

**(3 β ,5 α ,6 β ,7 β ,14 β)-Eudesmen-3,5,6,11-tetrol
methanol solvate: a new sesquiterpenoid
from *Chrysanthemum indicum* L.**Xiao-Lan Wang,^a Shu-Lin Peng,^b
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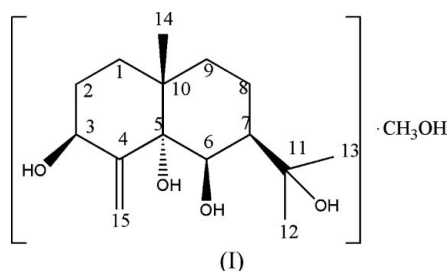
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Key indicatorsSingle-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.033
 wR factor = 0.068
Data-to-parameter ratio = 8.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound [systematic name: (3*S*,5*S*,6*R*,7*R*,10*S*)-7-(2-hydroxy-2-propyl)-10-methyl-4-methyleneperhydronaphthalene-3,5,6-triol methanol solvate], $\text{C}_{15}\text{H}_{26}\text{O}_4 \cdot \text{CH}_4\text{O}$, is a new sesquiterpenoid which was isolated from *Chrysanthemum indicum* L. The molecule contains two fused six-membered rings in chair conformations. There are methanol solvent molecules in the crystal structure. All molecules are linked by hydrogen bonds to give the three-dimensional structure.

Received 23 April 2006
Accepted 21 July 2006**Comment**

Chrysanthemum indicum L. is a traditional Chinese herb distributed widely in China. The inflorescence of *C. indicum* has been used for the treatment of vertigo, hypertensive symptoms, pneumonia, colitis, stomatitis, carbuncles and fever for a long time (Jiangsu New Medical College, 1993). Previous studies have reported that *C. indicum* possesses antibacterial, antiviral, anti-oxidant, anti-inflammatory and immunomodulatory properties (Wang *et al.*, 2000). Our present investigation of the inflorescence of this herb led to the isolation of (3 β ,5 α ,6 β ,7 β ,14 β)-eudesmen-3,5,6,11-tetrol methanol solvate, (I). The structure of (I) was elucidated by comprehensive spectroscopic analysis, and was confirmed by the single-crystal X-ray diffraction analysis reported here. The molecule contains two *trans* fused six-membered rings ($A = \text{atoms C1}-\text{C5/C10}$, $B = \text{C5}-\text{C10}$), which both adopt chair conformations (Fig. 1).



There are five kinds of hydrogen bonds in the crystal structure (Table 1). These form helices parallel to the c axis. Each helix is connected to six others by further hydrogen bonds, forming an extensive apiary-like crystal structure (Fig. 2)

Experimental

The dry inflorescence of *C. indicum* (10 kg) was collected in Hubei province, People's Republic of China. The methanol extract (600 g) was suspended in water (3.0 l) and partitioned successively with petroleum ether, EtOAc and *n*-butanol. The EtOAc extract (150 g)

was subjected to silica gel column chromatography (160–200 mesh, 1.8 kg) and eluted with CHCl_3 /methanol in increasing polarity. The fraction eluted with CHCl_3 /methanol (10:1) afforded the pure title compound (1) as colorless crystals (m.p. 470–473 K). Suitable crystals were obtained by slow evaporation of a methanol solution at room temperature. ^{13}C NMR (600 MHz, acetone, p.p.m.): δ 154.0 (C4), 106.4 (C15), 77.3 (C5), 75.2 (C6), 74.0 (C11), 69.5 (C3), 44.3 (C7), 37.7 (C10), 36.6 (1), 35.2 (C9), 33.1 (C2), 29.5 (C13), 28.9 (C12), 21.5 (C14), 17.3 (C8).

Crystal data

$\text{C}_{15}\text{H}_{26}\text{O}_4 \cdot \text{CH}_4\text{O}$
 $M_r = 302.40$
 Hexagonal, $P6_1$
 $a = 9.9315$ (8) Å
 $c = 29.113$ (5) Å
 $V = 2486.8$ (5) Å³
 $Z = 6$

$D_x = 1.212$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ (2) K
 Prism, colorless
 $0.56 \times 0.52 \times 0.48$ mm

Data collection

Siemens P4 diffractometer
 ω scans
 Absorption correction: none
 4581 measured reflections
 1881 independent reflections
 1495 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$
 $\theta_{\text{max}} = 27.2^\circ$
 3 standard reflections
 every 97 reflections
 intensity decay: 2.3%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.068$
 $S = 1.00$
 1881 reflections
 215 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0336P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.13$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.13$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0041 (6)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O1}-\text{H1O} \cdots \text{O2}^{\text{i}}$	0.810 (10)	2.080 (11)	2.884 (2)	171 (3)
$\text{O2}-\text{H2O} \cdots \text{O5}$	0.820 (10)	2.146 (11)	2.953 (3)	168 (2)
$\text{O3}-\text{H3O} \cdots \text{O4}$	0.822 (10)	1.959 (16)	2.702 (2)	150 (3)
$\text{O4}-\text{H4O} \cdots \text{O1}^{\text{ii}}$	0.818 (10)	1.982 (11)	2.795 (3)	172 (3)
$\text{O5}-\text{H5O} \cdots \text{O3}^{\text{iii}}$	0.823 (10)	1.968 (11)	2.789 (3)	175 (4)

Symmetry codes: (i) $y, -x+y, z - \frac{1}{6}$; (ii) $x+1, y+1, z$; (iii) $x-y, x, z + \frac{1}{6}$.

C-bound H atoms were positioned geometrically ($C-H = 0.93-0.98$ Å), and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The O-bound H atoms were located in difference Fourier syntheses and refined with the distance restraint $O-H = 0.82$ (1) Å. The absolute configuration could not be determined from the X-ray analysis, owing to the absence of strong anomalous scatterers, and Friedel pairs were averaged. However, the absolute configuration was assigned by reference to the chiral molecule of known absolute configuration which had been confirmed on a biogenetic basis (Southwell, 1977).

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Siemens, 1997); 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*.

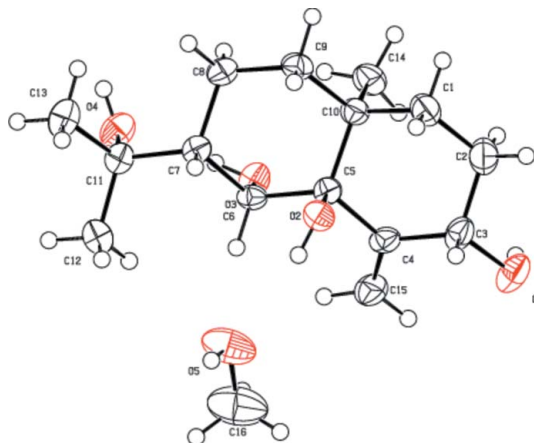


Figure 1

The asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

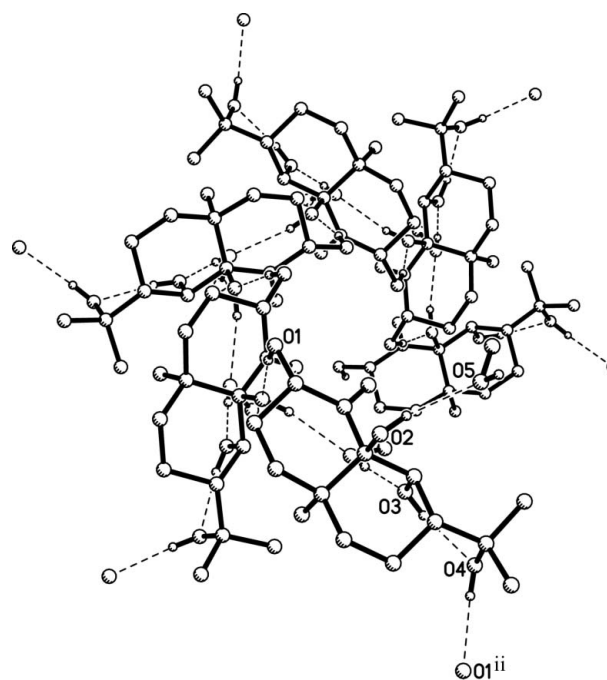


Figure 2

The apy-like crystal structure of (I), with hydrogen bonds indicated by dashed lines. The symmetry code is as in Table 1.

We are grateful to the staff of the Analytical Group of Chengdu Institute of Biology, Chinese Academy of Sciences, for measuring the NMR spectra.

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