Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl 7-*exo*-acetoxy-4-phenyltricyclo-[4.2.1.0^{2,5}]non-3-ene-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.007 Å
R factor = 0.051
wR factor = 0.142
Data-to-parameter ratio = 7.5

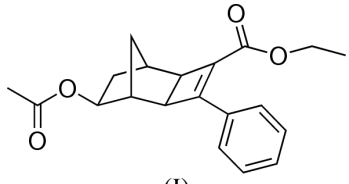
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 2 October 2003
Accepted 6 October 2003
Online 15 October 2003

The regio- and stereochemistry of the title compound, $C_{20}H_{22}O_4$, has been established by X-ray analysis. The bond lengths and angles are normal.

Comment

Recently, we studied the remote substituent effects on ruthenium-catalysed [2 + 2]-cycloaddition reactions between 2-substituted 5-norbornenes and unsymmetrically substituted alkynes. Four different regio- and stereoisomers could be formed in the cycloadditions. When the substituent of the norbornene is an *exo*-OAc group, two regioisomers were obtained in a ratio of 4:1. These regioisomers were separated by fractional recrystallization. The regio- and stereochemistry of the major isomer, (I), was established by a single-crystal X-ray diffraction analysis.



(I)

Experimental

Addition of *exo*-2-acetoxy-5-norbornene and ethyl 3-phenylpropiolate to Cp*RuCl(COD) in THF at 298 K provided two regioisomers in a ratio of 4:1. Fractional recrystallization in hexanes provided the major regioisomer (I). Suitable crystals were grown from hexanes.

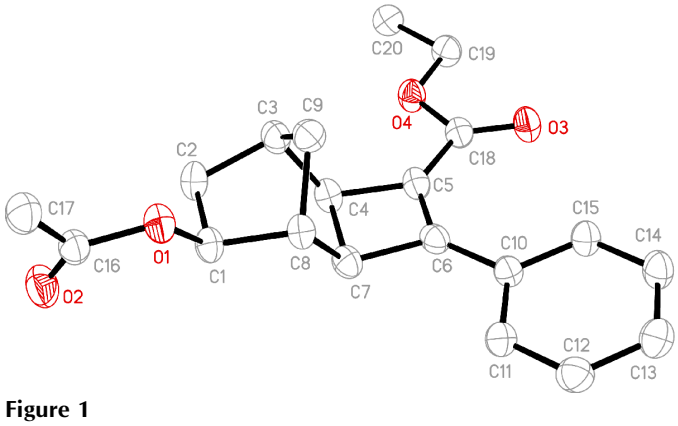


Figure 1
View of the molecule of (I), with the crystallographic labelling scheme. Displacement ellipsoids are at the 30% probability level and H atoms have been omitted.

Crystal data

$C_{20}H_{22}O_4$	D_x = 1.295 Mg m^{-3}
M_r = 326.38	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 18043 reflections
a = 9.7510 (14) Å	θ = 2.6–25.0°
b = 5.5920 (9) Å	μ = 0.09 mm^{-1}
c = 15.3550 (18) Å	T = 150 (1) K
β = 90.934 (10)°	Needle, colourless
V = 837.2 (2) Å^3	0.14 × 0.10 × 0.05 mm
Z = 2	

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.080
φ scans and ω scans with κ offsets	θ_{max} = 25.0°
Absorption correction: none	$h = -11 \rightarrow 11$
5502 measured reflections	$k = -6 \rightarrow 6$
1647 independent reflections	$l = -18 \rightarrow 18$
1182 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0749P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.051	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.142	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
S = 1.05	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
1647 reflections	Extinction correction: *SHELXTL*
220 parameters	Extinction coefficient: 0.058 (11)
H-atom parameters constrained	

All H atoms were placed in calculated positions, with C—H distances ranging from 0.98 to 1.00 Å, and included in the refinement in a riding-motion approximation, with $U_{iso} = 1.2U_{eq}$ ($1.5U_{eq}$ for methyl H atoms) of the carrier atom. In the absence of significant anomalous dispersion effects, Friedel pairs were merged.

Data collection: *COLLECT* (Nonius, 1997–2002); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2001); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors acknowledge NSERC Canada and the University of Toronto for funding.

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