Crystal structure of (*P*,1″*S*,2″*R*,5″*S*)-menthyl 4,4′-di-*tert*-butyl-2′-hydroxy-2,3,3′-trimethoxy-6′-methyl-1,1′-biphenyl-6-carboxylate, $C_6H(OCH_3)_2[C(CH_3)_3]CO_2(C_{10}H_{19})C_6H(CH_3)(OH)(OCH_3)[C(CH_3)_3]$

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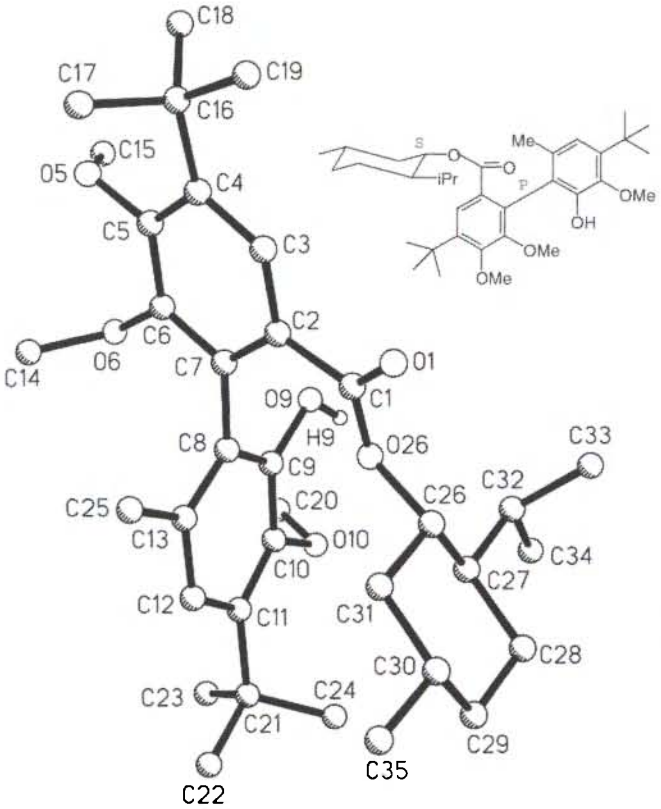

Abstract

$C_{35}H_{52}O_6$, orthorhombic, $P2_12_12_1$ (No. 19), a = 10.405(1) Å, b = 12.701(1) Å, c = 26.133(2) Å, V = 3453.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.059, $wR(F)$ = 0.060, T = 293 K.

Source of material

The title compound was prepared by atropodiasteroselective opening of the corresponding biaryl lactone with lithium (1*S*)-mentholate [1].

Discussion

Given the known configuration of the menthol part, the absolute configuration at the biaryl axis was elucidared by this X-ray analysis.

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Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.65 × 0.75 × 0.85 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.70 cm^{-1}
Diffractometer, scan mode:	Bruker AXS P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10446, 7921
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\,\sigma(F_{obs})$, 6834
$N(param)_{refined}$:	374
Program:	SHELXTL-plus [2]

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

Atom	Site	x	y	z	U_{iso}
H(3)	4*a*	0.9086(2)	0.1450(2)	0.37805(8)	0.08
H(9)	4*a*	0.855(2)	0.482(2)	0.218(1)	0.048(7)
H(12)	4*a*	0.5684(2)	0.1677(2)	0.14172(9)	0.08
H(14A)	4*a*	0.3663(3)	0.3866(3)	0.3027(2)	0.08
H(14B)	4*a*	0.4088(3)	0.3196(3)	0.3501(2)	0.08
H(14C)	4*a*	0.4260(3)	0.2745(3)	0.2947(2)	0.08
H(15A)	4*a*	0.5192(4)	0.4880(2)	0.4404(1)	0.08
H(15B)	4*a*	0.5639(4)	0.4899(2)	0.3831(1)	0.08
H(15C)	4*a*	0.6661(4)	0.4855(2)	0.4271(1)	0.08
H(17A)	4*a*	0.6436(3)	0.1216(3)	0.5063(1)	0.08
H(17B)	4*a*	0.6368(3)	0.0810(3)	0.4498(1)	0.08
H(17C)	4*a*	0.5692(3)	0.1868(3)	0.4649(1)	0.08
H(18A)	4*a*	0.7050(3)	0.3459(3)	0.48849(9)	0.08
H(18B)	4*a*	0.8552(3)	0.3361(3)	0.48752(9)	0.08
H(18C)	4*a*	0.7755(3)	0.2764(3)	0.52934(9)	0.08
H(19A)	4*a*	0.8775(3)	0.0709(3)	0.4478(1)	0.08
H(19B)	4*a*	0.8792(3)	0.1103(3)	0.5047(1)	0.08
H(19C)	4*a*	0.9589(3)	0.1701(3)	0.4628(1)	0.08
H(20A)	4*a*	0.7462(4)	0.6280(2)	0.1314(1)	0.08
H(20B)	4*a*	0.7117(4)	0.5817(2)	0.1853(1)	0.08
H(20C)	4*a*	0.6184(4)	0.5646(2)	0.1390(1)	0.08
H(22A)	4*a*	0.4974(5)	0.2038(3)	0.0677(1)	0.08
H(22B)	4*a*	0.6342(5)	0.1561(3)	0.0581(1)	0.08
H(22C)	4*a*	0.5670(5)	0.2230(3)	0.0154(1)	0.08
H(23A)	4*a*	0.5905(4)	0.4696(3)	0.0736(2)	0.08
H(23B)	4*a*	0.4708(4)	0.3947(3)	0.0756(2)	0.08
H(23C)	4*a*	0.5447(4)	0.4115(3)	0.0240(2)	0.08
H(24A)	4*a*	0.8096(4)	0.3880(4)	0.0586(1)	0.08
H(24B)	4*a*	0.7526(4)	0.3324(4)	0.0101(1)	0.08
H(24C)	4*a*	0.8198(4)	0.2655(4)	0.0528(1)	0.08
H(25A)	4*a*	0.6025(3)	0.1156(2)	0.2743(1)	0.08
H(25B)	4*a*	0.6212(3)	0.0510(2)	0.2237(1)	0.08
H(25C)	4*a*	0.4909(3)	0.1091(2)	0.2341(1)	0.08
H(26)	4*a*	1.0854(2)	0.0954(2)	0.20940(9)	0.08
H(27)	4*a*	0.9558(2)	0.2259(2)	0.13954(9)	0.08
H(28A)	4*a*	1.1788(3)	0.1047(3)	0.1222(1)	0.08

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(28B)	4a	1.1105(3)	0.1736(3)	0.0805(1)	0.08
H(29A)	4a	0.9377(3)	0.0600(3)	0.0796(1)	0.08
H(29B)	4a	1.0638(3)	-0.0036(3)	0.0684(1)	0.08
H(30)	4a	1.0666(3)	-0.0676(2)	0.1507(1)	0.08
H(31A)	4a	0.9150(3)	-0.0165(2)	0.2108(1)	0.08
H(31B)	4a	0.8437(3)	0.0517(2)	0.1697(1)	0.08
H(32)	4a	1.0611(3)	0.3226(3)	0.1950(1)	0.08
H(33A)	4a	1.2324(4)	0.2198(4)	0.2166(2)	0.08

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(33B)	4a	1.2759(4)	0.3348(4)	0.2036(2)	0.08
H(33C)	4a	1.2956(4)	0.2443(4)	0.1635(2)	0.08
H(34A)	4a	1.0371(4)	0.3863(3)	0.1113(2)	0.08
H(34B)	4a	1.1759(4)	0.3463(3)	0.0985(2)	0.08
H(34C)	4a	1.1563(4)	0.4368(3)	0.1385(2)	0.08
H(35A)	4a	0.9449(3)	-0.1684(3)	0.0969(1)	0.08
H(35B)	4a	0.8222(3)	-0.1033(3)	0.1116(1)	0.08
H(35C)	4a	0.8900(3)	-0.1739(3)	0.1527(1)	0.08

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)	4a	1.0034(2)	0.0893(2)	0.29915(7)	0.096(1)	0.104(2)	0.041(1)	0.059(1)	0.008(1)	0.006(1)
C(1)	4a	0.9307(2)	0.1539(2)	0.28151(9)	0.051(1)	0.048(1)	0.039(1)	0.012(1)	0.001(1)	-0.002(1)
C(2)	4a	0.8320(2)	0.2090(2)	0.31358(8)	0.045(1)	0.043(1)	0.0292(9)	0.004(1)	0.0022(9)	-0.0012(9)
C(3)	4a	0.8414(2)	0.1905(2)	0.36605(8)	0.045(1)	0.049(1)	0.033(1)	0.006(1)	0.0005(9)	0.0008(9)
C(4)	4a	0.7588(2)	0.2345(2)	0.40190(8)	0.049(1)	0.047(1)	0.0289(9)	-0.004(1)	0.0012(9)	-0.0030(9)
C(5)	4a	0.6657(2)	0.3026(2)	0.38267(9)	0.044(1)	0.042(1)	0.043(1)	-0.002(1)	0.0094(9)	-0.002(1)
O(5)	4a	0.5797(2)	0.3501(1)	0.41522(6)	0.0607(9)	0.0545(9)	0.0475(9)	0.0084(8)	0.0230(8)	-0.0015(8)
C(6)	4a	0.6506(2)	0.3185(2)	0.32951(9)	0.039(1)	0.041(1)	0.045(1)	0.0033(9)	0.0028(9)	0.0048(9)
O(6)	4a	0.5539(2)	0.3829(1)	0.31246(7)	0.0498(9)	0.0559(9)	0.056(1)	0.0133(8)	0.0049(8)	0.0124(8)
C(7)	4a	0.7326(2)	0.2720(2)	0.29421(7)	0.042(1)	0.037(1)	0.034(1)	-0.0031(9)	0.0011(9)	0.0007(8)
C(8)	4a	0.7037(2)	0.2850(2)	0.23835(8)	0.0377(9)	0.039(1)	0.038(1)	0.0027(9)	-0.0015(8)	0.0013(9)
C(9)	4a	0.7490(2)	0.3722(2)	0.21188(8)	0.042(1)	0.039(1)	0.035(1)	-0.001(1)	-0.0014(9)	-0.0016(8)
O(9)	4a	0.8209(2)	0.4430(1)	0.23844(7)	0.070(1)	0.054(1)	0.0386(8)	-0.0247(9)	-0.0012(8)	0.0031(8)
C(10)	4a	0.7238(2)	0.3855(2)	0.15973(8)	0.047(1)	0.042(1)	0.037(1)	0.002(1)	0.0012(9)	0.0044(9)
O(10)	4a	0.7725(2)	0.4745(1)	0.13641(6)	0.076(1)	0.0523(9)	0.0423(8)	-0.0049(9)	0.0034(8)	0.0122(8)
C(11)	4a	0.6605(2)	0.3073(2)	0.13162(9)	0.051(1)	0.053(1)	0.037(1)	0.003(1)	-0.007(1)	0.001(1)
C(12)	4a	0.6149(2)	0.2215(2)	0.15964(9)	0.058(1)	0.054(1)	0.042(1)	-0.007(1)	-0.015(1)	-0.001(1)
C(13)	4a	0.6336(2)	0.2093(2)	0.21224(9)	0.057(1)	0.043(1)	0.040(1)	-0.004(1)	-0.007(1)	0.002(1)
C(14)	4a	0.4284(3)	0.3371(3)	0.3152(2)	0.051(1)	0.086(2)	0.117(3)	0.010(2)	-0.009(2)	0.020(2)
C(15)	4a	0.5822(4)	0.4626(2)	0.4166(1)	0.114(2)	0.058(2)	0.076(2)	0.010(2)	0.040(2)	-0.004(2)
C(16)	4a	0.7653(2)	0.2032(2)	0.45855(8)	0.065(1)	0.062(1)	0.030(1)	0.001(1)	0.002(1)	0.004(1)
C(17)	4a	0.6423(3)	0.1425(3)	0.4710(1)	0.094(2)	0.088(2)	0.050(2)	-0.015(2)	0.010(1)	0.019(2)
C(18)	4a	0.7763(3)	0.2995(3)	0.49437(9)	0.083(2)	0.091(2)	0.037(1)	-0.003(2)	0.000(1)	-0.014(1)
C(19)	4a	0.8810(3)	0.1319(3)	0.4695(1)	0.102(2)	0.102(2)	0.036(1)	0.022(2)	-0.010(1)	0.013(2)
C(20)	4a	0.7068(4)	0.5701(2)	0.1491(1)	0.118(3)	0.053(2)	0.069(2)	0.008(2)	-0.006(2)	0.013(1)
C(21)	4a	0.6406(3)	0.3141(2)	0.0730(1)	0.078(2)	0.074(2)	0.036(1)	-0.004(2)	-0.014(1)	0.005(1)
C(22)	4a	0.5791(5)	0.2152(3)	0.0516(1)	0.180(4)	0.127(3)	0.052(2)	-0.039(3)	-0.032(2)	-0.001(2)
C(23)	4a	0.5537(4)	0.4058(3)	0.0604(2)	0.132(3)	0.136(3)	0.067(2)	0.029(3)	-0.040(2)	0.018(2)
C(24)	4a	0.7670(4)	0.3260(4)	0.0462(1)	0.111(3)	0.158(4)	0.047(2)	-0.002(3)	-0.002(2)	-0.002(2)
C(25)	4a	0.5824(3)	0.1125(2)	0.2384(1)	0.093(2)	0.058(2)	0.058(2)	-0.027(1)	-0.013(2)	0.009(1)
O(26)	4a	0.9307(2)	0.1833(1)	0.23362(6)	0.0661(9)	0.066(1)	0.0343(8)	0.0267(8)	0.0121(8)	0.0030(8)
C(26)	4a	1.0054(2)	0.1218(2)	0.19640(9)	0.060(1)	0.066(2)	0.034(1)	0.028(1)	0.011(1)	0.002(1)
C(27)	4a	1.0341(2)	0.1956(2)	0.15222(9)	0.060(1)	0.068(2)	0.042(1)	0.018(1)	0.007(1)	0.005(1)
C(28)	4a	1.0973(3)	0.1296(3)	0.1099(1)	0.089(2)	0.098(2)	0.047(2)	0.025(2)	0.026(1)	0.005(2)
C(29)	4a	1.0173(3)	0.0353(3)	0.0939(1)	0.094(2)	0.087(2)	0.038(1)	0.021(2)	0.012(1)	-0.007(1)
C(30)	4a	0.9875(3)	-0.0375(2)	0.1387(1)	0.071(2)	0.073(2)	0.047(1)	0.026(1)	0.003(1)	-0.009(1)
C(31)	4a	0.9261(3)	0.0276(2)	0.1813(1)	0.069(2)	0.074(2)	0.040(1)	0.019(1)	0.008(1)	0.000(1)
C(32)	4a	1.1113(3)	0.2931(3)	0.1677(1)	0.086(2)	0.083(2)	0.058(2)	0.009(2)	0.015(1)	0.003(2)
C(33)	4a	1.2402(4)	0.2711(4)	0.1899(2)	0.106(3)	0.146(4)	0.147(4)	-0.027(3)	-0.028(3)	0.034(4)
C(34)	4a	1.1210(4)	0.3727(3)	0.1250(2)	0.108(3)	0.103(3)	0.093(3)	-0.005(2)	0.018(2)	0.020(2)
C(35)	4a	0.9035(3)	-0.1291(3)	0.1236(1)	0.090(2)	0.088(2)	0.073(2)	0.009(2)	-0.006(2)	-0.019(2)

References

1. Bringmann, G.; Pabst, T.; Busemann, S.; Peters, K.; Peters, E.-M.: Atropo-Enantioselective Synthesis of a Simplified Analog of Mastigophorenes A und B. Tetrahedron **54** (1998) 1425-1438.
2. Sheldrick, G. M.: Program Package SHELXTL-Plus. Release 4.1, Siemens Analytical X-Ray Instruments Inc., Madison (WI 53719), USA 1990.