Crystal structure of 2-chloro-2-(chloromethyl)-5-(1-hydroxy-isopropyl)-cyclohexane-1-ol, $C_{10}H_{18}Cl_2O_2$

J. Zukerman-Schpector*[I], D. C. S. Coelho[I], C. M. A. Tanaka[II] and A. J. Marsaioli[II]

[I] Universidade Federal de São Carlos, Laboratório de Cristalografia, Estereodinâmica e Modelagem Molecular, Departamento de Química, C.P.676, 13565-905, São Carlos, SP, Brazil

[II] Universidade Estadual de Campinas, Instituto de Química, Campinas, SP, Brazil

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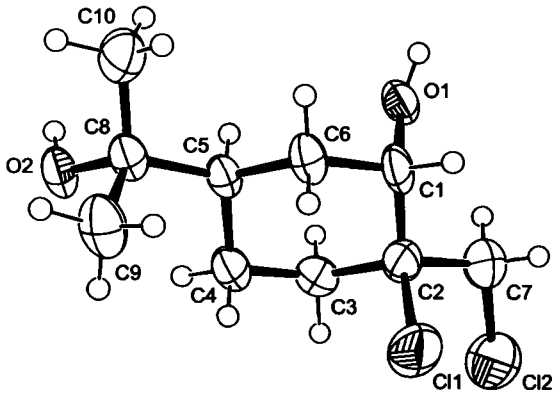

Abstract

$C_{10}H_{18}Cl_2O_2$, monoclinic, $P12_11$ (no. 4), a = 7.9492(9) Å, b = 7.8317(9) Å, c = 9.632(1) Å, β = 98.275(8)°, V = 593.4 Å^3, Z = 2, $R_{gt}(F)$ = 0.041, $wR_{ref}(F^2)$ = 0.127, T = 293 K.

Source of material

Aiming at the optimization of the oxidation of (–)-β-pinene, in order to obtain oxidated *ortho*-trujanes and products of oxidative clivage, several methods were tested. Among them, the reaction with $KMnO_4$/pyridine in aqueous solution was performed. During the neutralization of the base with HCl, the unexpected title compound was obtained and recrystallized from water [1].

Experimental details

Due to the fact that the chirality of the starting material was known, to the low diffraction quality of the crystal and the low speed of measuring of the diffractometer, the data collection was restricted to the minimum necessary to get the crystal structure.

Discussion

The six-membered ring is in a slightly distorted chair conformation, the Cremer and Pople's [2] ring-puckering parameters being q_2 = 0.040(5) Å, q_3 = –0.551(5) Å, φ_2 = 53(7)°, τ_2 = 175.8(5)° and Q = 0.5584(5) Å. The molecules are arranged via hydrogen bonds: d(O1···Cl2^i) = 3.556(4) Å, d(H1O1···Cl1^i) = 2.81 Å, ∠O1–H1O1···Cl2^i = 148°; d(O1···O2^{ii}) = 2.761(5) Å, d(H1O1···O2^{ii}) = 2.29 Å, ∠O1–H1O1···O2^{ii} = 115°; d(O2···O1^{iii}) = 2.874(4) Å, d(H2O2···O1^{iii}) = 1.96 Å, ∠O2–H2O2···O1^{iii} = 152° (symmetry codes: i = 1+x,y,z; ii = x,1+y,z; iii = 1–x,–½+y, 1–z).

Table 1. Data collection and handling.

Crystal:	colorless, irregular, size 0.05 × 0.10 × 0.15 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	5.21 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, $\theta/2\theta$
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1209, 1125
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 851
$N(param)_{refined}$:	129
Programs:	SIR92 [3], SHELXL-97 [4], PARST95 [5], PLATON [6], WinGX [7], ORTEP-3 [8]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*a*	0.3216	0.4052	0.1610	0.053
H(3A)	2*a*	0.1509	0.1633	0.4340	0.059
H(3B)	2*a*	–0.0135	0.1060	0.3355	0.059
H(4A)	2*a*	0.1456	–0.0651	0.2068	0.058
H(4B)	2*a*	0.1859	–0.1161	0.3658	0.058
H(5)	2*a*	0.4309	0.0516	0.3903	0.042
H(6A)	2*a*	0.5240	0.1821	0.1968	0.054
H(6B)	2*a*	0.3582	0.1228	0.1007	0.054
H(1O1)	2*a*	0.4825	0.4344	0.3524	0.055
H(7A)	2*a*	0.0525	0.5351	0.2180	0.078
H(7B)	2*a*	0.1007	0.4849	0.3763	0.078
H(9A)	2*a*	0.5014	–0.3275	0.1150	0.088
H(9B)	2*a*	0.4287	–0.1524	0.0545	0.088
H(9C)	2*a*	0.3143	–0.2751	0.1293	0.088
H(10A)	2*a*	0.7116	–0.0414	0.3649	0.092
H(10B)	2*a*	0.6965	–0.0361	0.2008	0.092
H(10C)	2*a*	0.7430	–0.2075	0.2814	0.092
H(2O2)	2*a*	0.5182	–0.2605	0.4736	0.055

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)	2*a*	0.3138(6)	0.3182(6)	0.2329(5)	0.065(3)	0.023(2)	0.044(3)	–0.002(2)	0.003(2)	0.011(2)
C(2)	2*a*	0.1234(7)	0.2838(6)	0.2437(6)	0.056(3)	0.034(3)	0.050(3)	0.004(2)	–0.013(2)	–0.006(2)
C(3)	2*a*	0.1062(6)	0.1323(7)	0.3383(6)	0.041(2)	0.042(3)	0.064(3)	–0.006(2)	0.005(2)	0.006(3)

* Correspondence author (e-mail: julio@power.ufscar.br)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(4)	2*a*	0.1973(6)	−0.0260(6)	0.2987(6)	0.049(3)	0.031(3)	0.064(3)	−0.004(2)	0.010(2)	0.003(2)
C(5)	2*a*	0.3848(6)	0.0107(6)	0.2963(4)	0.048(2)	0.025(2)	0.032(2)	−0.002(2)	0.004(2)	−0.001(2)
C(6)	2*a*	0.4042(7)	0.1575(6)	0.1953(5)	0.060(3)	0.029(2)	0.046(3)	−0.001(2)	0.010(2)	0.008(2)
Cl(1)	2*a*	0.0230(2)	0.2323(2)	0.0664(2)	0.090(1)	0.064(1)	0.0669(9)	0.0085(9)	−0.0329(8)	−0.0107(9)
O(1)	2*a*	0.3930(4)	0.3803(4)	0.3644(3)	0.058(2)	0.028(2)	0.046(2)	−0.007(2)	−0.005(1)	0.002(2)
C(7)	2*a*	0.0412(7)	0.4468(8)	0.2867(7)	0.065(3)	0.041(3)	0.084(4)	0.006(3)	−0.003(3)	−0.004(3)
Cl(2)	2*a*	−0.1790(2)	0.4189(3)	0.3008(2)	0.0677(9)	0.070(1)	0.142(2)	0.0208(9)	0.009(1)	−0.018(1)
C(8)	2*a*	0.4902(6)	−0.1486(6)	0.2684(5)	0.054(3)	0.029(3)	0.038(2)	−0.002(2)	0.004(2)	0.001(2)
C(9)	2*a*	0.4279(8)	−0.2337(7)	0.1290(5)	0.092(4)	0.043(3)	0.040(3)	0.004(3)	0.010(3)	−0.008(2)
C(10)	2*a*	0.6775(7)	−0.1044(8)	0.2799(7)	0.054(3)	0.048(4)	0.084(4)	0.007(3)	0.018(3)	0.002(3)
O(2)	2*a*	0.4675(4)	−0.2753(4)	0.3740(3)	0.070(2)	0.025(2)	0.040(2)	0.001(2)	−0.000(1)	0.001(2)

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References

1. Tanaka, C. M. A.: Constituentes químicos de cinco espécies de Echinodorus e avaliacão do β-pineno como substrato para obtencão de quirons mais elaborados. PhD thesis, Universidade Estadual de Campinas, UNICAMP, Campinas 2000.
2. Cremer, D.; Pople, J. A.: General definition of ring puckering coordinates. J. Am. Chem. Soc. **97** (1975) 1354-1358
3. Altomare, A.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Burla, M. C.; Polidori, G.; Camalli, M.: SIR92 – a program for automatic solution of crystal structures by direct methods. J. Appl. Crystallogr. **27** (1994) 435.
4. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1990.
5. Nardelli, M.: PARST95 – an update to PARST: a system of Fortran routines for calculating molecular structure parameters from the results of crystal structure analyses. J. Appl. Crystallogr. **28** (1995) 659.
6. Spek, A. L.: Single-crystal structure validation with the program PLATON. J. Appl. Crystallogr. **36** (2003) 7-13.
7. Farrugia, L. J.: WinGX suite for small-molecule single-crystal crystallography. J. Appl. Crystallogr. **32** (1999) 837-838.
8. Farrugia, L. J.: ORTEP-3 for Windows – a version of ORTEP-III with a Graphical User Interface (GUI). J. Appl. Crystallogr. **30** (1997) 565.