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4-{[3-(2-Chlorophenyl)-1,2,4-oxadiazol-5-yl]-methyl}-1-[(2,6-dimethylphenyl)aminocarbonyl-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 = 0.058
wR factor = 0.201
Data-to-parameter ratio = 13.7

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

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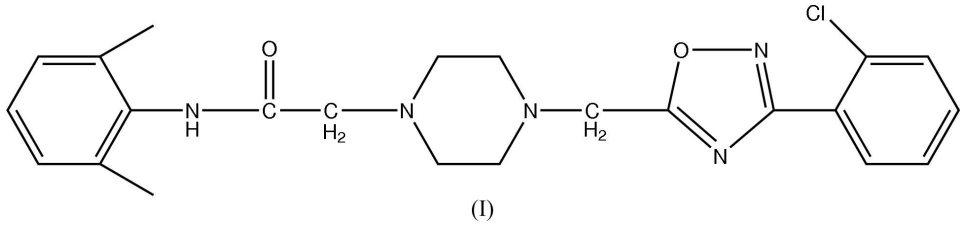
The title compound, $C_{23}H_{26}ClN_5O_2$, was synthesized by the reaction of 4-[(2,6-dimethylphenyl)aminocarbonylmethyl]-piperazine and 5-chloromethyl-3-(2-chlorophenyl)-1,2,4-oxadiazole. In the structure, there are intramolecular C—H···N, N—H···N and C—H···O hydrogen bonds, and intermolecular N—H···O hydrogen bonds.

Comment

Piperazine derivatives are of great interest because of their biological properties. Some derivatives of piperazine have antifilarial, antiamebic 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 also treat psychosis and bipolar disorders (Aicher *et al.*, 2004) or act as neurokinin antagonists (Janssens *et al.*, 2004).



(I)

The molecular structure of the title compound, (I), is shown in Fig.1. The dashed lines indicate intramolecular C—H···N, C—H···O and N—H···N hydrogen bonds (Table 2). The bond lengths and angles are given in Table 1. In the crystal structure, molecules are linked by N—H···O hydrogen bonds (Table 2 and Fig. 2), forming a three-dimensional network.

Experimental

4-[(2,6-Dimethylphenyl)aminocarbonylmethyl]piperazine (20 mmol) was dissolved in acetone (20 ml) and potassium carbonate (30 mmol) was added. 3-(2-Chlorophenyl)-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 8.58 (*m*, 1H), 7.92–7.94 (*m*, 1H), 7.51–7.53 (*m*, 1H), 7.40–7.44 (*m*, 1H), 7.35–7.39 (*m*, 1H), 7.05–7.09 (*m*, 3H), 3.99 (*s*, 2H), 3.21 (*m*, 2H), 2.78 (*m*, 8H), 2.21 (*s*, 6H).

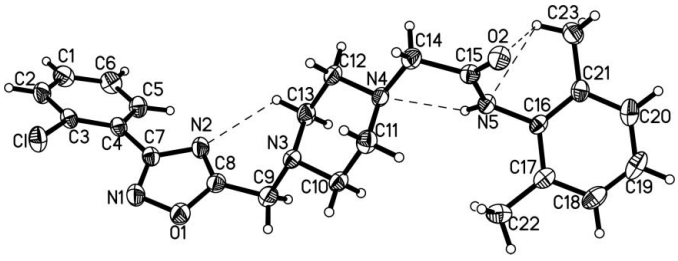


Figure 1
A view of the molecular structure of (I); the dashed lines indicate intramolecular C—H···O, C—H···N and N—H···N hydrogen bonds. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level

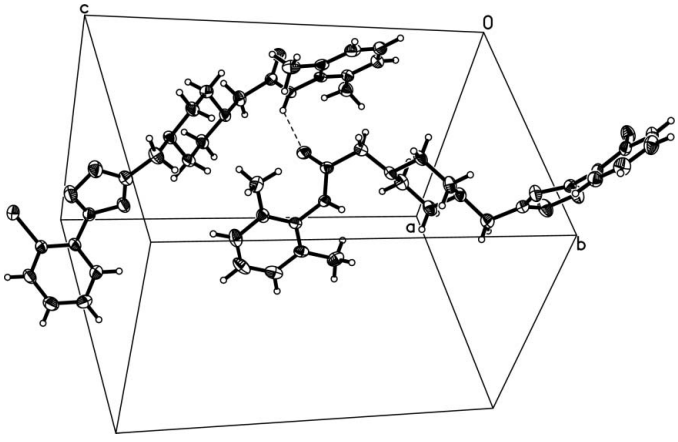


Figure 2
Part of the crystal structure of (I). The dashed line indicates the intermolecular N—H···O hydrogen bond.

Crystal data

$C_{23}H_{26}ClN_5O_2$
M_r = 439.94
Monoclinic, $P2_1/a$
a = 12.296 (1) Å
b = 10.587 (2) Å
c = 17.164 (2) Å
β = 99.77 (3)°
V = 2201.8 (6) Å^3
Z = 4
D_x = 1.327 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–13°
μ = 0.20 mm^{-1}
T = 293 (2) K
Block, colourless
0.4 × 0.3 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4061 measured reflections
3861 independent reflections
2427 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 25.0°
$h = -14 \rightarrow 0$
$k = 0 \rightarrow 12$
$l = -20 \rightarrow 20$
3 standard reflections every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.201
S = 1.01
3861 reflections
281 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1259P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.023 (4)

Table 1
Selected geometric parameters (Å, °).

Cl—C3	1.732 (3)	N4—C11	1.458 (4)
O1—C8	1.321 (4)	N4—C12	1.462 (4)
O1—N1	1.408 (4)	N5—C15	1.342 (4)
O2—C15	1.221 (4)	N5—C16	1.440 (4)
N1—C7	1.290 (4)	C4—C7	1.475 (4)
N2—C8	1.285 (4)	C8—C9	1.496 (5)
N2—C7	1.376 (4)	C10—C11	1.506 (5)
N3—C13	1.464 (4)	C12—C13	1.508 (5)
N3—C9	1.467 (4)	C14—C15	1.528 (4)
N3—C10	1.474 (4)	C17—C22	1.490 (5)
N4—C14	1.444 (4)	C21—C23	1.501 (5)
C8—O1—N1	106.5 (3)	N2—C8—C9	131.0 (3)
C7—N1—O1	103.5 (3)	O1—C8—C9	115.8 (3)
C8—N2—C7	103.1 (3)	N3—C9—C8	113.1 (3)
C13—N3—C9	111.7 (3)	N3—C10—C11	110.1 (3)
C13—N3—C10	109.5 (3)	N4—C11—C10	111.1 (3)
C9—N3—C10	109.2 (3)	N4—C12—C13	109.8 (3)
C14—N4—C11	111.3 (3)	N3—C13—C12	110.9 (3)
C14—N4—C12	112.2 (3)	N4—C14—C15	113.5 (3)
C11—N4—C12	108.4 (2)	O2—C15—N5	124.0 (3)
C15—N5—C16	124.2 (3)	O2—C15—C14	121.3 (3)
C2—C3—Cl	117.1 (3)	N5—C15—C14	114.6 (3)
C4—C3—Cl	121.6 (3)	C21—C16—N5	119.3 (3)
C5—C4—C7	118.1 (3)	C17—C16—N5	117.8 (3)
C3—C4—C7	124.4 (3)	C18—C17—C22	120.6 (3)
N1—C7—N2	113.7 (3)	C16—C17—C22	121.9 (3)
N1—C7—C4	123.2 (3)	C16—C21—C23	122.4 (3)
N2—C7—C4	123.1 (3)	C20—C21—C23	120.2 (3)
N2—C8—O1	113.2 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5A···N4	0.86	2.38	2.747 (4)	106
N5—H5A···O2^{i}	0.86	2.37	3.070 (3)	138
C13—H13A···N2	0.97	2.59	3.247 (5)	125
C23—H23A···O2	0.96	2.56	3.068 (4)	113
C23—H23A···N5	0.96	2.43	2.896 (4)	110

Symmetry code: (i) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+1$.

All H atoms were placed geometrically at C—H distances of 0.93–0.97 Å and an N—H distance of 0.86 Å, and included in the refinement in the riding-model approximation with U_{iso}(H) = 1.2U_{eq}(C,N) 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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