Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-Methyl-3-phenylpiperazine

Yue Meng,[a] Wei-Xiao Hu[a]* and Ke Wu[b]

[a]College of Pharmaceutical Science, Zhejiang University of Technology, Hangzhou 310032, People's Republic of China, and [b]Shanghai Jiubang Chemical Co. Ltd, Jinshan, Shanghai 201507, People's Republic of China
Correspondence e-mail: huyang@mail.hz.zj.cn

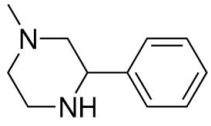
Received 16 April 2007; accepted 18 April 2007

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.122; data-to-parameter ratio = 19.0.

The title compound, $C_{11}H_{16}N_2$, is an important intermediate in the preparation of the antidepressant mirtazapine. The piperazine ring has a regular chair conformation and the benzene ring is attached at an equatorial position. In the crystal structure, there is only a weak N—H···N hydrogen bond.

Related literature

For related literature, see: Subba Rao & Subrahmanyam (2002); Munday (2001).



Experimental

Crystal data

$C_{11}H_{16}N_2$
M_r = 176.26
Monoclinic, $P2_1/c$
a = 10.6225 (18) Å
b = 5.9284 (10) Å
c = 18.392 (3) Å
β = 116.148 (7)°
V = 1039.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 296 (2) K
0.25 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.983, T_{max} = 0.987
6314 measured reflections
2350 independent reflections
1803 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.122
S = 1.03
2350 reflections
124 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···N4^{i}	0.881 (13)	2.646 (12)	3.5190 (16)	170.9 (13)

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

Data collection: *SMART* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2005); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2345).

References

Bruker (2005). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Munday, R. (2001). *Nutr. Cancer*, **40**, 205–210.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Subba Rao, K. V. & Subrahmanyam, M. (2002). *Photochem. Photobiol. Sci.* **1**, 597–599.

  doi:10.1107/S1600536807019241

supplementary materials

Acta Cryst. (2007). E**63**, o3048 [**doi:10.1107/S1600536807019241**]

1-Methyl-3-phenylpiperazine

Y. Meng, W.-X. Hu and K. Wu

Comment

Piperazine and its derivates are important intermediates because these compounds can be used as starting materials in pharmaceutial and agrochemical industries (Subba Rao, & Subrahmanyam, 2002). 1-Methyl-3-phenylpiperazine is an important preparation intermediate for mirtazapine, which is useful as an antidepressant (Munday, 2001). In our work on the preparation of piperazine derivates, the title compound was obtained.

The piperazaine ring has a regular chair conformation. The benzene ring is attached in an equatorial position at the piperazine ring. In the crystal structure, there is just a weak N—H···N hydrogen bond.

Experimental

4-Methyl-2-phenyl-1-tosylpiperazine (6 g, 0.018 mol) was dissolved in water (5 ml) and sulfuric acid (98%, 15 ml) while heating to 100°C. After half an hour at 100–110°C, the reaction mixture was poured into water (150 ml). Then, the solution was alkalized to pH 13 with sodium hydroxide (45%). The product was extracted twice with 30 ml e ther, and the collected organic layers were combined together. Thereafter, the solvent was removed *in vacuo* to give a white power (1.5 g). The solid product was dissolved in n-hexane, the solution was evaporated gradually at room temperature to afford single crystals of the title compound. *M*.p. 329.4–330.3 K.

Refinement

H atoms bonded to C were placed in calculated positions with C—H ranging from 0.093Å to 0.98 Å and with U_{iso}(H) = 1.2U_{eq}(C) or U_{iso}(H) = 1.5U_{eq}(C_{methyl}). The H atom bonded to N was refined freely with a distance restraint of 0.88 (1) Å.

Figures

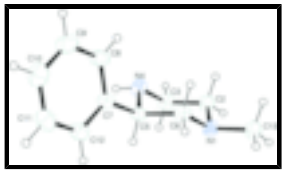


Fig. 1. The molecular structure of (I) with 30% probability displacement ellipsoids.

1-Methyl-3-phenylpiperazine

Crystal data

$C_{11}H_{16}N_2$

M_r = 176.26

F_{000} = 384

D_x = 1.126 Mg m^{-3}

Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2475 reflections
$a = 10.6225$ (18) Å	$\theta = 2.5$–27.5°
$b = 5.9284$ (10) Å	$\mu = 0.07$ mm^{-1}
$c = 18.392$ (3) Å	$T = 296$ (2) K
$\beta = 116.148$ (7)°	Block, colorless
$V = 1039.7$ (3) Å^3	0.25 × 0.20 × 0.15 mm
$Z = 4$	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	2350 independent reflections
Radiation source: fine-focus sealed tube	1803 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.023$
$T = 296(2)$ K	$\theta_{max} = 27.5^\circ$
φ and ω scans	$\theta_{min} = 2.1^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -12 \rightarrow 13$
$T_{min} = 0.983$, $T_{max} = 0.987$	$k = -7 \rightarrow 7$
6314 measured reflections	$l = -23 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.122$	$w = 1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.1409P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
2350 reflections	$\Delta\rho_{max} = 0.16$ e Å^{-3}
124 parameters	$\Delta\rho_{min} = -0.15$ e Å^{-3}
1 restraint	Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.062 (6)

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 $F^2 > 2\text{sigma}(F^2)$ is used only for calculat-

ing *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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.79168 (10)	−0.08650 (16)	0.44471 (6)	0.0466 (3)
C2	0.64369 (13)	−0.1219 (2)	0.42336 (8)	0.0530 (3)
H2A	0.6315	−0.1390	0.4723	0.064*
H2B	0.6118	−0.2595	0.3920	0.064*
C3	0.55695 (12)	0.0743 (2)	0.37478 (7)	0.0515 (3)
H3A	0.4586	0.0460	0.3596	0.062*
H3B	0.5842	0.2103	0.4074	0.062*
N4	0.57786 (10)	0.10558 (18)	0.30189 (6)	0.0475 (3)
H4	0.5293 (13)	0.224 (2)	0.2752 (8)	0.061 (4)*
C5	0.72649 (11)	0.14966 (19)	0.32436 (7)	0.0430 (3)
H5	0.7560	0.2815	0.3601	0.052*
C6	0.80955 (12)	−0.0534 (2)	0.37140 (7)	0.0467 (3)
H6A	0.7781	−0.1871	0.3377	0.056*
H6B	0.9081	−0.0312	0.3857	0.056*
C7	0.75175 (11)	0.19779 (19)	0.25148 (7)	0.0439 (3)
C8	0.69087 (14)	0.0681 (2)	0.18202 (8)	0.0589 (4)
H8	0.6319	−0.0504	0.1798	0.071*
C9	0.71643 (16)	0.1120 (3)	0.11590 (9)	0.0694 (4)
H9	0.6741	0.0236	0.0696	0.083*
C10	0.80360 (15)	0.2849 (3)	0.11811 (9)	0.0686 (4)
H10	0.8207	0.3144	0.0736	0.082*
C11	0.86525 (16)	0.4135 (3)	0.18661 (10)	0.0681 (4)
H11	0.9250	0.5306	0.1887	0.082*
C12	0.83970 (13)	0.3713 (2)	0.25268 (8)	0.0542 (3)
H12	0.8822	0.4608	0.2987	0.065*
C13	0.87606 (14)	−0.2752 (2)	0.49174 (8)	0.0638 (4)
H13A	0.8412	−0.4128	0.4621	0.096*
H13B	0.8708	−0.2837	0.5425	0.096*
H13C	0.9718	−0.2535	0.5016	0.096*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0440 (5)	0.0480 (6)	0.0442 (5)	0.0054 (4)	0.0161 (4)	0.0001 (4)
C2	0.0516 (7)	0.0573 (8)	0.0523 (7)	0.0003 (5)	0.0248 (6)	0.0050 (5)
C3	0.0451 (6)	0.0630 (8)	0.0525 (7)	0.0075 (5)	0.0272 (5)	0.0056 (6)
N4	0.0409 (5)	0.0579 (6)	0.0457 (6)	0.0068 (4)	0.0208 (4)	0.0047 (5)
C5	0.0430 (6)	0.0434 (6)	0.0448 (6)	−0.0020 (4)	0.0213 (5)	−0.0076 (5)
C6	0.0404 (6)	0.0514 (7)	0.0482 (6)	0.0036 (5)	0.0193 (5)	−0.0068 (5)
C7	0.0421 (6)	0.0442 (6)	0.0485 (6)	0.0011 (5)	0.0229 (5)	−0.0035 (5)
C8	0.0656 (8)	0.0626 (8)	0.0582 (8)	−0.0176 (6)	0.0360 (6)	−0.0153 (6)
C9	0.0757 (9)	0.0865 (11)	0.0552 (8)	−0.0110 (8)	0.0371 (7)	−0.0161 (7)

C10	0.0716 (9)	0.0850 (10)	0.0655 (9)	0.0016 (8)	0.0450 (7)	0.0075 (8)
C11	0.0677 (9)	0.0678 (9)	0.0827 (10)	−0.0114 (7)	0.0457 (8)	0.0045 (8)
C12	0.0529 (7)	0.0516 (7)	0.0619 (8)	−0.0055 (5)	0.0287 (6)	−0.0056 (6)
C13	0.0615 (8)	0.0569 (8)	0.0581 (8)	0.0116 (6)	0.0128 (6)	0.0034 (6)

Geometric parameters (Å, °)

N1—C6	1.4553 (15)	C6—H6B	0.9700
N1—C13	1.4559 (15)	C7—C12	1.3830 (16)
N1—C2	1.4580 (15)	C7—C8	1.3833 (17)
C2—C3	1.5082 (17)	C8—C9	1.3826 (18)
C2—H2A	0.9700	C8—H8	0.9300
C2—H2B	0.9700	C9—C10	1.370 (2)
C3—N4	1.4642 (15)	C9—H9	0.9300
C3—H3A	0.9700	C10—C11	1.368 (2)
C3—H3B	0.9700	C10—H10	0.9300
N4—C5	1.4686 (14)	C11—C12	1.3798 (19)
N4—H4	0.882 (12)	C11—H11	0.9300
C5—C7	1.5058 (16)	C12—H12	0.9300
C5—C6	1.5178 (16)	C13—H13A	0.9599
C5—H5	0.9800	C13—H13B	0.9599
C6—H6A	0.9700	C13—H13C	0.9599
C6—N1—C13	110.88 (10)	N1—C6—H6B	109.5
C6—N1—C2	109.59 (9)	C5—C6—H6B	109.5
C13—N1—C2	110.96 (10)	H6A—C6—H6B	108.1
N1—C2—C3	110.68 (10)	C12—C7—C8	117.76 (11)
N1—C2—H2A	109.5	C12—C7—C5	120.72 (10)
C3—C2—H2A	109.5	C8—C7—C5	121.51 (10)
N1—C2—H2B	109.5	C9—C8—C7	120.96 (12)
C3—C2—H2B	109.5	C9—C8—H8	119.5
H2A—C2—H2B	108.1	C7—C8—H8	119.5
N4—C3—C2	109.91 (10)	C10—C9—C8	120.48 (13)
N4—C3—H3A	109.7	C10—C9—H9	119.8
C2—C3—H3A	109.7	C8—C9—H9	119.8
N4—C3—H3B	109.7	C11—C10—C9	119.18 (13)
C2—C3—H3B	109.7	C11—C10—H10	120.4
H3A—C3—H3B	108.2	C9—C10—H10	120.4
C3—N4—C5	110.06 (9)	C10—C11—C12	120.65 (13)
C3—N4—H4	108.8 (9)	C10—C11—H11	119.7
C5—N4—H4	108.0 (9)	C12—C11—H11	119.7
N4—C5—C7	111.99 (9)	C11—C12—C7	120.97 (12)
N4—C5—C6	107.58 (9)	C11—C12—H12	119.5
C7—C5—C6	111.77 (9)	C7—C12—H12	119.5
N4—C5—H5	108.5	N1—C13—H13A	109.5
C7—C5—H5	108.5	N1—C13—H13B	109.5
C6—C5—H5	108.5	H13A—C13—H13B	109.5
N1—C6—C5	110.77 (9)	N1—C13—H13C	109.5
N1—C6—H6A	109.5	H13A—C13—H13C	109.5
C5—C6—H6A	109.5	H13B—C13—H13C	109.5

C6—N1—C2—C3	−57.31 (13)	C6—C5—C7—C12	−102.47 (13)
C13—N1—C2—C3	179.91 (10)	N4—C5—C7—C8	−44.73 (15)
N1—C2—C3—N4	57.50 (14)	C6—C5—C7—C8	76.06 (14)
C2—C3—N4—C5	−59.62 (13)	C12—C7—C8—C9	−0.5 (2)
C3—N4—C5—C7	−176.33 (9)	C5—C7—C8—C9	−179.09 (12)
C3—N4—C5—C6	60.47 (12)	C7—C8—C9—C10	0.4 (2)
C13—N1—C6—C5	−177.51 (9)	C8—C9—C10—C11	0.1 (2)
C2—N1—C6—C5	59.66 (12)	C9—C10—C11—C12	−0.4 (2)
N4—C5—C6—N1	−60.92 (11)	C10—C11—C12—C7	0.2 (2)
C7—C5—C6—N1	175.75 (9)	C8—C7—C12—C11	0.21 (19)
N4—C5—C7—C12	136.73 (11)	C5—C7—C12—C11	178.80 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···N4^{i}	0.881 (13)	2.646 (12)	3.5190 (16)	170.9 (13)

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2.

Fig. 1