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(1*RS*,2*RS*,2′*SR*,4*SR*)-2′-Methoxy-2′-phenylspiro[bicyclo[2.2.1]heptane-2,1′-cyclopropan]-3-one

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The title compound, $C_{16}H_{18}O_2$, an acylcylopropane, has no unusual bonding features. In the crystal structure, dimers are formed *via* a weak C—H···O hydrogen-bonding interaction involving a C—H of the phenyl ring and a carbonyl group of a neighbouring molecule.

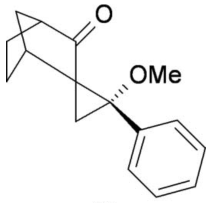
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.120
Data-to-parameter ratio = 14.3

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

Comment

The cyclopropanation reaction between Fischer carbene complexes and electron-deficient olefins has been widely studied. The carbene ligand from the complex has been transferred to the C═C double bond of alkenyl esters (Fischer & Dötz, 1970), amides (Wienand & Reissig, 1988), nitriles (Barluenga *et al.*, 2006), phosphonates and other electron-withdrawing groups, through a [2+1] cycloaddition process. The resulting cyclopropanes are considered valuable versatile building blocks in organic synthesis because of the ring strain [see Wong *et al.* (1989) for a review] and the fact that these are captodative (donor–aceptor) cyclopropanes [see Reissig & Zimmer (2003) for a review]. Recent work in our research group has demostrated that Fischer carbene complexes can also react with enones and enals to yield dihydrofurans. Exceptionally, reactions involving 3-methylene-2-norbornanone resulted in the formation of the corresponding acylcyclopropanes. The structural characterization of the title such compound, (I), is reported here.



(I)

The molecuar structure of compound (I) is shown in Fig. 1. There are no unusual bonding features.

In the crystal structure of (I), dimers are formed *via* a weak C—H···O hydrogen-bonding interaction. This involves C13—H13 and O1 of a neighbouring molecule at (1 + x, y, 1 + z) (Table 1).

Experimental

Pentacarbonyl[methoxy(phenyl)methylidene]chromium(0) (1 mmol, 312 mg) and 3-methylene-2-norbornanone (3 mmol, 366 mg) were added to a sealed flask with tetrahydrofuran (20 ml) under an N_2 atmosphere. The solution was stirred and heated at 373 K for 4 h in an oil bath. The disappearance of the red colour of the complex carbene was observed. The solution was allowed to reach room temperature and the solvent was removed *in vacuo*. The residue was dissolved in ethyl acetate (5 ml) and then diluted with hexanes (80 ml). The mixture was left in direct sunlight for 5 h to allow the metal complex to dissociate. The green precipitate (chromium species) was filtered over Celite, and the solvents were removed *in vacuo* to afford a mixture of diastereoisomers (yellow oil) that were separated by column chromatography (silica gel, hexanes–AcOEt, 3:1 *v*/*v*). Pure diastereoisomeric compounds were obtained in 41% and 37% yield. The minor diastereoisomer, (I), was successfully recrystallized as a white solid from a solution in a mixture of pentane and CH_2Cl_2 (20:1) at 243 K.

Crystal data

$C_{16}H_{18}O_2$
$M_r = 242.32$
Monoclinic, $P2_1/c$
$a = 9.4822$ (1) Å
$b = 20.6081$ (3) Å
$c = 6.9712$ (1) Å
$\beta = 109.246$ (1)°
$V = 1286.11$ (3) Å^3
$Z = 4$
Cu $K\alpha$ radiation
$\mu = 0.64$ mm^{-1}
$T = 293$ (2) K
0.37 × 0.37 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: part of the refinement model (ΔF) (*XABS2*; Parkin *et al.*, 1995)
$T_{\min} = 0.791$, $T_{\max} = 0.878$
17858 measured reflections
2350 independent reflections
2126 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.120$
$S = 1.03$
2350 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···O1$^{\rm i}$	0.93	2.79	3.416 (2)	126 (1)

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

The H atoms were included in calculated positions and treated as riding, with C—H = 0.93–0.98 Å and $U_{\rm iso}$(H) = 1.2 or 1.5$U_{\rm eq}$(C).

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

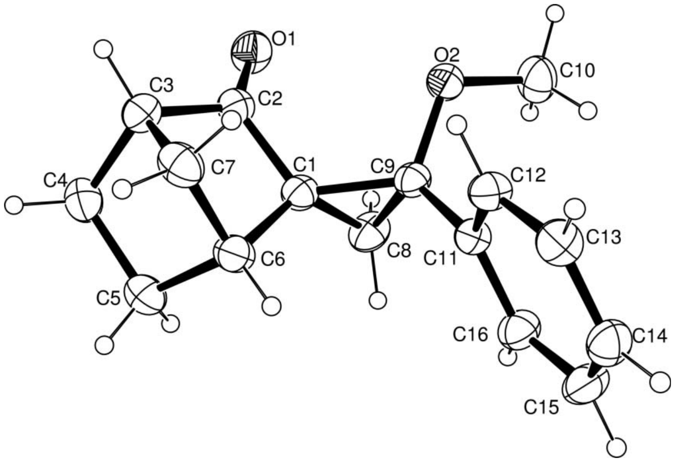


Figure 1
A view of the molecular structure of compound (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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