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2-Chloromethyl-1-methyl-1,3-benzimidazole

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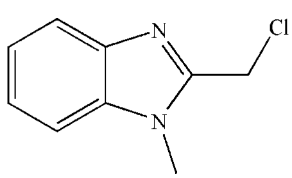
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.122; data-to-parameter ratio = 14.0.

The title compound, $C_9H_9ClN_2$, was prepared from the reaction of *N*-methylbenzene-1,2-diamine and 2-chloroacetic acid in boiling 6 *M* hydrochloric acid. The benzimidazole unit is approximately planar, the largest deviation from the mean plane being 0.008 (1) Å. The Cl atom is displaced by 1.667 (2) Å from this plane. The methyl group is statistically disordered with equal occupancy.

Related literature

For the biological activity of benzimidazoles, see: Refaat (2010); Laryea *et al.* (2010); Horton *et al.* (2003); Ries *et al.* (2003); Spasov *et al.* (1999); Matsui *et al.* (1994); Porcari *et al.* (1998); Rath *et al.* (1997); Migawa *et al.* (1998). For a description of the Cambridge Structural Database, see: Allen (2002).



1

Experimental

Crystal data

$C_9H_9ClN_2$
M_r = 180.63
Triclinic, $P\bar{1}$
a = 6.607 (2) Å
b = 8.168 (2) Å
c = 8.925 (3) Å
α = 84.566 (3)°
β = 79.682 (4)°
γ = 68.134 (4)°
V = 439.6 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 296 K
0.37 × 0.29 × 0.18 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.874, T_{max} = 0.937
2191 measured reflections
1523 independent reflections
1361 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.122
S = 1.06
1523 reflections
109 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2705).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bruker (2002). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Horton, D. A., Bourne, G. T. & Sinythe, M. L. (2003). *Chem. Rev.* **103**, 893–930.
Laryea, D., Gullbo, J., Isaksson, A., Larsson, R. & Nygren, P. (2010). *Anti-Cancer Drugs*, **21**, 33–42.
Matsui, T., Nakamura, Y., Ishikawa, H., Matsuura, A. & Kobayashi, F. (1994). *Jpn J. Pharmacol.* **64**, 115–124.
Migawa, M. T., Girardet, J. L., Walker, J. A., Koszalka, G. W., Chamberlain, S. D., Drach, J. C. & Townsend, L. B. (1998). *J. Med. Chem.* **41**, 1242–1251.
Porcari, A. R., Devivar, R. V., Kucera, L. S., Drach, J. C. & Townsend, L. B. (1998). *J. Med. Chem.* **41**, 1252–1262.
Rath, T., Morningstar, M. L., Boyer, P. L., Hughes, S. M., Buckheitjr, R. W. & Michejda, C. J. (1997). *J. Med. Chem.* **40**, 4199–4207.
Refaat, H. M. (2010). *Eur. J. Med. Chem.* **45**, 2949–2956.
Ries, U. J., Priepke, H. W. M., Hauel, N. H., Haaksma, E. E. J., Stassen, J. M., Wienen, W. & Nar, H. (2003). *Bioorg. Med. Chem. Lett.* **13**, 2297–2321.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spasov, A. A., Yozhitsa, I. N., Bugaeva, L. I. & Anisimova, V. A. (1999). *Pharm. Chem. J.* **33**, 232–243.

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2-Chloromethyl-1-methyl-1,3-benzimidazole

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S1. Comment

Benzimidazole and its derivatives are present in various bioactive compounds possessing antiparasitic, antimicrobial, and antifungal properties (Refaat, 2010; Laryea *et al.*, 2010; Horton *et al.*, 2003; Ries *et al.*, 2003; Spasov *et al.*, 1999; Matsui *et al.* 1994). They also play very important role in the synthesis of many natural products and synthetic drugs. Compounds possessing the benzimidazole moiety express significant activity against several viruses such as HIV (Porcari, *et al.*, 1998; Rath, *et al.*, 1997), Herpes (HSV-1) (Migawa, *et al.*, 1998), human cytomegalovirus (HCMV) and influenza. As a part of our ongoing investigations of benzimidazole derivatives, the title compound was synthesized and its crystal structure is reported herein.

The two fused rings forming the benzimidazole moiety are planar with the largest deviation from the mean plane being 0.008 (1)Å. The Cl atom is out of this plane by -1.667 (2)Å (Fig. 1). The methyl group is statistically disordered. The distances and angles within the methyl-benzimidazole agree with the values reported in the literature (43 hits found in the Cambridge Structural Database, Conquest, version 1.13; Allen, 2002).

The packing is only stabilized by electrostatic and van der Waals interactions.

S2. Experimental

For the preparation of the title compound *N*-methylbenzene-1,2-diamine (5.0 mmol) and 2-chloroacetic acid(6.0 mmol) was dissolved in 6 N hydrochloric acid (30.0 ml) and refluxed for 6 h. The reaction mixture was cooled in to room temperature, then neutralized with aqueous sodium hydroxide. The precipitate was filtered off and washed with cold water. The crude product was crystallized from ethanol to give white block-like crystals of the title compound.

S3. Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.96 Å (methyl), 0.97 Å (methylene) and 0.93 Å (aromatic) with $U_{iso}(H) = 1.2U_{eq}$(C, aromatic or methylene) and $U_{iso}(H) = 1.5U_{eq}$(C, methyl).

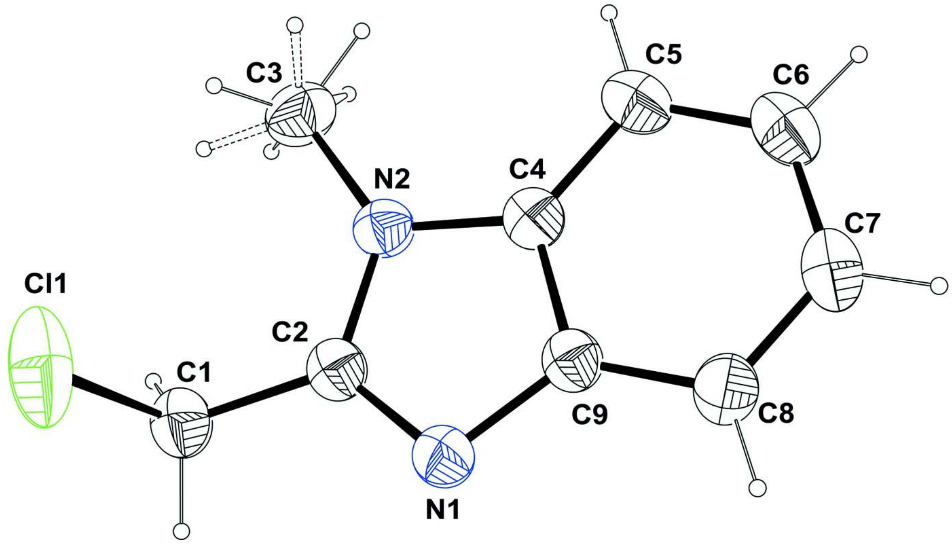

Figure 1

The asymmetric unit of (1) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

2-Chloromethyl-1-methyl-1,3-benzimidazole

Crystal data

$C_9H_9ClN_2$
M_r = 180.63
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.607 (2) Å
b = 8.168 (2) Å
c = 8.925 (3) Å
α = 84.566 (3)°
β = 79.682 (4)°
γ = 68.134 (4)°
V = 439.6 (2) Å^3
Z = 2
F(000) = 188
D_x = 1.365 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2191 reflections
θ = 2.3–25.1°
μ = 0.38 mm^{-1}
T = 296 K
Block, white
0.37 × 0.29 × 0.18 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.874, T_{max} = 0.937
2191 measured reflections
1523 independent reflections
1361 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 25.1°, θ_{min} = 2.3°
h = −7→5
k = −9→9
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.122
S = 1.06
1523 reflections
109 parameters
0 restraints
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

$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 0.1656P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.2259 (4)	0.4372 (3)	0.6995 (2)	0.0610 (6)	
H1A	0.3424	0.4781	0.7109	0.073*	
H1B	0.0985	0.5393	0.6790	0.073*	
C2	0.1665 (3)	0.3458 (2)	0.8432 (2)	0.0498 (5)	
C3	−0.2214 (4)	0.3954 (3)	0.8004 (3)	0.0666 (6)	
H3A	−0.3405	0.3638	0.8563	0.100*	0.50
H3B	−0.1788	0.3444	0.7017	0.100*	0.50
H3C	−0.2683	0.5215	0.7888	0.100*	0.50
H3D	−0.1846	0.4560	0.7082	0.100*	0.50
H3E	−0.3463	0.4754	0.8628	0.100*	0.50
H3F	−0.2568	0.2983	0.7757	0.100*	0.50
C4	−0.0294 (3)	0.2393 (2)	1.0212 (2)	0.0488 (5)	
C5	−0.1859 (4)	0.1843 (3)	1.1144 (3)	0.0600 (6)	
H5	−0.3242	0.2069	1.0885	0.072*	
C6	−0.1250 (4)	0.0947 (3)	1.2471 (3)	0.0675 (6)	
H6	−0.2258	0.0574	1.3137	0.081*	
C7	0.0842 (4)	0.0581 (3)	1.2848 (3)	0.0673 (6)	
H7	0.1198	−0.0041	1.3751	0.081*	
C8	0.2384 (4)	0.1119 (3)	1.1915 (2)	0.0605 (6)	
H8	0.3773	0.0871	1.2173	0.073*	
C9	0.1803 (3)	0.2046 (2)	1.0572 (2)	0.0495 (5)	
Cl1	0.31643 (17)	0.29247 (10)	0.54306 (8)	0.1088 (4)	
N1	0.3008 (3)	0.2749 (2)	0.94299 (19)	0.0535 (4)	
N2	−0.0342 (3)	0.3294 (2)	0.88264 (18)	0.0498 (4)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0686 (13)	0.0587 (12)	0.0545 (12)	−0.0238 (11)	−0.0075 (10)	0.0029 (10)
C2	0.0509 (11)	0.0472 (10)	0.0489 (11)	−0.0161 (8)	−0.0037 (8)	−0.0048 (8)
C3	0.0599 (13)	0.0680 (14)	0.0736 (15)	−0.0195 (11)	−0.0252 (11)	0.0030 (11)

C4	0.0505 (10)	0.0436 (10)	0.0495 (11)	−0.0146 (8)	−0.0031 (8)	−0.0077 (8)
C5	0.0550 (12)	0.0568 (12)	0.0676 (14)	−0.0235 (10)	0.0018 (10)	−0.0068 (10)
C6	0.0741 (15)	0.0634 (13)	0.0630 (14)	−0.0314 (12)	0.0101 (11)	−0.0041 (11)
C7	0.0840 (16)	0.0602 (13)	0.0512 (12)	−0.0235 (12)	−0.0033 (11)	0.0048 (10)
C8	0.0609 (13)	0.0639 (13)	0.0540 (12)	−0.0198 (10)	−0.0100 (10)	0.0006 (10)
C9	0.0513 (11)	0.0483 (10)	0.0474 (10)	−0.0173 (9)	−0.0040 (8)	−0.0042 (8)
Cl1	0.1675 (9)	0.0837 (5)	0.0523 (4)	−0.0326 (5)	0.0147 (4)	−0.0069 (3)
N1	0.0494 (9)	0.0588 (10)	0.0518 (10)	−0.0203 (8)	−0.0065 (7)	0.0006 (8)
N2	0.0465 (9)	0.0509 (9)	0.0520 (9)	−0.0170 (7)	−0.0082 (7)	−0.0028 (7)

Geometric parameters (Å, º)

C1—C2	1.488 (3)	C4—N2	1.377 (3)
C1—Cl1	1.786 (2)	C4—C5	1.389 (3)
C1—H1A	0.9700	C4—C9	1.398 (3)
C1—H1B	0.9700	C5—C6	1.373 (3)
C2—N1	1.310 (3)	C5—H5	0.9300
C2—N2	1.363 (3)	C6—C7	1.397 (4)
C3—N2	1.453 (3)	C6—H6	0.9300
C3—H3A	0.9600	C7—C8	1.373 (3)
C3—H3B	0.9600	C7—H7	0.9300
C3—H3C	0.9600	C8—C9	1.390 (3)
C3—H3D	0.9600	C8—H8	0.9300
C3—H3E	0.9600	C9—N1	1.393 (3)
C3—H3F	0.9600		
C2—C1—Cl1	110.86 (15)	H3B—C3—H3F	56.3
C2—C1—H1A	109.5	H3C—C3—H3F	141.1
Cl1—C1—H1A	109.5	H3D—C3—H3F	109.5
C2—C1—H1B	109.5	H3E—C3—H3F	109.5
Cl1—C1—H1B	109.5	N2—C4—C5	131.59 (19)
H1A—C1—H1B	108.1	N2—C4—C9	105.69 (17)
N1—C2—N2	114.14 (18)	C5—C4—C9	122.71 (19)
N1—C2—C1	123.36 (19)	C6—C5—C4	116.3 (2)
N2—C2—C1	122.49 (18)	C6—C5—H5	121.9
N2—C3—H3A	109.5	C4—C5—H5	121.9
N2—C3—H3B	109.5	C5—C6—C7	121.9 (2)
H3A—C3—H3B	109.5	C5—C6—H6	119.1
N2—C3—H3C	109.5	C7—C6—H6	119.1
H3A—C3—H3C	109.5	C8—C7—C6	121.5 (2)
H3B—C3—H3C	109.5	C8—C7—H7	119.3
N2—C3—H3D	109.5	C6—C7—H7	119.3
H3A—C3—H3D	141.1	C7—C8—C9	117.9 (2)
H3B—C3—H3D	56.3	C7—C8—H8	121.1
H3C—C3—H3D	56.3	C9—C8—H8	121.1
N2—C3—H3E	109.5	C8—C9—N1	130.36 (19)
H3A—C3—H3E	56.3	C8—C9—C4	119.77 (19)
H3B—C3—H3E	141.1	N1—C9—C4	109.87 (17)

H3C—C3—H3E	56.3	C2—N1—C9	104.17 (16)
H3D—C3—H3E	109.5	C2—N2—C4	106.13 (16)
N2—C3—H3F	109.5	C2—N2—C3	128.34 (18)
H3A—C3—H3F	56.3	C4—N2—C3	125.52 (17)