (3)
C3—C4—C5	119.4 (2)	O4—C20—C21	111.4 (2)
O1—C4—C5	116.6 (2)	C23—C21—C20	114.3 (2)
N3—C5—O2	113.0 (2)	C23—C21—C19	126.1 (2)
N3—C5—C4	128.8 (2)	C20—C21—C19	119.4 (2)
O2—C5—C4	118.2 (2)	N4—C23—C21	132.5 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2B···O1	0.96	2.19	2.963 (4)	136
C3—H3B···N3	0.93	2.54	2.922 (3)	105
C8—H8A···N3	0.93	2.48	2.839 (3)	103
C23—H23A···O3	0.93	2.35	2.771 (4)	107
C2—H2C···$Cg1^{i}$	0.96	2.78	3.667 (4)	153
C2—H2D···$Cg2^{ii}$	0.96	2.75	3.674 (3)	161
C16—H16A···$Cg2^{iii}$	0.93	2.71	3.531 (4)	148

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $x, y+1, z$; (iii) $-x+1, -y+1, -z+2$. Notes: $Cg1$ is the centroid of ring C5/O2/N2/C6/N3 and $Cg2$ is the centroid of ring C7–C12.

All H atoms were placed geometrically, with C—H distances in the range 0.93–0.96 Å, and included in the refinement in a riding-model approximation, with U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXL97*.

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