

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

1-Chloro-2-(4-phenylpiperazin-1-yl)-ethanone

Yong-Ji Xu* and Fei Jing

College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, Qingdao 266042, People's Republic of China

Correspondence e-mail: xuyj1960@qust.edu.cn

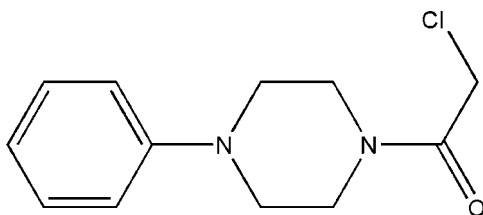
Received 17 December 2008; accepted 6 March 2009

Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.035; wR factor = 0.098; data-to-parameter ratio = 18.8.

The title compound, $\text{C}_{12}\text{H}_{15}\text{ClN}_2\text{O}$, is a piperazine derivative with the potential for use as a starting material for pharmaceutical and agrochemical applications. The structure is stabilized by $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, $\text{C}-\text{H}\cdots\pi$ interactions and $\pi-\pi$ stacking interactions [centroid-centroid distance = is 4.760 (2) Å].

Related literature

For the biological activity of piperazine and its derivatives, see: Berkheij (2005); Upadhayaya *et al.* (2004); Choudhary *et al.* (2006); Vacca *et al.* (1994); Hulme (1999). For reference structural data, see: Drew & Leslie (1986).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{15}\text{ClN}_2\text{O}$
 $M_r = 238.71$
 Monoclinic, $P2_1/c$
 $a = 9.4423$ (19) Å
 $b = 8.5629$ (17) Å

$c = 14.506$ (3) Å
 $\beta = 101.34$ (3)°
 $V = 1149.9$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 0.31$ mm⁻¹
 $T = 113$ K

0.20 × 0.18 × 0.12 mm

Data collection

Rigaku Saturn diffractometer
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.940$, $T_{\max} = 0.964$

9264 measured reflections
 2729 independent reflections
 2151 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.098$
 $S = 1.07$
 2729 reflections

145 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.25$ e Å⁻³
 $\Delta\rho_{\min} = -0.26$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C2}-\text{H2}\cdots\text{O1}^{\text{i}}$	0.93	2.47	3.1850 (16)	134
$\text{C9}-\text{H9A}\cdots\text{O1}$	0.97	2.38	2.7456 (17)	102
$\text{C12}-\text{H12B}\cdots\text{O1}^{\text{ii}}$	0.97	2.43	3.3426 (16)	157
$\text{C5}-\text{H5}\cdots\text{Cg2}^{\text{iii}}$	0.93	3.25	3.7651 (15)	117
$\text{C8}-\text{H8B}\cdots\text{Cg2}^{\text{iv}}$	0.97	3.09	4.0393 (12)	168
$\text{C12}-\text{H12A}\cdots\text{Cg2}^{\text{iv}}$	0.97	3.04	3.6878	125

Symmetry codes: (i) $x, -y - \frac{1}{2}, z + \frac{1}{2}$; (ii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (iii) $-x + 2, y - \frac{1}{2}, -z + \frac{1}{2}$; (iv) $x, -y - \frac{1}{2}, z - \frac{3}{2}$. Cg2 is the centroid of the C1-C6 ring.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

This work was supported by the Natural Science Foundation of Shandong Province, China (No. Y2007B50). The authors thank Professor Yong-Hong Wen for help with this paper.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2461).

References

- Berkheij, M. (2005). *Tetrahedron Lett.* **46**, 2369–2371.
 Choudhary, P., Kumar, R. & Verma, K. (2006). *Bioorg. Med. Chem.* **14**, 1819–1826.
 Drew, M. G. B. & Leslie, P. G. (1986). *Acta Cryst.* **C42**, 174–177.
 Hulme, C. (1999). *Tetrahedron Lett.* **40**, 5295–5299.
 Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Upadhayaya, P. S., Sinha, N. & Jain, S. (2004). *Bioorg. Med. Chem.* **12**, 2225–2238.
 Vacca, J. P., Dorsey, B. D., Schleif, W. A. & Levine, R. B. (1994). *J. Med. Chem.* **37**, 3443–3451.

supplementary materials

Acta Cryst. (2009). E65, o1211 [doi:10.1107/S1600536809008216]

1-Chloro-2-(4-phenylpiperazin-1-yl)ethanone

Y.-J. Xu and F. Jing

Comment

Piperazine and its derivatives are important pharmacores that can be found in biologically active compounds across a number of different therapeutic areas (Berkheij, 2005), such as antifungal (Upadhayaya *et al.*, 2004), anti-bacterial, anti-malarial, anti-psychotic agents (Choudhary *et al.*, 2006), HIV protease inhibitor (Vacca *et al.*, 1994), anti-depressant and anti-tumour activity colon, prostate, breast, lung and leukemia tumors (Hulme *et al.*, 1999). In an attempt to further synthesis piperazine derivatives, the title compound, 2-chloro-1-(4-phenylpiperazin-1-yl)ethanone, (I) (Fig. 1), was synthesized and its X-ray crystal structure determined.

In the structure of title compound (Fig. 1), the bond lengths and angles in the piperazine ring and the benzene ring are normal (Drew & Leslie, 1986) (Table 1). The dihedral angle between the piperazine ring N1/N2/C7—C10 and C1—C6 benzene ring is 36.8 (2)°. The molecular structure is stabilized by inter and intramolecular C—H···O interactions (Table 2). There exists π - π stacking interactions and C—H··· π interactions. The π - π stacking interaction between the two phenyl rings is observed in the structure. The centroid distance between the two rings is 4.760 Å. There are three types of C—H··· π interactions, C5—H5···C_g2, C8—H8B···C_g2 and C12—H12A···C_g2 (C_g2 is the C1—C6 ring centroid) (Table 2).

Experimental

To a solution of 1-phenylpiperazine hydrochloride (2.0 g, 10 mmol) triethylamine (2.8 ml, 2 mmol) in anhydrous dichloromethane (50 ml) was added chloroacetyl chloride (0.8 mL, 10 mmol) dropwise at 273 K. The reaction mixture was stirred at room temperature for 2 h and monitored by TLC, and then the mixture was diluted with dichloromethane (50 ml) and washed with water (200 ml). The organic phase was dried over anhydrous sodium sulfate and concentrated to yield a solid which was crystallized to obtain 2-chloro-1-(4-phenylpiperazin-1-yl)ethanone.

Refinement

H atoms were placed in calculated positions and treated using a riding model, with C—H = 0.93–0.98 Å and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ or $1.5 U_{\text{eq}}(\text{C})$ for methyl H atoms.

Figures

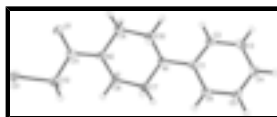


Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids.

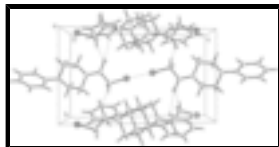


Fig. 2. The packing diagram of (I), viewed down the *c* axis, showing the intermolecular hydrogen bonds (dashed lines).

1-Chloro-2-(4-phenylpiperazin-1-yl)ethanone

Crystal data

$C_{12}H_{15}ClN_2O$

$M_r = 238.71$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.4423$ (19) Å

$b = 8.5629$ (17) Å

$c = 14.506$ (3) Å

$\beta = 101.34$ (3)°

$V = 1149.9$ (4) Å³

$Z = 4$

$F_{000} = 504$

$D_x = 1.379$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 3260 reflections

$\theta = 3.2$ – 27.9 °

$\mu = 0.31$ mm⁻¹

$T = 113$ K

Block, colourless

$0.20 \times 0.18 \times 0.12$ mm

Data collection

Rigaku Saturn
diffractometer

Radiation source: rotating anode

Monochromator: confocal

$T = 113$ K

ω scans

Absorption correction: multi-scan
(CrystalClear; Rigaku, 2005)

$T_{\min} = 0.940$, $T_{\max} = 0.964$

9264 measured reflections

2729 independent reflections

2151 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\max} = 27.9$ °

$\theta_{\min} = 3.2$ °

$h = -12 \rightarrow 12$

$k = -8 \rightarrow 11$

$l = -16 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

$wR(F^2) = 0.098$

$S = 1.07$

2729 reflections

145 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.0763P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.25$ e Å⁻³

$\Delta\rho_{\min} = -0.26$ e Å⁻³

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.62862 (4)	0.03256 (4)	0.08928 (2)	0.03631 (13)
O1	0.58185 (10)	-0.16571 (10)	0.24044 (7)	0.0297 (2)
N1	0.75320 (11)	0.03812 (12)	0.56187 (8)	0.0237 (2)
N2	0.66086 (12)	-0.00754 (12)	0.36497 (8)	0.0244 (2)
C1	0.82553 (13)	0.04592 (14)	0.65699 (10)	0.0230 (3)
C2	0.76046 (14)	-0.02277 (14)	0.72590 (10)	0.0276 (3)
H2	0.6724	-0.0739	0.7082	0.033*
C3	0.82561 (16)	-0.01541 (15)	0.81981 (11)	0.0312 (3)
H3	0.7815	-0.0627	0.8646	0.037*
C4	0.95649 (16)	0.06196 (16)	0.84812 (10)	0.0311 (3)
H4	1.0004	0.0664	0.9113	0.037*
C5	1.02012 (14)	0.13211 (15)	0.78058 (10)	0.0299 (3)
H5	1.1069	0.1854	0.7989	0.036*
C6	0.95647 (13)	0.12419 (15)	0.68604 (10)	0.0253 (3)
H6	1.0013	0.1714	0.6416	0.030*
C7	0.80673 (14)	0.13837 (14)	0.49496 (10)	0.0256 (3)
H7A	0.8961	0.0961	0.4821	0.031*
H7B	0.8263	0.2419	0.5215	0.031*
C8	0.69545 (14)	0.14856 (14)	0.40466 (10)	0.0266 (3)
H8A	0.6084	0.1975	0.4170	0.032*
H8B	0.7325	0.2128	0.3597	0.032*
C9	0.61886 (13)	-0.11941 (14)	0.43097 (10)	0.0250 (3)
H9A	0.6128	-0.2231	0.4035	0.030*
H9B	0.5242	-0.0921	0.4425	0.030*
C10	0.72706 (13)	-0.12050 (14)	0.52343 (10)	0.0256 (3)
H10A	0.6908	-0.1857	0.5683	0.031*
H10B	0.8174	-0.1650	0.5137	0.031*
C11	0.63200 (13)	-0.03982 (14)	0.27233 (10)	0.0237 (3)
C12	0.66787 (14)	0.08973 (15)	0.20873 (9)	0.0272 (3)
H12A	0.7695	0.1158	0.2266	0.033*
H12B	0.6124	0.1823	0.2169	0.033*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0485 (2)	0.0282 (2)	0.0289 (2)	0.00339 (14)	-0.00042 (16)	-0.00073 (13)
O1	0.0291 (5)	0.0214 (5)	0.0375 (6)	-0.0028 (4)	0.0034 (4)	-0.0060 (4)
N1	0.0219 (5)	0.0164 (5)	0.0314 (6)	-0.0042 (4)	0.0021 (4)	0.0009 (4)
N2	0.0241 (5)	0.0174 (5)	0.0309 (6)	-0.0036 (4)	0.0033 (4)	-0.0005 (4)
C1	0.0199 (6)	0.0161 (6)	0.0319 (7)	0.0025 (4)	0.0027 (5)	0.0008 (5)
C2	0.0236 (6)	0.0199 (6)	0.0396 (8)	-0.0006 (5)	0.0072 (6)	0.0022 (5)
C3	0.0358 (7)	0.0233 (7)	0.0360 (8)	0.0027 (6)	0.0105 (6)	0.0043 (6)
C4	0.0359 (7)	0.0263 (7)	0.0293 (8)	0.0046 (6)	0.0021 (6)	-0.0028 (6)
C5	0.0261 (7)	0.0254 (7)	0.0364 (8)	-0.0013 (5)	0.0016 (6)	-0.0042 (6)
C6	0.0222 (6)	0.0210 (6)	0.0325 (8)	-0.0023 (5)	0.0047 (5)	-0.0005 (5)
C7	0.0242 (6)	0.0181 (6)	0.0333 (8)	-0.0051 (5)	0.0028 (5)	0.0003 (5)
C8	0.0306 (7)	0.0164 (6)	0.0314 (7)	-0.0031 (5)	0.0026 (6)	-0.0005 (5)
C9	0.0228 (6)	0.0169 (6)	0.0348 (8)	-0.0049 (5)	0.0048 (5)	-0.0006 (5)
C10	0.0220 (6)	0.0160 (6)	0.0375 (8)	-0.0023 (5)	0.0031 (5)	0.0013 (5)
C11	0.0165 (6)	0.0193 (6)	0.0341 (7)	0.0031 (5)	0.0022 (5)	-0.0011 (5)
C12	0.0280 (7)	0.0220 (6)	0.0291 (7)	0.0016 (5)	-0.0004 (5)	-0.0014 (5)

Geometric parameters (\AA , $^\circ$)

C11—C12	1.7682 (14)	C5—C6	1.386 (2)
O1—C11	1.2304 (15)	C5—H5	0.9300
N1—C1	1.4157 (18)	C6—H6	0.9300
N1—C7	1.4586 (16)	C7—C8	1.5119 (18)
N1—C10	1.4705 (16)	C7—H7A	0.9700
N2—C11	1.3463 (18)	C7—H7B	0.9700
N2—C9	1.4632 (17)	C8—H8A	0.9700
N2—C8	1.4666 (15)	C8—H8B	0.9700
C1—C6	1.3965 (18)	C9—C10	1.5177 (18)
C1—C2	1.4015 (19)	C9—H9A	0.9700
C2—C3	1.381 (2)	C9—H9B	0.9700
C2—H2	0.9300	C10—H10A	0.9700
C3—C4	1.391 (2)	C10—H10B	0.9700
C3—H3	0.9300	C11—C12	1.5229 (18)
C4—C5	1.383 (2)	C12—H12A	0.9700
C4—H4	0.9300	C12—H12B	0.9700
C1—N1—C7	117.19 (10)	H7A—C7—H7B	108.2
C1—N1—C10	115.21 (10)	N2—C8—C7	110.53 (10)
C7—N1—C10	110.21 (11)	N2—C8—H8A	109.5
C11—N2—C9	119.41 (10)	C7—C8—H8A	109.5
C11—N2—C8	124.38 (11)	N2—C8—H8B	109.5
C9—N2—C8	114.07 (11)	C7—C8—H8B	109.5
C6—C1—C2	118.20 (13)	H8A—C8—H8B	108.1
C6—C1—N1	123.06 (12)	N2—C9—C10	111.12 (10)
C2—C1—N1	118.70 (11)	N2—C9—H9A	109.4

C3—C2—C1	120.76 (13)	C10—C9—H9A	109.4
C3—C2—H2	119.6	N2—C9—H9B	109.4
C1—C2—H2	119.6	C10—C9—H9B	109.4
C2—C3—C4	120.68 (13)	H9A—C9—H9B	108.0
C2—C3—H3	119.7	N1—C10—C9	111.28 (10)
C4—C3—H3	119.7	N1—C10—H10A	109.4
C5—C4—C3	118.83 (14)	C9—C10—H10A	109.4
C5—C4—H4	120.6	N1—C10—H10B	109.4
C3—C4—H4	120.6	C9—C10—H10B	109.4
C4—C5—C6	121.01 (13)	H10A—C10—H10B	108.0
C4—C5—H5	119.5	O1—C11—N2	122.85 (12)
C6—C5—H5	119.5	O1—C11—C12	121.70 (12)
C5—C6—C1	120.52 (13)	N2—C11—C12	115.44 (10)
C5—C6—H6	119.7	C11—C12—C11	111.30 (9)
C1—C6—H6	119.7	C11—C12—H12A	109.4
N1—C7—C8	109.74 (11)	C11—C12—H12A	109.4
N1—C7—H7A	109.7	C11—C12—H12B	109.4
C8—C7—H7A	109.7	C11—C12—H12B	109.4
N1—C7—H7B	109.7	H12A—C12—H12B	108.0
C8—C7—H7B	109.7		
C7—N1—C1—C6	-10.51 (17)	C11—N2—C8—C7	-143.92 (12)
C10—N1—C1—C6	121.65 (13)	C9—N2—C8—C7	52.87 (14)
C7—N1—C1—C2	166.96 (11)	N1—C7—C8—N2	-57.54 (14)
C10—N1—C1—C2	-60.88 (15)	C11—N2—C9—C10	145.88 (11)
C6—C1—C2—C3	-1.11 (18)	C8—N2—C9—C10	-50.00 (14)
N1—C1—C2—C3	-178.71 (11)	C1—N1—C10—C9	166.07 (10)
C1—C2—C3—C4	0.7 (2)	C7—N1—C10—C9	-58.57 (13)
C2—C3—C4—C5	0.3 (2)	N2—C9—C10—N1	52.08 (14)
C3—C4—C5—C6	-0.9 (2)	C9—N2—C11—O1	-6.05 (18)
C4—C5—C6—C1	0.5 (2)	C8—N2—C11—O1	-168.43 (12)
C2—C1—C6—C5	0.48 (19)	C9—N2—C11—C12	174.87 (11)
N1—C1—C6—C5	177.96 (11)	C8—N2—C11—C12	12.49 (17)
C1—N1—C7—C8	-164.61 (10)	O1—C11—C12—C11	-0.20 (15)
C10—N1—C7—C8	61.01 (13)	N2—C11—C12—C11	178.89 (9)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2 \cdots O1 ⁱ	0.93	2.47	3.1850 (16)	134
C9—H9A \cdots O1	0.97	2.38	2.7456 (17)	102
C12—H12B \cdots O1 ⁱⁱ	0.97	2.43	3.3426 (16)	157
C5—H5 \cdots Cg2 ⁱⁱⁱ	0.93	3.25	3.7651 (15)	117
C8—H8B \cdots Cg2 ^{iv}	0.97	3.09	4.0393 (12)	168
C12—H12A \cdots Cg2 ^{iv}	0.97	3.04	3.6878	125

Symmetry codes: (i) $x, -y-1/2, z+1/2$; (ii) $-x+1, y+1/2, -z+1/2$; (iii) $-x+2, y-1/2, -z+1/2$; (iv) $x, -y-1/2, z-3/2$.

Fig. 1

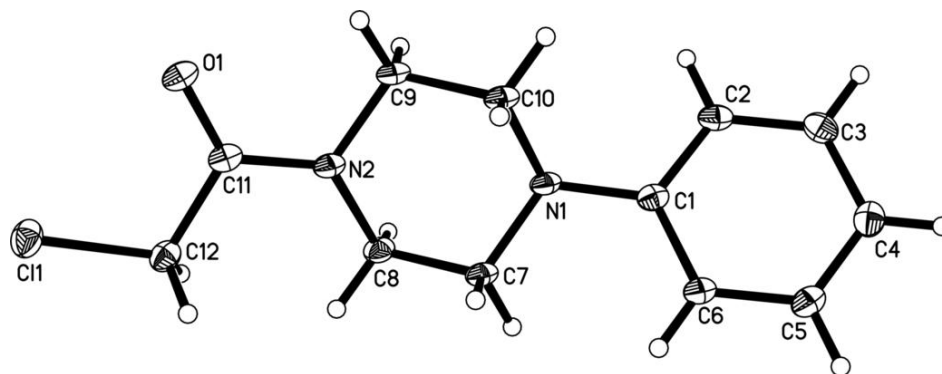


Fig. 2

