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Crystal structure of dipentyl 2,5-dihydroxycyclohexa-1,4-diene-1,4-dicarboxylate, $C_{18}H_{28}O_6$

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Abstract

$C_{18}H_{28}O_6$, monoclinic, $C2/c$ (no. 15), $a = 15.061(3)$ Å, $b = 13.791(3)$ Å, $c = 10.106(2)$ Å, $\beta = 118.60(3)^\circ$, $V = 1843.0(8)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0462$, $wR_{ref}(F^2) = 0.1315$, $T = 294.3$ K.

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The molecular structure is shown in the figure. 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 pentyl 2-bromo-3-oxobutanoate (0.26 mol) was mixed in anhydrous ethanol (100 mL) and 0.3 mol K_2CO_3 was added in batches under ice bath conditions and finally stirred under reflux for 6 h. The reaction mixture was filtered, washed with distilled water, and crystals of the title compound were obtained from ethanol.

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Table 1: Data collection and handling.

Crystal:	White block
Size:	$0.30 \times 0.25 \times 0.20$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.09 mm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD-4, ω
$\theta_{\max}$, completeness:	25.0° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4221, 1614, 0.025
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1214
$N(\text{param})_{\text{refined}}$:	113
Programs:	CAD-4 [1, 2], Olex2 [3]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.08761(9)	0.23562(9)	0.67948(13)	0.0601(4)
O2	0.16671(10)	0.12529(10)	0.86304(14)	0.0677(4)
O3	0.13287(10)	−0.05604(10)	0.79060(14)	0.0656(4)
C1	0.17126(19)	0.67624(16)	0.8382(3)	0.0860(7)
H1a	0.15139(19)	0.73588(16)	0.7825(3)	0.1290(11)*
H1b	0.14536(19)	0.67439(16)	0.9085(3)	0.1290(11)*
H1c	0.24371(19)	0.67216(16)	0.8921(3)	0.1290(11)*
C2	0.12899(17)	0.59120(14)	0.7299(2)	0.0685(6)
H2a	0.05589(17)	0.59652(14)	0.6744(2)	0.0821(7)*
H2b	0.15425(17)	0.59429(14)	0.6579(2)	0.0821(7)*
C3	0.15730(14)	0.49385(13)	0.8099(2)	0.0593(5)
H3a	0.23041(14)	0.48811(13)	0.8628(2)	0.0711(6)*
H3b	0.13434(14)	0.49221(13)	0.8846(2)	0.0711(6)*
C4	0.11323(15)	0.40696(13)	0.7056(2)	0.0604(5)
H4a	0.04019(15)	0.41313(13)	0.6505(2)	0.0724(6)*
H4b	0.13793(15)	0.40691(13)	0.6328(2)	0.0724(6)*
C5	0.14036(15)	0.31220(14)	0.7894(2)	0.0639(6)
H5a	0.11961(15)	0.31235(14)	0.8667(2)	0.0766(7)*
H5b	0.21286(15)	0.30189(14)	0.8374(2)	0.0766(7)*
C6	0.10769(13)	0.14440(13)	0.7301(2)	0.0521(5)
C7	0.05367(12)	0.07172(12)	0.61396(18)	0.0480(5)
C8	−0.01906(14)	0.10472(11)	0.45631(19)	0.0539(5)
H8a	−0.07061(14)	0.14504(11)	0.45998(19)	0.0647(6)*
H8b	0.01728(14)	0.14434(11)	0.41876(19)	0.0647(6)*
C9	0.06930(13)	−0.02349(13)	0.65126(18)	0.0496(5)
H3	0.1618(18)	−0.004(2)	0.847(3)	0.099(8)*

Experimental details

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

Comment

As an excellent oxygen-containing class of ligands, β -diketones have been a research hotspot in modern coordination chemistry, crystal engineering and material science [4–6]. The complexes composed of these ligands have certain application value in light, fluorescence device materials and so on [7–9]. Therefore, it is of great significance to study the effective method of synthesizing β -diketone-derived ligands.

The crystal structure of the title compound has not been previously reported. The asymmetric unit contains one half of a title molecule, located around an inversion center (see the figure). The single crystal structure verifies that all bond lengths and bond angles are within the normal range [10]. Two intramolecular OH...O hydrogen bonds are present.

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References

1. Enraf-Nonius: CAD-4 Software. Version 5.0. Enraf-Nonius, Delft, The Netherlands (1985).
2. Harms, K.; Wocadlo, S.: XCAD4. University of Marburg, Germany (1995).
3. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.
4. Aromí, G.; Gamez, P.; Reedijk, J.: Poly beta-diketones: prime ligands to generate supramolecular metallocusters. *Coord. Chem. Rev.* **252** (2008) 964–989.
5. Burke, A. N.: Treatment of neuropathic pain with 6*H*-pyrrolo [3,4-*d*]pyridazine compounds. WO2004006836(2004) 1–22.
6. Johnson, R. E.: 1-(Arylamino)pyrroles. US4163015(1979) 4–5.
7. Zhu, W. G.; Jiang, Q.; Lu, Z. Y.; Wei, X.; Xie, M.; Zou, D.; Tsutsui, T.: Novel binuclear europium β -diketone chelate used as red emitter in organic electroluminescent device. *Thin Solid Films* **363** (2000) 167–169.
8. Ma, D. Z.; Wu, Y. Q.; Zuo, X.: Rare earth doped β -diketone complexes as promising high-density optical recording materials for blue optoelectronics. *Mater. Lett.* **59** (2005) 3678–3681.
9. Poopathy, K.; Richard, P.: Electroluminescent materials and devices. EP1578756(2007) 6–13.
10. Mez, H.-C.; Rihs, G.: Chemistry of succinylsuccinic acid derivatives. part II. the crystal and molecular structure of diethyl succinylsuccinate. *Helv. Chim. Acta* **56** (1973) 2766–2772.