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Crystal structure of (2-(2-chlorophenyl)-5-ethyl-1,3-dioxan-5-yl)methanol hemihydrate, $C_{13}H_{17}ClO_3 \cdot 0.5 H_2O$

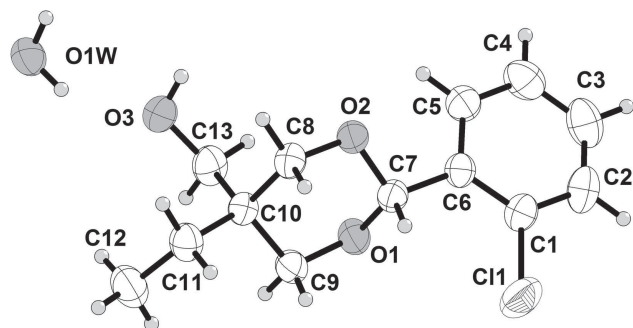


Table 1: Data collection and handling.

Crystal:	Colorless, block, size 0.18×0.38×0.45 mm
Wavelength:	Mo K_{α} radiation (0.71073 Å)
μ :	2.79 cm ⁻¹
Diffractometer, scan mode:	CCD area detector, φ and ω scans
$2\theta_{\max}$:	54.96°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	28223, 3119
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2485
$N(\text{param})_{\text{refined}}$:	167
Programs:	SHELX [13]

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Abstract

$C_{26}H_{36}Cl_2O_7$, orthorhombic, $Pccn$, $a = 14.2010(8)$ Å, $b = 24.4565(15)$ Å, $c = 7.8551(5)$ Å, $V = 2728.1(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0434$, $wR_{\text{ref}}(F^2) = 0.1291$, $T = 296(2)$ K.

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The crystal structure is shown in the figure, Tables 1–3 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) butanol (1.4759 g, 11 mmol), 2-chlorobenzaldehyde (1.4057 g, 10 mmol), cyclohexane (10 mL), N,N-dimethylformamide (2 mL) and p-toluene sulfonic acid (0.1003 g, 0.5825 mmol) were heated and stirred at 388 K for 3.5 hours, then sodium bicarbonate (0.05853 g, 0.6967 mmol) was added to dissolve the residue after the solvent was evaporated under reduced pressure.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	Site	x	y	z	U_{iso}
H(3)	8e	0.8188	0.7070	0.5508	0.096
H(7)	8e	1.0023	0.5734	0.9304	0.046
H(8A)	8e	0.8122	0.6338	0.7512	0.048
H(8B)	8e	0.8555	0.5770	0.8011	0.048
H(9A)	8e	1.0093	0.5470	0.6435	0.056
H(9B)	8e	1.0508	0.5876	0.5085	0.056
H(11A)	8e	0.8580	0.5356	0.5265	0.058
H(11B)	8e	0.7926	0.5848	0.4798	0.058
H(4)	8e	1.1115	0.7543	1.2213	0.072
H(13A)	8e	0.9602	0.6901	0.5880	0.060
H(13B)	8e	0.9650	0.6640	0.4060	0.060
H(5)	8e	1.0191	0.7135	1.0176	0.058
H(3A)	8e	1.2207	0.7032	1.3705	0.077
H(2)	8e	1.2420	0.6118	1.3066	0.070
H(12A)	8e	0.8795	0.6015	0.2360	0.099
H(12B)	8e	0.8468	0.5403	0.2355	0.099
H(12C)	8e	0.9506	0.5551	0.2849	0.099
H(1WA)	8e	0.718(4)	0.764(2)	0.303(4)	0.067
H(1WB)	8e	0.787(3)	0.725(2)	0.263(4)	0.067

The solution was washed with water (20 ml), and dried with anhydrous sodium sulfate. The resulting solution was filtered and evaporated, and the product was recrystallized from cyclohexane to afford colorless crystals (yield 70.2%; m. p. 344 K).

Discussion

Most 1,3-dioxane compounds are important part of organic chemistry. It has special activity [1] and widely existed in

Table 3: Atomic displacement parameters (Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Cl(1)	8e	1.16020(4)	0.53938(2)	1.07988(8)	0.0785(4)	0.0488(3)	0.0904(4)	0.0157(2)	−0.0235(3)	0.0054(2)
O(2)	8e	0.92981(7)	0.63994(5)	0.8845(1)	0.0356(6)	0.0497(7)	0.0375(6)	0.0046(5)	−0.0002(4)	−0.0024(5)
O(1)	8e	1.06598(8)	0.61321(5)	0.7461(1)	0.0323(6)	0.0658(8)	0.0394(6)	0.0027(5)	−0.0006(5)	−0.0064(5)
O(3)	8e	0.8389(1)	0.69035(6)	0.4679(2)	0.089(1)	0.0594(8)	0.0439(7)	0.0269(7)	−0.0078(7)	0.0043(6)
C(6)	8e	1.0741(1)	0.63775(6)	1.0355(2)	0.0395(8)	0.0411(8)	0.0344(8)	−0.0020(6)	−0.0003(6)	0.0046(6)
C(10)	8e	0.9174(1)	0.60975(6)	0.5868(2)	0.0339(7)	0.0391(8)	0.0352(7)	−0.0012(6)	−0.0012(6)	0.0028(6)
C(1)	8e	1.1419(1)	0.60841(7)	1.1245(2)	0.0430(9)	0.0458(9)	0.0457(9)	−0.0016(7)	−0.0030(7)	0.0074(7)
C(7)	8e	1.0150(1)	0.61154(6)	0.8996(2)	0.0381(8)	0.0401(8)	0.0360(8)	0.0022(6)	−0.0013(6)	0.0028(6)
C(8)	8e	0.8706(1)	0.61354(7)	0.7615(2)	0.0329(7)	0.0476(9)	0.0385(8)	−0.0020(6)	0.0001(6)	0.0052(6)
C(9)	8e	1.0154(1)	0.58532(8)	0.6138(2)	0.0407(9)	0.059(1)	0.0396(8)	0.0079(7)	−0.0023(7)	−0.0091(7)
C(11)	8e	0.8571(1)	0.57183(8)	0.4761(2)	0.0477(9)	0.0502(9)	0.0464(9)	−0.0052(7)	−0.0071(7)	−0.0014(8)
C(4)	8e	1.1187(2)	0.71732(8)	1.1977(3)	0.070(1)	0.050(1)	0.060(1)	−0.0101(9)	−0.001(1)	−0.0098(9)
C(13)	8e	0.9273(1)	0.66651(7)	0.5086(2)	0.059(1)	0.0464(9)	0.0435(9)	−0.0070(8)	−0.0006(8)	0.0091(7)
C(5)	8e	1.0639(1)	0.69282(7)	1.0751(2)	0.056(1)	0.0442(9)	0.0458(9)	0.0015(8)	−0.0028(8)	0.0003(7)
C(3)	8e	1.1846(2)	0.68680(9)	1.2858(3)	0.067(1)	0.073(1)	0.053(1)	−0.025(1)	−0.012(1)	−0.004(1)
C(2)	8e	1.1971(1)	0.63230(9)	1.2487(3)	0.049(1)	0.072(1)	0.054(1)	−0.0077(9)	−0.0150(8)	0.0124(9)
C(12)	8e	0.8862(2)	0.5667(1)	0.2912(3)	0.070(1)	0.077(1)	0.050(1)	−0.001(1)	−0.007(1)	−0.015(1)
O(1W)	4c	0.75	0.75	0.2195(2)	0.074(1)	0.056(1)	0.0385(9)	0.0056(9)	0	0

many natural product such as carbohydrate, vitamin, hormone and so on [2]. The synthesis of 1,3-dioxane compounds was mainly applied from aldol reaction through hydroxy containing polyfunctional group with aldehyde (ketone) [3]. 2,2-Bis(hydroxymethyl) butanol, which has typical neoamyl group, is a common chemical product [4–7]. The neoamyl group display a good heat resistance, hydrolysis resistance, and color stability [8–10]. Therefore, in this paper, a novel compound (2-(2-chlorophenyl)-5-ethyl-1,3-dioxan-5-yl)methanol was synthesized by the reaction of 2,2-bis(hydroxymethyl) butanol with 2-chlorobenzaldehyde, and p-toluene sulfonic acid as catalyst, DMF and cyclohexanone as solvent. The crystal structures of some similar 1,3-dioxanes have been reported [11, 12].

In the crystal, adjacent molecules are connected by O–H···O (2.7622(19) Å) hydrogen bonds between the oxygen atoms O3 and O1W into a one-dimensional chain.

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