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3-[2-(3-Chlorophenyl)ethyl]pyridine-2-carbonitrile

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.048
wR factor = 0.127
Data-to-parameter ratio = 13.5

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

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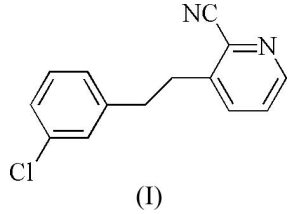
The title compound, $C_{14}H_{11}ClN_2$, is the key intermediate in the synthesis of the antihistaminic drug loratadine. The benzene and pyridine rings are connected through the ethylene bridge and are coplanar. There are intermolecular π–π and C—Cl···N interactions and weak hydrogen bonds stabilizing the packing.

Comment

The title compound, (I), which has been synthesized according to the literature method of Schumacher *et al.* (1989), is an intermediate in the synthesis of the antihistaminic drug loratadine. In the structure, shown in Fig. 1, the pyridine ring is connected to the benzene ring through the ethylene bridge. The benzene ring is coplanar with the pyridine ring. The benzene ring is planar, with an r.m.s. deviation of 0.003 Å, and the Cl atom deviates by only 0.004 (3) Å from this plane. The molecule adopts a stepped *trans* conformation, as shown by the torsion angles C7—C8—C9—C10 and C5—C4—C7—C8. The conformation of the molecule is similar to that of an earlier reported related compound, namely 2,2′-dinitrobenzyl (Yathirajan *et al.*, 2004). Several weak intermolecular bonds stabilize the packing, *viz.* the face-to-face contacts C_g(pyridine ring)···C_g(pyridine ring)^{i} at a distance of 4.240 (1) Å and C_g(pyridine ring)···C_g(benzene ring)^{ii} at a distance of 2.754 (1) Å [symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $-1+x, y, z$]. A further close contact is Cl···N1^{iii} [symmetry code: (iii) $x+\frac{3}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$] of 3.280 (2) Å (Fig. 2).



(I)

Experimental

A solution containing 3-[2-(3-chlorophenyl)ethyl]-*N*-(1,1-dimethylethyl)pyridine-2-carboxamide (35 g, 0.11 mol) and $POCl_3$ (150 ml) was heated under reflux for 3.5 h. Excess $POCl_3$ was removed by distillation and the remaining solution was poured into ice–water. The pH of the solution was adjusted to 8 with 60% aqueous NaOH at room temperature. The mixture was stirred for 2.5 h, during which time the pH was maintained at 8. The product was collected by filtration, washed with water and dried to yield 25 g (93.9%) of crystalline product.

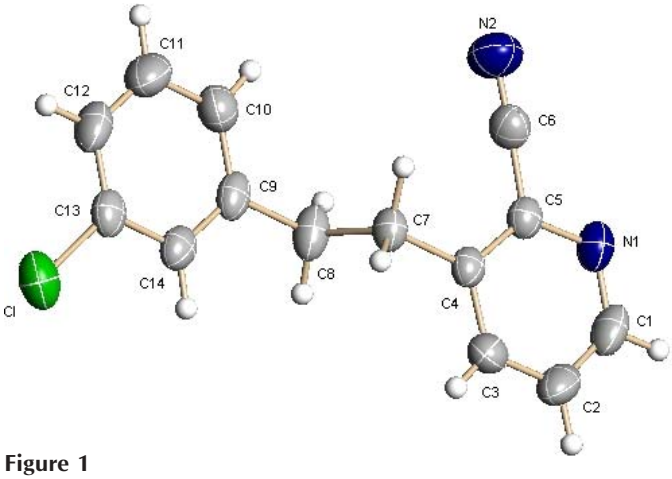


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as circles of arbitrary radius

Crystal data

$C_{14}H_{11}ClN_2$
M_r = 242.70
Monoclinic, $P2_1/n$
a = 7.5645 (8) Å
b = 12.2332 (12) Å
c = 13.5558 (14) Å
β = 102.867 (2)°
V = 1222.9 (2) Å^3
Z = 4
D_x = 1.318 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2106 reflections
θ = 5.7–52.7°
μ = 0.29 mm^{-1}
T = 293 (2) K
Prism, colorless
0.51 × 0.39 × 0.22 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.598, T_{max} = 0.939
7051 measured reflections
2668 independent reflections
1924 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 27.0°
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 15$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.127
S = 1.01
2668 reflections
198 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0674P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Selected torsion angles (°).

C5—C4—C7—C8	91.0 (2)	C7—C8—C9—C10	84.9 (2)

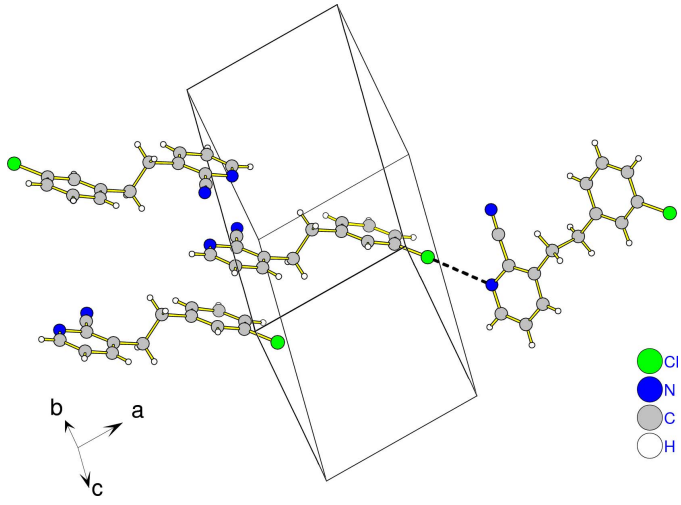


Figure 2
Part of the molecular packing of (I), showing the face-to-face contacts between the π ring systems. The short intermolecular Cl···N1 bond is indicated with a broken line.

All H-atom coordinates were refined, as were the individual isotropic displacement parameters.

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* (Sheldrick, 2000) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

X-ray data were collected at Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences.

References

Brandenburg, K. (1999). *DIAMOND*. Version 2.1c. Crystal Impact GbR, Bonn, Germany.
Bruker (1997). *SMART* (Version 5.622) and *SAINT* (Version 6.02a). Bruker AXS Inc., Madison, Wisconsin, USA.
Schumacher, D. P., Murphy, B. L., Clark, J. E., Tahbaz, P. & Mann, T. A. (1989). *J. Org. Chem.* **54**, 2242–2244.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2002). *SADABS*. Version 2.03. University of Göttingen, Germany.
Yathirajan, H. S., Nagaraj, B., Nagaraja, P. & Lynch, D. E. (2004). *Acta Cryst.* E**60**, o2455–o2456.