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

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.072
wR factor = 0.261
Data-to-parameter ratio = 15.5

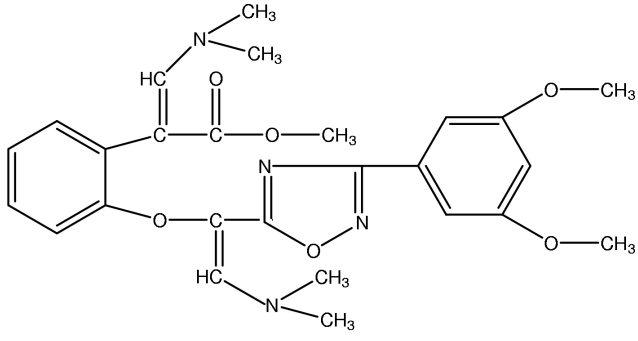
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{26}H_{30}N_4O_6$, was synthesized by the reaction of methyl (2-{[3-(3,5-dimethoxyphenyl)-1,2,4-oxadiazol-5-yl]methoxy}phenyl)acetate and *N*,*N*-dimethylformamide dimethyl acetal. In the molecular structure, there are intramolecular C—H···O and C—H···N hydrogen bonds, and also intermolecular C—H···π and C—H···O 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.* for angiotensin (Naka & Kubo, 1999) and adhesion (Juraszyk *et al.*, 1997)] for different receptors.

We report here the crystal structure of the title compound, (I). The molecular structure of (I) is shown in Fig. 1; dashed lines indicate intramolecular C—H···N and C—H···O hydrogen bonds (Table 2). Selected bond lengths and angles are given in Table 1. There are also intermolecular contacts, which indicate weak C—H···π interactions (Fig. 2) and C—H···O hydrogen bonds (Fig. 3). Full details of the hydrogen bonding are given in Table 2. The combination of both types of weak interactions generates a three-dimensional network.



(I)

Experimental

Methyl (2-{[3-(3,5-dimethoxyphenyl)-1,2,4-oxadiazol-5-yl]methoxy}-phenyl) acetate (14 mmol) was dissolved in DMF (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 petroleum ether (7.5 ml). Crystals of (I) suitable for

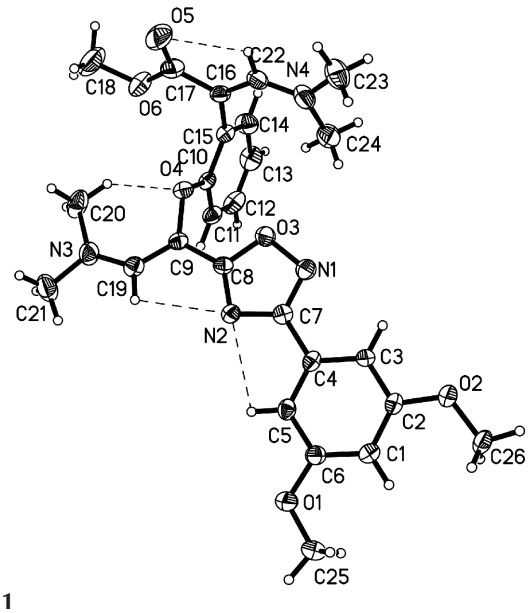


Figure 1
A view of the molecular structure of (I). Dashed lines indicate intramolecular C—H···N and C—H···O hydrogen bonds. Displacement ellipsoids are drawn at the 30% probability level.

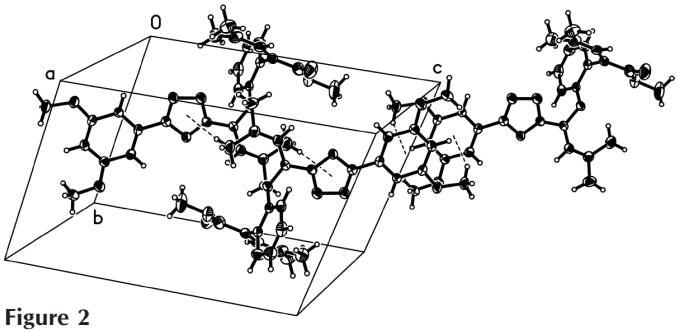


Figure 2
C—H···π interactions in (I), shown as dashed lines.

X-ray diffraction were obtained by slow evaporation of an ethanol solution. ^{1}H NMR ($CDCl_3$, p.p.m.): δ 7.69 (*m*, 1H), 7.33 (*m*, 1H), 7.15–7.26 (*m*, 4H), 6.94–6.95 (*m*, 1H), 6.87–6.89 (*m*, 1H), 6.54 (*m*, 1H), 3.81 (*s*, 6H), 3.56 (*s*, 3H), 3.01 (*s*, 6H), 2.85 (*m*, 6H).

Crystal data

$C_{26}H_{30}N_4O_6$
M_r = 494.54
Triclinic, $P\overline{1}$
a = 8.6500 (17) Å
b = 10.700 (2) Å
c = 14.817 (3) Å
α = 107.66 (3)°
β = 97.50 (3)°
γ = 95.15 (3)°
V = 1283.4 (4) Å^3
Z = 2
D_x = 1.280 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9–12°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.3 × 0.2 × 0.1 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
5386 measured reflections
5030 independent reflections
2308 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 26.0°
$h = 0 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -18 \rightarrow 18$
3 standard reflections
every 200 reflections
intensity decay: none

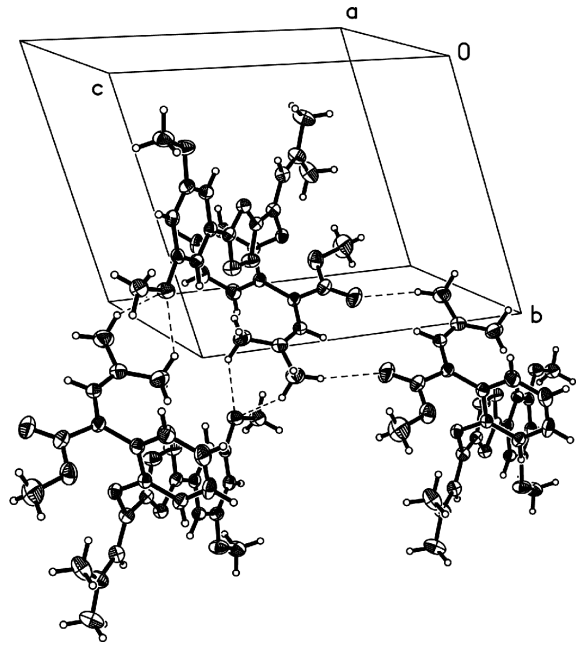


Figure 3
Short C—H···O contacts in (I), shown as dashed lines.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.261
S = 1.04
5030 reflections
325 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.13P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O4—C10	1.380 (5)	C19—N3	1.333 (5)
O4—C9	1.400 (5)	N3—C21	1.435 (6)
O3—C8	1.340 (5)	N3—C20	1.453 (6)
O3—N1	1.423 (4)	O6—C17	1.367 (6)
N2—C8	1.311 (5)	O6—C18	1.424 (6)
N2—C7	1.380 (5)	N4—C22	1.312 (6)
C4—C7	1.460 (6)	N4—C24	1.430 (6)
C8—C9	1.427 (6)	N4—C23	1.466 (6)
C7—N1	1.305 (5)	C16—C22	1.380 (7)
C15—C16	1.491 (6)	C16—C17	1.436 (7)
C9—C19	1.353 (6)	O5—C17	1.206 (6)
C10—O4—C9	116.1 (3)	N3—C19—C9	131.9 (4)
C8—O3—N1	106.4 (3)	C19—N3—C21	121.2 (4)
C8—N2—C7	102.4 (3)	C19—N3—C20	123.9 (4)
O2—C2—C1	124.2 (4)	C21—N3—C20	114.8 (4)
C5—C4—C7	120.3 (4)	C17—O6—C18	116.0 (5)
N2—C8—O3	113.2 (4)	C22—N4—C24	126.6 (5)
N2—C8—C9	129.7 (4)	C22—N4—C23	119.1 (5)
O3—C8—C9	117.1 (4)	C24—N4—C23	114.3 (5)
N1—C7—N2	114.9 (4)	O1—C6—C1	124.1 (4)
N1—C7—C4	120.9 (4)	C22—C16—C17	114.9 (4)
N2—C7—C4	124.2 (4)	C22—C16—C15	125.2 (4)
C7—N1—O3	103.1 (3)	C17—C16—C15	119.8 (4)
C10—C15—C16	120.6 (4)	O5—C17—O6	120.6 (5)
C11—C10—O4	123.7 (4)	O5—C17—C16	128.2 (6)
C19—C9—O4	123.7 (4)	O6—C17—C16	111.2 (4)
C19—C9—C8	120.5 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5A···N2	0.93	2.62	2.933 (5)	100
C19—H19A···N2	0.93	2.59	2.960 (5)	104
C20—H20A···O4	0.96	2.17	2.943 (5)	136
C22—H22A···O5	0.93	2.38	2.780 (6)	106
C20—H20B···$Cg1^{i}$	0.96	2.98	3.921 (6)	169
C26—H26C···$Cg2^{ii}$	0.96	2.91	3.723 (5)	143
C23—H23A···$O5^{iii}$	0.96	2.43	3.362 (7)	162
C23—H23B···$O2^{iv}$	0.96	2.56	3.339 (7)	138
C24—H24C···$O2^{iv}$	0.96	2.52	3.305 (7)	139

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $-1-x, 1-y, 2-z$; (iii) $-x, 2-y, 1-z$; (iv) $-x, 2-y, 2-z$. $Cg1$ and $Cg2$ are the centroid of the O3/N1/C7/N2/C8 and C1–C6 rings, respectively.

All H were positioned geometrically with C—H distances of 0.93–0.96 Å and included in the refinement in the 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: *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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