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The crystal structure of (2-(4-chlorophenyl)-5-methyl-1,3-dioxan-5-yl)methanol, C₁₂H₁₅ClO₃

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Abstract

C₁₂H₁₅ClO₃, orthorhombic, *P*2₁2₁2₁ (no. 19), *a* = 5.788(2) Å, *b* = 11.079(4) Å, *c* = 18.916(7) Å, *V* = 1213.0(8) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0559, *wR*_{ref}(*F*²) = 0.1898, *T* = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

2-(Hydroxymethyl)-2-methylpropane-1,3-diol (1.32 g, 11 mmol), 4-chlorobenzaldehyde (1.40 g, 10 mmol), *N,N*-dimethylformamide (5.0 mL), cyclohexane (10.0 mL) and *p*-toluene sulfonic acid (0.15 g) were heated and stirred at

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.45 × 0.40 × 0.36 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.30 mm ^{−1}
Diffractometer, scan mode:	φ and ω
θ _{max} , completeness:	27.6°, 99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	13,039, 2765, 0.041
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2291
<i>N</i> (<i>param</i>) _{refined} :	147
Programs:	Bruker [1], SHELX [2, 3]

388 K for 5 h, then sodium bicarbonate (0.09 g) was added to dissolve the residue. After the solution was washed with saturated salt water (10 mL*3), and dried with anhydrous sodium sulfate. The resulting solution was filtered and evaporated, and the product was recrystallized from cyclohexane and ethyl acetate to afford colorless crystals (1.94 g, yield 80%).

Experimental details

All hydrogen atoms were positioned using the standard options of the SHELX system. The *U*_{iso} values of the hydrogen atoms of methyl group was set to 1.5 *U*_{eq}(C) and the *U*_{iso} values of all other hydrogen atoms were set to 1.2 *U*_{eq}(C).

Comment

Polyfunctional branched compounds are a class of compounds with a wide range of applications [4–7]. Acetal compounds are important and were used widely in fragrances and food flavors [8, 9]. Acetal compounds are stable in neutral and alkaline conditions and will degrade and generate a material of small molecules under acidic conditions. Herein, a novel acetal compound was synthesized and characterized by single-crystal X-ray diffraction [1–3].

There is one molecule in the asymmetric unit (see the figure). The torsion angles of C(9)–O(2)–C(1)–O(1), C(10)–O(1)–C(1)–O(2) and C(1)–O(2)–C(9)–C(8) are 63.1(3), −63.5(3) and −57.4(3), respectively, indicating that the 1,3-dioxane

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.5084 (5)	0.8226 (2)	0.90060 (13)	0.0415 (6)
H1	0.4109	0.8460	0.9407	0.050*
C2	0.6044 (5)	0.6979 (2)	0.91251 (13)	0.0399 (6)
C3	0.8148 (5)	0.6631 (3)	0.88498 (16)	0.0481 (7)
H3	0.9044	0.7188	0.8604	0.058*
C4	0.8942 (6)	0.5456 (3)	0.89356 (17)	0.0495 (7)
H4	1.0343	0.5218	0.8739	0.059*
C5	0.7624 (6)	0.4651 (2)	0.93150 (15)	0.0480 (7)
C6	0.5547 (7)	0.4976 (3)	0.95972 (16)	0.0548 (8)
H6	0.4662	0.4419	0.9846	0.066*
C7	0.4779 (6)	0.6153 (3)	0.95061 (15)	0.0487 (7)
H7	0.3383	0.6388	0.9707	0.058*
C8	0.4492 (5)	1.0337 (3)	0.81919 (15)	0.0434 (6)
C9	0.6063 (7)	1.0251 (3)	0.88326 (15)	0.0506 (7)
H9A	0.7357	1.0798	0.8772	0.061*
H9B	0.5216	1.0501	0.9250	0.061*
C10	0.2685 (6)	0.9340 (3)	0.8254 (2)	0.0575 (8)
H10A	0.1637	0.9528	0.8638	0.069*
H10B	0.1791	0.9304	0.7820	0.069*
C11	0.5880 (6)	1.0152 (3)	0.75207 (16)	0.0533 (7)
H11A	0.6597	0.9361	0.7535	0.064*
H11B	0.4839	1.0168	0.7119	0.064*
C12	0.3282 (7)	1.1572 (3)	0.8195 (2)	0.0667 (9)
H12A	0.2507	1.1687	0.8638	0.100*
H12B	0.2174	1.1603	0.7817	0.100*
H12C	0.4409	1.2199	0.8130	0.100*
Cl1	0.8634 (2)	0.31890 (7)	0.94291 (6)	0.0785 (4)
O1	0.3740 (4)	0.81831 (18)	0.83805 (11)	0.0490 (5)
O2	0.6920 (4)	0.90435 (17)	0.89350 (10)	0.0465 (5)
O3	0.7614 (5)	1.1034 (3)	0.74217 (16)	0.0745 (8)
H3A	0.7036	1.1645	0.7253	0.112*

exhibits a chair conformation. The phenyl ring occupies the equatorial bond at C(1) atom (see the figure). All geometric parameters are in the expected ranges [10, 11].

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