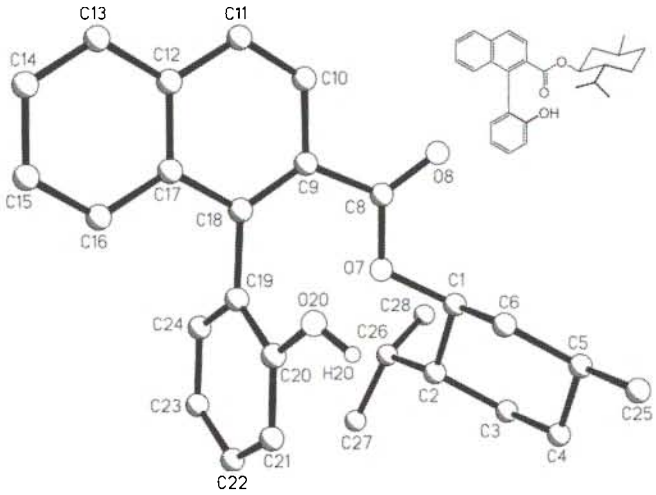
Crystal structure of (*P*)-1-(2-hydroxyphenyl)-2-naphthoic acid (1*S*,2*R*,5*S*)-(+)-menthyl ester, $C_{10}H_6[C_6H_4(OH)][COOC_6H_9(CH_3)(C_3H_7)]$

K. Peters*[I], E.-M. Peters[I], T. Hartung[II] and G. Bringmann[II]

[I] Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, D-70506 Stuttgart, Germany
[II] Universität Würzburg, Institut für Organische Chemie, Am Hubland, D-97074 Würzburg, Germany

Received April 22, 1999, CCDC-No. 1267/175



Abstract

$C_{27}H_{30}O_3$, orthorhombic, $P2_12_12_1$ (No. 19), a = 10.408(4) Å, b = 21.908(6) Å, c = 10.095(2) Å, V = 2301.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.067, $wR(F)$ = 0.049, T = 293 K.

Source of material

The title compound was synthesized by ring cleavage of the corresponding lactone with sodium-(*S*)-(+)-mentholate [1] and subsequent separation of the resulting atrop-diastereomers. Due to the stereochemically known menthol part, the absolute configuration at the biaryl axis was etablished to be *P* [2].

Discussion

An intermolecular hydrogen bridge was found with d[H(20)···O(8)] = 189 pm to form a zigzag chain parallel [001].

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.35 × 0.6 × 0.15 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.70 cm^{-1}
Diffractometer, scan mode:	Siemens R3m/V, Wyckoff
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3801, 3773
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 2\,\sigma(F_{obs})$, 2541
$N(param)_{refined}$:	276
Program:	SHELXTL-plus [3]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	0.3621(3)	−0.0114(1)	0.6024(3)	0.08
H(2)	4*a*	0.5766(3)	0.0321(2)	0.4626(4)	0.08
H(3A)	4*a*	0.5709(3)	−0.0601(2)	0.6574(5)	0.08
H(3B)	4*a*	0.6946(3)	−0.0399(2)	0.5804(5)	0.08
H(4A)	4*a*	0.6152(4)	−0.1319(2)	0.4986(5)	0.08
H(4B)	4*a*	0.6160(4)	−0.0816(2)	0.3882(5)	0.08
H(5)	4*a*	0.3998(4)	−0.1197(2)	0.5285(4)	0.08
H(6A)	4*a*	0.2838(3)	−0.0491(1)	0.4070(3)	0.08
H(6B)	4*a*	0.4093(3)	−0.0285(1)	0.3336(3)	0.08
H(10)	4*a*	−0.0149(3)	0.1070(1)	0.5119(3)	0.08
H(11)	4*a*	−0.1290(3)	0.1820(1)	0.4052(3)	0.08
H(13)	4*a*	−0.1422(4)	0.2606(2)	0.2282(4)	0.08
H(14)	4*a*	−0.0451(4)	0.3160(2)	0.0623(5)	0.08
H(15)	4*a*	0.1765(4)	0.3054(2)	0.0209(4)	0.08
H(16)	4*a*	0.2990(3)	0.2386(1)	0.1460(4)	0.08
H(20)	4*a*	0.342(4)	0.044(2)	0.131(4)	0.10(2)
H(21)	4*a*	0.5671(3)	0.0755(2)	0.1296(3)	0.08
H(22)	4*a*	0.7149(3)	0.1394(2)	0.2346(4)	0.08
H(23)	4*a*	0.6440(3)	0.2152(2)	0.3790(4)	0.08
H(24)	4*a*	0.4255(3)	0.2243(1)	0.4254(4)	0.08
H(25A)	4*a*	0.3209(5)	−0.1554(2)	0.3283(4)	0.08
H(25B)	4*a*	0.4497(5)	−0.1887(2)	0.3638(4)	0.08
H(25C)	4*a*	0.4491(5)	−0.1362(2)	0.2583(4)	0.08
H(26)	4*a*	0.5140(4)	0.0989(2)	0.6220(4)	0.08
H(27A)	4*a*	0.7269(4)	0.0982(2)	0.5420(5)	0.08
H(27B)	4*a*	0.7658(4)	0.0550(2)	0.6593(5)	0.08
H(27C)	4*a*	0.7219(4)	0.1222(2)	0.6882(5)	0.08
H(28A)	4*a*	0.4528(5)	0.0348(2)	0.7916(4)	0.08
H(28B)	4*a*	0.5527(5)	0.0831(2)	0.8421(4)	0.08
H(28C)	4*a*	0.5966(5)	0.0159(2)	0.8132(4)	0.08

* Correspondence author
(e-mail: karpet@vsibm1.mpi-stuttgart.mpg.de)

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}
C(1)	4*a*	0.3989(3)	0.0039(1)	0.5217(3)	0.044(2)	0.049(2)	0.060(2)	0.007(1)	0.002(2)	0.018(2)
C(2)	4*a*	0.5405(3)	0.0155(2)	0.5424(4)	0.040(2)	0.074(2)	0.070(2)	0.007(2)	0.002(2)	0.021(2)
C(3)	4*a*	0.6034(3)	–0.0459(2)	0.5740(5)	0.050(2)	0.107(3)	0.105(3)	0.026(2)	0.002(2)	0.031(3)
C(4)	4*a*	0.5775(4)	–0.0941(2)	0.4702(5)	0.098(3)	0.095(3)	0.100(4)	0.051(3)	0.028(3)	0.022(3)
C(5)	4*a*	0.4369(4)	–0.1037(2)	0.4487(4)	0.089(3)	0.064(2)	0.075(3)	0.017(2)	0.024(2)	0.009(2)
C(6)	4*a*	0.3748(3)	–0.0431(1)	0.4159(3)	0.067(2)	0.065(2)	0.058(2)	0.005(2)	0.003(2)	0.007(2)
O(7)	4*a*	0.3413(2)	0.06238(9)	0.4795(2)	0.040(1)	0.054(1)	0.067(1)	0.004(1)	–0.002(1)	0.017(1)
C(8)	4*a*	0.2152(3)	0.0687(1)	0.4908(3)	0.040(2)	0.056(2)	0.048(2)	0.005(2)	0.000(2)	0.003(2)
O(8)	4*a*	0.1468(2)	0.0327(1)	0.5480(3)	0.050(1)	0.075(2)	0.088(2)	0.006(1)	0.014(1)	0.035(1)
C(9)	4*a*	0.1597(3)	0.1235(1)	0.4234(3)	0.038(2)	0.046(2)	0.049(2)	0.002(1)	–0.001(2)	–0.002(2)
C(10)	4*a*	0.0278(3)	0.1324(1)	0.4482(3)	0.045(2)	0.060(2)	0.055(2)	0.003(2)	0.003(2)	0.005(2)
C(11)	4*a*	–0.0399(3)	0.1761(1)	0.3842(3)	0.038(2)	0.057(2)	0.076(2)	0.006(2)	–0.005(2)	–0.000(2)
C(12)	4*a*	0.0187(3)	0.2131(1)	0.2869(3)	0.046(2)	0.048(2)	0.069(2)	0.003(1)	–0.010(2)	–0.001(2)
C(13)	4*a*	–0.0522(4)	0.2552(2)	0.2111(4)	0.060(2)	0.063(2)	0.107(3)	0.008(2)	–0.017(2)	0.016(2)
C(14)	4*a*	0.0049(4)	0.2881(2)	0.1143(5)	0.075(3)	0.076(3)	0.121(4)	0.005(2)	–0.032(3)	0.035(3)
C(15)	4*a*	0.1366(4)	0.2818(2)	0.0897(4)	0.083(3)	0.074(2)	0.090(3)	–0.006(2)	–0.008(3)	0.036(2)
C(16)	4*a*	0.2086(3)	0.2420(1)	0.1631(4)	0.062(2)	0.059(2)	0.075(3)	–0.003(2)	–0.007(2)	0.014(2)
C(17)	4*a*	0.1530(3)	0.2060(1)	0.2633(3)	0.050(2)	0.039(2)	0.056(2)	–0.002(1)	–0.009(2)	–0.000(2)
C(18)	4*a*	0.2244(3)	0.1609(1)	0.3352(3)	0.039(2)	0.038(2)	0.049(2)	0.001(1)	–0.005(2)	–0.010(2)
C(19)	4*a*	0.3647(3)	0.1555(1)	0.3057(3)	0.039(2)	0.040(2)	0.045(2)	–0.001(1)	–0.001(1)	0.005(1)
C(20)	4*a*	0.4092(3)	0.1123(1)	0.2172(3)	0.046(2)	0.050(2)	0.045(2)	–0.000(2)	–0.007(2)	–0.002(2)
O(20)	4*a*	0.3168(2)	0.0771(1)	0.1606(3)	0.055(2)	0.080(2)	0.099(2)	0.008(1)	–0.013(1)	–0.048(2)
C(21)	4*a*	0.5379(3)	0.1061(2)	0.1906(3)	0.054(2)	0.068(2)	0.051(2)	0.008(2)	0.005(2)	–0.004(2)
C(22)	4*a*	0.6248(3)	0.1440(2)	0.2520(4)	0.038(2)	0.085(2)	0.071(3)	–0.003(2)	0.008(2)	0.007(2)
C(23)	4*a*	0.5834(3)	0.1882(2)	0.3378(4)	0.047(2)	0.072(2)	0.092(3)	–0.014(2)	–0.008(2)	–0.011(2)
C(24)	4*a*	0.4542(3)	0.1935(1)	0.3645(4)	0.050(2)	0.054(2)	0.070(2)	–0.006(2)	–0.002(2)	–0.013(2)
C(25)	4*a*	0.4118(5)	–0.1503(2)	0.3397(4)	0.176(5)	0.077(3)	0.115(4)	0.014(3)	0.032(4)	–0.003(3)
C(26)	4*a*	0.5706(4)	0.0658(2)	0.6439(4)	0.059(2)	0.090(3)	0.089(3)	–0.010(2)	–0.016(2)	0.023(3)
C(27)	4*a*	0.7090(4)	0.0873(2)	0.6323(5)	0.098(4)	0.163(4)	0.121(4)	–0.045(3)	–0.041(3)	0.033(4)
C(28)	4*a*	0.5404(5)	0.0483(2)	0.7857(4)	0.137(4)	0.110(3)	0.090(3)	–0.021(3)	–0.003(3)	–0.002(3)

References

1. Bringmann, G.; Breuning, M.; Walter, R.; Wuzik, A.; Peters, K.; Peters, E.-M.: Synthesis of Axially Chiral Biaryls by Atropo-Diastereoselective Cleavage of Configurally Unstable Biaryl Lactones with Menthol-Derived *O*-Nucleophiles. Eur. J. Org. Chem. 1999, in press.
2. Bringmann, G.; Hartung, T.: Atrop-Enantioselective Biaryl Synthesis by Stereocontrolled Cleavage of Configuratively Labile Lactone-Bridged Precursors using Chiral *H*-Nucleophiles. Tetrahedron **49** (1993) 7891-7902.
3. Sheldrick, G. M.: Program Package SHELXTL-plus. Release 4.1. Siemens Analytical X-Ray Instruments Inc. Madison (WI53719) USA 1990.