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Trilobolide-6-*O*-isobutyrate, a eudesmanolide from *Wedelia trilobata*

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
R factor = 0.051
wR factor = 0.153
Data-to-parameter ratio = 7.6

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

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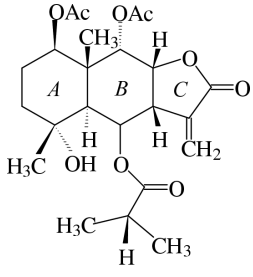
The structure of trilobolide-6-*O*-isobutyrate, $C_{23}H_{32}O_9$, isolated from the flower of *Wedelia trilobata*, shows an eudesmanolide sesquiterpene skeleton constructed from the fusion of two cyclohexane rings and a lactone ring.

Comment

Wedelia is a large genus of the Compositae family from which many eudesmanolide sesquiterpenes have been isolated in recent years (Bohlmann *et al.*, 1981; Farag *et al.*, 1996; Ferreira *et al.*, 1994; Ragasa *et al.*, 1993). *Wedelia trilobata*, a vigorous creeping herb native to tropical areas, was introduced into Hong Kong and utilized as a substitute for *W. chinensis*, a traditional Chinese medicine used for the treatment of the common cold, hepatitis, indigestion and infections (Jiangsu New Medical College, 1977). As part of our effort to search for antitumor agents from natural sources, the investigation of the chemical components of *W. trilobata* flowers has been undertaken. In this connection, the title compound, trilobolide-6-*O*-isobutyrate, (I), has been isolated and studied by X-ray crystallography. Compound (I) was previously isolated from *W. trilobata* (Bohlmann *et al.*, 1981) and *W. prostrata* (Farag *et al.*, 1996) and its chemical structure was elucidated by spectroscopic methods.



(I)

Compound (I) (Fig. 1 and Table 1) is characterized by the fusion of two cyclohexane rings (rings *A* and *B*) with a lactone ring (ring *C*). Rings *A* and *B* are *trans*-fused and rings *B* and *C* are *cis*-fused. Ring *A* exists in a chair conformation, with a mean torsion angle of 56° (ideal angle = 56°). Ring *B* exists in a twist-chair conformation, as indicated by the mean torsion angle of 47°. Finally, the five-membered lactone ring *C* adopts an envelope conformation, with atom C8 displaced by 0.42 Å from the least-squares plane of the remaining four atoms. The acetoxyl group at C1 and the hydroxy group at C4 adopt equatorial positions. The methyl groups at C4 and C10, the isobutyrate group at C6 and the acetoxyl group at C9 occupy axial positions.

An intermolecular hydrogen bond, involving O5—H and O9^{i}, with an O5···O9 distance of 2.986 (3) Å, is noted [symmetry code: (i) x, $y-1$, z].

Experimental

The flowers of *Wedelia trilobata* growing in Hong Kong were collected in April 2002. The pulverized dried flowers (500 g) were extracted with MeOH three times under reflux. The extract was concentrated *in vacuo* to give a residue (98.8 g). A large portion of the residue (90 g) was suspended in distilled water and partitioned with petroleum ether, $CHCl_3$, EtOAc and *n*-BuOH successively. The $CHCl_3$ fraction (4.58 g) was subjected to column chromatography over silica gel (Merck, 60 g) and eluted with a gradient hexane–EtOAc system (from 0:100 to 100:0) to afford 26 fractions. Trilobolide-6-*O*-isobutyrate (80 mg) was obtained from fraction 23 and further recrystallized from an acetone solution of the compound.

Crystal data

$C_{23}H_{32}O_9$	Mo $K\alpha$ radiation
M_r = 452.49	Cell parameters from 4824 reflections
Hexagonal, $P6_5$	θ = 2.4–20.8°
a = 9.8368 (8) Å	μ = 0.10 mm^{-1}
c = 42.455 (5) Å	T = 293 (2) K
V = 3557.6 (6) Å^3	Block, colorless
Z = 6	0.62 × 0.34 × 0.29 mm
D_x = 1.267 Mg m^{-3}	

Data collection

Siemens SMART/CCD diffractometer	R_{int} = 0.063
ω scans	θ_{max} = 25.0°
19275 measured reflections	$h = -11 \rightarrow 7$
4172 independent reflections	$k = -11 \rightarrow 11$
3029 reflections with $I > 2\sigma(I)$	$l = -50 \rightarrow 50$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.105P)^2 + 0.2647P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.051	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.153	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
S = 1.04	$\Delta\rho_{min}$ = −0.31 e Å^{-3}
2119 reflections	
280 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C16	1.359 (7)	O6—C20	1.355 (6)
O1—C1	1.464 (6)	O6—C6	1.463 (5)
O2—C16	1.185 (8)	O7—C20	1.201 (6)
O3—C18	1.346 (6)	O8—C12	1.338 (6)
O3—C9	1.465 (6)	O8—C8	1.456 (5)
O4—C18	1.186 (7)	O9—C12	1.211 (6)
O5—C4	1.439 (6)	C11—C13	1.299 (8)
C13—C11—C12	123.0 (5)	C12—C11—C7	105.5 (4)
C13—C11—C7	131.4 (5)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O9^{i}	0.82	2.19	2.986 (3)	163

Symmetry code: (i) x, $y-1$, z.

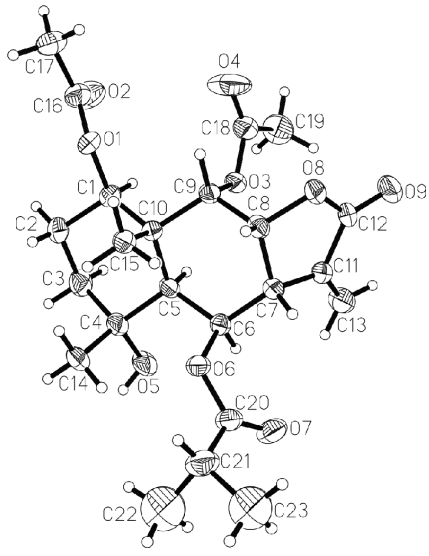


Figure 1
The molecular structure of (I), showing the atom-labeling scheme and ellipsoids drawn at the 30% probability level (Bruker, 1998).

H atoms were included in the riding model approximation and assigned $U_{iso}(H) = 1.2U_{eq}$ (CH and CH_2) and $1.5U_{eq}$ (CH_3) of the atom to which they were bonded. The hydroxy H atom was located from a difference map and its O—H distance was restrained to 0.82 Å. Terminal atoms C22 and C23 exhibit significant thermal motion and were refined isotropically. In the absence of atoms with a strong anomalous scattering contribution, reliable crystallographic determination of the absolute stereochemistry cannot be ascertained, and so Friedel pairs were merged. The enantiomers shown are chosen arbitrarily.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART* and *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL/PC*; molecular graphics: *XP* (Bruker, 1998); software used to prepare material for publication: *SHELXTL/PC*.

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