Open Access

Zhong-Yan Li, Lin Yuan, Min Zhang and Xian-You Yuan*

Crystal structure of (2-(2-chlorophenyl)-5-methyl-1,3-dioxan-5-yl)methanol, $C_{12}H_{15}ClO_3$

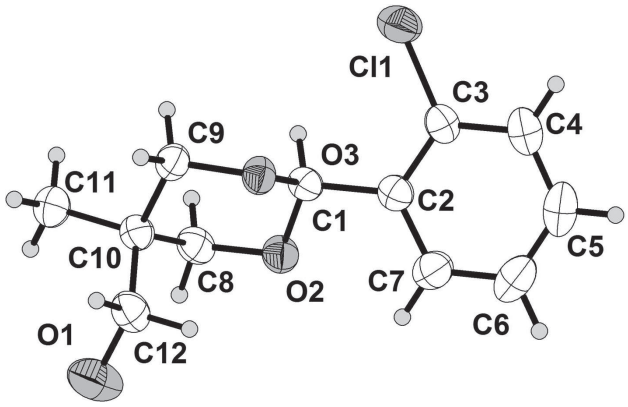

Table 1: Data collection and handling.

Crystal:	Colorless blocks size 0.45 × 0.42 × 0.36 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ	3.2 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness;	55.2°, >99 %,
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14420, 2025, 0.070
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1430
$N(param)_{refined}$:	155
Programs:	SHELX [10], Bruker programs [11], OLEX2 [12]

DOI 10.1515/ncrs-2016-0020
Received January 13, 2016; accepted April 25, 2016; available online May 12, 2016

Abstract

$C_{12}H_{15}ClO_3$, monoclinic, $P2_1/c$ (no. 14), $a = 10.0231(14)$ Å, $b = 10.7318(16)$ Å, $c = 11.2419(17)$ Å, $\beta = 106.637(7)°$, $V = 1158.6(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0493$, $wR_{ref}(F^2) = 0.1879$, $T = 296(2)$ K.

CCDC no.: 1445285

The crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

2,2-Bis(hydroxymethyl) propanol (0.82 g, 6.76 mmol), 2-chlorobenzaldehyde (1.40 g, 10.0 mmol), cyclohexane (10 mL), N,N-dimethytformamide (2 mL) and p-toluene sulfonic acid (0.10 g, 0.58 mmol) were refluxed with stirring at 373 K for four hours, then sodium bicarbonate (0.058 g, 0.70 mmol) was added and stirred at room temperature for half an hour. The solvent was evaporated under reduced pressure and then ethyl acetate was added to dissolve the residue. Subsequently, the solution was washed with saturated salt water (20 mL × 2) and water (20 mL × 2), respectively. Then the organic layer was dried with anhydrous sodium sulfate, filtered and evaporated, and the product was recrystallized from ethyl acetate to afford colorless crystals (yield 70%; m. p. 354.35 K).

Experimental details

Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C). The H atoms of the methyl group were allowed to rotate with a fixed angle around the C–C bond to best fit the experimental electron density, with U_{iso}(H) set to 1.5 U_{eq}(O). The H atoms of the hydroxyl group was allowed to rotate with a fixed angle around the C–O bond to best fit the experimental electron density, with U_{iso}(H) set to 1.5U_{eq}(O).

Discussion

Ketals are not only important structural units for natural products, bioactive compounds and chiral ligand [1, 2] but also parts of compounds with insecticidal effects as well as anti-foaming properties [3, 4]. The crystal structures of 1,3-dioxanes are in the focus of attention of chemists [5–9]. Herein, in this paper, a new compound (2-(2-chlorophenyl)-5-methyl-1,3-dioxan-5-yl)methanol was

***Corresponding author: Xian-You Yuan,** College of Chemistry and Bioengineering, Hunan University of Science and Engineering, Yongzhou Hunan 425199, P. R. China,
e-mail: yuanxianzhangmin@163.com
Zhong-Yan Li, Lin Yuan and Min Zhang: College of Chemistry and Bioengineering, Hunan University of Science and Engineering, Yongzhou Hunan 425199, P. R. China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.04575(7)	0.01475(6)	0.28544(8)	0.0590(4)
O3	0.32246(16)	−0.14102(12)	0.25111(16)	0.0398(5)
O2	0.23843(16)	−0.19771(13)	0.04365(16)	0.0423(6)
C1	0.2081(2)	−0.13461(17)	0.1417(2)	0.0366(7)
H1	0.1251	−0.1710	0.1576	0.044*
C2	0.1828(2)	0.00109(19)	0.1086(2)	0.0379(7)
C3	0.1111(2)	0.07519(18)	0.1700(2)	0.0405(7)
C10	0.3740(2)	−0.35153(17)	0.1898(2)	0.0397(7)
C5	0.1407(3)	0.2519(2)	0.0520(3)	0.0604(10)
H5	0.1264	0.3360	0.0324	0.072*
C8	0.2569(2)	−0.32810(19)	0.0708(2)	0.0424(8)
H8A	0.2783	−0.3703	0.0022	0.051*
H8B	0.1708	−0.3627	0.0796	0.051*
C9	0.3466(3)	−0.26883(19)	0.2908(2)	0.0434(7)
H9A	0.2660	−0.3001	0.3128	0.052*
H9B	0.4260	−0.2728	0.3643	0.052*
C12	0.5147(3)	−0.3190(2)	0.1711(3)	0.0489(8)
C4	0.0897(3)	0.20150(19)	0.1426(3)	0.0522(9)
H4	0.0418	0.2508	0.1848	0.063*
C7	0.2326(3)	0.0545(2)	0.0179(3)	0.0497(8)
H7	0.2803	0.0056	−0.0248	0.060*
C6	0.2125(3)	0.1800(2)	−0.0103(3)	0.0611(10)
H6	0.2472	0.2154	−0.0708	0.073*
C11	0.3707(3)	−0.4873(2)	0.2279(3)	0.0536(9)
H11A	0.2832	−0.5048	0.2433	0.080*
H11B	0.4451	−0.5026	0.3020	0.080*
H11C	0.3816	−0.5402	0.1625	0.080*
O1	0.5531(3)	−0.3938(2)	0.0839(3)	0.0765(8)
H1A	0.4854	−0.4048	0.0235	0.115*
H12A	0.592(3)	−0.325(2)	0.254(3)	0.061(8)*
H12B	0.513(3)	−0.228(2)	0.142(3)	0.044(6)*

synthesized by the reaction of 2,2-bis(hydroxymethyl) propanol with 2-chlorobenzaldehyde by using *p*-toluene sulfonic acid as catalyst, DMF and cyclohexanone as solvent, and its structure was studied by single-crystal structure analysis [10].

There is one molecule in the asymmetric unit of the title structure. All bond lengths and angles are in the expected ranges. The dioxanyl moiety is in a chair conformation with the chlorophenyl group approximately perpendicular to the central ring plane (*cf*. the figure).

Acknowledgements: This work was financially supported by the Science and Technology Planning Project of Hunan Province (2015GK3037), the Scientific Research Fund of Hunan Provincial Education Department (14A058, 13A030).

References

1. Ono, D.; Yamamura, S.; Nakamura, M.: Preparation and properties of bis (sodium sulfate) types of cleavable surfactants derived from diethyl tartrate. J. Oleo. Sci. **54** (2005) 51–57.
2. Marrian, S. F.: The chemical reactions of pentaerythritol and its derivatives. Chem. Rev. **43** (1948) 149–198.
3. Yuan, X. Y.; Yang, N. F.; Luo, H. A.; Liu, Y. J.: Synthesis of hetero diacetals and diketals of pentaerythritol under microwave irradiation. Chin. J. Org. Chem. **25** (2005) 1049–1052.
4. Wang, G. W.; Yuan, X. Y.; Liu, Y. C.; Lei, X.G.: Preparation and properties of sulfonate salt-type cleavable surfactants with a 1, 3-dioxane ring. J. Am. Oil. Chem. Soc. **71** (1994) 727–730.
5. Yuan, L.; Shi J. H.; Zhang M.: Crystal structure of 3, 3-dihydroxymethyl-1,6-dioxa-spiro[5.5]undecane, C11H20O4. Z. Kristallogr. NCS **227** (2012) 35–36.
6. Yuan, L.; Ou, G. C.; Shi, J. H.; Zhang, M.; Yuan, X. Y.: Synthesis and crystal structure of 12-(2-chlorophenyl)-7,11,13,16-tetraoxaspiro [5.2.5.2] cetane. Chin. J. Org. Chem. **33** (2013) 1817–1821.
7. Wang, X. Y.; Shi, J. H.; Zhang, M.; Ng, S. W.: 3, 9-Bis (2-chlorophenyl)-2, 4, 8, 10-tetraoxaspiro [5.5] undecane. Acta Crystallogr. **E66** (2010) o426.
8. Godard, A.; Thiebaud-Roux, S.; De Caro, P.; Vedrenne, E.; Mouloungui, Z.: New one-pot syntheses of ketals and acetals from oleic acid. Ind Crops Prod. **52** (2014) 111–117.
9. Zhang, M.; Jia, G. K.; Yuan, L.; Yuan, X. Y.: Crystal structure of 2- (4-chlorophenyl)-5-methyl-1, 3-dioxane-5-carboxylic acid, $C_{12}H_{13}ClO_4$. Z. Kristallogr. NCS **228** (2013) 393–394.
10. Sheldrick, G. M.: A short history of *SHELX*. Acta Crystallogr. **A64** (2008) 112–122.
11. Bruker (2014) APEX2, SAINT, and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
12. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete strudture solution, refinement and analysis programm. J. Appl. Cryst. **42** (2009) 339–341.