Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.069$
$S = 1.055$
3996 reflections
285 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0271P)^2 + 2.643P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.48$ e Å^{-3}
$\Delta\rho_{\min} = -0.33$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.422 (16)

Table 3. *Selected geometric parameters (Å, °) for (2)*

Ni—N4	1.835 (3)	Ni—N1	1.881 (3)
Ni—N11	1.839 (3)	Ni—N8	1.882 (3)
N4—Ni—N11	179.42 (16)	C5—N4—C3	120.4 (3)
N4—Ni—N1	87.35 (12)	C5—N4—Ni	126.8 (3)
N11—Ni—N1	92.22 (13)	C3—N4—Ni	112.8 (2)
N4—Ni—N8	92.84 (12)	C7—N8—C9	120.3 (3)
N11—Ni—N8	87.58 (13)	C7—N8—Ni	129.5 (2)
N1—Ni—N8	179.54 (14)	C9—N8—Ni	109.8 (2)
C14—N1—C2	119.9 (3)	C12—N11—C10	120.2 (3)
C14—N1—Ni	129.5 (2)	C12—N11—Ni	127.6 (3)
C2—N1—Ni	110.3 (2)	C10—N11—Ni	112.1 (2)
N4—Ni—N1—C2	−14.9 (2)	Ni—N4—C3—C2	31.5 (4)
N1—Ni—N4—C3	−9.8 (3)	N1—C2—C3—N4	−41.7 (4)
N11—Ni—N8—C9	13.2 (2)	C7—N8—C9—C10	151.3 (3)
N8—Ni—N11—C10	12.5 (3)	Ni—N8—C9—C10	−34.9 (3)
C14—N1—C2—C3	−151.5 (3)	C12—N11—C10—C9	143.3 (3)
Ni—N1—C2—C3	34.8 (3)	Ni—N11—C10—C9	−34.7 (3)
C5—N4—C3—C2	−147.5 (3)	N8—C9—C10—N11	43.9 (4)

Table 4. *Hydrogen-bonding geometry (Å, °) for (2)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C98—D98···O1	1.00	2.33	3.206 (6)	146.2
C99—D99···O2	1.00	2.14	3.094 (5)	159.7

H atoms were located from ΔF syntheses and refined with NH free, rigid methyls and other H atoms riding. For compound (2), which crystallizes in a polar space group, the origin was fixed according to the method of Flack & Schwarzenbach (1988). The structure was refined as a racemic twin with components 0.422 (16) and 0.578 (16), utilizing 1739 Friedel pairs, which were adequate to define the Flack (1983) parameter; no further Friedel opposites were measured as the nozzle of the low-temperature device obscured most of those remaining.

For both compounds, data collection: *XSCANS* (Fait, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994*b*); software used to prepare material for publication: *SHELXL*97.

We thank the Fonds der Chemischen Industrie for financial support and Mr A. Weinkauf for technical assistance.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1271). Services for accessing these data are described at the back of the journal.

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1,2:4,5-Di-*O*-isopropylidenegalactitol†

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Abstract

The title compound, $C_{12}H_{22}O_6$, is composed of two isopropylidene rings, one with approximate *m* symmetry and the other with approximate C_2 symmetry, joined by an extended backbone. Hydrogen bonds connect the molecules into ribbons parallel to the *y* axis.

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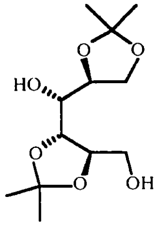
† IUPAC name: (2,2-dimethyl-1,3-dioxolan-4-yl)(5-hydroxymethyl-2,2-dimethyl-1,3-dioxolan-4-yl)methanol.

Comment

In the continuing course of our studies of the synthesis of macrocyclic compounds, we have prepared partially protected derivatives of dulcitol and mannitol that may serve as suitable building blocks for the synthesis of chiral macrocycles.

The structure determination of the title compound, 1,2:4,5-di-*O*-isopropylidenegalactitol, (1) (Fig. 1), confirms the expected nature of the product. Bond lengths and angles are normal. The O2—C2—C3—O3—C7 ring adopts an envelope (local *m*) conformation, with the O2 atom 0.399 (4) Å out of the plane of the other four atoms; the other five-membered ring has local twofold symmetry about O6 and the midpoint of the C5—O5 bond. The ring connection shows an extended conformation about the C3—C4 and C4—C5 bonds.



(1)

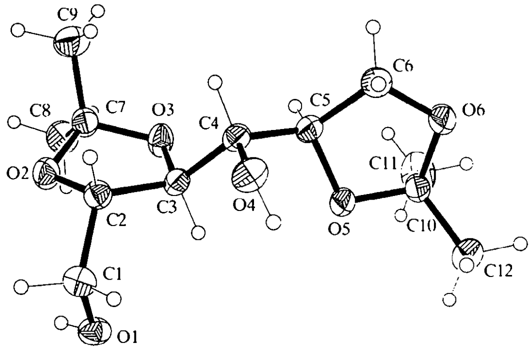


Fig. 1. The molecule of compound (1) in the crystal. Ellipsoids represent 50% probability levels and H-atom radii are arbitrary.

Both hydroxy groups function as hydrogen-bond donors, whereby the molecules are linked into ribbons parallel to the *y* axis (Fig. 2).

A search of the Cambridge Structural Database (Allen & Kennard, 1993) revealed only one other structure with two isopropylidene rings connected *via* a single carbon bridge (Herczegh *et al.*, 1995); however, large substituent groups (phenyl and modified piperidine) and the absence of hydrogen bonds render this structure not readily comparable with ours.

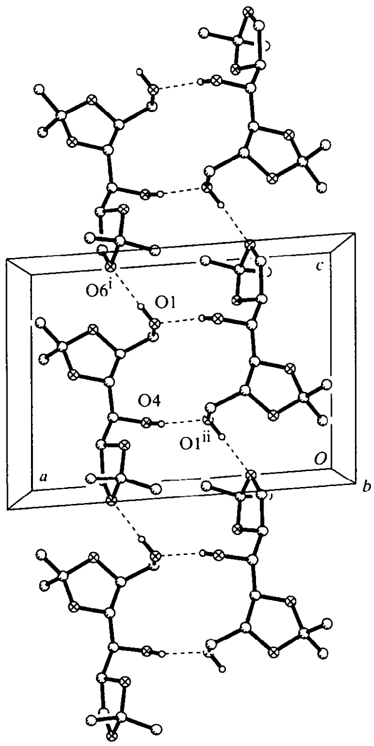


Fig. 2. Packing diagram of compound (1) viewed parallel to the *y* axis. Atomic radii are arbitrary and H atoms not involved in hydrogen bonding have been omitted for clarity. Hydrogen bonds are indicated by dashed lines. Symmetry codes are as in Table 2.

Experimental

The title compound (1) was obtained according to a known procedure (Hamm *et al.*, 1939). Single crystals were grown from ethanol.

Crystal data

$C_{12}H_{22}O_6$
$M_r = 262.30$
Monoclinic
$P2_1/c$
$a = 12.414\,(3)$ Å
$b = 11.891\,(3)$ Å
$c = 9.062\,(2)$ Å
$\beta = 94.23\,(2)^\circ$
$V = 1334.1\,(6)$ Å^3
$Z = 4$
$D_x = 1.306$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 50 reflections
$\theta = 10.0$–11.5°
$\mu = 0.104$ mm^{-1}
$T = 143\,(2)$ K
Prism
0.36 × 0.22 × 0.18 mm
Colourless

Data collection

Stoe Stadi-4 diffractometer
ω/θ scans
Absorption correction: none
3013 measured reflections
2351 independent reflections
1499 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$

$\theta_{\rm max} = 25.02^\circ$
$h = 0 \rightarrow 14$
$k = -3 \rightarrow 14$
$l = -10 \rightarrow 10$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.118$
$S = 1.063$
2351 reflections
175 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 0.2969P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected torsion angles (°)*

C7—O2—C2—C3	−26.5 (3)	C10—O6—C6—C5	−9.8 (3)
C7—O3—C3—C2	4.1 (3)	O5—C5—C6—O6	27.4 (3)
O2—C2—C3—O3	13.6 (3)	C2—O2—C7—O3	29.5 (3)
C2—C3—C4—C5	−179.8 (2)	C3—O3—C7—O2	−20.4 (3)
C10—O5—C5—C6	−35.5 (3)	C5—O5—C10—O6	30.1 (3)
C3—C4—C5—C6	178.8 (2)	C6—O6—C10—O5	−11.7 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1′···O6ⁱ	0.86 (3)	1.96 (4)	2.815 (3)	173 (4)
O4—H4′···O1ⁱⁱ	0.85 (3)	1.98 (3)	2.784 (3)	157 (3)

Symmetry codes: (i) $x, y, 1+z$; (ii) $1-x, 1-y, 1-z$.

H atoms were located from ΔF synthesis and refined with OH free, rigid methyls and other H atoms riding.

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *XP* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1272). Services for accessing these data are described at the back of the journal.

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A Tricyclic Trisiladiene

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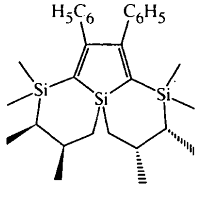
Abstract

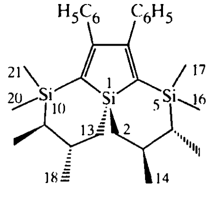
rel-(3*S*,4*R*,11*R*,12*S*)-7,8-Diphenyl-3,4,5,5,10,10,11,12-octamethyl-1,5,10-trisilatricyclo[7.4.0.0^{1,6}]trideca-6,8-diene, $C_{30}H_{42}Si_3$, crystallizes in the space group $P\bar{1}$ and has two independent molecules in the asymmetric unit. The methyl groups in both cyclohexane rings are in quasi-diequatorial positions.

Comment

As part of our interest in Si-containing polycyclic compounds we have investigated the Cp_2Zr-induced cyclizations (Cp is cyclopentadienyl) of diallyl divinyl siloles (Teng *et al.*, 1997). When silole (1), with two allyl substituents at the central Si atom and two vinyl groups at each of the peripheral Si atoms, was treated with a threefold excess of Cp_2ZrCl_2/*n*-BuLi at a temperature below 273 K, a tricyclic compound was obtained as a single stereoisomer. We had reported that the compound isolated has C_2 symmetry with the vicinal methyl groups *cis* with respect to each other. This stereochemical assignment was based on ^{1}H NMR spectra. In order to verify these conclusions, the X-ray structure analysis of this compound, (2), was carried out.



(1)



(2)

The structure analysis clearly shows that the vicinal methyl groups in both cyclohexane rings are in quasi-diequatorial, *i.e. trans* positions with respect to each other, rather than in axial-equatorial positions (Fig. 1). This is in contrast to the experience that Cp_2Zr-induced reactions of 1,7-octadienes lead preferentially, at low temperature, to *cis*-dimethylcyclohexanes (Rousset *et*

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