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The crystal structure of 4,10-diethoxy-6*H*,12*H*-6,12-epoxydibenzo[*b,f*][1,5]dioxocine, C₁₈H₁₈O₅

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.21 × 0.20 × 0.19 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffraction, scan mode:	Bruker,
$\theta_{\max}$, completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	14441, 3441, 0.039
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2032
$N(\text{param})_{\text{refined}}$:	208
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

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	0.47024(12)	0.66496(12)	0.75818(9)	0.0498(4)
O2	0.22425(12)	0.77132(11)	0.71009(8)	0.0450(3)
O4	−0.00790(13)	0.79578(10)	0.45279(9)	0.0500(4)
O5	−0.16820(14)	0.63813(10)	0.35135(9)	0.0499(4)
C1	0.6419(2)	0.5931(2)	0.89907(17)	0.0703(6)
H1A	0.731364	0.556087	0.918259	0.105*
H1B	0.646783	0.665328	0.932411	0.105*
H1C	0.571501	0.547490	0.919517	0.105*
C2	0.6045(2)	0.60943(19)	0.78539(16)	0.0571(5)
H2A	0.675457	0.655057	0.764068	0.068*
H2B	0.599923	0.536825	0.751131	0.068*
C3	0.42183(18)	0.69282(15)	0.65734(13)	0.0430(4)
C4	0.4875(2)	0.66558(18)	0.57871(15)	0.0537(5)
H4	0.571212	0.625068	0.592952	0.064*
C5	0.4286(2)	0.69866(19)	0.47823(16)	0.0602(6)
H5	0.471925	0.677826	0.425735	0.072*
C6	0.3079(2)	0.76129(18)	0.45594(14)	0.0541(5)
H6	0.271733	0.785724	0.389227	0.065*
C7	0.23956(19)	0.78823(15)	0.53361(13)	0.0437(4)
C8	0.29397(18)	0.75171(15)	0.63288(12)	0.0403(4)
C9	0.1071(2)	0.85819(15)	0.51168(14)	0.0495(5)
H9	0.123252	0.924383	0.471753	0.059*
C10	0.08058(18)	0.80300(15)	0.67050(14)	0.0457(4)
H10	0.040338	0.826132	0.728251	0.055*
C11	−0.00760(18)	0.70818(15)	0.61564(13)	0.0412(4)
C12	−0.05493(18)	0.62095(16)	0.66958(14)	0.0470(5)
H12	−0.028647	0.618714	0.740763	0.056*
C13	−0.14045(19)	0.53847(16)	0.61704(15)	0.0498(5)
H13	−0.171393	0.480219	0.653185	0.060*
C14	−0.18165(19)	0.54055(15)	0.51058(14)	0.0469(5)
H14	−0.239469	0.483827	0.476255	0.056*

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Abstract

C₁₈H₁₈O₅, monoclinic, $P2_1/c$ (no. 14), $a = 9.762(2)$ Å, $b = 11.832(2)$ Å, $c = 13.376(3)$ Å, $\beta = 102.81(3)^\circ$, $V = 1506.5(6)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0417$, $wR_{\text{ref}}(F^2) = 0.1380$, $T = 293(2)$ 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

0.1662 g 3-Ethoxysalicylaldehyde (1.0 mmol) and 0.1261 g furan-2-carbohydrazide (1.0 mmol) were dissolved in water-ethanol ($v/v = 2:3$) solution with stirring. After 0.5 h, 0.1195 g

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C15	−0.13678(18)	0.62685(14)	0.45560(13)	0.0416(4)
C16	−0.04902(17)	0.71074(14)	0.50948(13)	0.0401(4)
C17	−0.2611(2)	0.55634(17)	0.29346(15)	0.0517(5)
H17A	−0.352965	0.561521	0.309600	0.062*
H17B	−0.224724	0.480748	0.310232	0.062*
C18	−0.2723(2)	0.5797(2)	0.18205(15)	0.0660(6)
H18A	−0.334334	0.525563	0.141815	0.099*
H18B	−0.308798	0.654455	0.166098	0.099*
H18C	−0.181033	0.573982	0.166728	0.099*
O3	0.07534(14)	0.89692(11)	0.60408(10)	0.0529(4)

calcium perchlorate hydrate (0.5 mmol) was added. The mixture was stirred for 8 h at 70 °C. Then the reactant was filtered. The block crystals of 4,10-diethoxy-6*H*,12*H*-6,12-epoxydibenzo[*b,f*][1,5]dioxocine were obtained by slow evaporation of the filtrate in 25 days.

Experimental details

The hydrogen atoms were positioned geometrically (C–H = 0.93–0.97 Å). Their *U*_{iso} values were set to 1.2*U*_{eq} or 1.5*U*_{eq} of the parent atoms.

Comment

Design, synthesis and characterization of new oxygen containing compound have been received interesting, because they may coordinate metal ions [5, 6]. At the same time, they also have good applications in the synthesis of new bioactive molecules and drugs [7].

In the title compound, the bond lengths of C–O are in the range of 1.366(2) Å and 1.439(2) Å, which indicates that all the C–O bonds are single bonds similar to related ones [8, 9]. Some angles between mean planes may be of interest: 10.5° (plane 1: C3–C4–C5–C6–C7–C8 and plane 2: C7–C8–O2–C10–C9), 106.5° (plane 2: C7–C8–O2–C10–C9 and plane 3: C9–C10–C11–C16–O4) and 6.1° (plane 3: C9–C10–C11–C16–O4 and plane 4: C11–C12–C13–C14–C15–C16),

respectively, which show that the plane 1 and plane 2, plane 3 and plane 4 are approximately coplanar, whereas the whole molecule is not coplanar. The title molecules form a three-dimensional framework by π – π stacking interactions.

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