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3-Ethoxy-7-methoxycarbonyl-4-phenyltricyclo[4.2.1.$0^{2,5}$]non-3-ene

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.043
wR factor = 0.120
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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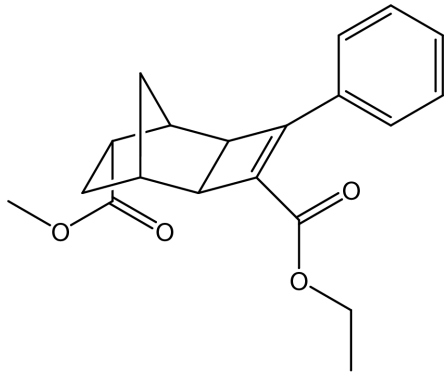
The regio- and stereochemistry of the title compound, $C_{20}H_{22}O_4$, has been established by X-ray analysis. The molecular dimensions are normal.

Comment

Recently, we reported the first examples of remote substituent effects on ruthenium-catalyzed [2+2] cycloaddition reactions between 2-substituted 5-norbornenes and unsymmetrically substituted alkynes (Jordan & Tam, 2000). Four different regio- and stereoisomers could be formed in the cycloadditions. When the substituent of the norbornene was an *endo* COOMe group, two regioisomers were obtained in a ratio of 2:1. These regioisomers were separated by fractional recrystallization. The regio- and stereochemistry of the major isomer, (I), was established by single-crystal X-ray diffraction analysis.



(I)

Experimental

Addition of *endo*-2-methoxycarbonyl-5-norbornene and ethyl 3-phenylpropiolate to Cp*RuCl(COD) (COD is 1,4-cyclooctadiene) in thf at 298 K yielded two regioisomers in a ratio of 2:1. Fractional recrystallization in an ethyl acetate/hexanes (1:4) mixture afforded the major regioisomer, (I). Suitable crystals were grown from an ethyl acetate/hexanes (1:4) mixture.

Crystal data

$C_{20}H_{22}O_4$
M_r = 326.38
Monoclinic, $P2_1/c$
a = 12.842 (8) Å
b = 9.831 (8) Å
c = 18.063 (6) Å
β = 131.44 (2)°
V = 1709.5 (18) Å^3
Z = 4
D_x = 1.268 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9.4–12.1°
μ = 0.09 mm^{-1}
T = 298 (2) K
Block, colourless
0.4 × 0.3 × 0.2 mm

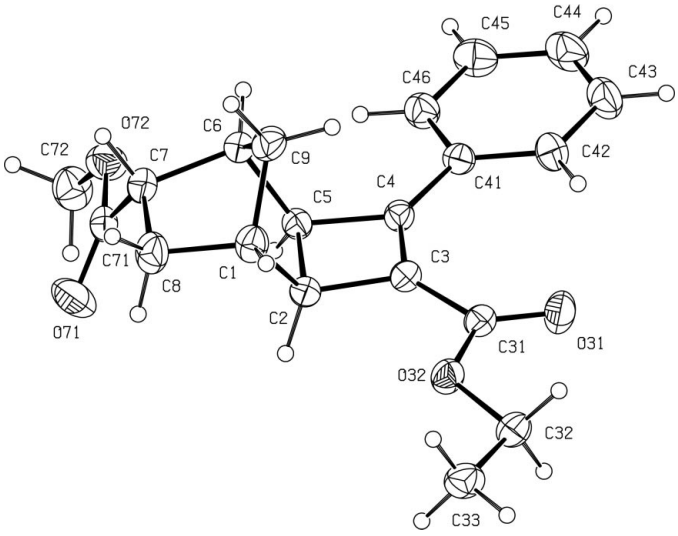


Figure 1
A view of (I). Displacement ellipsoids are drawn at the 30% probability level.

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
4180 measured reflections
3909 independent reflections
2433 reflections with $I > 2\sigma(I)$
$R_{int} = 0.010$
$\theta_{max} = 27.5^\circ$
$h = -16 \rightarrow 16$
$k = -12 \rightarrow 0$
$l = -23 \rightarrow 15$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.121$
$S = 1.00$
3909 reflections
220 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0662P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0112 (16)

The H atoms were all located in difference maps and were refined as riding atoms (C—H 0.93–0.98 Å). The methyl H atoms on C72 were obviously disordered and these were modelled as six half-occupancy H atoms spaced at 60° intervals. The methyl H atoms on C33 were not disordered and were allowed to rotate freely around the C—C bond.

Data collection: *CAD*-4-*PC Software* (Enraf–Nonius, 1992); cell refinement: *CAD*-4-*PC Software*; data reduction: *HELENA*/*PLATON*/*System S* (Spek, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON*; software used to prepare material for publication: *SHELXL*97.

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