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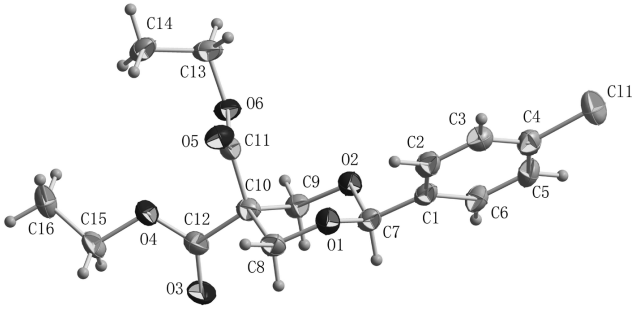
Crystal structure of diethyl 2-(4-chlorophenyl)-1, 3-dioxane-5,5-dicarboxylate, $C_{16}H_{19}ClO_6$



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Abstract

$C_{16}H_{19}ClO_6$, triclinic, $P\bar{1}$ (no. 2), $a = 7.905(3)$ Å, $b = 9.194(4)$ Å, $c = 13.094(5)$ Å, $\alpha = 99.928(4)°$, $\beta = 105.341(4)°$, $\gamma = 109.283(4)°$, $V = 830.2(6)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0343$, $wR_{ref}(F^2) = 0.0898$, $T = 296(2)$K.

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The asymmetric unit 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

Diethyl 2,2-bis(hydroxymethyl)malonate (2.64 g, 12 mmol), 4-chlorobenzaldehyde (1.41 g, 10 mmol) and nanosolid superacid SO_4^{2-}/Fe_2O_3 (0.1 g) were added into a mixture of *N*,*N*-dimethylformamide (12 mL) and cyclohexane (8 mL) and refluxed for 4 h at 378 K with stirring. After workup, the reaction mixture was filtrated and the organic layer was washed with water (10×3 mL) and then dried over Na_2SO_4. Then the white powder (2.43 g, yield 71%) was isolated after filtration

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Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.42 × 0.40 × 0.36 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.26 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7794, 2918, 0.018
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2685
$N(param)_{refined}$:	211
Programs:	Bruker programs [1], SHELX [2, 3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.07422(8)	0.25949(7)	0.94239(4)	0.0747(2)
O1	−0.45979(15)	0.05796(11)	1.25129(8)	0.0441(3)
O2	−0.42244(14)	0.32241(11)	1.26091(8)	0.0403(2)
O3	−0.86012(15)	0.13316(18)	1.38751(11)	0.0709(4)
O4	−0.63109(14)	0.21825(12)	1.55315(8)	0.0450(3)
O5	−0.26490(15)	0.17232(13)	1.50798(9)	0.0507(3)
O6	−0.26856(13)	0.41872(11)	1.52970(8)	0.0431(3)
C1	−0.3431(2)	0.18110(16)	1.12498(11)	0.0400(3)
C2	−0.1998(2)	0.12440(18)	1.14777(12)	0.0459(4)
H2	−0.1881	0.0713	1.2020	0.055*
C3	−0.0735(2)	0.14611(19)	1.09042(13)	0.0495(4)
H3	0.0223	0.1072	1.1055	0.059*
C4	−0.0910(2)	0.22587(18)	1.01089(12)	0.0479(4)
C5	−0.2339(3)	0.2809(2)	0.98523(13)	0.0546(4)
H5	−0.2455	0.3330	0.9304	0.066*
C6	−0.3604(2)	0.2577(2)	1.04230(13)	0.0513(4)
H6	−0.4584	0.2939	1.0252	0.062*
C7	−0.4760(2)	0.16675(16)	1.18987(11)	0.0405(3)
H7	−0.6078	0.1284	1.1393	0.049*
C8	−0.5823(2)	0.03951(17)	1.31508(12)	0.0435(3)
H8A	−0.5649	−0.0344	1.3575	0.052*
H8B	−0.7143	−0.0055	1.2661	0.052*
C9	−0.5454(2)	0.32040(17)	1.32363(12)	0.0398(3)
H9A	−0.6753	0.2874	1.2741	0.048*
H9B	−0.5059	0.4276	1.3714	0.048*
C10	−0.53787(18)	0.20298(16)	1.39407(11)	0.0358(3)
C11	−0.34170(18)	0.25997(16)	1.48304(11)	0.0353(3)
C12	−0.6956(2)	0.18094(17)	1.44417(13)	0.0426(3)
C13	−0.0854(2)	0.4846(2)	1.62007(14)	0.0542(4)
H13A	−0.0288	0.6004	1.6345	0.065*
H13B	0.0004	0.4412	1.5984	0.065*
C14	−0.1074(3)	0.4454(2)	1.72299(14)	0.0669(5)

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
H14A	−0.1483	0.3315	1.7117	0.100*
H14B	−0.2009	0.4798	1.7409	0.100*
H14C	0.0128	0.4999	1.7826	0.100*
C15	−0.7760(3)	0.1840(3)	1.60595(16)	0.0654(5)
H15A	−0.8399	0.0689	1.5921	0.078*
H15B	−0.8709	0.2251	1.5761	0.078*
C16	−0.6793(3)	0.2631(2)	1.72671(16)	0.0700(5)
H16A	−0.6218	0.3774	1.7398	0.105*
H16B	−0.5823	0.2248	1.7550	0.105*
H16C	−0.7712	0.2380	1.7632	0.105*

and evaporation. The product was recrystallized from ethyl acetate to afford colorless crystals.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{eq(C)}$ and the U_{iso} values of all other hydrogen atons were set to $1.2U_{eq(C)}$.

Discussion

1,3-Dioxane compounds of the main group elements are intriguing structurally due to the variety of 1,3-dioxane ring bonding modes which they display [4]. We previously synthesized a series of acetal compounds [5, 6].

A new acetal compound was synthesized and characterized by single-crystal X-ray diffraction [2, 3]. In the 1,3-dioxane ring, the bond lengths of O1–C7 and O2–C7 are 1.4039(17) Å and 1.4257(18) Å, respectively. All geometric parameters are in the expected ranges for this class of compounds [7, 8]

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References

1. Bruker. APEX2, SAINT and SADABS. Brucker AXS Inc., Madison, WI, USA (2009).
2. Sheldrick, G. M.: SHELXS-97: Program for the Solution of Crystal Structures. University of Göttingen, Germany (1997).
3. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
4. Kochhar, K. S.; Bal, B. S.; Deshpande, R. P.; Rajadhyaksha, S. N.; Pinnick, H. W.: Protecting groups in organic synthesis. Part 8. Conversion of aldehydes into geminal diacetates. J. Org. Chem. **48** (1983) 1765–1767.
5. Yuan, L.; Ou, G. C.; Shi, J. H.; Zhang, M.; Yuan, X. Y.: Synthesis and crystal structure of 12-(2-chlorophenyl)-7,11,13,16-tetraoxaspiro[5.2.5.2]cetane. Chin. J. Org. Chem. **33** (2013) 1817–1821.
6. Yuan, L.; Li, Z. Y.; Zhang, X. Y.; Li, W. Y.; Zhang, M.; Yuan, X. Y.: Synthesis, crystal structure and antibacterial activity of 3,3,9,9-tetraphenyl-2,4,8,10-tetraoxaspiro-[5.5]undecane with nanosolid Superacid. J. Bionanosci. **9** (2015) 226–230.
7. Jia, G.-K.; Yuan, L.; Yuan, X.-Y.; Li, Z.-Y.: Crystal structure of 5-ethyl-2-(*p*-tolyl)-1,3-dioxane-5-carboxylic acid, $C_{14}H_{18}O_4$. Z. Kristallogr. NCS **232** (2017) 819–820.
8. Jia, G.-K.; Yuan, L.; Zhang, M.; Yuan, X.-Y.; Liu, F.: Crystal structure of 2-(2-bromophenyl)-5-methyl-1,3-dioxane-5-carboxylic acid, $C_{12}H_{13}BrO_4$. Z. Kristallogr. NCS **231** (2016) 1207–1208.