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4,5,9,14-Tetramethyl-19-norcholest-24-en-3-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.137
Data-to-parameter ratio = 9.7

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

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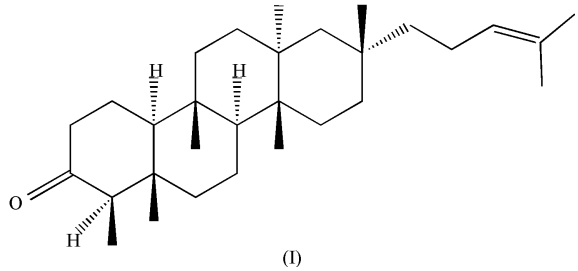
The title compound, $C_{30}H_{50}O$, was isolated from the Chinese medicinal plant *Aster tataricus* L., which has been used for the relief of coughs and as an expectorant. In the molecule, a tetracyclic ring system, with all six-membered rings in chair conformations, is linked to a C_6 side chain.

Comment

Triterpenoids represent an important class of natural product characterized by highly pronounced biological properties, such as analgesic activity and anti-mutagenic (Villasenor *et al.*, 2004) or anti-inflammatory activity (Matsuda *et al.*,1999; Janaki *et al.*,1999). In this paper, the structure of the title compound, (I), is reported.



(I)

Compound (I) was isolated from *Aster tataricus* L. It possesses a tetracyclic ring system, with all six-membered rings in chair conformations, and a C_6 side-chain.

Experimental

4,5,9,14-Tetramethyl-19-norcholest-24-en-3-one was isolated from *Aster tataricus* L. Column chromatography of the petroleum-ether-soluble portion of the ethanol extract of *A. tataricus* roots on silica gel (petroleum ether/ethyl acetate = 10:1) afforded a triterpenoid ketone fraction. Crystallization of the ketone fraction from acetone–

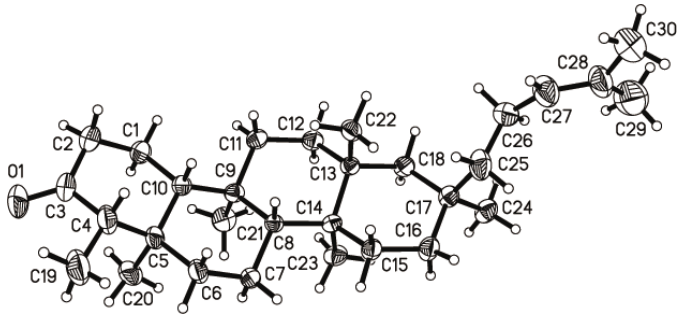


Figure 1
The molecular structure of (I), drawn with 30% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radii.

methanol (1:1), followed by preparative high-performance liquid chromatography of the filtrate portion, eventually yielded the title compound, (I) (Akihisa *et al.*, 1999). A quantity of (I) (30 mg) was dissolved in acetone (15 ml). The solution was kept at room temperature for 10 d and natural evaporation gave colourless single crystals of (I), suitable for X-ray analysis.

Crystal data

$C_{30}H_{50}O$	D_x = 1.057 Mg m^{-3}
M_r = 426.70	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 2200 reflections
a = 11.330 (2) Å	θ = 2.5–25.9°
b = 6.9142 (13) Å	μ = 0.06 mm^{-1}
c = 17.913 (3) Å	T = 293 (2) K
β = 107.145 (3)°	Block, colourless
V = 1341.0 (4) Å^3	0.24 × 0.20 × 0.16 mm
Z = 2	

Data collection

Bruker SMART CCD area-detector diffractometer	R_{int} = 0.032
φ and ω scans	θ_{max} = 27.3°
8242 measured reflections	$h = -14 \rightarrow 14$
3196 independent reflections	$k = -7 \rightarrow 8$
2017 reflections with $I > 2\sigma(I)$	$l = -13 \rightarrow 23$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0697P)^2 + 0.0717P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.048	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.137	$\Delta\rho_{max}$ = 0.12 e Å^{-3}
S = 1.02	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
3196 reflections	
330 parameters	
H-atom parameters constrained	

H atoms were positioned geometrically and treated as riding, with C—H distances in the range 0.92–0.98 Å and with U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for methyl groups. In the absence of significant anomalous scattering, Friedel pairs were merged, and the absolute configuration was assigned arbitrarily.

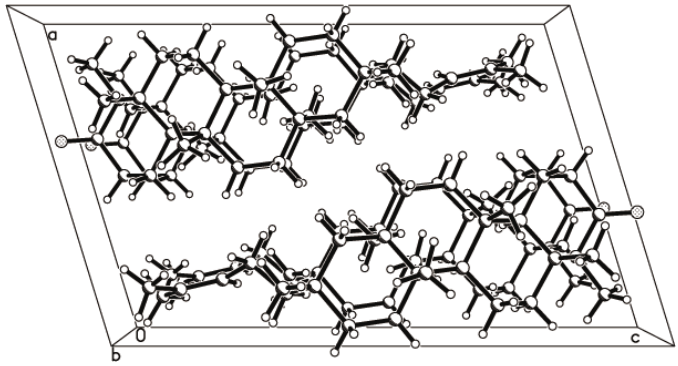


Figure 2
The crystal structure of (I), viewed along the *a* axis

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

References

Akihisa, T., Kimura, Y., Tai, T. & Arai, K. (1999). *Chem. Pharm. Bull.* **47**, 1161–1163.
Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Versions 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Janaki, S., Vijayasekaran, V., Viswanathan, S. & Balakrishna, K. (1999). *J. Ethnopharmacol.* **67**, 45–51.
Matsuda, H., Kageura, T., Toguchida, I., Murakami, T., Kishi, A. & Yoshikawa, M. (1999). *Bioorg. Med. Chem. Lett.* **9**, 3081–3086.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Villasenor, I. M., Canlas, A. P., Faustino, K. M. & Plana, K. G. (2004). *J. Ethnopharmacol.* **92**, 53–56.