Crystal structure of (±)-(2*R*)-2-[(3*S*,4*S*,5*S*)-4-benzoyl-5-hydroxymethyl-4-methyltetrahydro-3-furanyl]-1-phenylpropan-1-one, $C_{22}H_{24}O_4$

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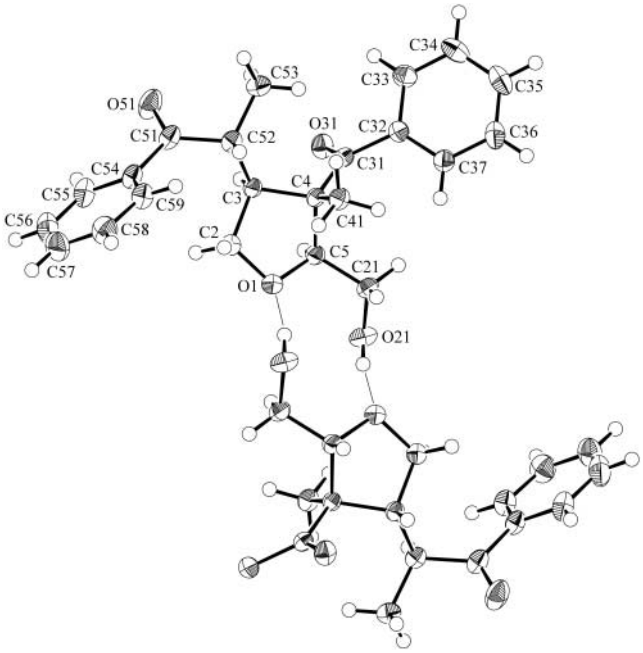

Abstract

$C_{22}H_{24}O_4$, triclinic, $P\bar{1}$ (No. 2), a = 6.1275(6) Å, b = 11.248(1) Å, c = 13.454(1) Å, α = 88.393(2)°, β = 81.420(2)°, γ = 79.313(2)°, V = 900.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.056, $wR_{ref}(F^2)$ = 0.153, T = 183 K.

Source of material

The title compound was prepared according to [1] and recrystallised from a dichloromethane/hexane solution of the compound; mp 373 K – 375 K.

Experimental details

The H atoms were placed in their geometrically calculated positions and included in the final refinement in the riding model approximation.

Discussion

The structure determination reveals the relative stereochemistry of the four chiral C3, C4, C5, and C52 atoms, as shown in the figure; the structure is an enantiomeric mixture. The THF-five-membered ring is slightly puckered as seen in the deviations of the O1, C2, C3, C4 and C5 atoms from their least-squares plane of –0.107(1) Å, –0.074(1) Å, 0.215(1) Å, –0.281(1) Å & 0.248(1) Å, respectively. Centrosymmetrically related molecules associate via H-bonding contacts involving the hydroxyl-H and the THF-ether O atoms. The geometric parameters defining these interactions are d(O21–H21o···O1^i) = 1.91 Å, d(O21···O1^i) = 2.7346(16) Å and the angle subtended at H21o is 166° for symmetry operation i: –1–x, –y, 1–z. This results in the formation of a 10-membered ring as highlighted in the figure.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.18 × 0.29 × 0.47 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	0.88 cm^{-1}
Diffractometer, scan mode:	Bruker AXS SMART CCD, ω
$2\theta_{max}$:	60°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7613, 5155
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3998
$N(param)_{refined}$:	235
Programs:	teXsan [2], SIR92 [3], SHELXL-97 [4], ORTEPII [5]

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

Atom	Site	x	y	z	U_{iso}
H(21o)	2*i*	–0.5292	0.1280	0.5436	0.035
H(2a)	2*i*	–0.1025	–0.2286	0.3482	0.034
H(2b)	2*i*	0.0811	–0.2106	0.4176	0.034
H(3)	2*i*	0.2314	–0.0813	0.3101	0.027
H(5)	2*i*	–0.0350	0.0670	0.4173	0.030
H(21a)	2*i*	–0.3563	0.2075	0.3762	0.036
H(21b)	2*i*	–0.4981	0.1006	0.3927	0.036
H(33)	2*i*	0.2540	0.1978	0.0696	0.039
H(34)	2*i*	0.1367	0.3461	–0.0454	0.048
H(35)	2*i*	–0.2350	0.4482	–0.0242	0.050
H(36)	2*i*	–0.4931	0.3975	0.1077	0.045
H(37)	2*i*	–0.3828	0.2393	0.2169	0.036
H(41a)	2*i*	–0.3257	–0.0807	0.2514	0.040
H(41b)	2*i*	–0.1976	–0.0362	0.1487	0.040
H(41c)	2*i*	–0.3784	0.0585	0.2190	0.040
H(52)	2*i*	0.0533	–0.2182	0.1735	0.032
H(53a)	2*i*	0.1664	–0.0490	0.0852	0.048
H(53b)	2*i*	0.3324	–0.1708	0.0452	0.048
H(53c)	2*i*	0.4086	–0.0838	0.1202	0.048
H(55)	2*i*	0.5487	–0.4242	0.3563	0.047
H(56)	2*i*	0.4554	–0.5932	0.4445	0.057
H(57)	2*i*	0.1483	–0.6722	0.4123	0.059
H(58)	2*i*	–0.0739	–0.5793	0.2954	0.052
H(59)	2*i*	0.0063	–0.4032	0.2131	0.041

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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*	–0.1881(2)	–0.07548(9)	0.43028(7)	0.0281(5)	0.0300(5)	0.0284(5)	–0.0040(4)	0.0012(4)	0.0034(4)
O(21)	2*i*	–0.4158(2)	0.1562(1)	0.51813(8)	0.0300(6)	0.0473(6)	0.0298(5)	–0.0097(5)	0.0030(4)	–0.0079(4)
O(31)	2*i*	0.2153(2)	0.12843(9)	0.25837(8)	0.0226(5)	0.0327(5)	0.0420(6)	–0.0083(4)	–0.0095(4)	0.0033(4)
O(51)	2*i*	0.5568(2)	–0.2845(1)	0.2032(1)	0.0240(6)	0.0388(6)	0.0768(9)	–0.0031(5)	–0.0042(6)	0.0025(6)
C(2)	2*i*	–0.0260(2)	–0.1684(1)	0.3742(1)	0.0275(7)	0.0272(6)	0.0296(6)	–0.0026(5)	–0.0019(5)	0.0024(5)
C(3)	2*i*	0.0980(2)	–0.1049(1)	0.28671(9)	0.0190(6)	0.0254(6)	0.0241(6)	–0.0032(5)	–0.0045(5)	0.0007(5)
C(4)	2*i*	–0.0755(2)	0.0126(1)	0.27443(9)	0.0167(5)	0.0245(6)	0.0239(6)	–0.0035(4)	–0.0037(4)	0.0008(5)
C(5)	2*i*	–0.1577(2)	0.0397(1)	0.3877(1)	0.0217(6)	0.0272(6)	0.0248(6)	–0.0041(5)	–0.0035(5)	0.0008(5)
C(21)	2*i*	–0.3709(2)	0.1316(1)	0.4136(1)	0.0256(7)	0.0338(7)	0.0272(6)	0.0004(5)	0.0000(5)	–0.0022(5)
C(31)	2*i*	0.0412(2)	0.1158(1)	0.2306(1)	0.0197(6)	0.0254(6)	0.0256(6)	–0.0035(5)	–0.0021(5)	–0.0012(5)
C(32)	2*i*	–0.0522(2)	0.2042(1)	0.1553(1)	0.0269(7)	0.0243(6)	0.0264(6)	–0.0059(5)	–0.0050(5)	0.0001(5)
C(33)	2*i*	0.1012(3)	0.2369(1)	0.0768(1)	0.0316(7)	0.0329(7)	0.0337(7)	–0.0117(6)	–0.0033(6)	0.0016(6)
C(34)	2*i*	0.0325(3)	0.3260(2)	0.0092(1)	0.051(1)	0.0421(9)	0.0331(7)	–0.0219(8)	–0.0072(7)	0.0100(6)
C(35)	2*i*	–0.1887(3)	0.3858(2)	0.0213(1)	0.062(1)	0.0297(7)	0.0382(8)	–0.0120(7)	–0.0186(8)	0.0094(6)
C(36)	2*i*	–0.3421(3)	0.3553(1)	0.0992(1)	0.0421(9)	0.0300(7)	0.0399(8)	0.0024(6)	–0.0153(7)	0.0009(6)
C(37)	2*i*	–0.2757(2)	0.2627(1)	0.1651(1)	0.0306(7)	0.0302(7)	0.0291(6)	–0.0020(5)	–0.0049(5)	0.0001(5)
C(41)	2*i*	–0.2610(2)	–0.0138(1)	0.2183(1)	0.0206(6)	0.0310(7)	0.0310(6)	–0.0059(5)	–0.0077(5)	–0.0006(5)
C(51)	2*i*	0.3583(2)	–0.2896(1)	0.2208(1)	0.0253(7)	0.0263(7)	0.0401(7)	–0.0027(5)	–0.0040(6)	–0.0059(6)
C(52)	2*i*	0.1819(2)	–0.1850(1)	0.1923(1)	0.0236(6)	0.0256(6)	0.0299(6)	–0.0047(5)	–0.0038(5)	–0.0030(5)
C(53)	2*i*	0.2812(3)	–0.1160(1)	0.1027(1)	0.0317(7)	0.0340(7)	0.0286(6)	–0.0073(6)	0.0006(5)	–0.0043(5)
C(54)	2*i*	0.2881(2)	–0.3973(1)	0.2753(1)	0.0280(7)	0.0237(6)	0.0373(7)	–0.0002(5)	–0.0045(6)	–0.0050(5)
C(55)	2*i*	0.4203(3)	–0.4548(1)	0.3445(1)	0.0388(9)	0.0317(8)	0.0471(9)	0.0025(6)	–0.0142(7)	–0.0047(7)
C(56)	2*i*	0.3664(3)	–0.5558(2)	0.3962(1)	0.060(1)	0.0339(8)	0.047(1)	0.0050(8)	–0.0182(8)	0.0004(7)
C(57)	2*i*	0.1837(4)	–0.6020(2)	0.3776(2)	0.063(1)	0.0280(8)	0.053(1)	–0.0042(7)	–0.0053(9)	0.0033(7)
C(58)	2*i*	0.0510(3)	–0.5465(2)	0.3086(1)	0.045(1)	0.0313(8)	0.055(1)	–0.0105(7)	–0.0062(8)	–0.0018(7)
C(59)	2*i*	0.1005(3)	–0.4429(1)	0.2587(1)	0.0330(8)	0.0301(7)	0.0402(8)	–0.0033(6)	–0.0080(6)	–0.0016(6)

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