Crystal structure of (2*S**,4*R**,5*R**)-2-*tert*-butyl-5-hydroxy-4-methoxy-5-methyl-1,3-dioxane, $C_{10}H_{20}O_4$

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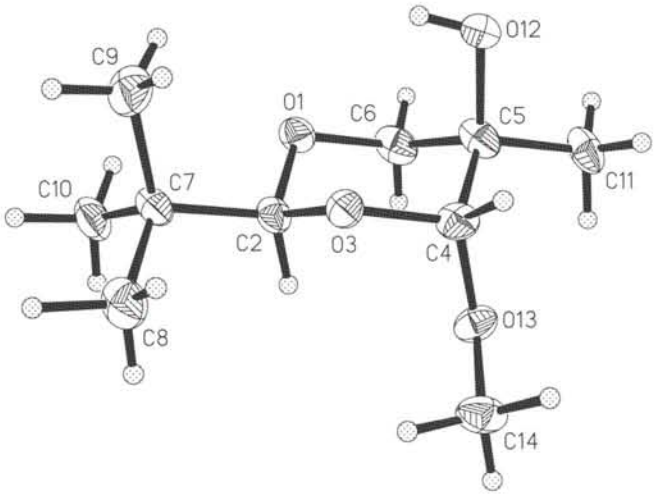

Abstract

$C_{10}H_{20}O_4$, orthorhombic, $P2_12_12_1$ (No. 19), $a = 5.822(1)$ Å, $b = 6.375(2)$ Å, $c = 29.932(7)$ Å, $V = 1110.9$ Å^3, $Z = 4$, $R_{gt}(F) = 0.057$, $wR_{ref}(F^2) = 0.169$, $T = 193$ K.

Source of material

m-Chloroperbenzoic acid (18.2 g, 79 mmol, 70 % in water) was dissolved in methanol (40 ml) and then slowly added at 281 K – 283 K to a stirred solution of 2-*tert*-butyl-5-methyl-4*H*-1,3-dioxin (11.25 g, 72 mmol) in 20 ml of methanol. After complete addition, the solution was allowed to warm up to room temperature, stirred for further 6 h, and then neutralized with a saturated K_2CO_3 solution. The aqueous solution was extracted three times with dichloromethane (20 ml), and the combined extracts were dried ($MgSO_4$). After filtration and evaporation of the solvent, the crude diastereomeric mixture was separated by fractionated distillation in vacuo to give a colourless oil. It crystallized on standing for 12 h at room temperature. Yield: 12.3 g (60.0 mmol, 76%, 75:15:15), mp 343 K – 345 K.

Experimental details

The relatively low ratio of observed numbers of reflections to parameters is due to many unobserved reflections at high θ values and high *k* indices.

Discussion

The crystal structure was determined to establish the relative configuration of the title compound. The title compound is a minor diastereomer obtained by oxidation of 2-*tert*-butyl-5-methyl-4*H*-1,3-dioxin with *m*-chloroperbenzoic acid in methanol [1, 2].

Table 1. Data collection and handling.

Crystal:	colourless, rectangular plate, size 0.13 × 0.23 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.93 cm^{-1}
Diffractometer, scan mode:	Enraf Nonius CAD4, ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1181, 1181
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 775
$N(param)_{refined}$:	207
Programs:	SHELX-97 [3], ZORTEP [4]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	4*a*	0.64(1)	–0.00(1)	0.110(2)	0.03(2)
H(4)	4*a*	0.83(1)	0.43(1)	0.169(2)	0.01(2)
H(6A)	4*a*	0.63(1)	–0.12(1)	0.185(2)	0.03(2)
H(6B)	4*a*	0.84(1)	–0.18(1)	0.210(2)	0.03(2)
H(8A)	4*a*	0.91(1)	0.082(8)	–0.001(2)	0.01(2)
H(8B)	4*a*	0.68(2)	0.09(1)	0.027(3)	0.06(3)
H(8C)	4*a*	0.91(1)	0.24(1)	0.037(2)	0.02(2)
H(9C)	4*a*	1.24(2)	–0.11(2)	0.098(3)	0.09(4)
H(9C)	4*a*	1.26(1)	–0.04(1)	0.044(2)	0.04(2)
H(9C)	4*a*	1.25(2)	0.12(2)	0.075(3)	0.06(3)
H(10A)	4*a*	0.93(2)	–0.35(1)	0.078(2)	0.05(3)
H(10B)	4*a*	0.71(1)	–0.30(1)	0.050(2)	0.04(2)
H(10C)	4*a*	0.94(1)	–0.29(1)	0.026(2)	0.04(2)
H(11A)	4*a*	0.75(1)	0.09(1)	0.269(2)	0.05(3)
H(11B)	4*a*	0.54(1)	0.19(1)	0.239(2)	0.04(2)
H(11C)	4*a*	0.74(1)	0.34(1)	0.253(2)	0.01(2)
H(12)	4*a*	1.13(2)	0.13(2)	0.190(3)	0.09(4)
H(14A)	4*a*	0.27(2)	0.45(2)	0.129(3)	0.08(4)
H(14B)	4*a*	0.50(1)	0.61(1)	0.144(3)	0.06(3)
H(14C)	4*a*	0.48(1)	0.45(1)	0.099(2)	0.04(2)

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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)	4*a*	0.8977(8)	–0.1191(7)	0.1463(1)	0.025(3)	0.029(3)	0.016(2)	0.001(2)	–0.001(2)	0.005(2)
C(2)	4*a*	0.816(1)	0.014(1)	0.1122(2)	0.019(4)	0.026(4)	0.019(3)	–0.005(3)	0.000(3)	–0.003(3)
O(3)	4*a*	0.8650(7)	0.2289(7)	0.1229(1)	0.019(2)	0.024(3)	0.021(2)	–0.002(2)	0.003(2)	0.001(2)
C(4)	4*a*	0.753(1)	0.294(1)	0.1633(2)	0.015(3)	0.031(5)	0.026(4)	–0.005(4)	0.003(3)	0.002(4)
C(5)	4*a*	0.823(1)	0.150(1)	0.2013(2)	0.024(4)	0.036(5)	0.018(3)	–0.010(4)	0.000(3)	0.003(3)
C(6)	4*a*	0.784(1)	–0.079(1)	0.1877(2)	0.031(5)	0.035(5)	0.018(4)	–0.006(4)	–0.001(3)	0.010(3)
C(7)	4*a*	0.932(1)	–0.041(1)	0.0679(2)	0.019(4)	0.035(4)	0.016(3)	–0.009(4)	0.001(3)	0.001(3)
C(8)	4*a*	0.841(2)	0.108(2)	0.0320(3)	0.032(5)	0.046(5)	0.025(4)	–0.007(5)	0.006(4)	0.004(4)
C(9)	4*a*	1.192(1)	–0.023(2)	0.0728(3)	0.026(4)	0.048(6)	0.035(5)	–0.008(4)	0.009(4)	–0.007(5)
C(10)	4*a*	0.867(2)	–0.265(1)	0.0561(2)	0.030(5)	0.051(6)	0.022(4)	–0.009(5)	–0.002(4)	–0.011(4)
C(11)	4*a*	0.702(2)	0.198(2)	0.2453(2)	0.035(5)	0.039(5)	0.016(4)	–0.011(5)	0.003(3)	–0.004(4)
O(12)	4*a*	1.0644(9)	0.1840(9)	0.2099(2)	0.022(3)	0.034(3)	0.025(2)	0.002(3)	–0.005(2)	–0.003(2)
O(13)	4*a*	0.5139(7)	0.2932(8)	0.1592(2)	0.018(3)	0.026(3)	0.029(3)	0.002(2)	0.004(2)	0.006(2)
C(14)	4*a*	0.431(2)	0.460(1)	0.1314(3)	0.032(5)	0.026(4)	0.033(4)	0.003(4)	–0.010(4)	0.008(3)

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