Crystal structure of 3*R*(*S*)-1′*S*(*R*)-2,2-dimethoxycarbonyl-3-(2′,6′,6′-trimethyl-2′-cyclohexen-1′-yl)-2,3-dihydrofuran, $C_{18}H_{26}O_5$

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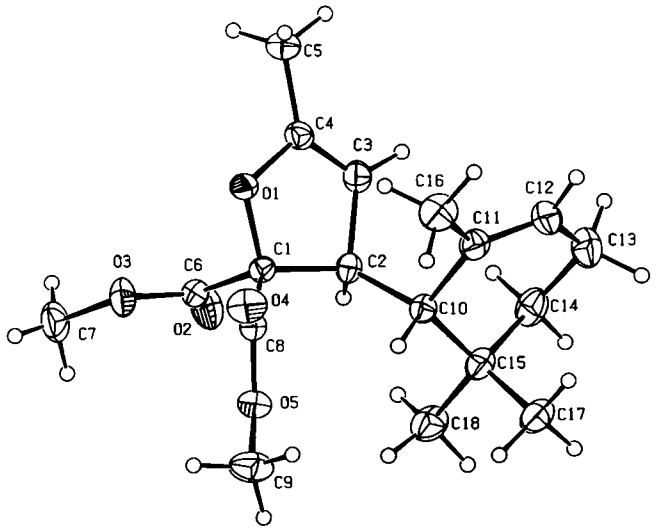

Abstract

$C_{18}H_{26}O_5$, triclinic, $P\bar{1}$ (No. 2), $a = 8.6823(7)$ Å, $b = 9.7285(7)$ Å, $c = 10.6426(8)$ Å, $\alpha = 80.779(6)°$, $\beta = 89.142(7)°$, $\gamma = 80.300(7)°$, $V = 874.6$ Å^3, $Z = 2$, $R_{gt}(F) = 0.056$, $wR_{ref}(F^2) = 0.150$, $T = 293$ K.

Source of material

The detailed synthesis of the title compound has already been published [1]. The crude solution containing both the title compound and its diastereomer was rapidly passed through a short column of neutral alumina to remove impurities and catalyst. Large colorless single crystals separated after concentrating the solution and allowing it to stay at room temperature for a week.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.25 × 0.30 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.88 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7S-CCD, 720 images, $\Delta\varphi = 0.5°$; 50° ω-scan, 100 images, $\Delta\omega = 0.5°$
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7608, 3131
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2882
$N(param)_{refined}$:	312
Programs:	SHELXS-97 [2], SHELXL-97 [3], PLATON [4]

Discussion

The stereochemistry of the title molecule has been first deduced from the ^{1}H-NMR and ^{13}C-NMR spectra and reported in [1]. The present X-ray single crystal measurements confirm the proposed topology depicted in the figure.

The molecule is built up by substituted dihydrofuran and cyclohexene rings which are linked directly. The cyclohexene ring adopts a distorted half-chair conformation. The dihydrofuran ring attached at C10 is twisted out of the plane of the six-membered ring with a torsion angle ∠C11–C10–C2–C3 = 17.9 °. The bond lengths within the puckered dihydrofuran ring are: d(C1—C2) = 1.559(2) Å, d(C2—C3) = 1.505(2) Å, d(C3—C4) = 1.309(2) Å and d(C1—O1) = 1.438(2) Å, d(C4—O1) = 1.397(2) Å, respectively, clearly indicating double bond character for C3—C4 while the remaining bonds reflect single bonding. The carbon-carbon distances within the six-membered ring (C10 to C15) vary between 1.495 Å – 1.551 Å and represent single bonds, except for the significantly shorter d(C11—C12) = 1.321(2) Å which is assigned as double bond.

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

Atom	Site	x	y	z	U_{iso}
H(21)	2*i*	–0.017(2)	0.081(2)	0.309(2)	0.041(4)
H(31)	2*i*	0.024(2)	0.178(2)	0.511(2)	0.050(5)
H(51)	2*i*	0.422(3)	0.071(3)	0.581(2)	0.071(7)
H(52)	2*i*	0.413(3)	0.232(3)	0.530(2)	0.076(7)
H(53)	2*i*	0.295(3)	0.175(2)	0.632(2)	0.069(6)
H(71)	2*i*	0.407(4)	–0.296(4)	0.270(4)	0.12(1)
H(72)	2*i*	0.318(4)	–0.278(3)	0.147(3)	0.11(1)
H(73)	2*i*	0.484(4)	–0.250(4)	0.131(3)	0.11(1)
H(91)	2*i*	0.137(4)	0.299(4)	–0.097(3)	0.096(9)
H(92)	2*i*	0.261(4)	0.161(4)	–0.126(3)	0.11(1)
H(93)	2*i*	0.069(4)	0.171(3)	–0.136(3)	0.100(9)
H(101)	2*i*	–0.016(2)	0.313(2)	0.151(2)	0.038(4)
H(121)	2*i*	–0.131(3)	0.552(3)	0.416(2)	0.068(6)
H(131)	2*i*	–0.312(3)	0.405(3)	0.500(3)	0.086(8)
H(132)	2*i*	–0.388(3)	0.496(3)	0.376(3)	0.084(8)
H(141)	2*i*	–0.254(3)	0.199(2)	0.417(2)	0.057(6)
H(142)	2*i*	–0.417(3)	0.272(3)	0.345(2)	0.077(7)
H(161)	2*i*	0.202(3)	0.418(3)	0.299(3)	0.080(8)
H(162)	2*i*	0.112(3)	0.565(3)	0.316(2)	0.076(7)
H(163)	2*i*	0.115(3)	0.507(3)	0.180(3)	0.077(7)
H(171)	2*i*	–0.296(3)	0.500(3)	0.154(2)	0.075(7)
H(172)	2*i*	–0.257(3)	0.407(2)	0.039(2)	0.065(6)
H(173)	2*i*	–0.422(3)	0.407(3)	0.121(2)	0.075(7)
H(181)	2*i*	–0.221(3)	0.064(3)	0.235(2)	0.071(7)
H(182)	2*i*	–0.363(4)	0.159(3)	0.147(3)	0.089(8)
H(183)	2*i*	–0.189(3)	0.144(3)	0.091(3)	0.081(8)

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Table 3. 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}
O(1)	2*i*	0.3230(1)	0.1267(1)	0.3382(1)	0.0316(6)	0.0484(6)	0.0361(6)	–0.0065(4)	–0.0014(4)	–0.0129(4)
O(2)	2*i*	0.1429(2)	–0.1342(1)	0.2914(1)	0.0655(9)	0.0384(6)	0.082(1)	–0.0153(6)	0.0243(7)	–0.0139(6)
O(3)	2*i*	0.3673(1)	–0.0980(1)	0.1994(1)	0.0424(7)	0.0410(6)	0.0717(8)	0.0004(5)	0.0092(6)	–0.0173(6)
O(4)	2*i*	0.3206(2)	0.2600(1)	0.0914(1)	0.0559(8)	0.0596(8)	0.0510(7)	–0.0270(6)	0.0025(6)	–0.0003(6)
O(5)	2*i*	0.1456(1)	0.1330(1)	0.0382(1)	0.0511(7)	0.0485(6)	0.0359(6)	–0.0125(5)	–0.0032(5)	–0.0088(5)
C(1)	2*i*	0.2058(2)	0.1038(1)	0.2533(1)	0.0291(7)	0.0341(7)	0.0351(7)	–0.0051(5)	–0.0013(6)	–0.0070(5)
C(2)	2*i*	0.0435(2)	0.1554(2)	0.3096(1)	0.0306(8)	0.0323(7)	0.0401(8)	–0.0049(6)	0.0028(6)	–0.0041(6)
C(3)	2*i*	0.0946(2)	0.1614(2)	0.4428(2)	0.0438(9)	0.0395(8)	0.0351(8)	0.0015(6)	0.0055(7)	–0.0011(6)
C(4)	2*i*	0.2468(2)	0.1474(2)	0.4519(1)	0.0456(9)	0.0365(7)	0.0318(7)	–0.0034(6)	0.0002(6)	–0.0049(6)
C(5)	2*i*	0.3533(3)	0.1546(2)	0.5579(2)	0.058(1)	0.060(1)	0.0384(9)	–0.0040(9)	–0.0078(8)	–0.0120(8)
C(6)	2*i*	0.2315(2)	–0.0570(2)	0.2518(1)	0.0372(8)	0.0351(7)	0.0349(7)	–0.0021(6)	–0.0021(6)	–0.0061(6)
C(7)	2*i*	0.4051(3)	–0.2460(2)	0.1879(3)	0.063(1)	0.042(1)	0.077(2)	0.0065(9)	0.005(1)	–0.022(1)
C(8)	2*i*	0.2352(2)	0.1765(2)	0.1199(1)	0.0340(8)	0.0378(7)	0.0367(8)	–0.0040(6)	0.0010(6)	–0.0076(6)
C(9)	2*i*	0.1524(4)	0.1979(3)	–0.0932(2)	0.091(2)	0.069(1)	0.038(1)	–0.020(1)	–0.012(1)	–0.0006(9)
C(10)	2*i*	–0.0570(2)	0.2958(1)	0.2387(1)	0.0326(8)	0.0322(7)	0.0388(8)	–0.0041(6)	–0.0002(6)	–0.0049(6)
C(11)	2*i*	–0.0394(2)	0.4215(2)	0.3023(2)	0.0462(9)	0.0313(7)	0.0492(9)	–0.0041(6)	–0.0070(7)	–0.0053(6)
C(12)	2*i*	–0.1479(2)	0.4725(2)	0.3788(2)	0.062(1)	0.0426(9)	0.058(1)	0.0038(8)	–0.0047(9)	–0.0187(8)
C(13)	2*i*	–0.2972(3)	0.4176(3)	0.4092(2)	0.055(1)	0.077(1)	0.066(1)	0.008(1)	0.012(1)	–0.020(1)
C(14)	2*i*	–0.3076(2)	0.2848(2)	0.3538(2)	0.0338(9)	0.065(1)	0.063(1)	–0.0057(8)	0.0070(8)	–0.0017(9)
C(15)	2*i*	–0.2318(2)	0.2849(2)	0.2237(2)	0.0331(8)	0.0407(8)	0.0524(9)	–0.0045(6)	–0.0046(7)	–0.0052(7)
C(16)	2*i*	0.1071(3)	0.4834(2)	0.2741(2)	0.060(1)	0.0424(9)	0.077(1)	–0.0200(9)	–0.006(1)	–0.0077(9)
C(17)	2*i*	–0.3111(2)	0.4117(2)	0.1278(2)	0.046(1)	0.053(1)	0.064(1)	–0.0007(8)	–0.0145(9)	–0.0034(9)
C(18)	2*i*	–0.2531(3)	0.1515(2)	0.1718(3)	0.050(1)	0.052(1)	0.085(2)	–0.0142(9)	–0.017(1)	–0.015(1)

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