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1-[(2,6-Dimethylphenyl)aminocarbonylmethyl]-4-{[3-(3-methoxyphenyl)-1,2,4-oxadiazol-5-yl]-methyl}piperazine

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Key indicators

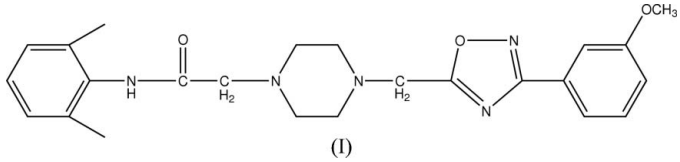
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.057
wR factor = 0.181
Data-to-parameter ratio = 15.2

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

The title compound, $C_{24}H_{29}N_5O_3$, contains intramolecular N—H···N hydrogen-bond and intermolecular C—H···π interactions.

Comment

Piperazine derivatives are of interest because of their biological properties. Some derivatives of piperazine have antifilarial, anti-amebic and spermicidal properties (Sonurlikar *et al.*, 1977). Some show high efficacy in treating or preventing neuronal damage or stimulating nerve growth (Tomlinson *et al.*, 2004). Some have also been used to treat psychosis and bipolar disorders (Aicher *et al.*, 2004) or act as neurokinin antagonists (Janssens *et al.*, 2004).



(I)

The molecular structure of (I) is shown in Fig. 1, where the dashed line indicates an intramolecular N—H···N hydrogen bond (Table 1). The bond lengths and angles are given in Table 1. In the crystal structure, there are also C—H···π interactions (Table 1 and Fig. 2).

Experimental

4-[(2,6-Dimethylphenyl)aminocarbonylmethyl]piperazine (20 mmol) was dissolved in acetone (20 ml) and potassium carbonate (30 mmol) was added. 3-(3-Methoxylphenyl)-5-chloromethyl-1,2,4-oxadiazole (20 mmol) in acetone (20 ml) was added to this mixture. The resulting mixture was refluxed for 6 h. Concentration of the mixture under reduced pressure afforded crude compound (I). Pure compound (I) was obtained by recrystallization from ethyl acetate. Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. ^{1}H NMR ($CDCl_3$, p.p.m.): δ 8.56 (*m*, 1H), 7.68–7.70 (*m*, 1H), 7.61–7.62 (*m*, 1H), 7.37–7.40 (*m*, 1H), 7.03–7.10 (*m*, 4H), 3.96 (*m*, 2H), 3.86 (*s*, 3H), 3.22 (*m*, 2H), 2.78 (*m*, 8H), 2.21 (*s*, 6H).

Crystal data

$C_{24}H_{29}N_5O_3$
M_r = 435.52
Monoclinic, $P2_1/c$
a = 17.120 (3) Å
b = 10.271 (2) Å
c = 13.042 (3) Å
β = 91.10 (3)°
V = 2292.9 (8) Å^3
Z = 4
D_x = 1.262 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
0.40 × 0.40 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.967, T_{max} = 0.992
4695 measured reflections
4484 independent reflections
2051 reflections with $I > 2\sigma(I)$

R_{int} = 0.032
θ_{max} = 26.0°
$h = -20 \rightarrow 20$
$k = -12 \rightarrow 0$
$l = 0 \rightarrow 15$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.181
S = 0.95
4484 reflections
295 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 0.15P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5A···N4	0.84 (3)	2.27 (3)	2.734 (4)	115 (3)
C15—H15B···$Cg4^{i}$	0.97	2.79	3.520 (4)	133

Symmetry code: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$. $Cg4$ is the centroid of atoms C17–C22.

Atom H5*A*, attached to atom N5, was located in a difference Fourier map and refined freely. Other H atoms were placed in calculated positions with C—H distances in the range 0.93–0.97 Å and refined using a riding model with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}).

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*.

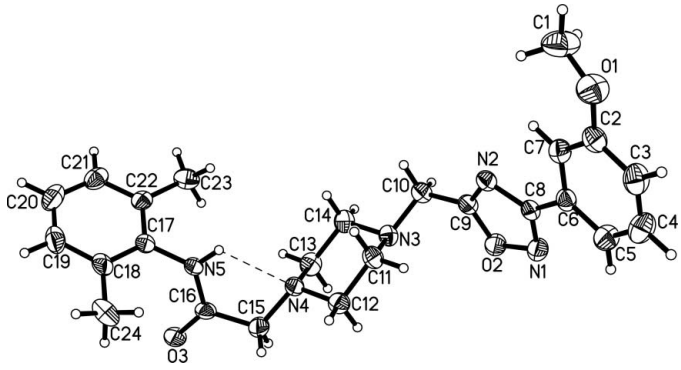


Figure 1
A view of the molecular structure of (I); the dashed line indicates an intramolecular N—H···N hydrogen bond. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

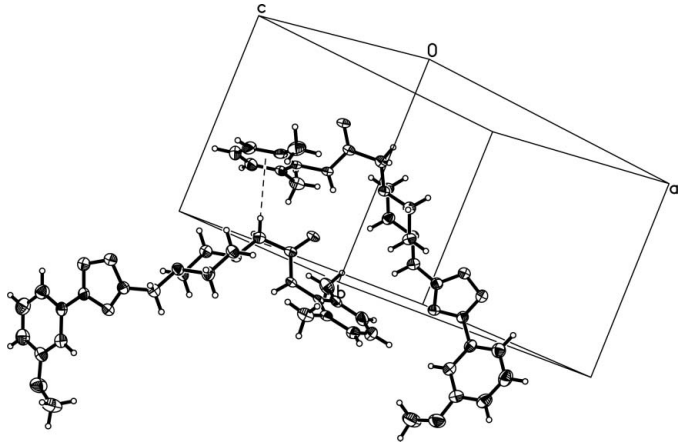


Figure 2
The C—H···π interaction in (I), shown as a dashed line.

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