Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry, least-squares-planes data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71161 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR1034]

References

Barnes, C. L., Larson, G. L., Santiago, A. & Rios Steiner, J. (1988). *Acta Cryst.* C**44**, 1323–1324.

Betancourt de Perez, R. M., Fuentes, L. M., Larson, G. L., Barnes, C. L. & Heeg, M. J. (1986). *J. Org. Chem.* **51**, 2039–2043.

Daly, J. J. & Sanz, F. (1974). *Acta Cryst.* B**30**, 2766–2767.

Eaborn, C., Hitchcock, P. B., Lickiss, P. D., Pidcock, A. & Safa, K. D. (1984). *J. Chem. Soc. Dalton Trans.* pp. 2015–2017.

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis.* Enraf-Nonius, Delft, The Netherlands.

Grieco, P. A. & Miyashita, M. (1974). *J. Org. Chem.* **39**, 120–122.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Sharma, H. K., Vincenti, S. P., Vicari, R., Cervantes, F. & Pannell, K. H. (1990). *Organometallics*, **9**, 2109–2116.

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Structure of (3*R**,6*S**)-3,6-Bis(methoxymethoxy)-2,2,7,7-tetramethyl-4-octene

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Abstract

The structure of the title compound has been established by X-ray analysis. The molecule has an inversion center and the two chiral C atoms have the opposite configuration to each other.

Comment

It has been found that in the presence of trialkylchlorosilane, addition of a cuprate reagent, R_2CuLi, to α,β-unsaturated γ-alkoxy aldehydes takes place in 1,2-addition mode with good diastereoselectivity (Arai, Nemoto, Ohashi & Nakamura, 1992). In order to ascertain the chirality of the newly introduced chiral center when ^{t}Bu$_2$CuLi is used as the cuprate reagent, the crystal structure has been determined. The molecule has an inversion center and is located on a crystallographic inversion center as shown in Fig. 1. This means the absolute configuration of the introduced chiral group is opposite to that of the γ-C atom of the reactant, although the product with the same configuration as that of the γ-C was obtained when Ph$_2$CuLi was used as the cuprate reagent. The central olefinic moiety is planar. The torsion angles are 116.6 (3), −66.5 (3), −63.0 (3) and −67.4 (4)° for O1—C2—C1—C1^i, C1—C2—O1—C3, C2—O1—C3—O2 and O1—C3—O2—C4, respectively.

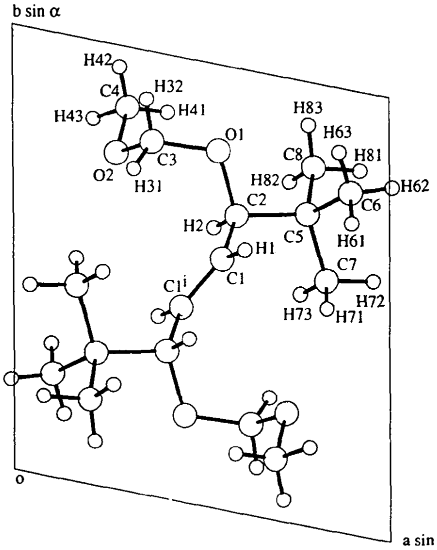

Fig. 1. Crystal structure viewed along the *c* axis with the numbering of the atoms. The C1^i atom is inverted by an inversion center at (1 − *x*, 1 − *y*, 1 − *z*).

Experimental

Crystal data

$C_{16}H_{32}O_4$
$M_r = 288.43$
Triclinic
$P\bar{1}$
$a = 8.087\ (3)$ Å
$b = 9.365\ (1)$ Å
$c = 6.200\ (1)$ Å
$\alpha = 98.45\ (1)^\circ$
$\beta = 98.19\ (3)^\circ$
$\gamma = 99.75\ (2)^\circ$
$V = 451.0\ (4)$ Å^3
$Z = 1$
$D_x = 1.06$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 20 reflections
$\theta = 20.80$–29.31°
$\mu = 0.69$ cm^{-1}
$T = 296$ K
Plate
0.4 × 0.3 × 0.25 mm
Colorless
Crystal source: AcOEt/CH$_2$Cl$_2$/hexane solution

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Data collection

Rigaku AFC-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1765 measured reflections
1604 independent reflections
1286 observed reflections [$F > 3\sigma(F)$]

$\theta_{\max} = 25.0°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 7$
3 standard reflections monitored every 50 reflections intensity variation: 5%

Refinement

Refinement on F
Final $R = 0.058$
$wR = 0.035$
$S = 3.01$
1286 reflections
155 parameters
All H-atom parameters refined

$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.0009$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Program(s) used to solve structure: *TEXSAN* (Molecular Structure Corporation, 1985). Molecular graphics: *PLUTO* (Motherwell & Clegg, 1978).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O1	0.5468 (2)	0.8058 (2)	0.4457 (3)	0.0499
O2	0.2754 (2)	0.7567 (2)	0.2255 (4)	0.0698
C1	0.5549 (3)	0.5625 (3)	0.5254 (5)	0.0431
C2	0.5940 (3)	0.6671 (3)	0.3704 (5)	0.0436
C3	0.3703 (4)	0.7974 (4)	0.4363 (7)	0.0623
C4	0.3061 (7)	0.8636 (7)	0.0881 (10)	0.0943
C5	0.7819 (3)	0.7013 (3)	0.3407 (5)	0.0465
C6	0.8973 (5)	0.7681 (5)	0.5611 (6)	0.0679
C7	0.8324 (5)	0.5595 (4)	0.2416 (8)	0.0707
C8	0.7993 (5)	0.8105 (5)	0.1797 (7)	0.0690

Table 2. *Geometric parameters* (Å, °)

O1—C2	1.445 (3)	C2—C5	1.542 (4)
O1—C3	1.409 (4)	C5—C6	1.522 (4)
O2—C3	1.382 (4)	C5—C7	1.524 (5)
O2—C4	1.420 (7)	C5—C8	1.534 (5)
C1—C2	1.497 (4)	C1—C1$^{\rm i}$	1.310 (4)
C2—O1—C3	114.1 (2)	C2—C5—C6	110.9 (3)
C3—O2—C4	113.6 (3)	C2—C5—C7	109.0 (2)
C2—C1—C1$^{\rm i}$	125.6 (3)	C2—C5—C8	108.5 (3)
O1—C2—C1	110.0 (2)	C6—C5—C7	110.1 (3)
O1—C2—C5	107.26 (19)	C6—C5—C8	109.5 (3)
C1—C2—C5	115.4 (2)	C7—C5—C8	108.9 (3)
O1—C3—O2	114.5 (3)		

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

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry including bond distances and angles involving H atoms and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71228 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1056]

References

Arai, M., Nemoto, T., Ohashi, Y. & Nakamura, E. (1992). *Synlett*, pp. 309-310

Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures*. Univ. of Cambridge, England.

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Structure of *endo*-(5*S**,6*S**,11*R**,12*R**)-5,6,11,12-Tetrahydro-5,6-dihydroxy-4,11,12-trimethoxy-9,13,13-trimethyl-6,10-methano-8(7*H*)-benzocyclodecenone

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Abstract

It has been confirmed that the title molecule has three fused rings consisting of a six-membered, an eight-membered and a benzene ring. The six-membered ring has a twisted C=C double bond; the dihedral angle between the planes of C10, C11, C15 and C13, C12, C18 is 22.1 (3)° [IUPAC numbering: C11—C10—C(methano) and C8—C9—C(methyl), respectively]. The eight-membered ring adopts a chair-boat conformation.

Comment

The title compound was obtained by Lewis acid promoted eight-membered ring cyclization between C9 and C10. The details of the synthesis will be published elsewhere (Tsuruta, Waizumi, Nakamura, Horiguchi & Kuwajima, 1993). In order to confirm the tricarbocyclic skeleton and the configuration of the substituents, the structure was determined by X-ray diffraction. The molecular structure is shown in Fig. 1.

The eight-membered ring adopts a chair-boat conformation possessing the same stereochemistry as natural taxanes. The six-membered ring has a C11=C12 double bond at a bridgehead site. This causes a substantial twist in the double bond and so the dihedral angle between planes 1 and 2, composed of C10, C11, C15 and C13, C12, C18, respectively, becomes 22.1 (3)°. The