Crystal structure of 2,2-dimethyl-5-(3,4,5-trimethoxybenzylidene)-1,3-dioxane-4,6-dione, $C_{16}H_{18}O_7$

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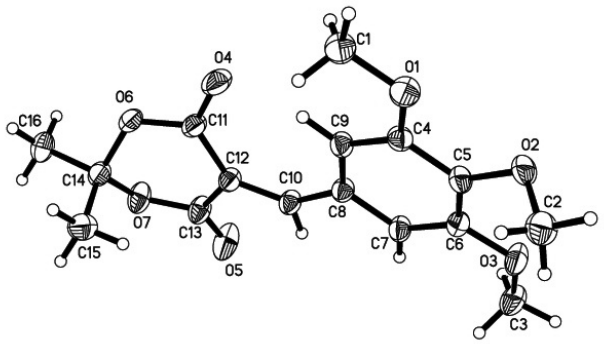

Abstract

$C_{16}H_{18}O_7$, triclinic, $P\bar{1}$ (no. 2), a = 5.334(2) Å, b = 11.674(3) Å, c = 12.510(5) Å, α = 96.41(3)°, β = 94.93(5)°, γ = 92.60(2)°, V = 770.1 Å^3, Z = 2, $R_{gt}(F)$ = 0.0537, $wR_{ref}(F^2)$ = 0.2641, T = 293 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.12×0.18×0.22 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.10 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ and ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7396, 3469
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2243
$N(param)_{refined}$:	208
Programs:	SHELXS-97 [4]

Source of material

A mixture of malonic acid (3.08 g, 0.02 mol) and acetic acid anhydride (3 ml) in strong sulfuric acid (0.25 ml) was stirred with water at 303K. After dissolving, propan-2-one (1.16 g, 0.02 mol) was added dropwise into solution for 1 h. The reaction was allowed to proceed for 2.5 h. The mixture was cooled and filtered, and then an ethanol solution of 3,4,5-trimethoxybenzaldehyde (6.36g,0.02 mol) was added. The solution was then filtered and concentrated. Single crystals were obtained by evaporation of an petroleum ether-acetone (1:1 *v/v*) solution of (I) at room temperature over a period of several days.

Discussion

Meldrum's acid and its derivatives have widely used in organic synthesis owing to their interesting conformational features [1,2]. In the title compound, bond lengths and angles are in agreement with those of the similar structures [3].The dioxane ring is in a distorted envelope conformation with the C14 atom bonded to the two methyl groups forming the flap. In the crystal structure it confirms that the title compound is linked by the 1,3-dioxane ring via a C10=C12 double bond [1.362(3)Å] and the phenyl ring via a C8–C10 single bond [1.458(3)Å], forming the C12–C10–C8 bond angle of 136.7(2)°. The crystal structure is stabilized by weak intermolecular C–H···O hydrogen bonds.

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

Atom	Site	x	y	z	U_{iso}
H(9A)	2*i*	0.3654	0.3365	0.4481	0.049
H(7A)	2*i*	0.8920	0.1403	0.3185	0.052
H(10A)	2*i*	0.8038	0.1119	0.4796	0.050
H(1A)	2*i*	0.0500	0.5616	0.3583	0.083
H(1B)	2*i*	0.2609	0.5232	0.4398	0.083
H(1C)	2*i*	0.0411	0.4349	0.3889	0.083
H(2A)	2*i*	0.7222	0.5168	0.0475	0.095
H(2B)	2*i*	0.9147	0.4458	0.1111	0.095
H(2C)	2*i*	0.7683	0.5414	0.1736	0.095
H(3A)	2*i*	1.1369	0.1283	0.0740	0.089
H(3B)	2*i*	0.9618	0.0685	0.1482	0.089
H(3C)	2*i*	1.1794	0.1575	0.1998	0.089
H(16A)	2*i*	0.3312	0.0978	0.8926	0.099
H(16B)	2*i*	0.3902	0.2256	0.9449	0.099
H(16C)	2*i*	0.5916	0.1334	0.9587	0.099
H(15A)	2*i*	0.8355	0.2967	0.7624	0.092
H(15B)	2*i*	0.9077	0.2600	0.8773	0.092
H(15C)	2*i*	0.7053	0.3514	0.8621	0.092

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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}
O(2)	2*i*	0.5612(3)	0.3993(2)	0.1235(1)	0.055(1)	0.063(1)	0.038(1)	0.0024(8)	–0.0026(7)	0.0204(8)
O(6)	2*i*	0.3629(3)	0.2211(2)	0.7409(2)	0.0409(9)	0.083(1)	0.041(1)	0.0150(8)	0.0138(7)	0.0113(9)
O(1)	2*i*	0.2948(3)	0.4641(2)	0.2886(2)	0.056(1)	0.056(1)	0.054(1)	0.0237(8)	0.0068(8)	0.0166(8)
O(7)	2*i*	0.6645(4)	0.0835(2)	0.7609(2)	0.080(1)	0.053(1)	0.042(1)	0.0203(9)	0.0177(9)	0.0170(8)
O(3)	2*i*	0.8878(4)	0.2295(2)	0.1344(2)	0.071(1)	0.069(1)	0.040(1)	0.0209(9)	0.0201(9)	0.0124(8)
C(9)	2*i*	0.4651(4)	0.3170(2)	0.3921(2)	0.045(1)	0.045(1)	0.034(1)	0.0095(9)	0.0030(8)	0.0076(9)
C(6)	2*i*	0.7561(4)	0.2558(2)	0.2217(2)	0.044(1)	0.048(1)	0.036(1)	0.0042(9)	0.0080(9)	0.0046(9)
C(7)	2*i*	0.7794(4)	0.1985(2)	0.3137(2)	0.049(1)	0.049(1)	0.035(1)	0.014(1)	0.0070(9)	0.0086(9)
C(4)	2*i*	0.4479(4)	0.3757(2)	0.3014(2)	0.040(1)	0.042(1)	0.040(1)	0.0093(9)	0.0012(9)	0.0052(9)
C(5)	2*i*	0.5930(4)	0.3451(2)	0.2154(2)	0.046(1)	0.046(1)	0.034(1)	0.0041(9)	0.0002(9)	0.0091(9)
C(8)	2*i*	0.6314(4)	0.2292(2)	0.3994(2)	0.042(1)	0.041(1)	0.036(1)	0.0078(9)	0.0068(8)	0.0079(9)
C(10)	2*i*	0.6800(4)	0.1643(2)	0.4913(2)	0.046(1)	0.046(1)	0.037(1)	0.0139(9)	0.0087(9)	0.0079(9)
O(4)	2*i*	0.2263(3)	0.2719(2)	0.5852(2)	0.051(1)	0.105(2)	0.061(1)	0.036(1)	0.0195(9)	0.031(1)
C(12)	2*i*	0.5916(4)	0.1602(2)	0.5899(2)	0.043(1)	0.045(1)	0.038(1)	0.0111(9)	0.0077(9)	0.0105(9)
O(5)	2*i*	0.8484(5)	0.0042(2)	0.6232(2)	0.115(2)	0.072(1)	0.057(1)	0.056(1)	0.031(1)	0.026(1)
C(14)	2*i*	0.5688(4)	0.1872(2)	0.8094(2)	0.046(1)	0.055(1)	0.040(1)	0.011(1)	0.0111(9)	0.008(1)
C(13)	2*i*	0.7138(5)	0.0786(2)	0.6561(2)	0.064(2)	0.048(1)	0.042(1)	0.018(1)	0.016(1)	0.014(1)
C(11)	2*i*	0.3843(4)	0.2238(2)	0.6345(2)	0.040(1)	0.057(1)	0.044(1)	0.013(1)	0.0113(9)	0.010(1)
C(1)	2*i*	0.1499(5)	0.4988(2)	0.3760(2)	0.059(2)	0.055(1)	0.054(2)	0.025(1)	0.005(1)	0.004(1)
C(2)	2*i*	0.7575(5)	0.4826(3)	0.1131(3)	0.064(2)	0.069(2)	0.060(2)	–0.002(1)	0.003(1)	0.028(1)
C(3)	2*i*	1.0550(5)	0.1386(2)	0.1395(3)	0.065(2)	0.063(2)	0.054(2)	0.016(1)	0.024(1)	0.002(1)
C(16)	2*i*	0.4607(6)	0.1584(3)	0.9105(2)	0.071(2)	0.092(2)	0.040(2)	0.002(2)	0.018(1)	0.015(1)
C(15)	2*i*	0.7728(5)	0.2824(3)	0.8296(3)	0.051(1)	0.066(2)	0.066(2)	0.000(1)	0.007(1)	0.003(1)

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