organic papers

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Key indicators

Single-crystal X-ray study

$T = 298$ K

Mean σ(C–C) = 0.002 Å

R factor = 0.040

wR factor = 0.112

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.

Methyl 4-(4-chlorophenyl)-1-methylpiperidine-4-carboxylate

The title compound, $C_{14}H_{18}ClNO_2$, was synthesized by the reaction of arecoline with 4-chlorophenylmagnesium bromide. In the crystal structure, a single weak intermolecular C—H···O hydrogen bond links molecules into one-dimensional chains along the *a* axis.

Comment

Arecoline derivatives which are muscarinic agonists have attracted attention recently because of their useful profiles in the treatment of Alzheimer's disease (Hu *et al.*, 2002). The title compound, (I), was synthesized by the reaction of arecoline with 4-chlorophenylmagnesium bromide (Bosch *et al.*, 1985), and the crystal structure is presented here.

(I)
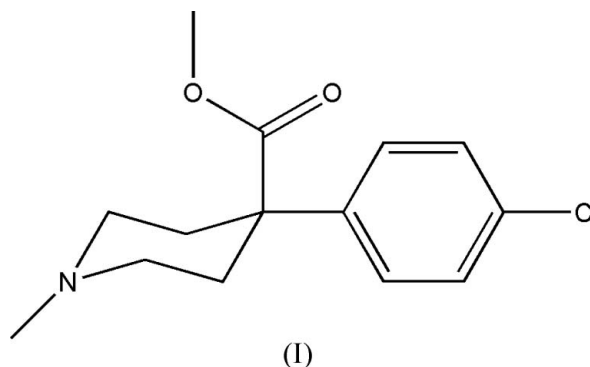
The molecular structure of (I) is shown in Fig. 1. The piperidine ring adopts a chair conformation. All bond distances and angles are normal (Allen *et al.*, 1987). In the crystal structure, a single weak intermolecular C—H···O hydrogen bond links molecules into one-dimensional chains along the *a* axis (Table 1).

Experimental

To a solution of 166 ml of 1 *M* 4-chlorophenylmagnesium bromide in 700 ml diethyl ether was added 12.9 g of arecoline freebase in 300 ml diethyl ether at 263 K. The mixture was stirred at 263 K for 30 min, poured on to ice and treated with 200 ml of 10% HCl. The aqueous layer was separated, washed with 200 ml diethyl ether, cooled in an ice bath, and 100 ml of saturated sodium bicarbonate solution was added. The solution was extracted with 2 × 100 ml of diethyl ether, washed with brine, dried and concentrated *in vacuo*. The crude mixture was purified by column chromatography on alumina to give the title compound as a white solid. The solid was crystallized from an EtOAc/hexane (1:1) mixture to yield crystals suitable for X-ray analysis.

Crystal data

$C_{14}H_{18}ClNO_2$
$M_r = 267.74$
Triclinic, $P\bar{1}$
$a = 5.4546$ (11) Å
$b = 10.238$ (2) Å
$c = 13.615$ (3) Å
$\alpha = 69.01$ (3)°
$\beta = 84.09$ (3)°
$\gamma = 83.24$ (3)°

$V = 703.4$ (3) Å^3
$Z = 2$
$D_x = 1.264$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.27$ mm^{-1}
$T = 298$ (2) K
Prism, colorless
$0.25 \times 0.22 \times 0.18$ mm

Data collection

Bruker SMART CCD APEX-II diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.93$, $T_{max} = 0.95$

6981 measured reflections
3187 independent reflections
2350 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.112$
$S = 1.05$
3187 reflections
235 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2 + 0.0857P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C5—H12⋯O1^i	0.970 (12)	2.516 (17)	3.2518 (17)	132.7 (13)

Symmetry code: (i) $x + 1, y, z$.

All H atoms were located in difference maps and refined independently with isotropic displacement parameters.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); 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, 2003); software used to prepare material for publication: *SHELXTL*.
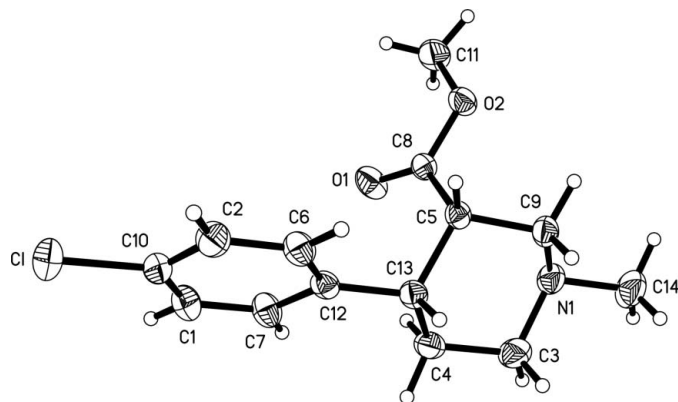

Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level and H atoms shown as small spheres of arbitrary radii.

References

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