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Cyclobuxophyllinine M, an alkaloid from *Buxus microphylla*

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The title compound (systematic name: 17-ethylidene-4,4,13,14-tetramethyl-3-methylaminododecahydrocyclopropa[9,10]cyclopenta[*a*]phenanthren-16-one), $C_{25}H_{39}NO$, was semi-synthesized from cyclovirobuxine D. It has a four-ring triterpenoid nucleus in a *trans–cis–trans* configuration. The carbonyl and methyl groups have a *trans* configuration about the C=C bond in the α,β-unsaturated ketone group.

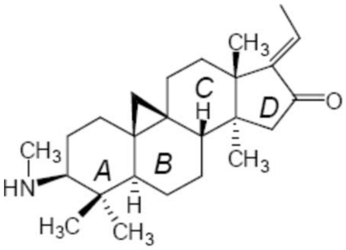
Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.005 Å
R factor = 0.058
wR factor = 0.119
Data-to-parameter ratio = 8.9

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

Comment

Buxus alkaloids are known for their interesting physiological activities. As part of a continuing structural study of the fused four-ring triterpenoid compounds, the structural analysis of the title compound, (I), was undertaken to confirm the conclusion of chemical work and to provide further structural data for the *Buxus* alkaloid obtained from the natural source. We have semi-synthesized the title compound, (I), from naturally occurring cyclovirobuxine D.



(I)

The title compound, cyclobuxophyllinine M, was obtained from the leaves and twigs of *Buxus microphylla* Sieb, *et* Zucc. *var. suffrulicosa Makinoforma major* Maino (Nakano *et al.*, 1966). The genus *Buxus* is known to possess anticholinesterase properties (Choudhary *et al.*, 2003).

In the molecule of (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The short C1—C2 bond of 1.491 (5) Å is ascribed to delocalization of the C2=C19 double bond. The structure of (I) contains a fused four-ring triterpenoid system: *A*, *B*, *C* and *D*. Similar to cyclovirobuxine D (Choudhary *et al.*, 2003), the triterpenoid nucleus has a *trans–cis–trans* configuration for ring junctions *A*/*B*, *B*/*C* and *C*/*D*. Rings *A*, *B* and *C* are not planar, having total puckering amplitudes, Q_T of 0.572 (4), 0.526 (4) and 0.661 (3) Å, respectively, and chair conformations [Φ = 1(6)°, θ = 3.8 (4)°; Φ = 91.2 (5)°, θ = 134.9 (3); and Φ = 33.7 (3)°, θ = 72.8 (3)°; Cremer & Pople, 1975]. The conformation of ring *D* is an envelope, with atom C4 at the flap position, 0.609 (3) Å from the mean plane through the other four atoms.

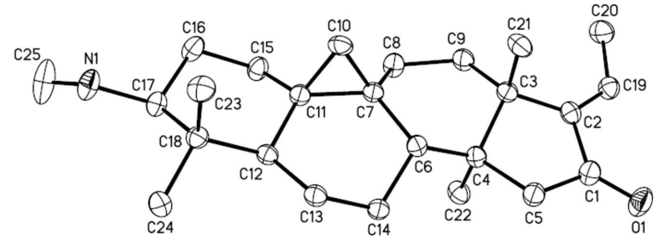


Figure 1
Molecular structure of (I) with the numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted.

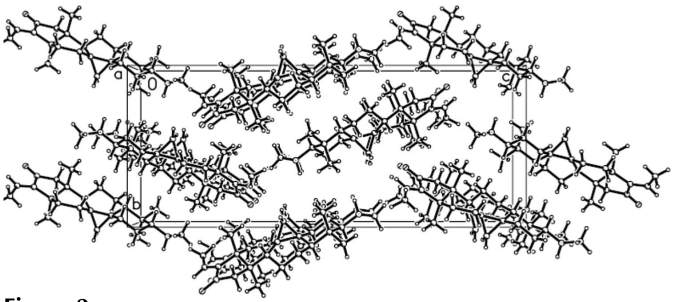


Figure 2
Projection of the structure down the *a* axis.

The dimethylamino substituent is attached to ring *A*; the torsion angles C15—C16—C17—N1 [−178.3 (3)°] and C25—N1—C17—C16 [77.2 (5)°] are indicative of a (+)-synclinal conformation. The sum of the bond angles around N1 (333.4°) indicates sp^3 character.

Experimental

The crude alkaloid of cyclovirobuxine D was purified and recrystallized (Liu *et al.*, 2006). The title compound was then semi-synthesized from cyclovirobuxine D according to the literature method (Desai *et al.*, 1981) followed by recrystallization from acetone. The melting point of the purified compound is 434–435 K (acetone, decomp.).

Crystal data

$C_{25}H_{39}NO$
M_r = 369.57
Orthorhombic, $P2_12_12_1$
a = 7.1102 (13) Å
b = 11.054 (2) Å
c = 27.294 (5) Å
V = 2145.2 (7) Å^3
Z = 4
Mo *Kα* radiation
μ = 0.07 mm^{-1}
T = 193 (2) K
0.42 × 0.40 × 0.20 mm

Data collection

Rigaku Mercury diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.776, T_{max} = 0.987
20717 measured reflections
2269 independent reflections
2128 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.119
S = 1.01
2269 reflections
255 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

The H atom on N1 was located in a difference map and refined. All other H atoms were positioned geometrically, with C—H = 1.00, 0.99 and 0.98 Å for methine, methylene and methyl H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.2$ for methine and methylene H, and $x = 1.5$ for all other H atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration was assigned consistent with the starting material.

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1994); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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