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1-(3-Chloro-10,11-dihydrodibenzo[*b*,*f*]azepin-5-yl)ethanone

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.061
wR factor = 0.119
Data-to-parameter ratio = 13.6

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

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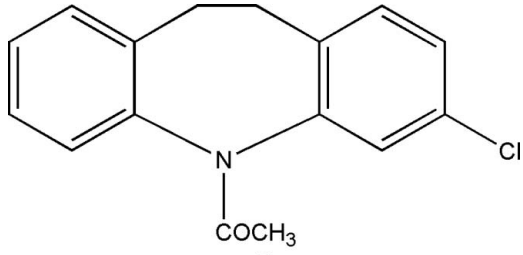
In the title compound, $C_{16}H_{14}ClNO$, the central seven-membered azepine ring adopts a half-boat conformation. A C—H$\cdots\pi$ interaction is observed in the crystal structure.

Comment

The tricyclic ring system 10,11-dihydro-5*H*-dibenz[*b*,*f*]azepine, *i.e.* iminodibenzyl and its substituted derivatives, are important pharmaceutical intermediates of antidepressant and antipsychotic drugs (Craig *et al.*, 1961; Doyle *et al.*, 1976; Kricka & Ledwith, 1974; Melloni *et al.*, 1979). The presence of certain electron-withdrawing substituents on the aromatic ring at the 3 position (as in *e.g.* chloroimipramine, clocapramine, clospipramine) generally increases the pharmacological activity (Kohegyi & Galamb, 1994). In a search for new iminodibenzyl compounds with potentially high bioactivity, we have synthesized the title compound, (I), and its crystal structure is reported here.



(I)

The molecular structure of (I) is shown in Fig. 1. The central seven-membered azepine ring adopts a half-boat conformation (Table 1). The overall structure of the molecule is similar to a butterfly shape, having a dihedral angle of 61.2 (1)° between the two benzene rings. The N1—C1, N1—C14 and N1—C15 bonds lie in a plane, implying that the N atom is sp^2-hybridized. The bond lengths and angles in (I) are comparable to those observed for 1-(10,11-dihydrodibenz[*b*,*f*]azepin-5-yl)ethanone (Nagaraj *et al.*, 2005).

In the crystal structure, a C—H$\cdots\pi$ interaction involving the C4—H4 group and the C9–C14 benzene ring (centroid *Cg*) of the molecule at the symmetry position $(\frac{1}{2}+x, \frac{3}{2}-y, 2-z)$ (H4$\cdots$*Cg* = 2.66 Å) is observed.

Experimental

The title compound was prepared according to the methods described by Csende & Hosztafi (1997) and Hosztafi *et al.* (1995). Anhydrous $CuCl_2$ (0.06 mol), isoamyl nitrite (0.075 mol) and dry acetonitrile (150 ml) were added to a three-necked round-bottomed flask and the

mixture was stirred for 30 min. 3-Amino-5-acetyliminodibenzyl (12.30 g) was slowly added over a period of 10 min to the reaction solution at room temperature. The reaction was monitored by thin-layer chromatography, then the mixture was poured into 20% aqueous hydrochloric acid (100 ml) and extracted with chloroform and washed with water and dried over anhydrous Na_2SO_4 and evaporated under reduced pressure. The product was purified by crystallization from a mixture of benzene–*n*-hexane **(ratio?)**. Single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethyl acetate–toluene (1:8) solution (m.p. 397–398 K).

Crystal data

$C_{16}H_{14}ClNO$
$M_r = 271.73$
Orthorhombic, $P2_12_12_1$
$a = 7.1885$ (7) Å
$b = 9.1504$ (9) Å
$c = 20.251$ (2) Å
$V = 1332.1$ (2) Å^3
$Z = 4$
$D_x = 1.355$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.28$ mm^{-1}
$T = 298$ (2) K
Block, colourless
0.24 × 0.19 × 0.13 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.937$, $T_{max} = 0.960$
7020 measured reflections
2357 independent reflections
2258 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.119$
$S = 1.25$
2357 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0338P)^2 + 0.6638P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Absolute structure: Flack (1983), 969 Friedel pairs
Flack parameter: 0.08 (1)

Table 1
Selected geometric parameters (Å, °).

O1—C15	1.210 (4)	N1—C1	1.435 (4)
N1—C15	1.373 (4)	N1—C14	1.442 (4)
C15—N1—C1	119.9 (3)	C1—N1—C14	117.3 (3)
C15—N1—C14	122.8 (3)		
C14—N1—C1—C6	−51.2 (4)	C7—C8—C9—C14	−69.5 (4)
N1—C1—C6—C7	−3.5 (6)	C8—C9—C14—N1	−5.1 (4)
C1—C6—C7—C8	−7.0 (6)	C1—N1—C14—C9	73.7 (4)
C6—C7—C8—C9	63.9 (4)		

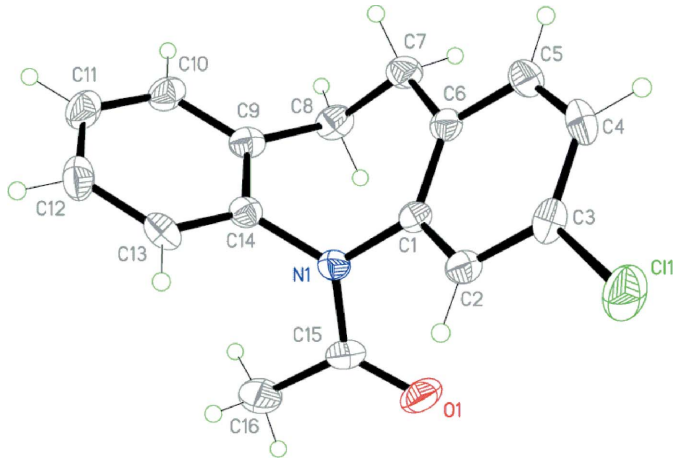


Figure 1
The molecular structure of (I), showing the atom-numbereing scheme. Displacement ellipsoids are drawn at the 50% probability level.

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å and with $U_{iso}(H) = 1.2_{eq}(C)$ or 1.5_{eq}(methyl C).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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, 2002); software used to prepare material for publication: *SHELXL97*.

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