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# 5,6-[2α-Acetyl-1β-(ethoxycarbonyl)ethylidene]-1,3,3a,4,5,6,7,7a-octahydro-4,7-epoxyisobenzofuran-1,3-dione

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**Key indicators**

Single-crystal X-ray study
*T* = 150 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.043
*wR* factor = 0.119
Data-to-parameter ratio = 16.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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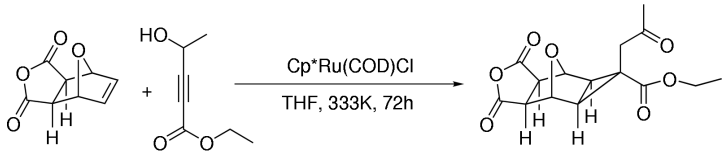
The regio- and relative stereochemistry of the title compound, $C_{15}H_{16}O_7$, has been established by X-ray analysis. The molecular dimensions are normal. Molecules form centrosymmetric hydrogen-bonded dimers through two independent weak C—H···O interactions.

## Comment

Recently, we studied the ruthenium-catalyzed [2 + 2]-cycloadditions between bicyclic alkenes and propargyl alcohols (Villeneuve *et al.*, 2003). When the bicylic alkene (I) (see scheme) was used as the bicyclic alkene component and propargylic alcohol (II) was used as the alkyne component, the product obtained was not the expected [2 + 2] cyclobutene cycloadduct. An unknown product was formed instead and its structure was not determined by NMR experiments. The structure of the title compound, (III), was established by the present single-crystal X-ray diffraction analysis.




(I) (II) (III)

A view of (III) is shown in Fig. 1. Molecules form centrosymmetric hydrogen-bonded dimers through two independent weak C—H···O interactions (see Fig. 2). The graph-set descriptor (Bernstein *et al.*, 1995) for the C3—H3*A*···O5$^{i}$ [symmetry code: (i) $1-x$, $1-y$, $1-z$] interaction gives $R_2^2(12)$ rings and the C4—H4*A*···O7$^{i}$ interaction gives $R_2^2(16)$

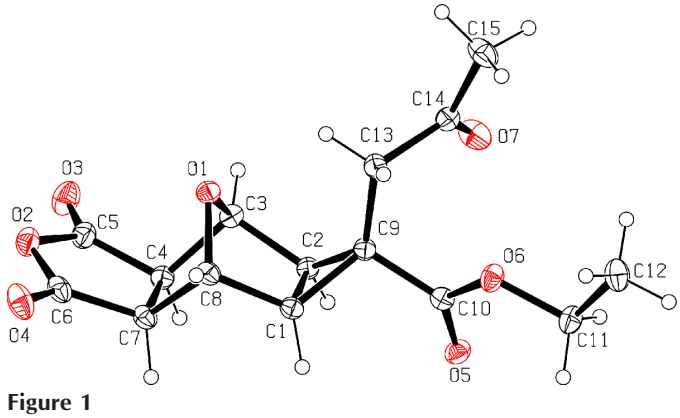



**Figure 1**
View of the molecule of (III), showing the crystallographic labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented by small spheres.

rings. Two other rings are formed [$R_2^2(10)$ and $R_2^2(14)$] using combinations of both interactions; see Table 1 for the hydrogen-bonding geometries.

## Experimental

Addition of *exo*-3,6-epoxy-1,2,3,6-tetrahydrophthalic anhydride (142.3 mg, 0.8566 mmol), (I), and ethyl 4-hydroxy-2-pentynoate (60.7 mg, 0.427 mmol), (II), to Cp*RuCl(COD) (4.6 mg, 0.012 mmol) in THF (1 ml) at 333 K followed by column chromatography with pentane/diethyl ether provided the product (III). Suitable crystals were grown from 40% pentane/diethyl ether.

### *Crystal data*

$C_{15}H_{16}O_7$
$M_r$ = 308.28
Monoclinic, $P2_1/n$
$a$ = 11.2150 (6) Å
$b$ = 12.2282 (3) Å
$c$ = 11.5439 (5) Å
$\beta$ = 115.7530 (19)°
$V$ = 1425.88 (10) Å$^3$
$Z$ = 4
$D_x$ = 1.436 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 5791 reflections
$\theta$ = 2.6–27.5°
$\mu$ = 0.12 mm$^{-1}$
$T$ = 150 (1) K
Slab, colourless
0.44 × 0.20 × 0.12 mm

### *Data collection*

Nonius KappaCCD diffractometer
$\varphi$ scans and $\omega$ scans with $\kappa$ offsets
Absorption correction: none
11434 measured reflections
3262 independent reflections
2381 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.048
$\theta_{max}$ = 27.5°
$h = -14 \rightarrow 13$
$k = -15 \rightarrow 15$
$l = -14 \rightarrow 15$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.119
$S$ = 1.01
3262 reflections
202 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0613P)^2 + 0.3395P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.23 e Å$^{-3}$
Extinction correction: *SHELXL*97
Extinction coefficient: 0.010 (2)

**Table 1**
Hydrogen-bonding geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C3—H3$A$···O5$^{i}$ | 0.98 | 2.51 | 3.3547 (19) | 145 |
| C4—H4$A$···O7$^{i}$ | 0.98 | 2.29 | 3.1659 (19) | 148 |

Symmetry code: (i) $1-x, 1-y, 1-z$.

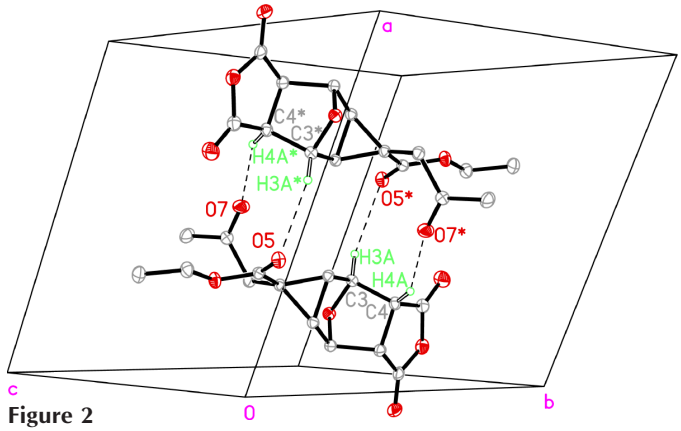



**Figure 2**
View of the hydrogen-bonded dimer of (III), showing weak intermolecular C—H···O interactions as dashed lines. O atoms are shown in red and H atoms are green. Only the H atoms involved in the hydrogen bonds are shown. Displacement ellipsoids are drawn at the 30% probability level. The suffix * in atom labels corresponds to symmetry code (i) in Table 1.

All H atoms were placed in calculated positions, with C—H distances ranging from 0.96 to 0.98 Å and included in the refinement in a riding-model approximation with $U_{iso}$ = 1.2$U_{eq}$ (1.5$U_{eq}$ for methyl) of the carrier atom.

Data collection: *COLLECT* (Nonius, 1997–2002); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2004).

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