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3,6-Di-*O*-acetylswietenolide 0.25-hydrate

Robert G. Fowles,[a] Baldwin S. Mootoo,[a] Russel Ramsewak,[a] William Reynolds[b] and Alan J. Lough[b]*

[a]Department of Chemistry, University of the West Indies, St Augustine, Trinidad and Tobago, and [b]Department of Chemistry, University of Toronto, Toronto, Ontario, Canada M5S 3H6

Correspondence e-mail:
alough@chem.utoronto.ca

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
Disorder in solvent or counterion
R factor = 0.039
wR factor = 0.098
Data-to-parameter ratio = 9.6

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

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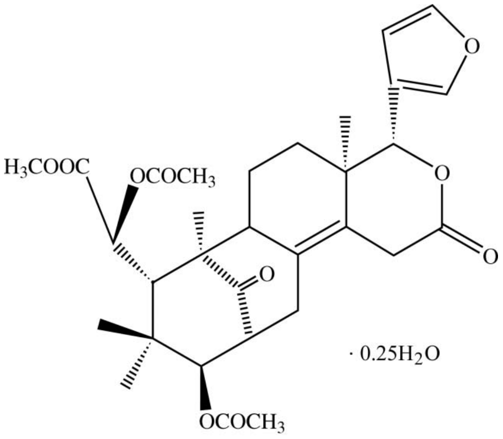
The title compound, $C_{31}H_{38}O_{10}\cdot0.25H_2O$, was extracted from *Swietenia macrophylla*, a plant belonging to the Meliaceae family. The crystal structure is stabilized by O—H···O and weak C—H···O hydrogen bonds.

Comment

The title compound, (I), a B,D-*seco*-limonoid (bicyclononalide), has been isolated from the ethyl acetate extract of the seeds of *Swietenia macrophylla* belonging to the Meliaceae family. The seeds of *Swietenia macrophylla* are eaten by the natives of Malaysia and are thought to be a treatment for high blood pressure (Chan *et al.*, 1976). The compound exhibits antifeedant activity against *Spodoptera frugiperda* (Mootoo *et al.*, 1999). Preliminary studies conducted on this compound involved the use of the following bioassays: antifeedant (neonate larvae of *Helicoverpa zea*, *Heliothis virescens*, *Manduca sexta*), toxicity (*Artemia salina*), anti-oxidant, antibacterial, antifungal, anti-inflammatory and mosquitocidal (*Ochlerotatus triseriatus*). The crystal structure of the related compound swietenine, also extracted from *Swietenia macrophylla*, has recently been determined (Solomon *et al.*, 2003).



(I)

The molecular structure of (I) is shown in Fig. 1. The four six-membered rings, C1–C6, C1/C6–C9/O1, C4/C5/C15–C18 and C16–C18/C20–C22 are in sofa, half-chair, chair and boat conformations, respectively, giving values for the total puckering amplitudes (Cremer & Pople, 1975) of 0.460 (2), 0.635 (3), 0.639 (3) and 0.764 (3), respectively. The five-membered ring, O3/C11–C13, is essentially planar, with an r.m.s. deviation of fitted atoms of 0.003 Å.

The crystal structure of (I) is stabilized by O—H···O and weak C—H···O hydrogen bonds (Table 2).

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Experimental

Crude ethyl acetate extracts (17.98 g) were obtained from the seeds of *Swietenia macrophylla*. The sample was dissolved in chloroform–ethyl acetate (50:1), placed on a silica-gel column (Merck, 70–230 mesh, 350 g) and eluted with chloroform–ethyl acetate (50:1) followed by increasing amounts of ethyl acetate (25:1, 10:1, 5:1, 1:1, 100% ethyl acetate). The fractions were grouped on the basis of thin-layer chromatographic analysis into eight major fractions (*A*–*H*). Crystallization of fraction *D* (4.67 g) from methanol afforded compound (I) (364 mg). The title compound was identified as 3,6-*O*,*O*-diacetylswietenolide on the basis of one- and two-dimensional ^{1}H and ^{13}C NMR data, which agrees with the data reported for this compound by Kadota *et al.* (1990).

Crystal data

$C_{31}H_{38}O_{10}\cdot 0.25H_2O$
$M_r = 575.12$
Orthorhombic, $P2_12_12_1$
$a = 12.5030$ (2) Å
$b = 13.7366$ (2) Å
$c = 16.7164$ (4) Å
$V = 2871.02$ (9) Å^3
$Z = 4$
$D_x = 1.331$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 150$ (1) K
Block, colourless
$0.28 \times 0.20 \times 0.18$ mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min} = 0.905$, $T_{max} = 0.988$
24546 measured reflections
3660 independent reflections
2970 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.098$
$S = 1.03$
3660 reflections
382 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0534P)^2 + 0.3768P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXTL/PC* (Sheldrick, 2001)
Extinction coefficient: 0.0103 (14)

Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
O1*W*—H1*W*$\cdots$O2	0.84	1.82	2.662 (11)	180
O1*W*—H2*W*$\cdots$O6^{i}	0.84	2.34	3.180 (11)	180
C2—H2*A*$\cdots$O9	0.99	2.53	3.515 (3)	172
C3—H3*B*$\cdots$O6^{ii}	0.99	2.42	3.260 (3)	142
C7—H7*A*$\cdots$O8^{iii}	0.99	2.51	3.372 (3)	146
C12—H12*A*$\cdots$O2^{iv}	0.95	2.42	3.355 (3)	170
C15—H15*A*$\cdots$O8^{iii}	0.99	2.43	3.396 (3)	164
C20—H20*A*$\cdots$O5	1.00	2.54	3.000 (3)	108
C22—H22*A*$\cdots$O6	1.00	2.24	2.707 (4)	107
C23—H23*A*$\cdots$07	0.98	2.43	3.000 (3)	116
C24—H24*C*$\cdots$O10	0.98	2.59	3.271 (3)	126
C26—H26*A*$\cdots$O8^{iii}	0.98	2.60	3.087 (4)	111

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+\frac{1}{2}, -y+1, z-\frac{1}{2}$; (iii) $-x+\frac{1}{2}, -y+1, z+\frac{1}{2}$; (iv) $-x+\frac{1}{2}, -y, z-\frac{1}{2}$.

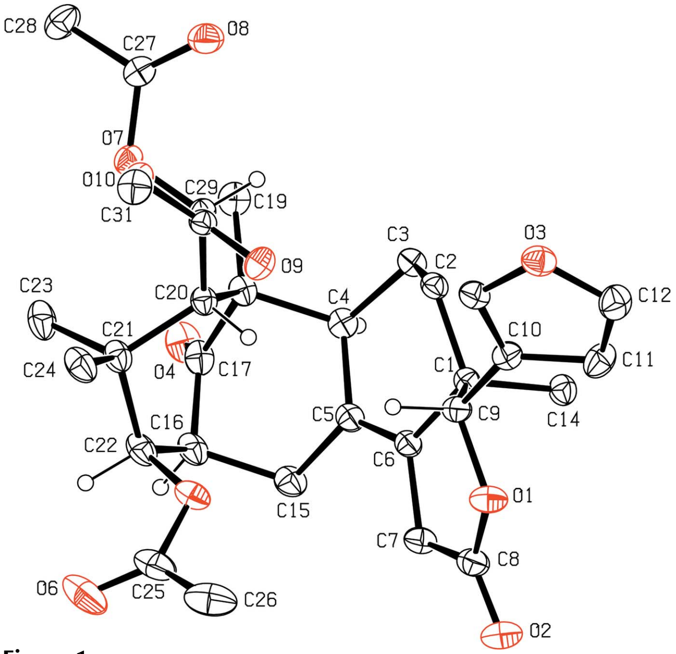

Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms bonded to asymmetric C atoms have been included in order to illustrate the stereochemisty. Other H atoms and the partially occupied solvent water molecule are not shown.

In the absence of significant anomalous dispersion effects, Friedel pairs were merged and the absolute stereochemistry was assigned on the basis of the investigations of McPhail & Sim (1964, 1966). The occupancy of the solvent water O atom refined to close to 0.25 and was eventually fixed at this value. H atoms bonded to C atoms were placed in calculated positions, with C—H = 0.95–1.00 Å. They were included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C_{methyl})$. The H atoms of the partially occupied solvent water molecule were placed in positions most likely in terms of hydrogen bonding and then refined in the riding-model approximation, with $U_{iso}(H) = 1.5U_{eq}(O)$.

Data collection: *COLLECT* (Bruker Nonius, 2002); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL/PC* (Sheldrick, 2001); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXTL/PC*.

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