Structure of 6-Chloro-4-methyl-*N*-(3-piperidinopropyl)furo[3,2-*b*]indole-2-carboxamide

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Abstract. $C_{20}H_{24}ClN_3O_2$, $M_r = 373.88$, monoclinic, $P2_1/n$, $a = 10.052$ (1), $b = 20.872$ (2), $c = 9.885$ (1) Å, $\beta = 110.023$ (8)°, $V = 1948.4$ (3) Å^3, $Z = 4$, $D_x = 1.275$ Mg m^{-3}, $\lambda(Cu\,K\alpha) = 1.5418$ Å, $\mu = 1.90$ mm^{-1}, $F(000) = 792$, $T = 285$ K, final $R = 0.062$, $wR = 0.060$ for 1859 observed reflections. The furo[3,2-*b*]-indole ring system is planar with a maximum deviation from a least-squares plane through the ring of 0.04 Å. The piperidine ring adopts a chair conformation similar to the morpholine ring of a furo-[3,2-*b*]indole analog [Mizuno, Kawashima, Sota & Kitamura (1987). *Acta Cryst.* C43, 525–527]; however, the ring N atom is sp^3 hybridized, accepting an intramolecular N⋯N hydrogen bond (2.880 Å).

Experimental. Colorless needle-shaped crystal from hexane and chloroform. Crystal $0.1 \times 0.1 \times 0.4$ mm.

Rigaku AFC-5 automated four-circle diffractometer, graphite-monochromated Cu $K\alpha$ radiation; cell dimensions by least-squares refinement of 20 reflections with $40 < 2\theta < 55°$; intensity data $2\theta_{max} = 124°$, ω–2θ scan technique. Three standard reflections (060, 45$\bar{4}$, 51$\bar{4}$) showed 2% intensity fluctuation. 3565 reflections ($-11 < h < 10$, $0 < k < 23$, $0 < l < 11$), 1859 observed $|F_o| > 3\sigma(F_o)$. No correction for absorption. Structure solved by direct methods. Positional and thermal parameters for all non-H atoms refined by block-diagonal least squares. H atoms located in a difference Fourier map or added in geometrically reasonable positions; their positional and isotropic thermal parameters included in the subsequent refinement. $R = 0.062$, $wR = 0.060$ for 1859 reflections, $S = 1.35$; $\sum w(|F_o| - |F_c|)^2$ minimized, unit weights.

Table 1. *Final positional and equivalent isotropic thermal parameters for the non-H atoms with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\sum_i \sum_j \beta_{ij}(\mathbf{a}_i \cdot \mathbf{a}_j).$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Cl(6)	0.8333 (2)	0.03803 (9)	0.7611 (2)	6.0
O(1)	0.3942 (3)	0.1064 (2)	1.1733 (3)	4.2
O(9)	0.3282 (4)	0.0918 (2)	1.5039 (4)	5.9
N(4)	0.7163 (4)	0.0321 (2)	1.2327 (4)	4.8
N(10)	0.2029 (5)	0.1417 (2)	1.2970 (5)	5.3
N(14)	0.0662 (5)	0.2205 (2)	1.0489 (5)	5.4
C(2)	0.4182 (6)	0.0888 (2)	1.3156 (5)	4.2
C(3)	0.5430 (6)	0.0570 (3)	1.3747 (6)	4.8
C(3a)	0.6018 (5)	0.0547 (3)	1.2651 (5)	4.3
C(4a)	0.6933 (5)	0.0475 (3)	1.0903 (5)	4.2
C(5)	0.7783 (6)	0.0351 (3)	1.0091 (6)	4.8
C(6)	0.7292 (6)	0.0544 (3)	0.8677 (6)	4.6
C(7)	0.6020 (6)	0.0858 (3)	0.8053 (6)	5.0
C(8)	0.5170 (6)	0.0991 (3)	0.8853 (6)	4.6
C(8a)	0.5623 (5)	0.0804 (2)	1.0292 (5)	4.0
C(8b)	0.5105 (5)	0.0840 (2)	1.1450 (5)	4.1
C(9)	0.3120 (6)	0.1079 (3)	1.3806 (6)	4.6
C(11)	0.0912 (7)	0.1630 (3)	1.3486 (7)	6.8
C(12)	0.0452 (9)	0.2317 (4)	1.2900 (9)	10.3
C(13)	−0.0280 (7)	0.2361 (3)	1.1307 (7)	6.6
C(15)	−0.0184 (7)	0.2000 (4)	0.9045 (8)	8.4
C(16)	0.0758 (8)	0.1811 (4)	0.8189 (8)	8.5
C(17)	0.1642 (7)	0.2365 (4)	0.8056 (7)	7.7
C(18)	0.2475 (7)	0.2607 (4)	0.9560 (7)	7.5
C(19)	0.1488 (7)	0.2759 (3)	1.0391 (7)	6.5
C(20)	0.8367 (6)	−0.0023 (3)	1.3301 (7)	6.5

Table 2. *Bond distances (Å) and angles (°)*

O(1)–C(2)	1.393 (7)	C(2)–O(1)–C(8b)	104.5 (4)
O(1)–C(8b)	1.373 (6)	O(1)–C(2)–C(3)	112.2 (5)
C(2)–C(3)	1.362 (8)	O(1)–C(2)–C(9)	117.8 (5)
C(2)–C(9)	1.476 (8)	C(3)–C(2)–C(9)	130.0 (5)
C(3)–C(3a)	1.401 (8)	C(2)–C(3)–C(3a)	104.8 (5)
C(3a)–N(4)	1.380 (7)	C(3)–C(3a)–C(8b)	108.7 (5)
C(3a)–C(8b)	1.371 (8)	N(4)–C(3a)–C(8b)	108.4 (5)
N(4)–C(4a)	1.385 (7)	C(3a)–N(4)–C(4a)	107.2 (4)
N(4)–C(20)	1.452 (8)	N(4)–C(4a)–C(8a)	110.5 (5)
C(4a)–C(5)	1.381 (8)	C(5)–C(4a)–C(8a)	120.7 (5)
C(4a)–C(8a)	1.423 (7)	C(4a)–C(5)–C(6)	117.3 (5)
C(5)–C(6)	1.374 (8)	C(5)–C(6)–C(7)	123.3 (5)
C(6)–Cl(6)	1.755 (6)	C(6)–C(7)–C(8)	120.0 (5)
C(6)–C(7)	1.380 (8)	C(7)–C(8)–C(8a)	118.7 (5)
C(7)–C(8)	1.376 (8)	C(8)–C(8a)–C(4a)	119.9 (5)
C(8)–C(8a)	1.393 (8)	C(8b)–C(8a)–C(4a)	103.1 (5)
C(8a)–C(8b)	1.412 (8)	C(8a)–C(8b)–C(3a)	110.8 (5)
C(9)–O(9)	1.220 (7)	O(1)–C(8b)–C(3a)	109.8 (5)
C(9)–N(10)	1.328 (8)	C(2)–C(9)–O(9)	119.4 (5)
N(10)–C(11)	1.453 (9)	C(2)–C(9)–N(10)	116.1 (5)
C(11)–C(12)	1.556 (12)	O(9)–C(9)–N(10)	124.6 (5)
C(12)–C(13)	1.495 (12)	C(9)–N(10)–C(11)	121.1 (5)
C(13)–N(14)	1.476 (9)	N(10)–C(11)–C(12)	108.5 (6)
N(14)–C(15)	1.454 (9)	C(11)–C(12)–C(13)	115.1 (7)
N(14)–C(19)	1.447 (9)	C(12)–C(13)–N(14)	112.8 (6)
C(15)–C(16)	1.522 (12)	C(13)–N(14)–C(15)	109.5 (5)
C(16)–C(17)	1.493 (12)	C(13)–N(14)–C(19)	110.6 (5)
C(17)–C(18)	1.521 (11)	C(15)–N(14)–C(19)	109.0 (5)
C(18)–C(19)	1.523 (10)	N(14)–C(15)–C(16)	110.9 (6)
		C(15)–C(16)–C(17)	110.5 (7)
		C(16)–C(17)–C(18)	108.5 (7)
		C(17)–C(18)–C(19)	110.7 (6)
		C(18)–C(19)–N(14)	111.4 (6)
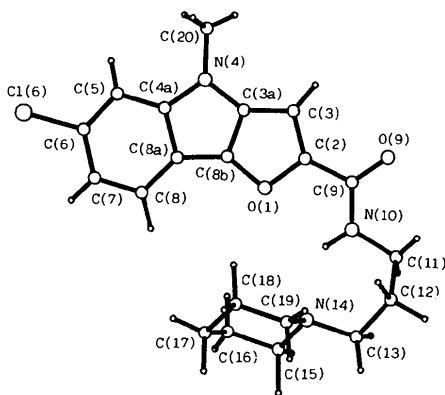
Fig. 1. Molecular structure and atomic numbering system.

Fig. 2. Stereoscopic view along the a axis showing the cell packing.

$(\Delta/\sigma)_{max} = 0.36$ for non-H atoms. Final $\Delta\rho$ excursions ± 0.2 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Programs: *MULTAN*84 (Main, Germain & Woolfson, 1984) and *HBLSV* (Ashida, 1979). Calculations on a PANAFACOM U-1200 and ACOS850 at the Computing Center for Research in Agriculture, Foresty and Fishery. The final atomic parameters are given in Table 1. Bond distances and angles are listed in Table 2.* Fig. 1 shows the molecule and the numbering

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44365 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

scheme, and Fig. 2 the packing of the molecules in the cell.

Related literature. This structure is one of a series of furo[3,2-b]indoles. The previous structure of the series is listed in Mizuno, Kawashima, Sota & Kitamura (1987).

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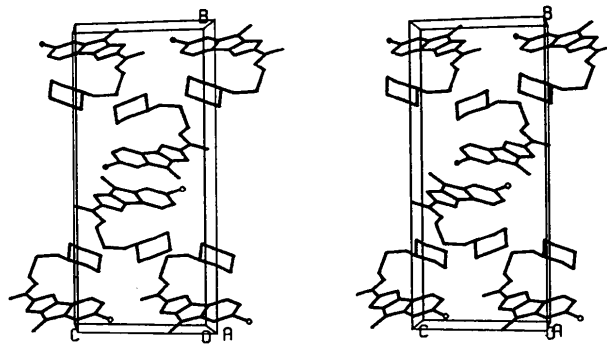Mizuno, H., Kawashima, Y., Sota, K. & Kitamura, K. (1987). *Acta Cryst.* C43, 525–527.

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anti-5,16:10,15-Bis(*tert*-butylimino)-1,2,3,4,11,12,13,14-octamethyl-5,10,15,16-tetrahydrobenzo[*h*]pentaphene

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Abstract. $C_{42}H_{50}N_2$, $M_r = 582.88$, triclinic, $P\bar{1}$, $a = 11.701$ (9), $b = 11.796$ (15), $c = 15.538$ (8) Å, $\alpha = 69.12$ (7), $\beta = 62.27$ (7), $\gamma = 67.15$ (7)°, $V = 1708$ Å^3, $Z = 2$, $D_x = 1.133$ Mg m^{-3}, $F(000) = 632$, λ(Mo $K\alpha$) $= 0.71069$ Å, $\mu = 0.06$ mm^{-1}, $T = 291$ (1) K, final $R = 0.062$ for 3466 unique observed $[F \geq 4.0\sigma(F)]$ diffractometer data and 398 variables. The constitution and configuration of the hitherto unknown Diels–Alder adduct of 2,5-di-*tert*-butyl-2,5-dihydrobenzo[e]-pyrrolo[3,4-g]isoindole with 3,4,5,6-tetramethyl-1,2-dehydrobenzene has been elucidated *via* the crystal-structure analysis. The *tert*-butyl groups of the annelated cyclic compound are in *anti* position. The perpendicular to the plane of the naphthalene ring and the direction through the position of the N atom and the central C atom of the *tert*-butyl groups are nearly parallel and therefore there is ample space at the N atoms for the free electron pairs.