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Crystal structure of diisopropyl 2,5-dihydroxycyclohexa-1,4-diene-1,4-dicarboxylate, $C_{14}H_{20}O_6$

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Abstract

$C_{14}H_{20}O_6$, triclinic, $P\bar{1}$ (no. 2), $a = 6.4270(13)$ Å, $b = 8.3458(17)$ Å, $c = 8.4120(17)$ Å, $\alpha = 107.08(3)^\circ$, $\beta = 106.43(3)^\circ$, $\gamma = 107.37(3)^\circ$, $V = 376.2(2)$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.0521$, $wR_{\text{ref}}(F^2) = 0.1541$, $T = 294.0$ K.

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The molecular structure is shown in the figure (only the asymmetric unit is labelled). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of isopropyl 2-bromo-3-oxobutanoate (0.26 mol) was mixed in anhydrous ethanol (100 mL) and added 0.3 mol K_2CO_3 in batches under ice bath conditions and stirred in

Table 1: Data collection and handling.

Crystal:	White block
Size:	$0.32 \times 0.22 \times 0.18$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	Enraf-Nonius CAD-4, ω
θ_{max} , completeness:	25.0°, 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	1764, 1305, 0.028
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1039
$N(\text{param})_{\text{refined}}$:	115
Programs:	CAD-4 [1, 2], Olex2 [3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.1202(2)	0.47484(13)	0.75546(15)	0.0549(4)
O2	0.3775(2)	0.48955(15)	0.62176(19)	0.0711(5)
O3	0.3800(2)	0.17846(17)	0.43275(19)	0.0632(5)
C1	0.4379(5)	0.7394(3)	1.0179(4)	0.0907(8)
C2	0.0258(4)	0.7218(3)	0.8817(3)	0.0745(6)
H2a	0.0817(4)	0.8531(3)	0.9422(3)	0.1118(9)*
H2b	−0.1073(4)	0.6760(3)	0.7678(3)	0.1118(9)*
H2c	−0.0230(4)	0.6652(3)	0.9569(3)	0.1118(9)*
C3	0.2230(3)	0.6768(2)	0.8475(2)	0.0581(5)
H3a	0.2695(3)	0.7307(2)	0.7672(2)	0.0697(6)*
C4	0.2133(3)	0.3986(2)	0.6485(2)	0.0487(4)
C5	0.1035(2)	0.1968(2)	0.5705(2)	0.0445(4)
C6	0.1951(3)	0.1012(2)	0.4713(2)	0.0461(4)
C7	−0.1006(3)	0.1013(2)	0.6074(3)	0.0536(5)
H7a	−0.053(4)	0.144(3)	0.740(3)	0.094(7)*
H7b	−0.230(5)	0.138(4)	0.565(4)	0.111(8)*
H3	0.427(5)	0.306(4)	0.486(4)	0.120(9)*
H1a	0.500(4)	0.878(3)	1.089(3)	0.088(6)*
H1b	0.570(6)	0.702(4)	0.980(4)	0.139(10)*
H1c	0.384(5)	0.684(5)	1.090(4)	0.128(12)*

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refluxing for 7 h. The mixture after the reaction was filtered, washed with distilled water, and finally white crystals were obtained from ethanol.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Comment

As an unstable compound, the synthesis of enols has been paid much attention in the field of organic chemistry [4–6]. β -Diketones are involved in almost all monographs on coordination chemistry [7]. Because the methylene hydrogen in the β -diketone-derived molecule is affected by two carbonyl groups, the compound is composed of keto – type and enol – type interchangeable isomers. These molecules and metal ions can form compounds with peculiar structures through a variety of coordination methods [8, 9]. As a result, attention has been paid to the investigations on the structure and property of the new β -diketone-derived ligands [10, 11]. The title compound is an intermediate for the synthesis of β -diketone derivatives. According to previous reports [12, 13], we synthesized the title compound.

The molecular structure of the title compound is shown in the figure, bond lengths and angles are within normal ranges. The title molecule is arranged around an inversion center (see the Figure). The length of the C5–C6 bond is 1.359 Å, indicating a C=C double bond.

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