A crystal of approximate dimensions 0·25 × 0·30 × 0·42 mm was cut from a large block and used for data collection on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. The cell constants and orientation matrix were determined by least-squares refinement of the setting angles of 25 reflections in the 10–15° range. Intensity data were collected in the range $2 < \theta < 25°$ using the $\omega/2\theta$ scan method and variable scan speed (1·10–5·50° min^{-1}). The intensities of three standard reflections, monitored at regular intervals, did not show significant variations. 3095 unique reflections were collected (h 0→9, k −10→10, l −18→18), of which 2518 with $I > 3\sigma(I)$ were considered observed. Data were corrected for Lorentz and polarization effects; absorption was ignored.

The structure was solved by direct methods (*MULTAN*11/82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least-squares calculations on F's. H atoms were located from a difference map and included in the refinement fixed at these positions with the overall isotropic temperature factor B_{iso} = 5·0 Å^2; C, O and N had anisotropic temperature factors. The refinement converged completely with R = 0·048 and wR = 0·062, where $w = [\sigma^2(F_o) + (0{\cdot}010F_o)^2]^{-1}$; max. shift/e.s.d. in the last cycle of refinement was < 0·01 and goodness of fit, S = 1·383. A final difference map was devoid of significant features with $\Delta\rho$ in the range −0·40 to 0·48 e Å^{-3}. Scattering factors used in the calculations were taken from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965). Computer programs used in this study were from the Enraf–Nonius *Structure Determination Package* (B. A. Frenz & Associates, Inc., 1985) and *ORTEP*II (Johnson, 1976).

Final fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s are listed in Table 1.* Table 2 contains bond lengths and bond angles. Fig. 1 shows the molecular structure of the title compound. Fig. 2 is a stereoview of the unit-cell packing.

Related literature. 8-Methoxy- and 8-nitronaphthonitrile (Procter, Britton & Dunitz, 1981), *N,N*-dimethyl-8-nitro-1-naphthaleneamine (Egli, Wallis & Dunitz, 1986), and 8-dimethylamino-1-naphthonitrile (Parvez & Schuster, 1990).

* Lists of structure amplitudes, anisotropic temperature factors, least-squares-planes data, H-atom parameters and molecular dimensions involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53372 (35 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (±)-Cycleatjehenine, a New Bisbenzylisoquinoline Alkaloid from *Cyclea atjehensis*

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Abstract. 6,10,25-Trimethoxy-30-methyl-8,2,3-dioxa-15,30-diazaheptacylco[22.6.2.2^{9,6}.2^{18,21}.1^{3,7}.0^{12,35}.0^{27,31}]heptatriaconta-3,5,7(37),9,11,13,15,18,20,24,26,31,33,35-undecaen-32-ol–methanol (1/2), $C_{37}H_{36}N_2O_6.2CH_3OH$, M_r = 668·79, monoclinic, $P2_1/n$, a = 11·034 (3), b = 33·399 (3), c = 10·416 (2) Å, β = 114·25 (1)°, V = 3500 (2) Å^3, Z =

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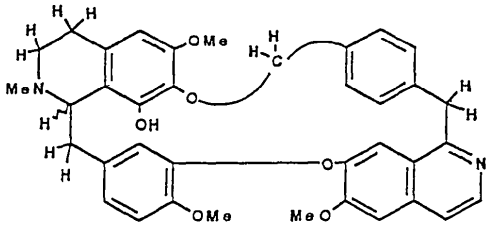
Table 1. *Summary of data collection and structure refinement*

Crystal size (mm)	0·18 × 0·30 × 0·70
Diffractometer	Enraf–Nonius CAD-4
Monochromator	Graphite
Cell constants (θ°)	23 reflections, $15 < \theta < 25$
θ_{max}(°)	50
Scan method	$\omega/2\theta$
ω-Scan width (°)	$(1{\cdot}00 + 0{\cdot}14 \tan\theta)$
Variable scan speed (°min^{-1})	2·06–5·50
Scan ranges of h, k, l	0→10, 0→33, −10→10
Intervals of standard reflections (s)	7200
Crystal decay* (%)	35·4
Data corrections applied†	Lorentz and polarization
Unique data measured, R_{int}	3578, 0·015
Data used [$I > 3\sigma(I)$]	2801
Parameters refined	442
R, wR	0·0594, 0·0816
Weighting scheme	$w = [\sigma^2(F_o) + (0{\cdot}080F_o)^2]^{-1}$
$(\Delta/\sigma)_{max}$ in last cycle	< 0·1
$\Delta\rho$, e Å^{-3} in final ΔF map	−0·294, 0·307
S	1·753

* Linear decay, corrected for by appropriate scaling.
† Absorption ignored.

4, $D_x = 1{\cdot}269$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, μ = 0·684 mm^{-1}, $F(000) = 1424$, $T = 293$ (1) K, R = 0·0594 for 2801 observed data with $I > 3\sigma(I)$. The N-containing ring of the tetrahydroisoquinoline group adopts a twist-boat conformation. The mean planes of the isoquinoline moieties are inclined at 47·2 (1)°. The two halves of the molecule comprising the benzyltetrahydroisoquinoline and benzylisoquinoline residues are joined by a methyleneoxy bridge on one side and an ether linkage on the other side. The dihedral angle between the two benzyl groups is 12·7 (5)°.

Experimental. Our investigations on the alkaloids from the leaves of *Cyclea atjehensis* Forman (Menispermaceae) of Thai origin have yielded the title compound which appears to be a minor component of the alkaloids reported from this plant (Tantisewie, Pharadai, Amnauypol, Freyer, Guinaudeau & Shamma, 1990). This alkaloid incorporates the unusual methyleneoxy bridge and belongs to a new subgroup of bisbenzylisoquinolines. Presently, more than 500 naturally occurring bisbenzylisoquinolines are known. However, cycleatjehenine (1) is the first to be obtained as a racemate.



(1)

Table 2. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$B_{eq} = (1/3)\sum_i\sum_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
O1	0·7161 (3)	0·2154 (1)	0·5564 (3)	4·40 (7)
O2	0·9659 (2)	0·2425 (1)	0·5997 (2)	4·17 (6)
O3	1·0503 (2)	0·3147 (1)	0·7188 (3)	4·23 (6)
O4	0·3375 (2)	0·1054 (1)	0·5136 (2)	3·28 (6)
O5	0·2412 (2)	0·0698 (1)	0·2788 (2)	3·62 (6)
O6	0·2649 (2)	0·0866 (1)	0·7184 (2)	4·35 (6)
O7	0·5767 (3)	0·5277 (1)	0·2754 (3)	6·18 (9)
O8	0·1914 (3)	0·4780 (1)	0·4875 (4)	8·3 (1)
N1	0·4522 (3)	0·2850 (1)	0·6531 (3)	3·89 (8)
N2	0·8464 (3)	0·0155 (1)	0·6011 (3)	3·74 (8)
C1	0·5473 (4)	0·2542 (1)	0·6545 (4)	3·39 (9)
C2	0·6732 (3)	0·2739 (1)	0·6606 (3)	3·13 (9)
C3	0·7596 (4)	0·2516 (1)	0·6197 (3)	3·39 (9)
C4	0·8848 (3)	0·2658 (1)	0·6423 (3)	3·32 (9)
C5	0·9233 (3)	0·3035 (1)	0·6996 (3)	3·33 (9)
C6	0·8387 (4)	0·3264 (1)	0·7349 (4)	3·67 (9)
C7	0·7116 (3)	0·3116 (1)	0·7149 (3)	3·32 (9)
C8	0·6183 (4)	0·3376 (1)	0·7480 (4)	4·8 (1)
C9	0·5106 (4)	0·3137 (1)	0·7669 (4)	4·7 (1)
C10	0·3295 (4)	0·2681 (1)	0·6526 (5)	5·4 (1)
C11	1·0962 (4)	0·3524 (1)	0·7797 (5)	5·6 (1)
C12	1·0623 (4)	0·2189 (1)	0·7121 (4)	4·5 (1)
C13	1·0045 (4)	0·1796 (1)	0·7270 (4)	3·65 (9)
C14	0·9634 (4)	0·1726 (1)	0·8334 (4)	4·1 (1)
C15	0·9084 (4)	0·1360 (1)	0·8440 (4)	3·9 (1)
C16	0·8921 (3)	0·1058 (1)	0·7488 (3)	3·04 (9)
C17	0·9305 (4)	0·1133 (1)	0·6398 (4)	4·0 (1)
C18	0·9857 (4)	0·1496 (1)	0·6305 (4)	4·4 (1)
C19	0·8438 (3)	0·0647 (1)	0·7673 (4)	3·47 (9)
C20	0·7747 (3)	0·0422 (1)	0·6340 (4)	3·20 (9)
C21	0·6382 (3)	0·0498 (1)	0·5417 (3)	2·86 (8)
C22	0·5573 (3)	0·0766 (1)	0·5776 (3)	2·88 (8)
C23	0·4267 (3)	0·0815 (1)	0·4883 (3)	2·76 (8)
C24	0·3725 (3)	0·0613 (1)	0·3577 (3)	2·81 (8)
C25	0·4479 (3)	0·0358 (1)	0·3205 (3)	3·20 (9)
C26	0·5834 (3)	0·0293 (1)	0·4129 (3)	2·83 (8)
C27	0·6641 (4)	0·0022 (1)	0·3815 (4)	3·8 (1)
C28	0·7908 (4)	−0·0042 (1)	0·4766 (4)	4·2 (1)
C29	0·1818 (4)	0·0525 (1)	0·1414 (4)	5·0 (1)
C30	0·3818 (3)	0·1299 (1)	0·6331 (3)	2·75 (8)
C31	0·4583 (3)	0·1629 (1)	0·6424 (3)	3·08 (8)
C32	0·4976 (3)	0·1882 (1)	0·7583 (3)	3·17 (9)
C33	0·4533 (4)	0·1790 (1)	0·8623 (4)	3·8 (1)
C34	0·3755 (4)	0·1456 (1)	0·8524 (4)	3·77 (9)
C35	0·3403 (3)	0·1205 (1)	0·7378 (4)	3·11 (8)
C36	0·2335 (4)	0·0741 (1)	0·8328 (4)	5·3 (1)
C37	0·5888 (4)	0·2232 (1)	0·7749 (4)	3·9 (1)
C38	0·5652 (5)	0·5652 (1)	0·2103 (5)	6·6 (1)
C39	0·1075 (6)	0·4495 (2)	0·4991 (6)	9·4 (2)

Crystals of (1) in the form of thin needles were obtained from methanol solution at room temperature. Details of data collection and structure refinement are given in Table 1. The structure was solved by direct methods (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least-squares calculations of F's. A difference Fourier synthesis calculated at an intermediate stage of the refinement revealed all H atoms. These were included in the refinement with idealized geometry (C—H and O—H 0·95 Å) and fixed isotropic temperature factor B_{iso} = 5·0 Å^2; C, N and O had anisotropic temperature factors. Scattering factors used in the calculations were taken

Table 3. *Bond distances* (Å) *and bond angles* (°)

O1	C3	1·366 (4)	C6	C7	1·419 (6)
O2	C4	1·389 (5)	C7	C8	1·492 (6)
O2	C12	1·451 (5)	C8	C9	1·511 (6)
O3	C5	1·383 (5)	C12	C13	1·495 (5)
O3	C11	1·409 (5)	C13	C14	1·379 (6)
O4	C23	1·374 (4)	C13	C18	1·373 (5)
O4	C30	1·399 (4)	C14	C15	1·387 (6)
O5	C24	1·371 (4)	C15	C16	1·375 (6)
O5	C29	1·428 (4)	C16	C17	1·388 (7)
O6	C35	1·370 (4)	C16	C19	1·511 (5)
O6	C36	1·432 (6)	C17	C18	1·379 (6)
O7	C38	1·408 (6)	C19	C20	1·486 (5)
O8	C39	1·367 (8)	C20	C21	1·439 (4)
N1	C1	1·465 (5)	C21	C22	1·419 (5)
N1	C9	1·454 (5)	C21	C26	1·404 (4)
N1	C10	1·465 (6)	C22	C23	1·366 (4)
N2	C20	1·326 (6)	C23	C24	1·414 (4)
N2	C28	1·356 (5)	C24	C25	1·354 (5)
C1	C2	1·515 (5)	C25	C26	1·424 (4)
C1	C37	1·544 (5)	C26	C27	1·399 (6)
C2	C3	1·407 (6)	C27	C28	1·358 (5)
C2	C7	1·373 (5)	C30	C31	1·368 (5)
C3	C4	1·386 (5)	C30	C35	1·381 (6)
C4	C5	1·385 (5)	C31	C32	1·389 (5)
C5	C6	1·368 (6)	C32	C33	1·393 (7)
C32	C37	1·505 (5)	C34	C35	1·380 (5)
C33	C34	1·385 (5)			

C4	O2	C12	113·7 (3)	C2	C7	C6	119·9 (4)
C5	O3	C11	117·5 (3)	C2	C7	C8	120·2 (3)
C23	O4	C30	119·7 (2)	C6	C7	C8	119·8 (3)
C24	O5	C29	117·0 (3)	C7	C8	C9	112·2 (3)
C35	O6	C36	117·2 (3)	N1	C9	C8	110·5 (4)
C1	N1	C9	112·3 (3)	O2	C12	C13	111·1 (3)
C1	N1	C10	112·7 (3)	C12	C13	C14	121·8 (4)
C9	N1	C10	110·9 (4)	C12	C13	C18	120·5 (5)
C20	N2	C28	119·4 (3)	C14	C13	C18	117·6 (4)
N1	C1	C2	109·6 (3)	C13	C14	C15	120·8 (4)
N1	C1	C37	116·9 (4)	C14	C15	C16	121·4 (4)
C2	C1	C37	107·3 (3)	C15	C16	C17	117·7 (3)
C1	C2	C3	118·7 (4)	C15	C16	C19	121·5 (4)
C1	C2	C7	122·6 (4)	C17	C16	C19	120·8 (3)
C3	C2	C7	118·5 (3)	C16	C17	C18	120·5 (4)
O1	C3	C2	117·9 (3)	C13	C18	C17	121·9 (4)
O1	C3	C4	120·8 (4)	C16	C19	C20	114·4 (4)
C2	C3	C4	121·3 (4)	N2	C20	C19	116·8 (3)
O2	C4	C3	118·9 (3)	N2	C20	C21	121·2 (3)
O2	C4	C5	121·5 (3)	C19	C20	C21	122·0 (3)
C3	C4	C5	119·5 (4)	C20	C21	C22	122·3 (3)
O3	C5	C4	114·8 (3)	C20	C21	C26	118·4 (3)
O3	C5	C6	125·0 (4)	C22	C21	C26	119·3 (3)
C4	C5	C6	120·1 (4)	C21	C22	C23	119·9 (3)
C5	C6	C7	120·5 (3)	O4	C23	C22	125·3 (3)
O4	C23	C24	114·1 (3)	O4	C30	C35	117·5 (3)
C22	C23	C24	120·6 (3)	C31	C30	C35	121·5 (4)
O5	C24	C23	113·8 (3)	C30	C31	C32	121·0 (4)
O5	C24	C25	125·6 (3)	C31	C32	C33	117·3 (3)
C23	C24	C25	120·7 (3)	C31	C32	C37	121·8 (4)
C24	C25	C26	120·1 (3)	C33	C32	C37	120·9 (4)
C21	C26	C25	119·5 (3)	C32	C33	C34	121·6 (3)
C21	C26	C27	118·3 (3)	C33	C34	C35	120·0 (4)
C25	C26	C27	122·3 (3)	O6	C35	C30	116·7 (3)
C26	C27	C28	119·7 (3)	O6	C35	C34	124·7 (4)
N2	C28	C27	123·1 (4)	C30	C35	C34	118·7 (3)
O4	C30	C31	121·0 (3)	C1	C37	C32	117·8 (3)

from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965). Computer programs used in this study were from the Enraf–Nonius *Structure Determination Package* (Frenz, 1985) and *ORTEP*II (Johnson, 1976).

Final fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s are listed in

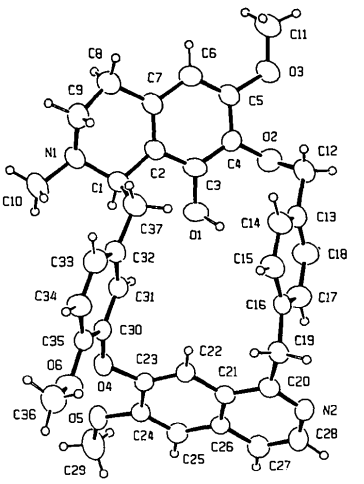


Fig. 1. Molecular structure of (±)-cycleatjehenine with the crystallographic numbering scheme.

Table 2.* Table 3 contains bond lengths and angles. Fig. 1 shows the molecular structure of (1).

Related literature. The crystal structures of a few bisbenzylisoquinoline alkaloids have been reported, *e.g.* methylwarifteine (Borkakoti & Palmer, 1978*a*), dimethylwarifteine (Borkakoti & Palmer, 1978*b*) and tetrandrine (Gilmore, Bryan & Kupchan, 1976).

This research was supported by NSF grant INT-8512266.

*Lists of structure factors, anisotropic thermal parameters, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53385 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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