Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-[(2,6-Dimethylphenyl)aminocarbonylmethyl]-4-{[3-(4-nitrophenyl)-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) = 0.006 Å
R factor = 0.070
wR factor = 0.229
Data-to-parameter ratio = 15.0

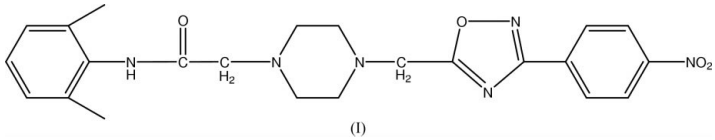
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 October 2004
Accepted 11 October 2004
Online 22 October 2004

The title compound, $C_{23}H_{26}N_6O_4$, was synthesized by the reaction of 4-[(2,6-dimethylphenyl)aminocarbonylmethyl]-piperazine and 5-chloromethyl-3-(4-nitrophenyl)-1,2,4-oxadiazole. Its molecular structure is characterized by a weak intramolecular C—H···N interaction. The crystal packing is stabilized by weak intermolecular N—H···O and C—H···O hydrogen bonds and a C—H···π interaction.

Comment

Piperazine derivatives are of great interest because of their physiological activity. Derivatives of piperazine have anti-filarial, anti-amoebic and spermicidal properties (Sonurlikar *et al.*, 1977); they also show high efficacy in treating or preventing neuronal damage or stimulating nerve growth (Tomlinson *et al.*, 2004). Some of them are used to treat psychosis and bipolar disorders (Aicher *et al.*, 2004), while others are neurokinin antagonists (Janssens *et al.*, 2004).



(I)

The molecular structure of the title compound, (I) (Fig. 1), shows normal bond lengths and angles (Table 1) and a weak intramolecular C3—H3*B*···N2 interaction (Table 2). The crystal packing (Fig. 2) is stabilized by weak intermolecular N—H···O and C—H···O hydrogen bonds and by a C—H···π interaction with the C16–C21 ring (Table 2).

Experimental

4-[(2,6-Dimethylphenyl)aminocarbonylmethyl]piperazine (20 mmol) and 5-chloromethyl-3-(4-nitrophenyl)-1,2,4-oxadiazole (20 mmol) were dissolved in anhydrous ethanol (80 ml). The resulting mixture was refluxed for 6 h. The mixture was then concentrated under reduced pressure to afford 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 ($CDCl_3$, δ): 8.56 (*m*, 1H), 8.33–8.34 (*m*, 2H), 8.27–8.29 (*m*, 2H), 7.06–7.10 (*m*, 3H), 4.00 (*s*, 2H), 3.27 (*s*, 2H), 2.80 (*m*, 8H), 2.21 (*s*, 6H).

Crystal data

$C_{23}H_{26}N_6O_4$
M_r = 450.50
Monoclinic, $P2_1/c$
a = 7.2240 (14) Å
b = 9.5650 (19) Å
c = 33.245 (7) Å
β = 95.22 (3)°
V = 2287.6 (8) Å^3
Z = 4
D_x = 1.308 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 25 reflections
θ = 9–12°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.4 × 0.2 × 0.1 mm

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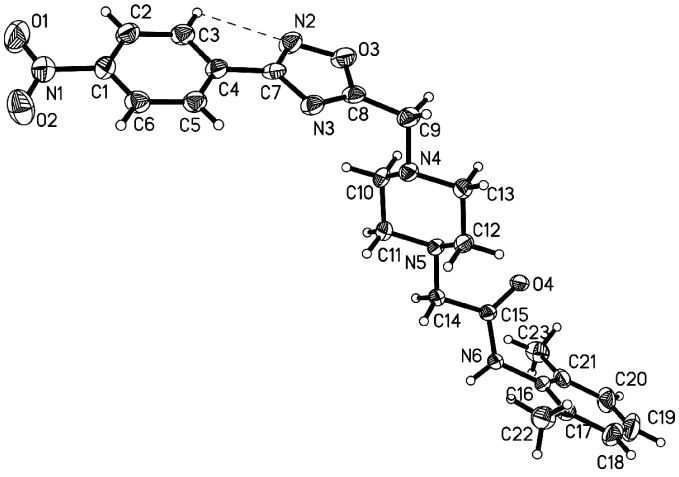

Figure 1
A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. The dashed line indicates the intramolecular C—H···N interaction.

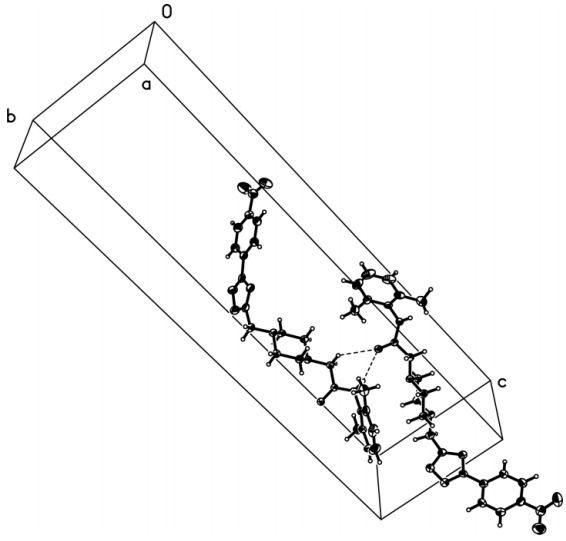

Figure 2
A part of the crystal structure of (I). Dashed lines indicate intermolecular N—H···O and C—H···O hydrogen bonds.

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4833 measured reflections
4466 independent reflections
1964 reflections with $I > 2\sigma(I)$
$R_{int} = 0.096$
$\theta_{max} = 26.0^\circ$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 11$
$l = -39 \rightarrow 39$
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.229$
$S = 1.06$
4466 reflections
298 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.25P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O3—C8	1.345 (5)	N5—C14	1.465 (5)
O3—N2	1.416 (5)	N6—C15	1.341 (4)
O4—C15	1.235 (4)	N6—C16	1.447 (4)
N2—C7	1.294 (5)	C4—C7	1.474 (6)
N3—C8	1.306 (5)	C8—C9	1.494 (6)
N3—C7	1.379 (5)	C10—C11	1.503 (5)
N4—C9	1.449 (5)	C14—C15	1.504 (5)
N4—C13	1.462 (5)	C17—C22	1.495 (6)
N5—C12	1.459 (5)		
C8—O3—N2	106.2 (3)	N3—C8—O3	113.2 (4)
C7—N2—O3	103.2 (3)	N4—C9—C8	113.0 (4)
C8—N3—C7	102.0 (3)	N5—C14—C15	112.2 (3)
C9—N4—C13	108.9 (3)	O4—C15—N6	122.8 (3)
C13—N4—C10	108.1 (3)	O4—C15—C14	122.5 (3)
C11—N5—C14	110.2 (3)	N6—C15—C14	114.7 (3)
C15—N6—C16	123.6 (3)	C17—C16—N6	116.5 (4)
C6—C1—N1	119.7 (4)	C16—C21—C20	117.8 (5)
C5—C4—C7	121.7 (4)	C16—C21—C23	123.0 (4)
N2—C7—N3	115.4 (4)	C20—C21—C23	119.2 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6A···O4^{i}	0.86	2.22	3.055 (4)	163
C3—H3B···N2	0.93	2.48	2.798 (6)	100
C14—H14B···O4^{i}	0.97	2.57	3.302 (5)	132
C10—H10A···Cg^{ii}	0.96	2.61	3.563 (7)	166

Symmetry codes: (i) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$. Cg is the centroid of the C16–C21 ring.

All H atoms bonded to the C atoms were positioned geometrically, with C—H = 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} of the carrier atom. The H atom bonded to the N atom was positioned geometrically, with N—H = 0.86 Å, and included in the refinement in a riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(N).

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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