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(2*R*,4*S*,7*R*,9*S*)-5,6-Diisopropyl-1,10-dimethoxy-3,8-dimethyldeca-3,4,6,7-tetraene-2,9-diol

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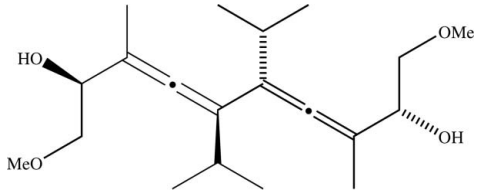
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.064; data-to-parameter ratio = 15.6.

The molecule of the title compound, $C_{20}H_{34}O_4$, is centrosymmetric. In the crystal structure, a network of O—H···O hydrogen bonds result in each molecule being linked to four neighbouring molecules and a two-dimensional net is formed.

Related literature

For details of the synthesis, see: Krause & Hoffmann-Röder (2004).



Experimental

Crystal data

$C_{20}H_{34}O_4$
M_r = 338.47
Monoclinic, $P2_1/n$
a = 7.1583 (11) Å
b = 7.1326 (8) Å
c = 19.575 (2) Å
β = 97.956 (7)°
V = 989.8 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 173 (1) K
0.15 × 0.15 × 0.13 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
12747 measured reflections
1814 independent reflections
815 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.064
S = 0.93
1814 reflections
116 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O1^{i}	0.881 (16)	1.953 (16)	2.8304 (18)	173.8 (17)

Symmetry code: (i) $-x-\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2599).

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supplementary materials

Acta Cryst. (2007). E**63**, o4402 [**doi:10.1107/S1600536807051835**]

(2*R*,4*S*,7*R*,9*S*)-5,6-Diisopropyl-1,10-dimethoxy-3,8-dimethyldeca-3,4,6,7-tetraene-2,9-diol

M. Poonoth, M. Schürmann, H. Preut and N. Krause

Comment

The title compound, (I), is one of two diastereomeric bisallenes produced from the S_N2'-substitution of a bis-propargyl oxirane with a diisopropyl magnesiumcuprate. The crystal structure determination of (I) has been carried out to establish the relative configuration of the stereogenic elements.

The complete molecule (Fig. 1) is generated by inversion symmetry. In the crystal, a network of O—H···O hydrogen bonds (Table 1) leads to a two-dimensional network.

Experimental

In a dry flask (Krause & Hoffmann-Röder, 2004) equipped with a magnetic stirring bar, a suspension of CuCN (386 mg, 4.3 mmol) in dry THF (20 ml) was cooled to 223 K under argon. At this temperature, isopropylmagnesium chloride (4.3 ml, 8.6 mmol, 2.0 *M* solution in THF) was added dropwise, and the mixture was stirred at 223 K for 30 minutes. Then a solution of 3-(methoxymethyl)-2-[4-(3-methoxymethyl-2-methyloxiran-2-yl] buta-1,3-diynyl)-2-methyloxirane in THF (4 ml) was added dropwise over 15 min at 223 K, and stirring was continued for 2 h at this temperature. The reaction mixture was then hydrolyzed with aq. satd. NH_4Cl (4 ml) and filtered through a short pad of Celite; the filtrate was dried with Na_2SO_4 and the solvent was removed *in vacuo*. The residue was purified by column chromatography on silica gel (cyclohexane/ethyl acetate, 2:1) to give the title bisallene as a colourless solid (258 mg, 42.3%) along with the second diastereomer (257 mg, 42.2%) which was a colourless oil. The title compound was taken up in ethyl acetate, and dichloromethane was added dropwise until it was completely dissolved. Colourless blocks of (I) were obtained by slow evaporation at ambient temperature; m.p. 389 K.

^{1}H NMR (400 MHz, $CDCl_3$): δ 4.26–4.2.4 (m, 2 H), 3.50 (dd, J = 3.2/9.6 Hz 2 H), 3.40 (s, 6 H), 3.40 (t, J = 8.4 Hz, 2 H), 2.34–2.27 (m, 2 H), 2.21 (d, J = 3.2 Hz, 2H), 1.78 (s, 6 H), 1.00 (t, J = 6.4 Hz, 12 H). ^{13}C NMR (100 MHz, $CDCl_3$): d 198.3, 113.2, 104.8, 75.7, 71.3, 58.9, 29.6, 22.9, 22.3, 15.3. HRMS (ESI): calcd for $C_{20}H_{35}O_4$ (*M*+H): 339.2457, found 339.2531.

Refinement

H atoms at C atoms were placed in calculated positions, with C—H = 0.98–1.00 Å and were refined as riding, with U_{iso}(H)= 1.5U_{eq}(C) for methyl and 1.2U_{eq}(C) for all other H atoms; the methyl groups were allowed to rotate but not to tip. The position of H2A was taken from a difference map and the coordinates were refined with U_{iso}(H)= 1.5U_{eq}(O).

Figures

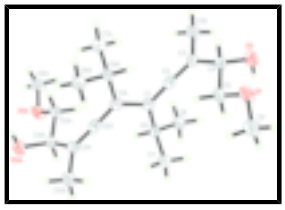


Fig. 1. : The molecular structure of (I) showing displacement ellipsoids for the non-hydrogen atoms at the 50% probability level. The atoms with suffix A are generated by the symmetry operation (−1 − *x*, 1 − *y*, −1 − *z*).

(2*R*,4*S*,7*R*,9*S*)-5,6-Diisopropyl-1,10-dimethoxy-3,8-dimethyldeca- 3,4,6,7-tetraene-2,9-diol

Crystal data

$C_{20}H_{34}O_4$	F_{000} = 372
M_r = 338.47	D_x = 1.136 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 12747 reflections
a = 7.1583 (11) Å	θ = 2.9–25.4º
b = 7.1326 (8) Å	μ = 0.08 mm^{-1}
c = 19.575 (2) Å	*T* = 173 (1) K
β = 97.956 (7)º	Block, colourless
V = 989.8 (2) Å^3	0.15 × 0.15 × 0.13 mm
Z = 2	

Data collection

Nonius KappaCCD diffractometer	1814 independent reflections
Radiation source: fine-focus sealed tube	815 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.036
Detector resolution: 19 vertical, 18 horizontal pixels mm^{-1}	θ_{max} = 25.4º
T = 173(1) K	θ_{min} = 2.9º
464 frames via ω–rotation (Δω=1°) and two times 50 s per frame (five sets at different κ–angles) scans	*h* = −8→8
Absorption correction: none	*k* = −8→8
12747 measured reflections	*l* = −23→23

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difmap and geom
$R[F^2 > 2\sigma(F^2)]$ = 0.037	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.064	$w = 1/[\sigma^2(F_o^2) + (0.0075P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$S = 0.93$	$(\Delta/\sigma)_{max} < 0.001$
1814 reflections	$\Delta\rho_{max} = 0.15$ e Å^{-3}
116 parameters	$\Delta\rho_{min} = -0.14$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.44166 (17)	0.76006 (17)	0.25645 (6)	0.0387 (4)
O2	−0.07509 (18)	0.65573 (18)	0.23350 (6)	0.0380 (4)
H2A	−0.077 (3)	0.533 (2)	0.2385 (9)	0.046*
C1	−0.4326 (3)	0.4524 (3)	0.02719 (9)	0.0273 (5)
C2	−0.3061 (3)	0.5491 (3)	0.06830 (10)	0.0281 (5)
C3	−0.1788 (3)	0.6388 (3)	0.11019 (9)	0.0283 (5)
C4	−0.2167 (3)	0.7141 (3)	0.17929 (9)	0.0303 (5)
H4A	−0.2100	0.8539	0.1768	0.036*
C5	−0.4122 (2)	0.6645 (3)	0.19503 (9)	0.0333 (5)
H5A	−0.5085	0.7032	0.1563	0.040*
H5B	−0.4226	0.5274	0.2014	0.040*
C6	−0.4500 (2)	0.2422 (2)	0.03559 (9)	0.0286 (5)
H6A	−0.4812	0.1862	−0.0114	0.034*
C7	−0.6130 (2)	0.1963 (2)	0.07667 (9)	0.0403 (6)
H7A	−0.7289	0.2568	0.0547	0.060*
H7B	−0.5821	0.2427	0.1240	0.060*
H7C	−0.6318	0.0602	0.0774	0.060*
C8	−0.2682 (3)	0.1514 (2)	0.07011 (9)	0.0385 (6)
H8A	−0.1640	0.1858	0.0450	0.058*
H8B	−0.2830	0.0148	0.0697	0.058*
H8C	−0.2407	0.1953	0.1179	0.058*
C9	−0.6108 (3)	0.7024 (3)	0.28127 (9)	0.0484 (6)
H9A	−0.6250	0.7717	0.3235	0.073*
H9B	−0.6046	0.5677	0.2913	0.073*
H9C	−0.7192	0.7280	0.2461	0.073*
C10	0.0172 (2)	0.6760 (3)	0.09233 (9)	0.0394 (6)
H10A	0.0279	0.6231	0.0468	0.059*

H10B	0.1110	0.6173	0.1270	0.059*
H10C	0.0395	0.8115	0.0916	0.059*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0282 (9)	0.0535 (10)	0.0352 (9)	−0.0076 (7)	0.0077 (7)	−0.0123 (7)
O2	0.0298 (9)	0.0478 (9)	0.0341 (8)	0.0007 (9)	−0.0040 (7)	0.0025 (9)
C1	0.0288 (14)	0.0291 (13)	0.0249 (13)	−0.0010 (11)	0.0067 (10)	−0.0027 (11)
C2	0.0313 (14)	0.0288 (12)	0.0251 (12)	0.0072 (11)	0.0069 (11)	0.0049 (11)
C3	0.0282 (13)	0.0316 (13)	0.0249 (12)	0.0012 (11)	0.0025 (11)	0.0011 (10)
C4	0.0267 (13)	0.0357 (14)	0.0268 (12)	−0.0006 (10)	−0.0024 (10)	0.0031 (10)
C5	0.0291 (13)	0.0407 (14)	0.0284 (12)	−0.0029 (11)	−0.0014 (10)	−0.0043 (11)
C6	0.0291 (13)	0.0286 (13)	0.0269 (12)	−0.0024 (11)	−0.0009 (10)	−0.0018 (10)
C7	0.0402 (15)	0.0389 (15)	0.0418 (13)	−0.0062 (11)	0.0059 (11)	0.0029 (11)
C8	0.0374 (14)	0.0329 (13)	0.0435 (13)	−0.0005 (11)	−0.0009 (11)	0.0033 (11)
C9	0.0293 (14)	0.0703 (18)	0.0467 (14)	−0.0060 (12)	0.0092 (11)	−0.0071 (12)
C10	0.0300 (15)	0.0500 (15)	0.0385 (14)	−0.0041 (11)	0.0054 (11)	−0.0048 (11)

Geometric parameters (Å, °)

O1—C5	1.4230 (17)	C6—C7	1.541 (2)
O1—C9	1.4266 (18)	C6—H6A	1.0000
O2—C4	1.424 (2)	C7—H7A	0.9800
O2—H2A	0.881 (16)	C7—H7B	0.9800
C1—C2	1.319 (2)	C7—H7C	0.9800
C1—C1^{i}	1.498 (3)	C8—H8A	0.9800
C1—C6	1.515 (2)	C8—H8B	0.9800
C2—C3	1.306 (2)	C8—H8C	0.9800
C3—C4	1.514 (2)	C9—H9A	0.9800
C3—C10	1.516 (2)	C9—H9B	0.9800
C4—C5	1.516 (2)	C9—H9C	0.9800
C4—H4A	1.0000	C10—H10A	0.9800
C5—H5A	0.9900	C10—H10B	0.9800
C5—H5B	0.9900	C10—H10C	0.9800
C6—C8	1.525 (2)		
C5—O1—C9	112.43 (14)	C7—C6—H6A	107.9
C4—O2—H2A	110.6 (12)	C6—C7—H7A	109.5
C2—C1—C1^{i}	121.1 (2)	C6—C7—H7B	109.5
C2—C1—C6	120.72 (17)	H7A—C7—H7B	109.5
C1^{i}—C1—C6	118.1 (2)	C6—C7—H7C	109.5
C3—C2—C1	177.8 (2)	H7A—C7—H7C	109.5
C2—C3—C4	122.63 (18)	H7B—C7—H7C	109.5
C2—C3—C10	121.93 (18)	C6—C8—H8A	109.5
C4—C3—C10	115.45 (16)	C6—C8—H8B	109.5
O2—C4—C3	111.40 (15)	H8A—C8—H8B	109.5
O2—C4—C5	111.23 (14)	C6—C8—H8C	109.5
C3—C4—C5	112.53 (15)	H8A—C8—H8C	109.5

O2—C4—H4A	107.1	H8B—C8—H8C	109.5
C3—C4—H4A	107.1	O1—C9—H9A	109.5
C5—C4—H4A	107.1	O1—C9—H9B	109.5
O1—C5—C4	107.99 (14)	H9A—C9—H9B	109.5
O1—C5—H5A	110.1	O1—C9—H9C	109.5
C4—C5—H5A	110.1	H9A—C9—H9C	109.5
O1—C5—H5B	110.1	H9B—C9—H9C	109.5
C4—C5—H5B	110.1	C3—C10—H10A	109.5
H5A—C5—H5B	108.4	C3—C10—H10B	109.5
C1—C6—C8	112.96 (16)	H10A—C10—H10B	109.5
C1—C6—C7	110.27 (15)	C3—C10—H10C	109.5
C8—C6—C7	109.88 (14)	H10A—C10—H10C	109.5
C1—C6—H6A	107.9	H10B—C10—H10C	109.5
C8—C6—H6A	107.9		
C2—C3—C4—O2	128.7 (2)	C3—C4—C5—O1	−173.06 (14)
C10—C3—C4—O2	−51.8 (2)	C2—C1—C6—C8	−24.8 (3)
C2—C3—C4—C5	3.0 (3)	C1^{i}—C1—C6—C8	157.52 (19)
C10—C3—C4—C5	−177.50 (17)	C2—C1—C6—C7	98.6 (2)
C9—O1—C5—C4	−171.66 (15)	C1^{i}—C1—C6—C7	−79.1 (2)
O2—C4—C5—O1	61.14 (19)		

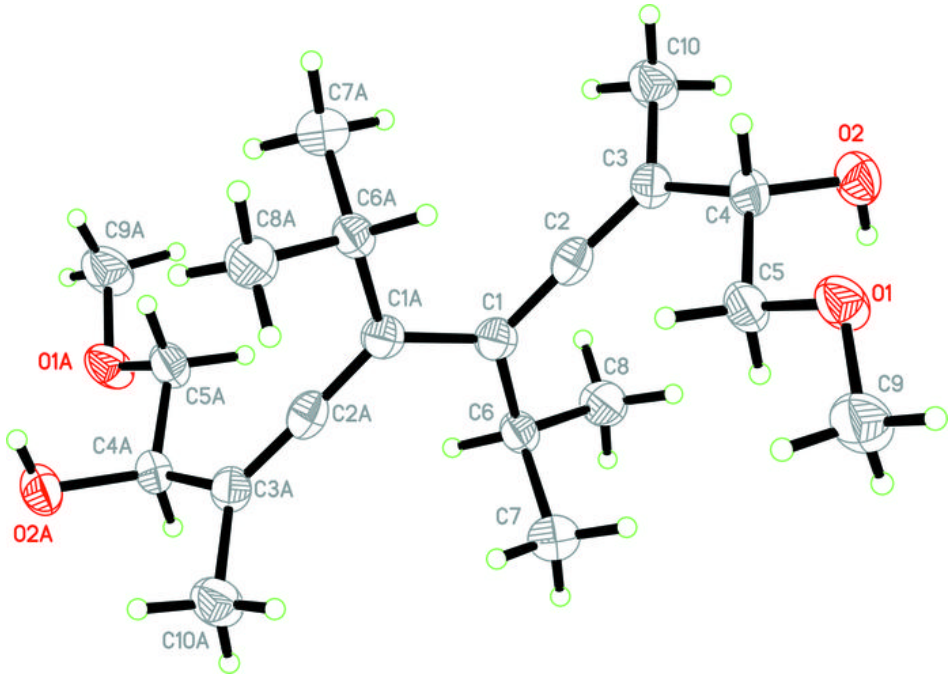
Symmetry codes: (i) −*x*−1, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2A···O1^{ii}	0.881 (16)	1.953 (16)	2.8304 (18)	173.8 (17)

Symmetry codes: (ii) −*x*−1/2, *y*−1/2, −*z*+1/2.

Fig. 1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C5A
C9
C2A
C6
C4A
C3A
C7
O2A
C10A