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Chasmanthinine†

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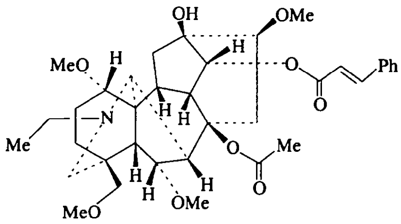
Abstract

The crystal structure of chasmanthinine, $C_{36}H_{49}NO_9$, a C_{19} norditerpenoid alkaloid which has been isolated from the roots of *Aconitum chasmanthum* Stapf ex Holmes, contains independent molecules separated by normal van der Waals distances. The mean bond distances are: N—C_{sp^3} 1.468 (11), C_{sp^3}—C_{sp^3} 1.54 (2), C_{sp^2}—C_{sp^2} 1.471 (2), O—C_{sp^3} 1.428 (18), O—C_{sp^2} 1.338 (2) and C═O 1.205 (4) Å. The fused-ring system contains one boat, one half-chair, two chair and two envelope conformations.

Comment

Continuing our studies on the diterpenoid alkaloids of *Aconitum chasmanathum* Stapf ex Holmes of Pakistani origin (Parvez *et al.*, 1998), we have isolated the title compound, (1). We report here the structure of (1), which contains discrete molecules of chasmanthinine separated by normal van der Waals distances. The absolute configuration (Fig. 1) was not determined by this X-ray analysis, but was chosen to be the same as that known for chasmanine 14-α-benzoate hydrochloride (De Camp & Pelletier, 1977). The molecular dimensions in (1) are normal and lie within expected values for the corresponding bond distances and angles, with mean bond distances: N—C_{sp^3} 1.468 (11), C_{sp^3}—C_{sp^3} 1.54 (2), C_{sp^2}—C_{sp^2} 1.471 (2), O—C_{sp^3} 1.428 (18), O—C_{sp^2} 1.338 (2) and C═O 1.205 (4) Å, while the C_{sp^3}—C_{sp^2} and C_{sp^2}═C_{sp^2} distances are 1.482 (5) and 1.318 (5) Å, respectively.



(1)

The six-membered rings *A* (C1–C5, C11) and *E* (C4, C5, C11, C17, N1, C19) adopt chair conformations. Ring *A* is slightly flattened at C1 due to the methoxy substituent attached to C1 as reported in the structures of a chasmanine intermediate (Przybylska & Ahmed, 1980), aconitine (Codding, 1982) and chasmaconitine (Parvez *et al.*, 1998). The six-membered ring *D* (C8, C9, C13–C16) has a half-chair conformation, with C14 0.899 (4) Å out of the plane of the remaining ring atoms. The seven-membered ring *B* (C5–C11) adopts a boat conformation. The five-membered rings *C* (C9, C10, C12–C14) and *F* (C5–C7, C11, C17) have C14- and C17-envelope conformations, respectively, with C14 0.714 (5) and C17 0.742 (4) Å out of the planes formed by the remaining atoms of their respective rings. The 3-phenyl-2-propenoate moiety attached to C14 is essentially planar, with O8 lying 0.290 (4) Å below and O9 0.189 (5) Å above the plane formed by atoms C26–C34. The conformations of rings *A*–*F* in (1) are identical to the corresponding rings in chasmaconitine (Parvez *et al.*, 1998). There is a short intramolecular O6—H···O7 hydrogen bond [O6···O7 2.588 (4), H···O7 2.02 Å and O6—H···O7 116°]. A diagram of the molecular packing in the unit cell of (1) has been deposited.

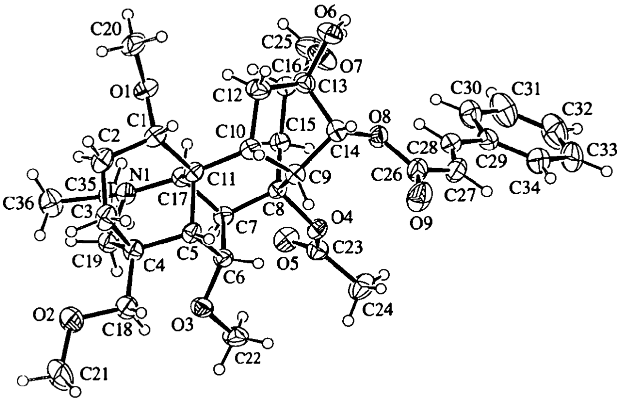


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (1) with the atomic numbering scheme. Displacement ellipsoids are plotted at the 30% probability level and H atoms are drawn as small circles of arbitrary radii.

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† Alternative name: [1α,6α,14α(*E*),16β]-20-ethyl-13-hydroxy-1,6,16-trimethoxy-4-methoxymethylaconitane-8,14-diyl 8-acetate 14-(3-phenyl-2-propenoate).

Experimental

Chasmanthinine was isolated from the roots of *A. chasmanthum*, collected from Rescuta Top in Azad Jammu Kashmir, using a method described previously (Parvez *et al.*, 1998). The pure compound was recrystallized from ethanol at room temperature by slow evaporation.

Crystal data

$C_{36}H_{49}NO_9$
$M_r = 639.78$
Orthorhombic
$P2_12_12_1$
$a = 8.300\,(2)$ Å
$b = 14.813\,(5)$ Å
$c = 27.654\,(6)$ Å
$V = 3400.0\,(16)$ Å^3
$Z = 4$
$D_x = 1.250$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 20$–$30°$
$\mu = 0.727$ mm^{-1}
$T = 293\,(1)$ K
Prismatic
0.50 × 0.43 × 0.40 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
5751 measured reflections
5037 independent reflections
4727 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.041$

$\theta_{\rm max} = 67.6°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 17$
$l = -30 \rightarrow 33$
3 standard reflections every 200 reflections frequency: 120 min intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.051$
$wR(F^2) = 0.126$
$S = 1.045$
5002 reflections
459 parameters
H atoms riding
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 2.05P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.010$
$\Delta\rho_{\rm max} = 0.237$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.201$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute configuration: Flack (1983)
Flack parameter = 0.1 (3)

Based on the systematic absences of: $h00$, $h = 2n + 1$; $0k0$, $k = 2n + 1$, and $00l$, $l = 2n + 1$, the space group was uniquely determined to be $P2_12_12_1$ (No. 19). Friedel pairs (2383) were collected to $\theta_{\rm max} = 60°$ and were not merged. Two methoxy groups attached to C4 and C16 were disordered and their atoms were allowed to refine at two locations with site-occupancy factors of 0.50. The phenyl ring was refined as a regular hexagon. The H atoms were included at geometrically idealized positions with C—H and O—H distances of 0.95 Å. Non-H atoms were given anisotropic displacement parameters.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) in *TEXSAN*. Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1328). Services for accessing these data are described at the back of the journal. A unit-cell packing diagram has also been deposited.

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$\alpha\alpha'$-Diselenocyanato-*ortho*-xylene†

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Abstract

Molecules of the title compound, $C_{10}H_8N_2Se_2$, stack in a one-dimensional ladder configuration, with relatively short Se···N interactions between neighbours.

Comment

The title compound, (I), is commonly used in the synthesis of cyclophanes incorporating selenium (Akabori

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† Alternative name: 1,2-phenylenebis(methylselenocyanate).