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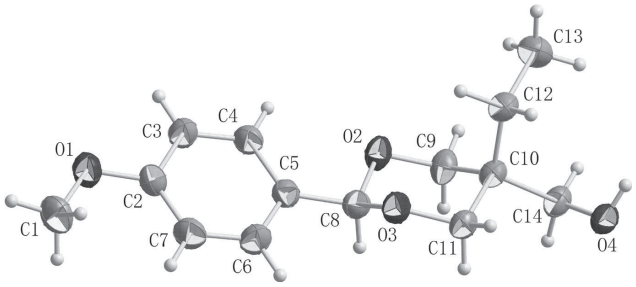
Crystal structure of (5-ethyl-2-(4-methoxyphenyl)-1,3-dioxan-5-yl)methanol, $C_{14}H_{20}O_4$



https://doi.org/10.1515/ncrs-2018-0114
Received April 15, 2018; accepted June 29, 2018; available online July 23, 2018

Abstract

$C_{14}H_{20}O_4$, triclinic, $P\bar{1}$, $a = 6.1028(13)$ Å, $b = 10.885(2)$ Å, $c = 20.737(4)$ Å, $\alpha = 100.673(2)°$, $\beta = 92.465(2)°$, $\gamma = 98.973(2)°$, $V = 1333.5(5)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0431$, $wR_{ref}(F^2) = 0.1225$, $T = 296(2)$ K.

CCDC no.: 1852454

One of two crystallographically independent molecules of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

Trimethylolpropane (1.47 g, 11 mmol), 4-methoxybenzaldehyde (1.36 g, 10 mmol), cyclohexane (10.0 mL), *N*,*N*-dimethytformamide (5.0 mL) and *p*-toluene sulfonic acid (0.15 g) were heated and stirred at 388 K for 5 h. Now sodium bicarbonate (0.09 g) was added to dissolve the residue after the solvent was evaporated. The solution was washed with brine (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 colourless crystals (1.89 g, yield 75%).

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size	0.42 × 0.37 × 0.33 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEXII, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12893, 4683, 0.024
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3531
$N(param)_{refined}$:	329
Programs:	Bruker [8], SHELX [9], Olex2 [10]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.5588(2)	0.83716(11)	0.43992(6)	0.0607(3)
O2	0.44859(17)	0.34516(10)	0.21887(5)	0.0503(3)
O3	0.82794(17)	0.41785(10)	0.22107(5)	0.0509(3)
O4	0.8917(2)	0.09975(11)	0.07063(6)	0.0587(3)
H4	0.8670	0.1112	0.0332	0.088*
C1	0.7448(4)	0.90499(19)	0.48291(10)	0.0767(6)
H1A	0.8653	0.9303	0.4573	0.115*
H1B	0.7908	0.8515	0.5110	0.115*
H1C	0.7035	0.9789	0.5093	0.115*
C2	0.5931(3)	0.73228(15)	0.39629(8)	0.0474(4)
C3	0.4165(3)	0.67435(16)	0.35137(8)	0.0549(4)
H3	0.2868	0.7093	0.3513	0.066*
C4	0.4302(3)	0.56577(16)	0.30683(8)	0.0532(4)
H4A	0.3088	0.5272	0.2775	0.064*
C5	0.6233(3)	0.51324(15)	0.30519(7)	0.0451(4)
C6	0.7988(3)	0.57326(17)	0.34948(9)	0.0588(5)
H6	0.9301	0.5398	0.3488	0.071*
C7	0.7862(3)	0.68148(18)	0.39481(9)	0.0594(5)
H7	0.9074	0.7200	0.4242	0.071*
C8	0.6450(3)	0.39293(16)	0.25922(8)	0.0480(4)
H8	0.6744	0.3299	0.2852	0.058*
C9	0.4655(3)	0.22577(15)	0.17870(8)	0.0506(4)
H9A	0.3289	0.1942	0.1510	0.061*
H9B	0.4839	0.1653	0.2066	0.061*
C10	0.6617(2)	0.23656(14)	0.13540(7)	0.0410(4)
C11	0.8667(3)	0.30232(16)	0.18084(8)	0.0511(4)
H11A	0.9061	0.2457	0.2088	0.061*
H11B	0.9909	0.3208	0.1545	0.061*
C12	0.6232(3)	0.31489(15)	0.08293(8)	0.0480(4)
H12A	0.5997	0.3980	0.1050	0.058*
H12B	0.7578	0.3269	0.0600	0.058*
C13	0.4300(3)	0.25958(19)	0.03219(10)	0.0706(5)
H13A	0.4576	0.1811	0.0067	0.106*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H13B	0.4141	0.3182	0.0036	0.106*
H13C	0.2958	0.2443	0.0541	0.106*
C14	0.6938(3)	0.10291(15)	0.10514(8)	0.0516(4)
H14A	0.6999	0.0542	0.1397	0.062*
H14B	0.5663	0.0627	0.0749	0.062*
O5	0.9048(2)	0.36610(14)	0.44044(7)	0.0781(4)
O6	0.81422(18)	−0.08568(11)	0.20359(5)	0.0524(3)
O7	1.1217(2)	−0.12335(12)	0.26251(6)	0.0614(3)
O8	1.1052(2)	−0.10886(12)	0.06112(6)	0.0663(4)
H8A	1.0315	−0.0515	0.0681	0.100*
C15	0.7140(4)	0.3921(2)	0.47389(12)	0.0964(8)
H15A	0.5942	0.3924	0.4424	0.145*
H15B	0.7480	0.4735	0.5027	0.145*
H15C	0.6708	0.3280	0.4992	0.145*
C16	0.8904(3)	0.25229(18)	0.39823(9)	0.0593(5)
C17	0.7061(3)	0.15755(19)	0.38699(10)	0.0675(5)
H17	0.5777	0.1694	0.4082	0.081*
C18	0.7135(3)	0.04527(18)	0.34414(10)	0.0648(5)
H18	0.5881	−0.0176	0.3365	0.078*
C19	0.9009(3)	0.02380(17)	0.31245(8)	0.0537(4)
C20	1.0828(3)	0.1214(2)	0.32277(9)	0.0664(5)
H20	1.2101	0.1101	0.3008	0.080*
C21	1.0775(3)	0.2342(2)	0.36490(10)	0.0706(5)
H21	1.2004	0.2986	0.3710	0.085*
C22	0.9028(3)	−0.09792(17)	0.26591(8)	0.0557(4)
H22	0.8091	−0.1669	0.2811	0.067*
C23	0.8096(3)	−0.19931(16)	0.15622(9)	0.0537(4)
H23A	0.7472	−0.1887	0.1142	0.064*
H23B	0.7139	−0.2683	0.1699	0.064*
C24	1.0409(2)	−0.23339(14)	0.14809(8)	0.0446(4)
C25	1.1350(3)	−0.23750(17)	0.21695(8)	0.0573(5)
H25A	1.0526	−0.3092	0.2320	0.069*
H25B	1.2891	−0.2493	0.2153	0.069*
C26	1.0175(3)	−0.36285(16)	0.10254(10)	0.0623(5)
H26A	0.9007	−0.4199	0.1174	0.075*
H26B	0.9683	−0.3538	0.0588	0.075*
C27	1.2224(4)	−0.42525(19)	0.09700(12)	0.0772(6)
H27A	1.1892	−0.5041	0.0660	0.116*
H27B	1.2675	−0.4410	0.1392	0.116*
H27C	1.3405	−0.3703	0.0823	0.116*
C28	1.1906(3)	−0.13095(15)	0.12186(8)	0.0497(4)
H28A	1.2104	−0.0525	0.1542	0.060*
H28B	1.3357	−0.1556	0.1162	0.060*

Experimental details

All hydrogen atoms were positioned geometrically. The U_{iso} values of the hydrogen atoms of methyl groups were 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

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

In the structure, the torsion angles of C(9)—O(2)—C(8)—O(3), C(11)—O(3)—C(8)—O(2) and C(8)—O(3)—C(11)—C(10) are 63.56(16), −63.37(16) and 58.19(16), respectively, indicating that the 1,3-dioxane ring exhibits a chair conformation. The torsion angle of C(11)—O(3)—C(8)—C(5) is 174.60(12), suggesting that the phenyl ring occupies the equatorial bond of C(8) atom. The geometry of the two crystallographically independent molecules are very similar.

Acknowledgements: This work was financially supported by the Natural Science Foundation of Hunan Province of China (2017JJ3093).

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