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Ethyl 7-oxo-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) = 0.002 Å
R factor = 0.041
wR factor = 0.113
Data-to-parameter ratio = 17.8

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

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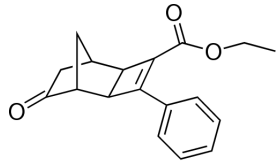
The regio- and stereochemistry of the title compound, $C_{18}H_{18}O_3$, has been established by X-ray analysis. The bond lengths and angles are normal.

Comment

Recently, we studied the remote substituent effects on ruthenium-catalysed [2 + 2]-cycloaddition reactions between 2-substituted 5-norbornenes and unsymmetrically substituted alkynes. Two different regio- and stereoisomers could be formed in the cycloadditions. When the substituent of the norbornene is a ketone group, two regioisomers were obtained in a ratio of 7.5:1. These regioisomers were separated by fractional recrystallization. The regio- and stereochemistry of the major isomer was established by a single-crystal X-ray diffraction analysis. The structure of ethyl 7-oxo-4-phenyltricyclo[4.2.1.$0^{2,5}$]non-3-ene-3-carboxylate, (I), is reported.



(I)

Experimental

Addition of 2-norbornenone and ethyl 3-phenylpropiolate to Cp*RuCl(COD) in THF at 298 K provided two regioisomers in a ratio of 7.5:1. Fractional recrystallization in an ethyl acetate/hexanes (1:19) mixture provided the major regioisomer (I). Suitable crystals were grown from an ethyl acetate/hexanes (1:19) mixture.

Crystal data

$C_{18}H_{18}O_3$
M_r = 282.32
Orthorhombic, *Pccn*
a = 16.2196 (4) Å
b = 18.2187 (4) Å
c = 10.0604 (2) Å
V = 2972.85 (11) Å^3
Z = 8
D_x = 1.262 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 16712 reflections
θ = 2.6–27.5°
μ = 0.09 mm^{-1}
T = 150 (1) K
Block, colourless
0.34 × 0.32 × 0.30 mm

Data collection

Nonius KappaCCD diffractometer
φ scans and ω scans with κ offsets
Absorption correction: none
27619 measured reflections
3398 independent reflections
2591 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 27.5°
$h = -20 \rightarrow 21$
$k = -23 \rightarrow 22$
$l = -11 \rightarrow 13$

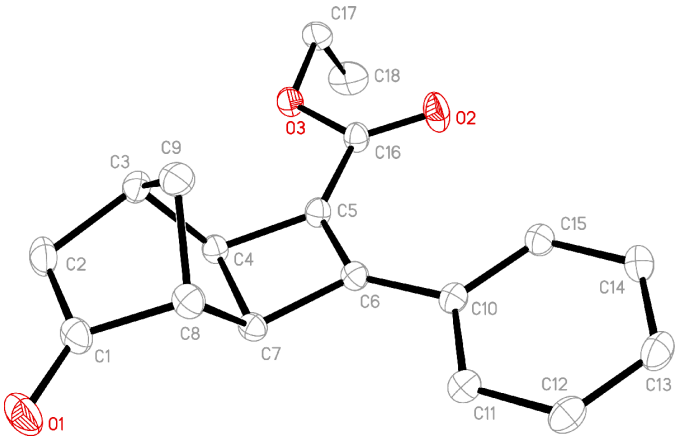


Figure 1
View of the molecule of (I), with the crystallographic labelling scheme. Displacement ellipsoids are at the 30% probability level and H atoms have been omitted.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.113$
$S = 1.03$
3398 reflections
191 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.8216P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0048 (13)

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-model approximation, with $U_{iso} = 1.2U_{eq}$ ($1.5U_{eq}$ for methyl H atoms) 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: *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*.

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