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Racemic 2,10-dibenzyloxy-12-chloro-3,9-dimethoxy-5,8,13,13a-tetrahydro-6*H*-dibenzo[*a*,*g*]quinolizine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.063
wR factor = 0.177
Data-to-parameter ratio = 17.0

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

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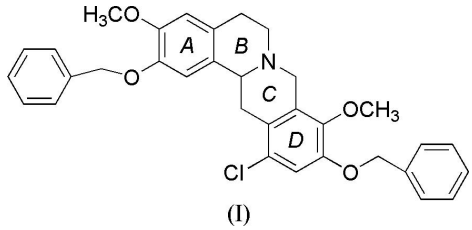
The title compound, $C_{33}H_{32}ClNO_4$, prepared by methylation of 12-chloro-2,10-dibenzyloxy-9-hydroxy-3-methoxytetrahydroprotoberberine with diazomethane, contains a fused four-ring system in which the *trans*-fused rings both have half-chair conformations. No significant intermolecular interactions are observed in the structure.

Comment

Protoberberine compounds, obtained either from plants or synthesis, possess a variety of biological and pharmacological properties, such as insecticidal (Miyazawa *et al.*, 1998), antalgic (Jin *et al.*, 1964), antimalarial, antibacterial and antitumor (Lin *et al.*, 2002, and references therein). Stepholidine and its analogs have been reported to have a unique pharmacological characteristic as potential novel antipsychotic agents (Jin *et al.*, 2002). As part of our ongoing studies of stepholidine derivatives, the crystal structure analysis of the title compound, (I), has been carried out and the results are presented here.



(I)

The asymmetric unit of (I) consists of one independent molecule (Fig.1). The molecular skeleton contains a fused four-ring system. Rings *B* and *C*, both having half-chair conformations, exist in a *B*/*C*-*trans* form, which is consistent with the presence of the Bohlmann bands (2733–2920 cm^{-1}) in the IR spectrum of (I) (Takao & Iwasa, 1976). Ring *B* is puckered in such a manner that the four atoms C5, C14, C15 and C16 are coplanar to within 0.008 (2) Å, while atoms C6 and N7 are unequally displaced from this plane on opposite sides, with out-of-plane displacements of 0.496 (5) and 0.267 (4) Å, respectively. The plane defined by C5/C14/C15/C16 in ring *B* is nearly parallel to benzene ring *A*, and the dihedral angle formed by these planes is 1.1 (2)°. Benzene rings *A* and *D* are twisted by 30.81 (9)° with respect to one another, which is very similar to the situation found in an analog of (I) (Ding *et al.*, 2005). The methoxy group at atom C3 is nearly coplanar with ring *A*, as indicated by the C26—O4—C3—C4 torsion angle [−3.1 (4)°]; while C34 of the methoxy group at atom C9 is rotated out of the plane *D*, the C34—O2—C9—C10 torsion angle being −60.5 (3)° compared with 88.8 (3)° in the case of stepholidine monohydrate (II)

(Wu *et al.*, 1987). No significant intermolecular interactions are observed in the crystal structure.

Experimental

The title compound, (I), was prepared by the reaction of 2,10-dibenzyloxy-12-chloro-9-hydroxy-3-methoxytetrahydroprotoberberine (80 mg) and diazomethane [prepared from nitrosomethylurea (1 g)] in tetrahydrofuran (1 ml) at room temperature for 72 h (yield 65%, m.p. 422–424 K). Crystals of (I) suitable for single-crystal X-ray diffraction were grown from a methanol solution at room temperature by slow evaporation. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48–7.28 (*m*, 10H), 6.92 (*s*, 1H), 6.65 (*s*, 1H), 6.79 (*s*, 1H), 5.17 (*d*, 2H, J = 2.4 Hz), 5.08 (*d*, 2H, J = 5.6 Hz), 4.21 (*d*, 1H, J = 15.9 Hz), 3.89 (*s*, 3H), 3.87 (*s*, 3H), 3.50–3.44 (*m*, 2H), 3.19–3.09 (*m*, 3H), 2.70–2.59 (*m*, 2H), 2.51 (*dd*, 1H, J = 11.3, 16.6 Hz). Analysis calculated for $C_{33}H_{32}ClNO_4$: C 73.12, H 5.95, N 2.58%; found: C 73.15, H 6.07, N 2.47%.

Crystal data

$C_{33}H_{32}ClNO_4$	D_x = 1.262 Mg m^{-3}
M_r = 542.05	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 3475 reflections
a = 20.4833 (18) Å	θ = 5.0–44.9°
b = 21.6089 (18) Å	μ = 0.17 mm^{-1}
c = 16.0523 (14) Å	T = 293 (2) K
β = 126.549 (2)°	Block, yellow
V = 5707.9 (9) Å^3	0.51 × 0.42 × 0.40 mm
Z = 8	

Data collection

Bruker SMART CCD area-detector diffractometer	6226 independent reflections
	3798 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.069
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	θ_{max} = 27.0°
	$h = -26 \rightarrow 22$
T_{min} = 0.705, T_{max} = 0.930	$k = -27 \rightarrow 25$
16679 measured reflections	$l = -13 \rightarrow 20$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.063	$w = 1/[\sigma^2(F_o^2) + (0.0924P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.177	$(\Delta/\sigma)_{max}$ = 0.087
S = 0.96	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
6226 reflections	$\Delta\rho_{min}$ = −0.33 e Å^{-3}
366 parameters	

All H atoms were located in a difference Fourier map, but they were introduced in calculated positions and treated as riding on their parent atoms [C—H = 0.93–0.97 Å, and U_{iso}(H) = 1.2U_{eq}(C) for CH and 1.5U_{eq}(C) for CH_3 groups].

Data collection: *SMART* (Bruker,2000); cell refinement: *SAINT* (Bruker,2000); data reduction: *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

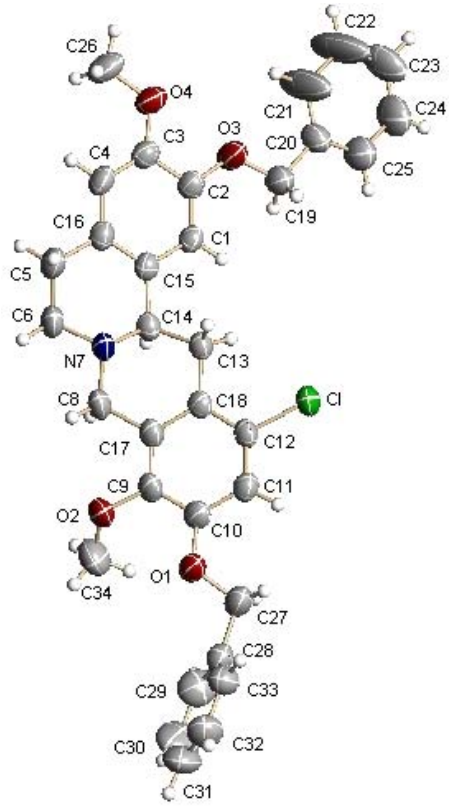


Figure 1
View of (I), showing the atom-labeling scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

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