Crystal structure of (1′*S*)-camphanic acid (2*S*,4*S*)-2-*tert*-butyl-4-methyl-1,3-dioxolan-4-ylmethyl ester, $C_{19}H_{30}O_6$

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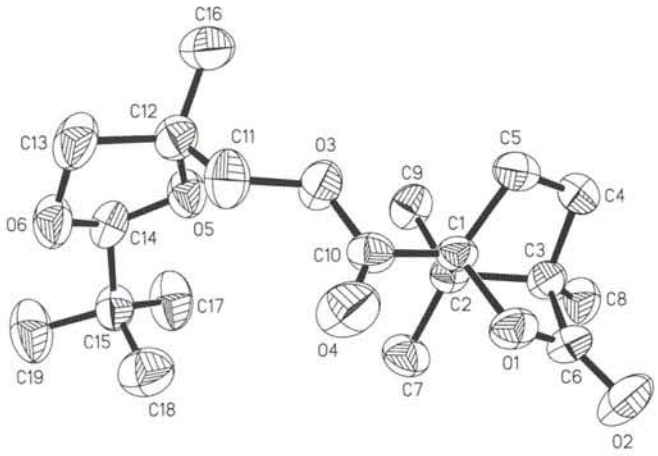

Abstract

$C_{19}H_{30}O_6$, monoclinic, $P12_11$ (No. 4), $a = 6.241(1)$ Å, $b = 10.588(1)$ Å, $c = 14.697(1)$ Å, $\beta = 93.95(1)°$, $V = 968.9$ Å^3, $Z = 2$, $R_{gt}(F) = 0.044$, $wR_{ref}(F^2) = 0.100$, $T = 297$ K.

Source of material

Pyridine (238 mg, 3.01 mmol) and (–)-camphanic acid chloride (621 mg, 2.87 mmol) were added at room temperature to a stirred solution of (2*S*,4*R*)-2-*tert*-butyl-4-hydroxymethyl-4-methyl-1,3-dioxolane (500 mg, 2.87 mmol) in 8 ml of THF. The mixture was stirred for 3 h and concentrated to yield 925 mg (91 %) of colourless crystals, mp 373 K.

Discussion

The crystal structure was determined to establish the relative configuration of the title compound and the absolute configuration of its alcohol moiety. It was prepared from enantiomerically pure (*S*)-camphanic acid chloride and (2*S*,4*R*)-2-*tert*-butyl-4-hydroxymethyl-4-methyl-1,3-dioxolane, thus fixing the absolute configuration [1]. For the structure of a mixed crystal of the (2*R*,4*S*) and (2*S*,4*S*) isomers see [2].

Table 1. Data collection and handling.

Crystal:	colourless needle, size 0.18 × 0.33 × 0.93 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.89 cm^{-1}
Diffractometer, scan mode:	Enraf Nonius CAD4, ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2681, 1815
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1365
$N(param)_{refined}$:	346
Programs:	SHELX-97 [3], ZORTEP [4]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(4A)	2*a*	–0.265(7)	0.120(5)	0.339(3)	0.08(1)
H(4B)	2*a*	–0.119(7)	–0.014(4)	0.369(3)	0.06(1)
H(5A)	2*a*	–0.098(7)	0.149(4)	0.218(3)	0.06(1)
H(5B)	2*a*	0.041(6)	0.008(4)	0.226(2)	0.037(9)
H(7A)	2*a*	0.285(8)	0.383(5)	0.452(4)	0.09(2)
H(7B)	2*a*	0.397(7)	0.394(4)	0.362(3)	0.06(1)
H(7C)	2*a*	0.432(7)	0.281(5)	0.421(3)	0.07(2)
H(8A)	2*a*	–0.051(9)	0.103(6)	0.531(4)	0.10(2)
H(8B)	2*a*	0.091(8)	0.214(5)	0.535(3)	0.07(2)
H(8C)	2*a*	–0.120(9)	0.246(6)	0.503(4)	0.10(2)
H(9A)	2*a*	–0.104(8)	0.399(4)	0.367(3)	0.07(1)
H(9B)	2*a*	–0.160(8)	0.313(5)	0.286(3)	0.07(1)
H(9C)	2*a*	0.010(7)	0.415(4)	0.277(3)	0.06(1)
H(11A)	2*a*	0.298(8)	0.316(5)	0.028(3)	0.07(1)
H(11B)	2*a*	0.480(8)	0.382(4)	0.104(3)	0.07(1)
H(13A)	2*a*	0.183(7)	0.637(4)	–0.040(3)	0.07(1)
H(13B)	2*a*	0.361(7)	0.532(4)	–0.043(3)	0.06(1)
H(14A)	2*a*	0.219(7)	0.738(5)	0.119(3)	0.06(1)
H(16A)	2*a*	–0.119(9)	0.563(7)	0.027(4)	0.11(2)
H(16B)	2*a*	–0.035(9)	0.432(6)	–0.016(4)	0.08(2)
H(16C)	2*a*	–0.131(6)	0.419(4)	0.073(3)	0.05(1)
H(17A)	2*a*	0.160(8)	0.794(5)	0.263(3)	0.08(2)
H(17B)	2*a*	0.41(1)	0.781(7)	0.354(5)	0.13(2)
H(17C)	2*a*	0.252(7)	0.658(5)	0.310(3)	0.06(1)
H(18A)	2*a*	0.702(8)	0.604(5)	0.194(4)	0.08(2)
H(18B)	2*a*	0.555(9)	0.529(6)	0.255(4)	0.08(2)
H(18C)	2*a*	0.69(1)	0.638(6)	0.303(5)	0.12(2)
H(19A)	2*a*	0.426(9)	0.904(6)	0.175(4)	0.09(2)
H(19B)	2*a*	0.629(9)	0.840(6)	0.131(4)	0.11(2)
H(19C)	2*a*	0.69(2)	0.88(1)	0.272(7)	0.21(4)

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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*a*	0.3464(4)	0.0817(2)	0.3306(2)	0.038(1)	0.047(1)	0.055(2)	0.012(1)	–0.002(1)	0.001(1)
O(2)	2*a*	0.3435(6)	0.0000(4)	0.4703(2)	0.080(2)	0.087(2)	0.070(2)	0.025(2)	–0.009(2)	0.029(2)
O(3)	2*a*	0.2021(4)	0.2973(3)	0.1502(2)	0.041(2)	0.064(2)	0.059(2)	0.011(1)	0.013(1)	0.021(1)
O(4)	2*a*	0.5092(5)	0.2007(4)	0.1906(2)	0.042(2)	0.128(3)	0.072(2)	0.023(2)	0.020(1)	0.027(2)
O(5)	2*a*	0.2009(5)	0.5582(3)	0.1483(2)	0.065(2)	0.057(2)	0.039(1)	–0.015(2)	0.014(1)	–0.002(1)
O(6)	2*a*	0.4376(5)	0.6500(3)	0.0603(2)	0.080(2)	0.063(2)	0.039(1)	–0.021(2)	0.014(1)	0.001(1)
C(1)	2*a*	0.2033(5)	0.1688(3)	0.2785(2)	0.028(2)	0.038(2)	0.042(2)	0.005(2)	–0.002(1)	0.000(2)
C(2)	2*a*	0.1370(5)	0.2605(3)	0.3538(2)	0.035(2)	0.037(2)	0.041(2)	0.006(2)	0.003(2)	–0.001(2)
C(3)	2*a*	0.0637(6)	0.1487(4)	0.4129(2)	0.039(2)	0.047(2)	0.039(2)	0.003(2)	0.003(2)	0.004(2)
C(4)	2*a*	–0.0948(7)	0.0772(4)	0.3457(3)	0.041(2)	0.051(2)	0.057(2)	–0.008(2)	–0.001(2)	0.005(2)
C(5)	2*a*	–0.0007(6)	0.0936(4)	0.2527(3)	0.039(2)	0.042(2)	0.052(2)	0.003(2)	–0.006(2)	–0.007(2)
C(6)	2*a*	0.2640(6)	0.0687(4)	0.4138(3)	0.047(2)	0.052(2)	0.050(2)	0.005(2)	–0.012(2)	0.010(2)
C(7)	2*a*	0.3257(8)	0.3342(5)	0.3989(3)	0.059(3)	0.059(3)	0.054(3)	–0.013(3)	–0.006(2)	–0.012(2)
C(8)	2*a*	–0.016(1)	0.1759(7)	0.5057(3)	0.067(3)	0.077(3)	0.051(2)	–0.001(3)	0.011(2)	0.014(3)
C(9)	2*a*	–0.0430(7)	0.3524(4)	0.3233(3)	0.050(3)	0.047(2)	0.054(2)	0.016(2)	0.010(2)	0.003(2)
C(10)	2*a*	0.3250(6)	0.2220(4)	0.2024(2)	0.032(2)	0.053(2)	0.041(2)	0.003(2)	0.004(2)	–0.004(2)
C(11)	2*a*	0.3028(8)	0.3665(4)	0.0789(3)	0.066(3)	0.058(3)	0.055(2)	0.002(3)	0.025(2)	0.003(2)
C(12)	2*a*	0.1828(7)	0.4892(4)	0.0637(2)	0.059(3)	0.056(2)	0.034(2)	–0.007(2)	0.005(2)	–0.003(2)
C(13)	2*a*	0.3012(9)	0.5753(5)	–0.0005(3)	0.102(4)	0.067(3)	0.034(2)	–0.014(3)	0.010(2)	0.002(2)
C(14)	2*a*	0.3139(7)	0.6715(4)	0.1362(2)	0.062(3)	0.042(2)	0.042(2)	0.007(2)	0.010(2)	0.006(2)
C(15)	2*a*	0.4503(7)	0.7109(4)	0.2187(2)	0.065(3)	0.045(2)	0.039(2)	–0.009(2)	0.008(2)	–0.004(2)
C(16)	2*a*	–0.0517(9)	0.4711(6)	0.0332(4)	0.066(3)	0.077(4)	0.062(3)	–0.003(3)	–0.012(3)	–0.001(3)
C(17)	2*a*	0.300(1)	0.7299(6)	0.2971(4)	0.104(5)	0.068(3)	0.058(3)	–0.013(3)	0.033(3)	–0.012(3)
C(18)	2*a*	0.616(1)	0.6110(6)	0.2443(4)	0.074(4)	0.089(4)	0.061(3)	0.005(3)	–0.002(3)	–0.003(3)
C(19)	2*a*	0.558(1)	0.8372(5)	0.1987(4)	0.119(5)	0.065(3)	0.076(3)	–0.036(4)	0.025(4)	–0.009(3)

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