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Dasypogalactone–methanol–water (1/2/1)

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Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.008 Å
H-atom completeness 97%
R factor = 0.050
wR factor = 0.085
Data-to-parameter ratio = 11.1

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

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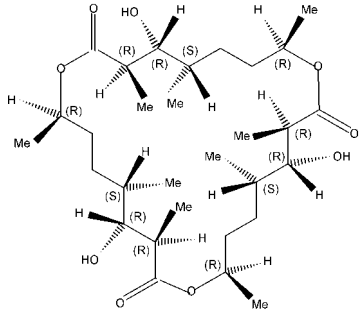
In the crystal structure of the title compound, 4,12,20-trihydroxy-3,5,8,11,13,16,19,21,24-nonamethyl-1,9,17-trioxacyclotetracosane-2,10,18-trione–methanol–water (1/2/1), $C_{30}H_{54}O_9\cdot 2CH_4O\cdot H_2O$, the molecules are packed parallel to [100], with various hydrogen bonds to trapped CH_3OH and H_2O solvent molecules.

Comment

In an earlier study, we reported the isolation of dasypogalactone from the Indonesian lichen *Usnea Dasypoga* Rohl. and proposed its molecular structure from mass spectra and NMR data (Suwarso *et al.*, 1999). However, the configuration of dasypogalactone was not fully established. Now, we have succeeded in growing crystals, still of poor quality and scattering power [only 1961 intensities with $I > 2\sigma(I)$], but at least suitable for a single-crystal X-ray analysis. It was not possible to determine the absolute configuration, but the former structure was confirmed with the correct relative configuration assigned as (2,3,7*R**)(4*S**)-3,7-dihydroxy-2,4-dimethyloctanoic acid for the monomeric acid unit (see *Scheme*).



(I)

The molecule of the title compound, (I), exhibits non-crystallographic C_3 symmetry, with the OH groups (O3, O6, O9; for numbering see Fig. 1) lying in the ring plane and directed towards the ring centre. Their intramolecular non-bonding distances are O3···O6 = 3.966 (5), O3···O9 = 3.544 (5) and O6···O9 = 3.978 (5) Å. Carboxylic acid O atoms O2, O5 and O8 point to one side of the ring plane and the three methyl groups C6, C16 and C26 to the other. The three parts of the ring show almost equal bond geometries at the 3σ level. In the crystal structure, the molecules are packed head-to-tail along [100] (Fig. 2), with a plane-to-plane distance of 8.898 (1) Å. Enclosed solvent molecules, one water and two methanol per asymmetric unit, are linked to this host lattice by various hydrogen bonds. The MeOH solvent molecule 2 (C200/O200) is situated, sandwich-like, midway between two dasypogalactone rings (Fig. 3), with C200 and O200 4.390 (7)

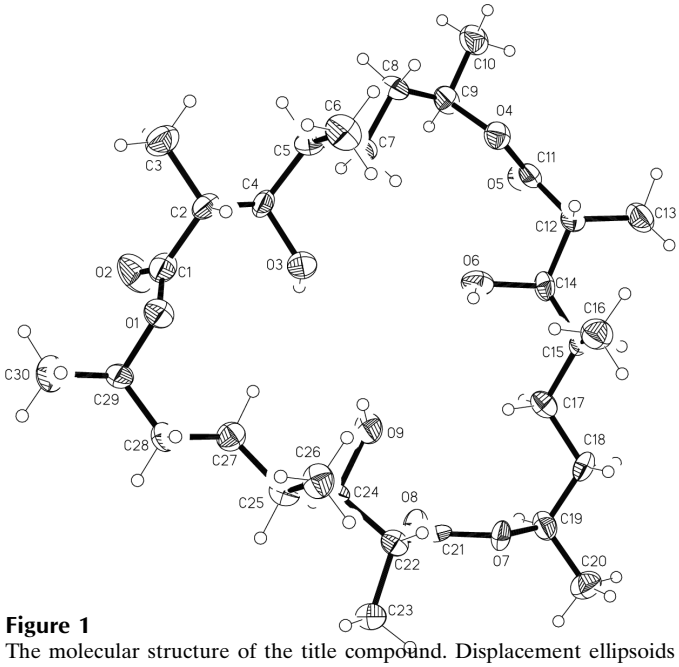


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level, and solvent molecules are omitted.

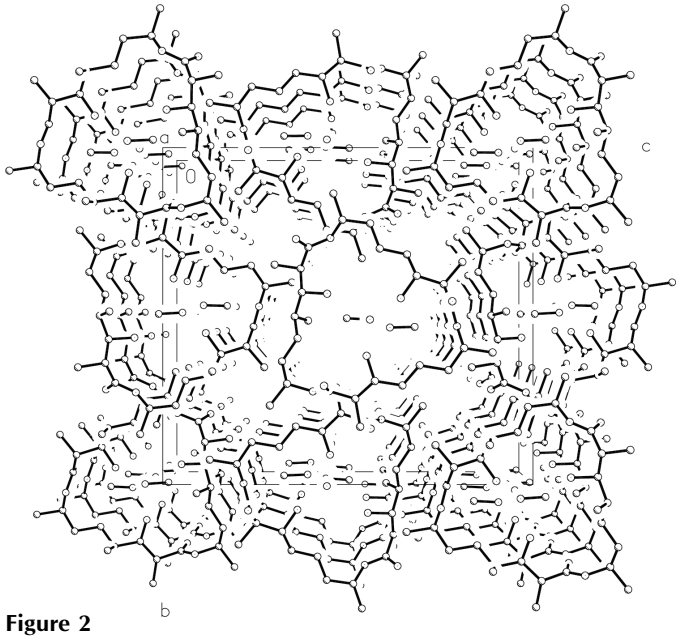


Figure 2
Packing diagram, viewed along [100], with H atoms omitted.

and 4.327 (5) Å, respectively, above the plane defined by O3, O6 and O9. This solvent molecule shows an O200—H200···O300 hydrogen bond to the water atom O300, with H200···O300 = 1.84 Å and an angle of 160°, and a weak C200—H20*E*···O2 interaction (2.43 Å; 158°). Atoms C100 and O100 of methanol solvent molecule 1 lie 2.601 (7) and 2.181 (5) Å above the O3/O6/O9 plane. The corresponding hydrogen bonds are O100—H100···O9 (1.95 Å, 149°) and C100—H10*D*···O6 (2.37 Å, 167°). The water atom O300 is located 6.967 (4) Å above the O3/O6/O9 plane; additional hydrogen bonds are O6—H6···O300($x+1$, y, z) (2.08 Å, 154°) and O9—H9···O300($x+1$, y, z) (2.12 Å, 126°). It was not possible to locate the water H atoms. All the above values are normalized for C—H = 1.08 Å and O—H = 0.938 Å. A somewhat similar packing of C_3- symmetric macrolides, with trapped water and methanol solvent molecules, was described recently (Burke & Zhao, 2000).

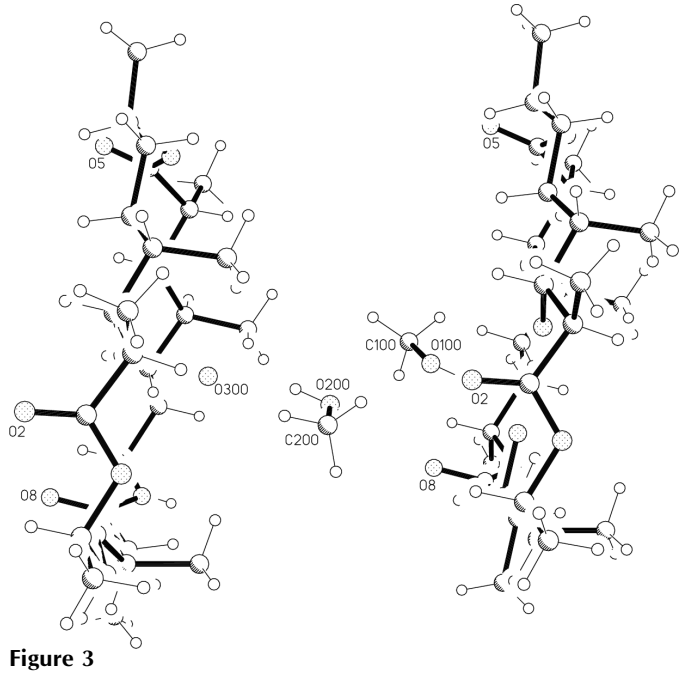


Figure 3
The position of the solvent molecules between two dasypogalactone rings.

Experimental

Isolation of the compound has already been described by Suwarso *et al.* (1999). The product was crystallized from a CH_2Cl_2/CH_3OH solution.

Crystal data

$C_{30}H_{54}O_9\cdot 2CH_4O\cdot H_2O$
M_r = 640.83
Orthorhombic, $P2_12_12_1$
a = 8.8977 (14) Å
b = 19.524 (3) Å
c = 21.445 (3) Å
V = 3725.5 (10) Å^3
Z = 4
D_x = 1.143 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 713 reflections
θ = 2.5–12.7°
μ = 0.09 mm^{-1}
T = 153 (2) K
Block, colourless
0.32 × 0.25 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.906, T_{max} = 0.953
22975 measured reflections
4594 independent reflections
1359 reflections with $I > 2\sigma(I)$
R_{int} = 0.108
θ_{max} = 27.1°
$h = -11 \rightarrow 8$
$k = -24 \rightarrow 24$
$l = -27 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.085
S = 0.86
4594 reflections
413 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0003P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.330 (7)	C4—C5	1.526 (7)
O1—C29	1.455 (6)	C5—C6	1.508 (7)
O2—C1	1.203 (7)	C5—C7	1.516 (7)
O3—C4	1.464 (5)	C7—C8	1.503 (6)
C1—C2	1.502 (8)	C8—C9	1.504 (7)
C2—C4	1.527 (6)	C9—C10	1.523 (6)
C2—C3	1.528 (6)		
C1—O1—C29	117.2 (5)	C2—C4—C5	115.4 (5)
O2—C1—O1	124.4 (7)	C6—C5—C7	109.3 (6)
O2—C1—C2	122.7 (7)	C6—C5—C4	113.4 (5)
O1—C1—C2	112.8 (6)	C7—C5—C4	110.1 (5)
C1—C2—C4	108.8 (5)	C8—C7—C5	116.6 (5)
C1—C2—C3	111.0 (5)	C7—C8—C9	113.3 (5)
C4—C2—C3	111.7 (5)	O4—C9—C8	105.5 (5)
O3—C4—C2	106.1 (5)	O4—C9—C10	109.4 (5)
O3—C4—C5	109.8 (5)	C8—C9—C10	113.5 (5)

H atoms were placed at calculated positions riding on the C or O atoms, with isotropic displacement parameters $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(CH_3/OH)$. All CH_3 and OH groups were allowed to rotate but not to tip. The H atoms of the water molecule could not be located and were, therefore, not included in the refinement. The title compound crystallizes in the non-centrosymmetric space group $P2_12_12_1$; however, in the absence of significant anomalous scattering effects, the Flack (1983) parameter is essentially meaningless. Accordingly, Friedel pairs were merged.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2002); program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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