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8-Chloro-6,11-dihydro-5*H*-benzo[5,6]-cyclohepta[1,2-*b*]pyridine

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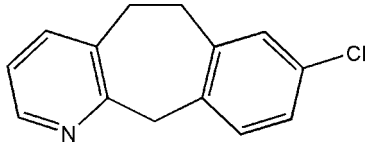
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.115; data-to-parameter ratio = 14.0.

The title compound, $C_{14}H_{12}ClN$, has a tricyclic fused-ring system composed of a benzene ring, a pyridine ring and a central nonplanar seven-membered ring.

Related literature

For related literature, see: Haria *et al.* (1994); Lin *et al.* (2005); Stampa *et al.* (2000).



Experimental

Crystal data

$C_{14}H_{12}ClN$
M_r = 229.70
Monoclinic, $P2_1/c$
a = 10.609 (3) Å
b = 13.588 (3) Å
c = 8.1997 (18) Å
β = 103.841 (4)°
V = 1147.8 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 294 (2) K
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.937, T_{max} = 0.948
8106 measured reflections
2027 independent reflections
1638 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.115
S = 1.03
2027 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2293).

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supplementary materials

Acta Cryst. (2007). E**63**, o4131 [**doi:10.1107/S1600536807045813**]

8-Chloro-6,11-dihydro-5*H*-benzo[5,6]cyclohepta[1,2-*b*]pyridine

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Comment

Loratadine is a long-acting tricyclic antihistamine with selective peripheral histamine H1-receptor antagonistic activity (Haria *et al.*, 1994). The title compound, (I), was unexpectedly obtained in the preparation of loratadine by the cross reductive coupling between 8-chloro-10,11-dihydro-4-aza-5*H*- dibenzo[a,d] cyclohepten-5-one and ethyl 4-oxopiperidine-1-carboxylate. We report here the crystal structure of (I) (Fig. 1).

The molecule contains a tricyclic fused-ring system composed of a benzene ring, a pyridine ring and a central non-planar seven-membered ring whose conformation was found in a similar system in 8-chloro-10,11-dihydro-4-aza-5*H*- dibenzo[a,d] cyclohepten-5-one (Lin *et al.*, 2005).

Experimental

The title compound was synthesized according to the method described by Stampa *et al.* (2000). Colorless blocks of (I) were grown by slow evaporation of a methanol solution (m.p. 372–374 K).

Refinement

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å), and refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier) or 1.5$_{eq}$(methyl groups).

Figures

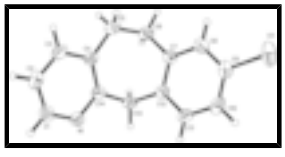

Fig. 1. A view of the molecular of (I). Displacement ellopsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

8-Chloro-6,11-dihydro-5*H*-benzo[5,6]cyclohepta[1,2-*b*]pyridine

Crystal data

$C_{14}H_{12}ClN$	F_{000} = 480
M_r = 229.70	D_x = 1.329 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 372-374 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.609 (3) Å	Cell parameters from 3781 reflections
b = 13.588 (3) Å	θ = 2.5–26.2º

c = 8.1997 (18) Å	μ = 0.30 mm^{-1}
β = 103.841 (4)º	T = 294 (2) K
V = 1147.8 (5) Å^3	Block, colorless
Z = 4	0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2027 independent reflections
Radiation source: fine-focus sealed tube	1638 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.021
T = 294(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -12 \rightarrow 12$
T_{min} = 0.937, T_{max} = 0.948	$k = -16 \rightarrow 16$
8106 measured reflections	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.043	H-atom parameters constrained
$wR(F^2)$ = 0.115	$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.6828P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.005
2027 reflections	$\Delta\rho_{max}$ = 0.44 e Å^{-3}
145 parameters	$\Delta\rho_{min}$ = −0.55 e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl1	0.41484 (7)	0.65806 (6)	0.24910 (9)	0.0848 (3)

N1	1.09198 (17)	0.58490 (13)	0.8124 (2)	0.0509 (5)
C1	1.1725 (2)	0.65826 (18)	0.8727 (3)	0.0582 (6)
H1	1.2574	0.6547	0.8611	0.070*
C2	1.1368 (3)	0.73863 (18)	0.9510 (3)	0.0632 (6)
H2	1.1952	0.7893	0.9897	0.076*
C3	1.0125 (3)	0.74217 (18)	0.9706 (3)	0.0606 (6)
H3	0.9862	0.7960	1.0242	0.073*
C4	0.9251 (2)	0.66698 (15)	0.9120 (2)	0.0485 (5)
C5	0.7909 (2)	0.6772 (2)	0.9426 (3)	0.0687 (7)
H5A	0.7602	0.7428	0.9069	0.082*
H5B	0.8000	0.6742	1.0630	0.082*
C6	0.6847 (2)	0.60588 (19)	0.8626 (3)	0.0593 (6)
H6A	0.7029	0.5426	0.9180	0.071*
H6B	0.6030	0.6294	0.8813	0.071*
C7	0.6700 (2)	0.59178 (15)	0.6771 (3)	0.0456 (5)
C8	0.5626 (2)	0.62711 (16)	0.5612 (3)	0.0524 (5)
H8	0.4978	0.6605	0.5976	0.063*
C9	0.5514 (2)	0.61297 (17)	0.3926 (3)	0.0550 (6)
C10	0.6447 (2)	0.56352 (18)	0.3355 (3)	0.0592 (6)
H10	0.6358	0.5537	0.2211	0.071*
C11	0.7528 (2)	0.52821 (16)	0.4512 (3)	0.0534 (5)
H11	0.8171	0.4950	0.4135	0.064*
C12	0.76631 (19)	0.54171 (14)	0.6219 (2)	0.0436 (5)
C13	0.8840 (2)	0.50542 (16)	0.7484 (3)	0.0511 (5)
H13A	0.9334	0.4621	0.6932	0.061*
H13B	0.8565	0.4676	0.8341	0.061*
C14	0.9696 (2)	0.58905 (15)	0.8304 (2)	0.0440 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0748 (5)	0.0773 (5)	0.0845 (5)	−0.0125 (3)	−0.0162 (4)	0.0282 (4)
N1	0.0510 (10)	0.0547 (11)	0.0462 (10)	0.0076 (9)	0.0102 (8)	0.0002 (8)
C1	0.0524 (13)	0.0702 (16)	0.0489 (12)	−0.0005 (12)	0.0059 (10)	0.0044 (11)
C2	0.0681 (16)	0.0580 (14)	0.0560 (14)	−0.0079 (12)	0.0000 (12)	−0.0047 (11)
C3	0.0727 (16)	0.0530 (13)	0.0503 (13)	0.0083 (12)	0.0033 (11)	−0.0131 (10)
C4	0.0569 (13)	0.0498 (12)	0.0368 (10)	0.0080 (10)	0.0068 (9)	−0.0032 (9)
C5	0.0668 (16)	0.0806 (17)	0.0610 (15)	0.0077 (13)	0.0202 (12)	−0.0243 (13)
C6	0.0608 (14)	0.0718 (15)	0.0504 (13)	0.0045 (12)	0.0237 (11)	−0.0025 (11)
C7	0.0499 (12)	0.0434 (11)	0.0461 (11)	−0.0013 (9)	0.0165 (9)	0.0010 (9)
C8	0.0491 (12)	0.0462 (12)	0.0627 (14)	−0.0005 (10)	0.0150 (11)	0.0015 (10)
C9	0.0557 (13)	0.0485 (12)	0.0563 (13)	−0.0113 (10)	0.0045 (11)	0.0103 (10)
C10	0.0721 (16)	0.0648 (15)	0.0396 (11)	−0.0208 (12)	0.0110 (11)	0.0002 (10)
C11	0.0591 (13)	0.0521 (13)	0.0531 (13)	−0.0086 (10)	0.0221 (11)	−0.0125 (10)
C12	0.0505 (12)	0.0356 (10)	0.0456 (11)	−0.0033 (9)	0.0135 (9)	−0.0015 (8)
C13	0.0559 (13)	0.0407 (11)	0.0567 (13)	0.0070 (9)	0.0135 (10)	−0.0045 (9)
C14	0.0513 (12)	0.0434 (11)	0.0358 (10)	0.0082 (9)	0.0075 (9)	0.0043 (8)

Geometric parameters (Å, °)

Cl1—C9	1.745 (2)	C6—H6A	0.9700
N1—C1	1.329 (3)	C6—H6B	0.9700
N1—C14	1.342 (3)	C7—C8	1.384 (3)
C1—C2	1.365 (3)	C7—C12	1.390 (3)
C1—H1	0.9300	C8—C9	1.373 (3)
C2—C3	1.367 (4)	C8—H8	0.9300
C2—H2	0.9300	C9—C10	1.368 (3)
C3—C4	1.386 (3)	C10—C11	1.387 (3)
C3—H3	0.9300	C10—H10	0.9300
C4—C14	1.394 (3)	C11—C12	1.385 (3)
C4—C5	1.510 (3)	C11—H11	0.9300
C5—C6	1.511 (3)	C12—C13	1.503 (3)
C5—H5A	0.9700	C13—C14	1.509 (3)
C5—H5B	0.9700	C13—H13A	0.9700
C6—C7	1.504 (3)	C13—H13B	0.9700
C1—N1—C14	118.67 (19)	C8—C7—C6	121.29 (19)
N1—C1—C2	123.3 (2)	C12—C7—C6	119.02 (19)
N1—C1—H1	118.3	C9—C8—C7	120.1 (2)
C2—C1—H1	118.3	C9—C8—H8	120.0
C1—C2—C3	117.9 (2)	C7—C8—H8	120.0
C1—C2—H2	121.1	C10—C9—C8	121.2 (2)
C3—C2—H2	121.1	C10—C9—Cl1	119.61 (18)
C2—C3—C4	121.2 (2)	C8—C9—Cl1	119.18 (19)
C2—C3—H3	119.4	C9—C10—C11	118.9 (2)
C4—C3—H3	119.4	C9—C10—H10	120.5
C3—C4—C14	116.8 (2)	C11—C10—H10	120.5
C3—C4—C5	117.0 (2)	C12—C11—C10	120.9 (2)
C14—C4—C5	126.2 (2)	C12—C11—H11	119.5
C4—C5—C6	120.24 (19)	C10—C11—H11	119.5
C4—C5—H5A	107.3	C11—C12—C7	119.17 (19)
C6—C5—H5A	107.3	C11—C12—C13	121.49 (19)
C4—C5—H5B	107.3	C7—C12—C13	119.32 (18)
C6—C5—H5B	107.3	C12—C13—C14	111.88 (17)
H5A—C5—H5B	106.9	C12—C13—H13A	109.2
C7—C6—C5	113.98 (19)	C14—C13—H13A	109.2
C7—C6—H6A	108.8	C12—C13—H13B	109.2
C5—C6—H6A	108.8	C14—C13—H13B	109.2
C7—C6—H6B	108.8	H13A—C13—H13B	107.9
C5—C6—H6B	108.8	N1—C14—C4	122.2 (2)
H6A—C6—H6B	107.7	N1—C14—C13	114.49 (17)
C8—C7—C12	119.68 (19)	C4—C14—C13	123.27 (19)
C14—N1—C1—C2	−0.7 (3)	C10—C11—C12—C7	−0.3 (3)
N1—C1—C2—C3	1.4 (4)	C10—C11—C12—C13	−178.9 (2)
C1—C2—C3—C4	−0.4 (4)	C8—C7—C12—C11	0.1 (3)
C2—C3—C4—C14	−1.0 (3)	C6—C7—C12—C11	179.7 (2)
C2—C3—C4—C5	179.0 (2)	C8—C7—C12—C13	178.75 (19)

C3—C4—C5—C6	171.7 (2)	C6—C7—C12—C13	−1.6 (3)
C14—C4—C5—C6	−8.3 (4)	C11—C12—C13—C14	109.3 (2)
C4—C5—C6—C7	−49.7 (3)	C7—C12—C13—C14	−69.4 (2)
C5—C6—C7—C8	−110.3 (2)	C1—N1—C14—C4	−0.8 (3)
C5—C6—C7—C12	70.1 (3)	C1—N1—C14—C13	176.47 (18)
C12—C7—C8—C9	−0.2 (3)	C3—C4—C14—N1	1.7 (3)
C6—C7—C8—C9	−179.8 (2)	C5—C4—C14—N1	−178.3 (2)
C7—C8—C9—C10	0.5 (3)	C3—C4—C14—C13	−175.4 (2)
C7—C8—C9—Cl1	179.90 (16)	C5—C4—C14—C13	4.6 (3)
C8—C9—C10—C11	−0.7 (3)	C12—C13—C14—N1	−122.11 (19)
Cl1—C9—C10—C11	179.93 (16)	C12—C13—C14—C4	55.2 (3)
C9—C10—C11—C12	0.6 (3)		

Fig. 1