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Ethyl 5-methyl-2,2-diphenylcyclopropanespiro-3(4*H*)-furan-4-carboxylate

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The title compound, $C_{22}H_{22}O_3$, was synthesized by a reaction of ethyl acetoacetate and diphenylmethylenecyclopropane. The five-membered dihydrofuran ring adopts an envelope conformation.

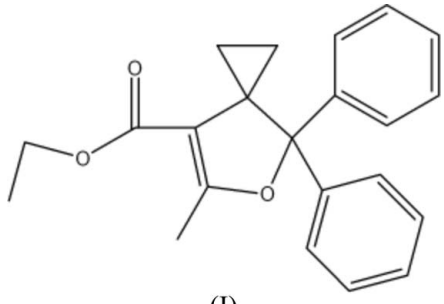
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
R factor = 0.051
wR factor = 0.152
Data-to-parameter ratio = 18.4

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

Comment

Compounds containing a dihydrofuran ring system and cyclopropane have become generally accepted as useful intermediates in organic synthesis. The chemistry of these compounds has been extensively studied and widely exploited in organic synthesis (Lee *et al.*, 1992; Lipshutz, 1986; Pietruszka, 2003). The title compound, (I), is a new compound containing a dihydrofuran ring system and cyclopropane. We report here the synthesis and crystal structure of (I).



(I)

The molecular structure of (I) is shown in Fig. 1. All bond lengths and angles in (I) are normal. The five-membered ring adopts an envelope conformation, with atom C7 lying at the flap position. The two benzene rings make a dihedral angle of 76.9 (2)°.

Experimental

A CH_3CN–THF solution (5:1, 10 ml) of ceric(IV) ammonium nitrate (0.60 g, 1.1 mmol) was added dropwise to a CH_3CN–THF solution (5:1, 2 ml) of ethyl acetoacetate (0.6 mmol) and diphenylmethylenecyclopropane (0.5 mmol) with stirring at ice-bath temperature. The mixture was warmed slowly to room temperature and stirring continued for 3 h. After the solvent of the reaction mixture had been evaporated, water (15 ml) was added. The aqueous layer was extracted with diethyl ether (3 times, 15 ml). The organic layer was dried over anhydrous $MgSO_4$. The residue was subjected to preparative thin-layer chromatography (eluant: petroleum ether–ethyl acetate, 10:1) to afford (I). Single crystals of (I) were obtained by evaporation of an ethanol–chloroform (1:1) mixed solution.

Crystal data

$C_{22}H_{22}O_3$
M_r = 334.40
Monoclinic, $P2_1/n$
a = 12.590 (3) Å
b = 9.979 (2) Å
c = 15.193 (3) Å
β = 105.29 (3)°
V = 1841.2 (7) Å^3
Z = 4
D_x = 1.206 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 295 (2) K
Chunk, colourless
0.31 × 0.27 × 0.24 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: none
17549 measured reflections
4203 independent reflections
3026 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.152
S = 1.09
4203 reflections
229 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0784P)^2 + 0.1896P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.013 (2)

Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å, and torsion angles were refined to fit the electron density; U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were placed in calculated positions with C—H = 0.97 (methylene) or 0.93 Å (aromatic), and refined in riding mode with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

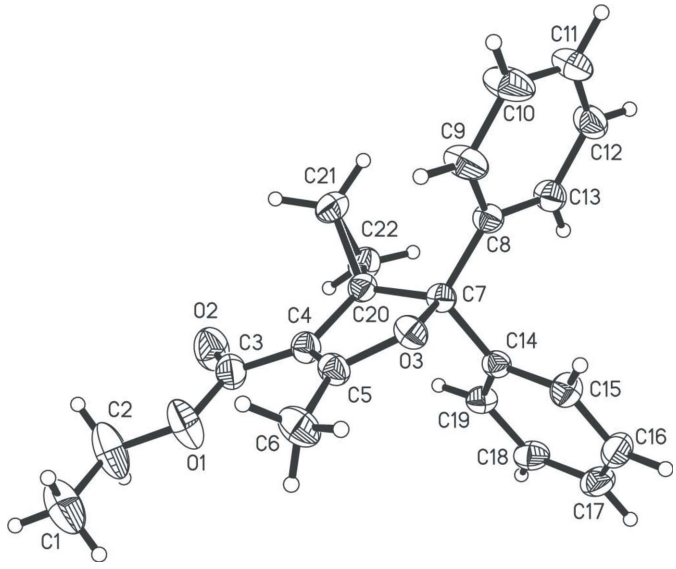


Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids (arbitrary spheres for H atoms).

References

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Lee, J., Li, J. H., Oya, S. & Snyder, J. K. (1992). *J. Org. Chem.* **57**, 5301–5312.
Lipshutz, B. H. (1986). *Chem. Rev.* **86**, 795–819.
Pietruszka, J. (2003). *Chem. Rev.* **103**, 1051–1070.
Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure.* Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.