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tert-Butyl 3-oxospiro[indan-1,4′-piperidine]-1′-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.150
Data-to-parameter ratio = 14.4

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

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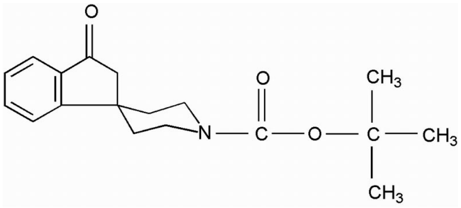
The title compound, $C_{18}H_{23}NO_3$, crystallizes with the piperidine ring in a chair conformation. A spiro junction links the indanone ring system and the *N*-*tert*-butoxycarbonylpiperidine ring; the spiro C atom has distorted tetrahedral geometry.

Comment

Spiro-substituted piperidines are an important class of compounds as subunits in a number of biologically active compounds (Takemoto *et al.*, 1999; Nishi *et al.*, 1998). A large number of substituted piperidine analogues have been synthesized and investigated (Chambers *et al.*, 1992). The title compound, *tert*-butyl 3-oxospiro[indan-1,4′-piperidine]-1′-carboxylate, (I), was prepared from *tert*-butyl 3-hydroxyspiro[indan-1,4′-piperidine]-1′-carboxylate and its crystal structure is reported here.



(I)

The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. In this structure, the piperidine ring has a chair conformation. Due to π–π conjugation, the Csp^2—O bond (C14—O3) is significantly shorter than the Csp^3—O bond (C15—O3), similar to the effect observed in the distances of C14—N1 and C11—N1. Because of the steric hindrance between the indanone and piperidine rings, atom C8 has a distorted tetrahedral geometry with angles C13—C8—C9 [112.03 (19)°] and C7—C8—C9 [102.34 (19)°] deviating significantly from the ideal tetrahedral value. The indanone moiety is almost planar, with atoms C8 and C9 deviating by 0.0570 (3) and −0.0711 (3) Å, respectively, from the indanone ring plane.

Experimental

tert-Butyl 3-hydroxyspiro[indan-1,4′-piperidine]-1′-carboxylate (2.7 mmol) was dissolved in CH_2Cl_2 (16 ml). A molecular sieve (0.81 g, 4 Å, powder) and pyridinium chlorochromate (PCC; 1.2 g, 5.4 mmol)) were added to the resulting solution with ice-cooling. The mixture was stirred at room temperature for 1 h and extracted with diethyl ether, evaporated and separated by flash chromatography (eluant: ethyl acetate–petroleum ether). A white powder was obtained (yield: 86%) and single crystals suitable for crystallographic

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analysis were obtained by slow evaporation from an ethyl acetate–petroleum ether solution (m.p. 393–395 K). IR (KBr, ν cm^{-1}): 2931, 1749, 1686, 1417, 1366, 1178, 1150, 1031, 757; ^{1}H NMR (CDCl$_3$): δ 7.55–7.19 (*m*, 4H), 3.89 (*s*, 2H), 3.59 (broad *s*, 2H), 3.47 (broad *s*, 2H), 1.81–1.78 (4H, *m*), 1.45 (*s*, 9H); analysis calculated for $C_{18}H_{23}NO_3$: C 71.74, H 7.69, N 4.65%; found: C 71.75, H 7.79, N 4.57%.

Crystal data

$C_{18}H_{23}NO_3$
$M_r = 301.37$
Monoclinic, $P2_1/c$
$a = 12.998$ (6) Å
$b = 15.198$ (6) Å
$c = 8.543$ (4) Å
$\beta = 102.850$ (8)°
$V = 1645.4$ (12) Å^3
$Z = 4$

$D_x = 1.217$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 757 reflections
$\theta = 2.9$–21.5°
$\mu = 0.08$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.25 × 0.20 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.981$, $T_{\max} = 0.988$
6761 measured reflections

2913 independent reflections
1547 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.050$
$\theta_{\max} = 25.0°$
$h = -12 \rightarrow 15$
$k = -15 \rightarrow 18$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.150$
$S = 1.00$
2913 reflections
202 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0729P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.12$ e Å^{-3}
$\Delta\rho_{\min} = -0.12$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.219 (3)	N1—C14	1.353 (3)
O2—C14	1.212 (3)	N1—C11	1.460 (3)
O3—C14	1.349 (3)	N1—C12	1.459 (3)
O3—C15	1.474 (3)		
C14—O3—C15	121.7 (2)	O1—C1—C9	125.0 (3)
C14—N1—C11	118.6 (2)	O2—C14—O3	125.0 (3)
C14—N1—C12	123.6 (2)	O2—C14—N1	124.2 (3)
C11—N1—C12	114.8 (2)	O3—C14—N1	110.8 (2)
O1—C1—C2	127.2 (3)		
O1—C1—C2—C3	2.0 (5)	C15—O3—C14—O2	3.4 (4)
O1—C1—C2—C7	−177.7 (3)	C15—O3—C14—N1	−177.8 (2)

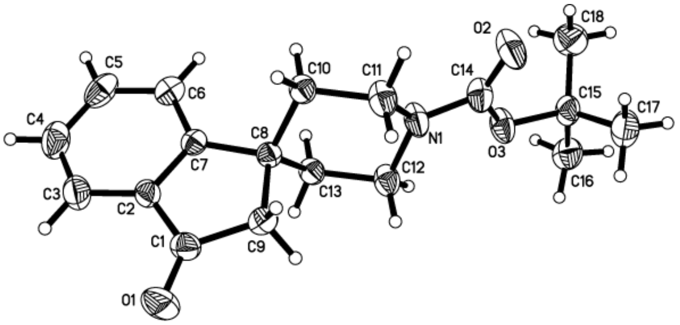

Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding. For the CH and CH$_2$ groups, $U_{\rm iso}$(H) values were set equal to $1.2U_{\rm eq}$(carrier atom) and for the methyl groups they were set equal to $1.5U_{\rm eq}$(carrier atom).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997; data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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References

Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

Chambers, M. S., Baker, R., Billington, D. C., Knight, A. K., Middlemiss, D. N. & Wong, E. H. F. (1992). *J. Med. Chem.* **35**, 2033–2039.

Nishi, T., Nakajima, K., Iio, Y., Ishibashi, K. & Fukazawa, T. (1998). *Tetrahedron Asymmetry*, **9**, 2567–2570.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Takemoto, T., Nakajima, K., Iio, Y., Tamura, M. & Nishi, T. (1999). *Tetrahedron Asymmetry*, **10**, 1787–1793.