Crystal structure of tetramethyl(1*R*,1′*S*)-1,1′-bi-(1,4,6,7,8,9,10,11-octahydro-1,4-epoxy-benzocyclooctene-2,3-dicarboxylate), $[C_{12}H_{13}O(COOCH_3)_2]_2$

K. Peters, E.-M. Peters

Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, D-70506 Stuttgart, Germany

B. Vanselow and W. Tochtermann

Christian-Albrechts-Universität Kiel, Institut für Organische Chemie, Olshausenstraße 40, D-24098 Kiel, Germany

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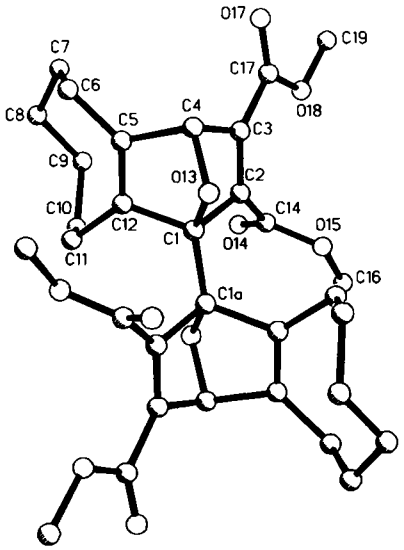

Source of material: The title compound was prepared from 2,2′-bi-(3,4-hexamethylenefuran as described in ref. 1.

$C_{32}H_{38}O_{10}$, monoclinic, $P12_1/n1$ (No. 14), a =12.006(6) Å, b =12.891(5) Å, c =9.883(5) Å, β =101.20(4)°, V =1500.5 Å^3, Z =2, $R(F)$ =0.085, $R_w(F)$ =0.082.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless prism, size 0.4 x 0.9 x 1.35 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.00 cm^{-1}
Diffractometer:	Siemens P4
Scan mode:	ω
$T_{measurement}$:	293 K
$2\theta_{max}$:	55°
$N(hkl)_{unique}$:	3454
Criterion for F_o:	$F_o > 3\,\sigma(F_o)$
$N(param)_{refined}$:	191
Program:	SHELXTL-plus

Table 2. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	x	y	z	U_{iso}
H(4A)	4*e*	0.1481(3)	0.7529(2)	0.0754(4)	0.08
H(6A)	4*e*	0.0604(4)	0.8257(3)	0.2658(5)	0.08
H(6B)	4*e*	–0.0016(4)	0.7466(3)	0.3464(5)	0.08
H(7A)	4*e*	–0.0966(5)	0.8953(4)	0.2960(7)	0.08
H(7B)	4*e*	–0.1079(5)	0.8696(4)	0.1389(7)	0.08
H(8A)	4*e*	–0.2759(4)	0.8458(4)	0.2266(6)	0.08
H(8B)	4*e*	–0.2163(4)	0.7508(4)	0.3095(6)	0.08
H(9A)	4*e*	–0.3423(4)	0.7388(3)	0.0780(6)	0.08
H(9B)	4*e*	–0.2220(4)	0.7402(3)	0.0386(6)	0.08
H(10A)	4*e*	–0.2762(3)	0.5746(3)	0.0414(5)	0.08
H(10B)	4*e*	–0.3069(3)	0.5871(3)	0.1876(5)	0.08
H(11A)	4*e*	–0.1400(3)	0.4912(3)	0.1874(4)	0.08
H(11B)	4*e*	–0.1240(3)	0.5842(3)	0.2916(4)	0.08
H(16A)	4*e*	–0.1583(5)	0.4735(4)	–0.5049(5)	0.08
H(16B)	4*e*	–0.2438(5)	0.5579(4)	–0.4737(5)	0.08
H(16C)	4*e*	–0.2294(5)	0.4539(4)	–0.3900(5)	0.08
H(19A)	4*e*	–0.1489(4)	0.8923(3)	–0.4444(5)	0.08
H(19B)	4*e*	–0.0249(4)	0.9295(3)	–0.3835(5)	0.08
H(19C)	4*e*	–0.1242(4)	0.9569(3)	–0.3074(5)	0.08

Table 3. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	0.0021(3)	0.5581(2)	0.0106(3)	0.036(2)	0.038(2)	0.060(2)	0.001(1)	0.008(1)	0.004(1)
C(2)	4*e*	–0.0426(3)	0.6275(2)	–0.1176(3)	0.043(2)	0.040(2)	0.058(2)	0.003(1)	0.009(1)	0.007(1)
C(3)	4*e*	0.0122(3)	0.7166(2)	–0.0896(4)	0.043(2)	0.044(2)	0.067(2)	0.001(1)	0.011(2)	0.007(1)
C(4)	4*e*	0.0889(3)	0.7021(2)	0.0529(4)	0.045(2)	0.039(2)	0.076(2)	–0.004(1)	0.007(2)	0.003(2)
C(5)	4*e*	0.0105(3)	0.6961(2)	0.1580(4)	0.047(2)	0.046(2)	0.067(2)	–0.001(1)	0.008(2)	–0.002(2)
C(6)	4*e*	–0.0032(4)	0.7794(3)	0.2589(5)	0.070(3)	0.057(2)	0.101(3)	–0.002(2)	0.001(2)	–0.022(2)

Table 3. (Continued)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(7)	4*e*	−0.1051(5)	0.8405(4)	0.2289(7)	0.131(5)	0.107(4)	0.187(7)	−0.024(4)	0.049(5)	−0.085(4)
C(8)	4*e*	−0.2216(4)	0.7911(4)	0.2268(6)	0.091(4)	0.073(3)	0.163(5)	0.014(3)	0.038(4)	−0.022(3)
C(9)	4*e*	−0.2634(4)	0.7229(3)	0.1094(6)	0.068(3)	0.085(3)	0.142(5)	0.021(2)	0.016(3)	−0.006(3)
C(10)	4*e*	−0.2545(3)	0.6068(3)	0.1301(5)	0.057(2)	0.072(2)	0.102(3)	−0.007(2)	0.029(2)	−0.013(2)
C(11)	4*e*	−0.1388(3)	0.5654(3)	0.1958(4)	0.069(2)	0.045(2)	0.085(3)	−0.002(2)	0.032(2)	0.002(2)
C(12)	4*e*	−0.0438(3)	0.6067(2)	0.1326(4)	0.046(2)	0.042(2)	0.061(2)	0.005(1)	0.011(2)	0.004(2)
O(13)	4*e*	0.1180(2)	0.5937(2)	0.0420(2)	0.038(1)	0.042(1)	0.073(2)	−0.0002(9)	0.005(1)	0.005(1)
C(14)	4*e*	−0.1387(3)	0.5983(3)	−0.2296(4)	0.062(2)	0.043(2)	0.064(2)	0.006(2)	0.004(2)	0.011(2)
O(14)	4*e*	−0.2361(2)	0.6115(2)	−0.2253(3)	0.049(2)	0.097(2)	0.090(2)	0.002(1)	−0.001(1)	−0.002(2)
O(15)	4*e*	−0.1014(2)	0.5523(2)	−0.3320(3)	0.083(2)	0.090(2)	0.064(2)	0.010(2)	0.005(2)	−0.004(2)
C(16)	4*e*	−0.1908(5)	0.5054(4)	−0.4338(5)	0.155(6)	0.134(5)	0.069(3)	−0.001(4)	−0.005(3)	−0.022(3)
C(17)	4*e*	−0.0002(3)	0.8169(3)	−0.1636(4)	0.053(2)	0.046(2)	0.079(3)	0.002(2)	0.017(2)	0.013(2)
O(17)	4*e*	0.0491(3)	0.8936(2)	−0.1217(3)	0.113(3)	0.049(2)	0.105(2)	−0.019(2)	−0.002(2)	0.016(2)
O(18)	4*e*	−0.0741(2)	0.8109(2)	−0.2830(3)	0.076(2)	0.059(2)	0.096(2)	−0.005(1)	−0.005(2)	0.031(2)
C(19)	4*e*	−0.0947(4)	0.9052(3)	−0.3610(5)	0.076(3)	0.077(3)	0.115(4)	0.007(2)	0.004(3)	0.046(3)

References

1. Vanselow, B.: Unpublished results.
2. Sheldrick, G. M.: Program Package SHELXTL-plus. Release 4.1. Siemens Analytical X-Ray Instruments Inc., Madison (WI 53719), USA 1990.