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Crystal structure of (2-(4-bromophenyl)-5-ethyl-1,3-dioxan-5-yl)methanol, $C_{13}H_{17}BrO_3$

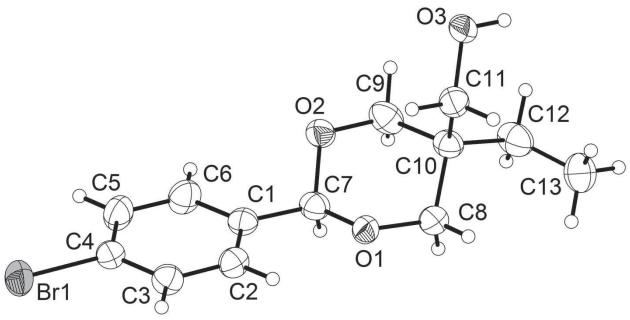

Table 1: Data collection and handling.

Crystal:	Colorless blocks
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	31.2 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}:	19045, 4611, 0.037
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3983
$N(param)_{refined}$:	312
Programs:	SHELX [12], Bruker programs [13]

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Abstract

$C_{13}H_{17}BrO_3$, triclinic, $P\bar{1}$ (No. 2), $a = 9.8235(7)$ Å, $b = 10.3354(7)$ Å, $c = 13.9981(10)$ Å, $\alpha = 99.853(3)°$, $\beta = 95.106(3)°$, $\gamma = 108.139(3)°$, $V = 1315.22(16)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0340$, $wR_{ref}(F^2) = 0.1245$, $T = 296(2)$ K.

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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

Trimethylolpropane (1.6100 g, 12.0000 mmol), 4-bromobenzaldehyde (1.8502 g, 10.0001 mmol), cyclohexane (10.0 mL), N,N-dimethylformamide (5.0 mL) and p-toluene sulfonic acid (0.1730 g, 1.0047 mmol) were heated and stirred at 388.15 K for 3.5 h, then sodium bicarbonate (0.0856 g, 1.0190 mmol) was added to dissolve the residue after the solvent was evaporated under reduced pressure. 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 colourless crystals (2.2159 g, 7.3613 mmol; yield 73.62%; m. p. 368.95–369.85 K).

Experimental details

All H atoms were placed at calculated positions and were included in the riding model approximation. 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 (HFIX 137 in the SHELX program [11]), with U(H) set to $1.5U_{eq}$(O). The H atoms of the hydroxyl groups were allowed to rotate with a fixed angle around the C—O bond to best fit the experimental electron density (HFIX 147 in the SHELX program [11]), with U(H) set to $1.5U_{eq}$(O).

Discussion

Acetals have many properties such as aroma or chemical stability [1], which were used widely in fragrances and food flavors [2–4]. 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 have a wide range of applications. These compounds are important for the synthesis of fine chemicals and other industrial applications and have great potential for development [5–7]. The crystal structures of some similar 1,3-dioxanes have been reported [8–11]. The bond lengths and angles are in the expected

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	−0.45008(3)	0.23532(4)	0.13884(2)	0.05915(18)
O1	0.1697(2)	0.40478(19)	0.45860(14)	0.0422(5)
O2	0.0425(2)	0.1912(2)	0.49107(14)	0.0433(5)
O3	0.3743(2)	0.1087(2)	0.50954(17)	0.0520(6)
H3	0.4478	0.1243	0.5485	0.078*
C4	−0.2981(3)	0.2598(3)	0.2412(2)	0.0390(6)
C1	−0.0785(3)	0.2967(3)	0.3900(2)	0.0362(6)
C7	0.0365(3)	0.3209(3)	0.4760(2)	0.0377(6)
H7	0.0104	0.3686	0.5347	0.045*
C8	0.2781(3)	0.4380(3)	0.5424(2)	0.0443(7)
H8A	0.2520	0.4918	0.5972	0.053*
H8B	0.3700	0.4950	0.5283	0.053*
C2	−0.0520(3)	0.3676(3)	0.3151(2)	0.0387(6)
H2	0.0407	0.4280	0.3151	0.046*
C11	0.3621(3)	0.2371(3)	0.4925(2)	0.0413(6)
H11A	0.3030	0.2199	0.4294	0.050*
H11B	0.4577	0.2998	0.4896	0.050*
C3	−0.1612(3)	0.3499(3)	0.2400(2)	0.0407(6)
H3A	−0.1430	0.3978	0.1895	0.049*
C12	0.3893(4)	0.3476(4)	0.6726(2)	0.0530(8)
H12A	0.3354	0.3793	0.7209	0.064*
H12B	0.4044	0.2646	0.6880	0.064*
C10	0.2957(3)	0.3073(3)	0.57119(19)	0.0372(6)
C6	−0.2169(3)	0.2064(3)	0.3887(2)	0.0488(7)
H6	−0.2356	0.1583	0.4391	0.059*
C9	0.1444(3)	0.2112(3)	0.5766(2)	0.0469(7)
H9A	0.1498	0.1215	0.5846	0.056*
H9B	0.1100	0.2503	0.6338	0.056*
C5	−0.3267(3)	0.1869(3)	0.3146(2)	0.0492(7)
H5	−0.4190	0.1254	0.3139	0.059*
C13	0.5361(4)	0.4592(4)	0.6835(3)	0.0689(10)
H13A	0.5925	0.4282	0.6379	0.103*
H13B	0.5856	0.4770	0.7492	0.103*
H13C	0.5232	0.5432	0.6704	0.103*
Br2	0.25069(4)	0.01295(3)	−0.07613(3)	0.06352(18)
O4	0.34139(18)	0.67823(18)	0.13075(13)	0.0375(4)
O5	0.09608(19)	0.58298(19)	0.13708(14)	0.0418(5)
C23	0.2212(3)	0.8284(3)	0.21837(18)	0.0327(5)
C20	0.2052(3)	0.5893(3)	0.07821(18)	0.0363(6)
H20	0.1822	0.6255	0.0208	0.044*
O6	0.3878(2)	0.8940(2)	0.37328(16)	0.0555(6)
H6A	0.3724	0.9567	0.4104	0.083*
C19	0.1386(3)	0.3334(3)	0.0785(2)	0.0416(6)
H19	0.0797	0.3436	0.1257	0.050*
C21	0.3406(3)	0.8181(3)	0.1573(2)	0.0410(6)
H21A	0.3266	0.8524	0.0981	0.049*
H21B	0.4339	0.8768	0.1945	0.049*
C22	0.0794(3)	0.7163(3)	0.1646(2)	0.0422(7)
H22A	0.0055	0.7095	0.2068	0.051*
H22B	0.0470	0.7440	0.1062	0.051*
C17	0.2367(3)	0.1902(3)	−0.0261(2)	0.0402(6)
C14	0.2154(3)	0.4475(3)	0.04421(18)	0.0350(6)
C24	0.2535(3)	0.7986(3)	0.31896(19)	0.0403(6)
H24A	0.1756	0.8039	0.3558	0.048*
H24B	0.2568	0.7046	0.3112	0.048*
C15	0.3035(3)	0.4308(3)	−0.0251(2)	0.0448(7)
H15	0.3559	0.5076	−0.0482	0.054*
C16	0.3155(3)	0.3032(3)	−0.0604(2)	0.0462(7)
H16	0.3759	0.2934	−0.1067	0.055*
C18	0.1484(3)	0.2036(3)	0.0432(2)	0.0441(6)
H18	0.0961	0.1264	0.0662	0.053*
C25	0.2091(3)	0.9744(3)	0.2247(2)	0.0446(7)
H25A	0.2991	1.0423	0.2607	0.054*
H25B	0.1995	0.9911	0.1588	0.054*
C26	0.0850(4)	1.0002(3)	0.2729(3)	0.0564(8)
H26A	−0.0053	0.9358	0.2369	0.085*
H26B	0.0872	1.0937	0.2731	0.085*
H26C	0.0946	0.9872	0.3391	0.085*

ranges. There are two crystallographically independent molecules in the asymmetric unit. One of them is shown in the figure.

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