Crystal structure of 2-(4-chlorophenyl)-5-methyl-1,3-dioxane-5-carboxylic acid, $C_{12}H_{13}ClO_4$

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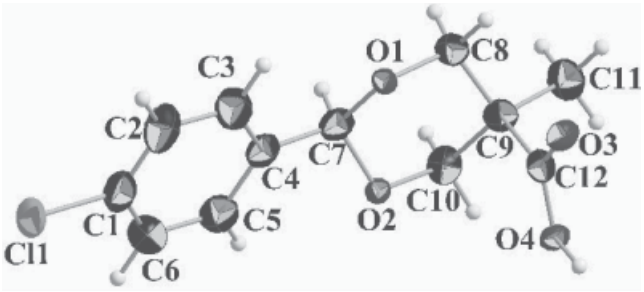

Abstract

$C_{12}H_{13}ClO_4$, monoclinic, *C*2/*c* (no. 15), a = 35.310(2) Å, b = 5.9993(2) Å, c = 22.7422(8) Å, β = 98.692(4)°, V = 4762.3 Å^3, Z = 16, $R_{gt}(F)$ = 0.0397, $wR_{ref}(F^2)$ = 0.1048, T = 153 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.35×0.37×0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	3.21 cm^{-1}
Diffractometer, scan mode:	CrysAlis CCD, φ and ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10335, 4190
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3398
$N(param)_{refined}$:	316
Programs:	SHELX [7]

Source of material

2,2-bis(hydroxymethyl) propionic acid (0.5061 g, 3.77 mol), 4-chlorobenzaldehyde (0.5816 g, 4.15 mol), *N*,*N*-dimethylformamide (2 mL), cyclohexane (15 mL) and *p*-toluene sulfonic acid (0.0326 g) were heated and stirred at 373 K for six hours. Sodium bicarbonate (0.0351 g, 0.42 mol) was added to dissolve the residue after the solvent was evaporated under reduced pressure. The organic 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 *n*-hexane to afford colourless crystals (yield 71%; **m.p.** 453 K).

Discussion

The title compound was synthesized to be used as a protection of carbonyl or synthetic intermediate in organic syntheses [1]. This class of compounds has useful insecticidal as well as anti-foaming properties [2, 3]. The crystal structures of some similar 1,3-dioxanes have been reported [4-6]. In the title structure, there are two independent molecules in the asymmetric unit. The 1,3-dioxane ring adopts a chair conformation and the 4-chlorophenyl substituent occupies an equatorial site. In the crystal structure, adjacent crystallographic independant molecules are connected by two O–H···O hydrogen bonds (O···O distances: 2.618(18) and 2.681(18) Å) into a dimer.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(8D)	8*f*	0.7757(5)	0.443(3)	–0.0334(8)	0.036
H(4D)	8*f*	0.7897(5)	0.529(3)	0.8683(8)	0.032
H(19)	8*f*	0.9255	0.2263	0.0653	0.027
H(3)	8*f*	0.6069	0.3915	0.8113	0.035
H(5)	8*f*	0.6621	0.4054	0.6685	0.034
H(8A)	8*f*	0.6798	0.8407	0.8986	0.026
H(8B)	8*f*	0.6634	0.9691	0.8383	0.026
H(10A)	8*f*	0.7033	0.9570	0.7516	0.027
H(10B)	8*f*	0.7433	0.8257	0.7620	0.027
H(21A)	8*f*	0.9032	0.0105	–0.0163	0.029
H(21B)	8*f*	0.8824	0.1255	–0.0762	0.029
H(7)	8*f*	0.6466	0.7681	0.7506	0.025
H(20A)	8*f*	0.8302	0.1748	0.0724	0.026
H(20B)	8*f*	0.8700	0.0415	0.0780	0.026
H(6)	8*f*	0.6246	0.1087	0.6262	0.038
H(15)	8*f*	0.9116	0.6311	0.1417	0.039
H(14)	8*f*	0.9531	0.9250	0.1749	0.048
H(11A)	8*f*	0.7616	1.1043	0.8477	0.040
H(11B)	8*f*	0.7370	1.1098	0.9012	0.040
H(11C)	8*f*	0.7193	1.2066	0.8373	0.040
H(23A)	8*f*	0.8067	–0.1232	–0.0052	0.041
H(23B)	8*f*	0.8489	–0.2246	–0.0021	0.041
H(23C)	8*f*	0.8261	–0.1402	–0.0642	0.041
H(17)	8*f*	0.9650	0.5579	–0.0024	0.042
H(2)	8*f*	0.5689	0.0980	0.7690	0.038
H(18)	8*f*	1.0065	0.8493	0.0312	0.051

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(1)	8*f*	0.56600(2)	–0.14619(9)	0.66394(3)	0.0374(3)	0.0353(3)	0.0568(4)	–0.0102(2)	–0.0026(3)	–0.0096(3)
Cl(2)	8*f*	1.01330(2)	1.1347(1)	0.12909(4)	0.0410(4)	0.0344(3)	0.1135(6)	–0.0113(3)	–0.0205(4)	–0.0017(3)
O(1)	8*f*	0.66110(3)	0.6379(2)	0.83087(5)	0.0211(7)	0.0256(7)	0.0180(6)	–0.0010(6)	0.0035(5)	–0.0011(5)
O(2)	8*f*	0.69750(3)	0.6259(2)	0.75373(5)	0.0191(7)	0.0250(7)	0.0209(7)	–0.0015(5)	0.0040(5)	–0.0010(5)
O(5)	8*f*	0.87570(3)	0.3709(2)	0.07001(5)	0.0185(7)	0.0250(7)	0.0232(7)	–0.0005(5)	0.0036(5)	–0.0004(5)
O(7)	8*f*	0.82284(4)	0.3408(2)	–0.09411(6)	0.0287(8)	0.0345(8)	0.0235(7)	–0.0011(6)	0.0012(6)	0.0084(6)
O(8)	8*f*	0.79306(4)	0.3572(2)	–0.01447(6)	0.0238(8)	0.0336(8)	0.0309(8)	0.0116(6)	0.0004(6)	0.0006(6)
O(6)	8*f*	0.90520(4)	0.3426(2)	–0.01451(6)	0.0222(7)	0.0276(7)	0.0248(7)	–0.0012(6)	0.0054(5)	0.0006(6)
O(4)	8*f*	0.77408(4)	0.6237(2)	0.84885(6)	0.0221(7)	0.0292(8)	0.0282(7)	0.0072(6)	0.0032(6)	0.0012(6)
C(9)	8*f*	0.71937(5)	0.8599(3)	0.83965(8)	0.021(1)	0.0170(9)	0.0213(9)	0.0013(8)	0.0025(8)	0.0011(7)
C(19)	8*f*	0.91107(5)	0.3605(3)	0.04814(8)	0.0181(9)	0.027(1)	0.023(1)	0.0053(8)	0.0008(8)	0.0033(8)
C(3)	8*f*	0.61044(6)	0.3350(3)	0.77350(9)	0.027(1)	0.038(1)	0.022(1)	–0.0034(9)	0.0034(8)	0.0003(9)
O(3)	8*f*	0.74044(4)	0.6246(2)	0.92420(6)	0.0262(7)	0.0352(8)	0.0236(7)	0.0049(6)	0.0054(6)	0.0092(6)
C(5)	8*f*	0.64315(6)	0.3428(3)	0.68910(9)	0.024(1)	0.033(1)	0.028(1)	–0.0008(9)	0.0059(8)	–0.0013(9)
C(8)	8*f*	0.67883(5)	0.8397(3)	0.85482(8)	0.022(1)	0.0211(9)	0.0219(9)	0.0031(8)	0.0015(8)	–0.0023(8)
C(10)	8*f*	0.71708(5)	0.8293(3)	0.77252(8)	0.022(1)	0.022(1)	0.022(1)	–0.0025(8)	0.0023(8)	0.0032(8)
C(21)	8*f*	0.88657(5)	0.1363(3)	–0.03228(8)	0.024(1)	0.024(1)	0.026(1)	0.0037(8)	0.0041(8)	–0.0018(8)
C(7)	8*f*	0.66003(5)	0.6318(3)	0.76846(8)	0.0185(9)	0.027(1)	0.0182(9)	0.0036(8)	0.0026(7)	0.0024(8)
C(22)	8*f*	0.84802(5)	0.1205(3)	–0.00934(8)	0.021(1)	0.0196(9)	0.022(1)	0.0019(8)	0.0032(8)	0.0014(7)
C(20)	8*f*	0.85508(5)	0.1660(3)	0.05732(8)	0.021(1)	0.0217(9)	0.023(1)	–0.0013(8)	0.0017(8)	0.0033(8)
C(12)	8*f*	0.74591(5)	0.6881(3)	0.87312(8)	0.0182(9)	0.0177(9)	0.0202(9)	–0.0025(8)	0.0006(7)	–0.0028(7)
C(24)	8*f*	0.81977(5)	0.2875(3)	–0.04129(8)	0.0190(9)	0.0168(9)	0.021(1)	–0.0032(8)	0.0018(7)	–0.0019(7)
C(6)	8*f*	0.62100(6)	0.1667(3)	0.66385(9)	0.030(1)	0.034(1)	0.031(1)	0.0010(9)	0.0032(9)	–0.0080(9)
C(16)	8*f*	0.93434(5)	0.5642(3)	0.06670(9)	0.0166(9)	0.026(1)	0.032(1)	0.0039(8)	–0.0040(8)	0.0050(9)
C(15)	8*f*	0.93084(6)	0.6750(3)	0.11915(9)	0.026(1)	0.031(1)	0.038(1)	0.0010(9)	–0.0024(9)	–0.0004(9)
C(4)	8*f*	0.63814(5)	0.4290(3)	0.74397(8)	0.0178(9)	0.0235(9)	0.0203(9)	0.0043(8)	–0.0007(7)	0.0034(8)
C(14)	8*f*	0.95536(6)	0.8497(4)	0.1389(1)	0.034(1)	0.033(1)	0.050(1)	0.005(1)	–0.011(1)	–0.008(1)
C(1)	8*f*	0.59361(5)	0.0772(3)	0.69421(9)	0.022(1)	0.024(1)	0.035(1)	0.0017(9)	–0.0063(8)	–0.0012(9)
C(11)	8*f*	0.73581(6)	1.0911(3)	0.85813(9)	0.031(1)	0.020(1)	0.029(1)	–0.0012(9)	0.0008(9)	0.0001(8)
C(23)	8*f*	0.83089(6)	–0.1130(3)	–0.02128(9)	0.030(1)	0.022(1)	0.029(1)	0.0004(9)	0.0029(9)	0.0010(8)
C(13)	8*f*	0.98307(6)	0.9124(4)	0.1053(1)	0.023(1)	0.026(1)	0.071(2)	–0.0009(9)	–0.014(1)	0.007(1)
C(17)	8*f*	0.96253(6)	0.6316(3)	0.0339(1)	0.023(1)	0.037(1)	0.044(1)	0.0001(9)	0.0040(9)	0.004(1)
C(2)	8*f*	0.58796(6)	0.1602(3)	0.74871(9)	0.025(1)	0.038(1)	0.032(1)	–0.0078(9)	0.0012(9)	0.0058(9)
C(18)	8*f*	0.98707(6)	0.8051(4)	0.0535(1)	0.025(1)	0.040(1)	0.062(2)	–0.005(1)	0.002(1)	0.010(1)

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