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Methyl 3,3,7,7,9-pentamethyl-1,5-dioxaspiro-[5.5]undec-8-ene-8-carboxylate

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.120
Data-to-parameter ratio = 16.8

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

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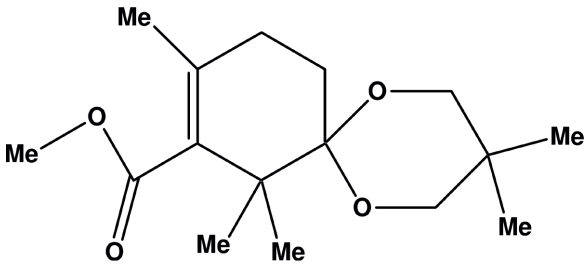
The crystal structure of the title ester, $C_{16}H_{26}O_4$, consists of discrete spirocyclic molecules. The six-membered 1,3-dioxane fragment is in a chair conformation and the cyclohexene fragment is in a distorted half-chair conformation. In the crystal structure, the molecules are connected by weak C—H···O contacts, with C···O distances ranging from 2.642 (1) to 2.762 (1) Å, forming a three-dimensional network.

Comment

In connection with a project aimed at the synthesis of taxol and its analogues, we developed a highly efficient Pd-catalysed carbonylation of an enol trifluoromethanesulfonate to produce the title ester, (I) (Toivola & Koskinen, 1996). As the reaction is sensitive to particular conditions, sometimes leading to dimerized products in yields nearly equal to that for the desired compound, we have subsequently investigated the reaction more closely (Toivola *et al.*, 2000). In this communication, we report the crystal structure of (I), whose molecular structure is illustrated in Fig. 1.



(I)

Selected bond lengths and angles are given in Table 1, and it can be seen that they are within the normal ranges. The crystal

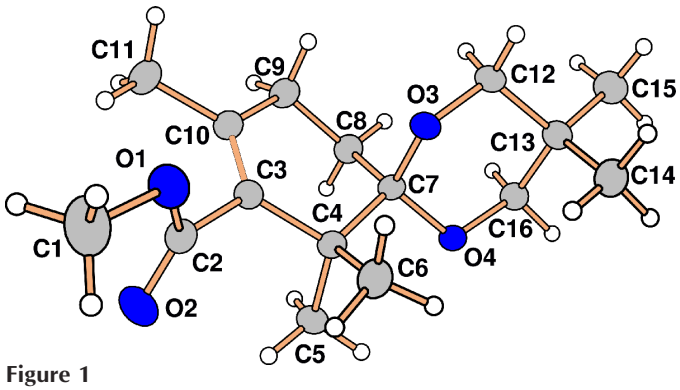


Figure 1
The molecular structure of (I), indicating the numbering of the atoms. Displacement ellipsoids have been drawn at the 50% probability level.

structure consists of discrete spirocyclic molecules. The six-membered 1,3-dioxane fragment is in the chair conformation and the cyclohexene fragment is in a distorted half-chair conformation. A similar spirocyclic framework to that forming the skeleton of (I) is also found in two other crystal structures (Kosela *et al.*, 1999; Nicolaou *et al.*, 2003). However, in both of these structures the ring framework is only a small part of a larger molecule. Therefore, steric effects and different ring substituents render neither the ring conformations nor the bond parameters directly comparable.

In the crystal structure of (I), there are some weak intermolecular C—H···O contacts, with C···O distances of 2.642 (1)–2.762 (1) Å, that lead to the formation of a three-dimensional network, as illustrated in Fig. 2.

Experimental

Compound (I) was synthesized as described earlier (Toivola & Koskinen, 1996). Colourless crystals of (I), suitable for crystal structure analysis, were grown from methanol.

Crystal data

$C_{16}H_{26}O_4$	D_x = 1.165 Mg m^{-3}
M_r = 282.37	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2855 reflections
a = 10.735 (2) Å	θ = 2.5–26.0°
b = 9.4311 (19) Å	μ = 0.08 mm^{-1}
c = 16.610 (3) Å	T = 150 (2) K
β = 106.81 (3)°	Block, colourless
V = 1609.7 (6) Å^3	0.20 × 0.20 × 0.15 mm
Z = 4	

Data collection

Bruker–Nonius KappaCCD diffractometer	2855 reflections with $I > 2\sigma(I)$
φ scans, and ω scans with κ offsets	R_{int} = 0.039
Absorption correction: none	θ_{max} = 26.0°
20 225 measured reflections	$h = -13 \rightarrow 13$
3133 independent reflections	$k = -11 \rightarrow 11$
	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 0.4495P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.120	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
S = 1.08	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
3133 reflections	Extinction correction: none
187 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.3394 (15)	C3—C4	1.5328 (15)
O1—C1	1.4481 (16)	C4—C5	1.5416 (17)
O2—C2	1.2094 (16)	C4—C6	1.5409 (17)
C2—C3	1.4984 (17)	C9—C10	1.5026 (17)
C3—C10	1.3364 (17)	C10—C11	1.5078 (17)
O2—C2—C3—C10	107.29 (15)		

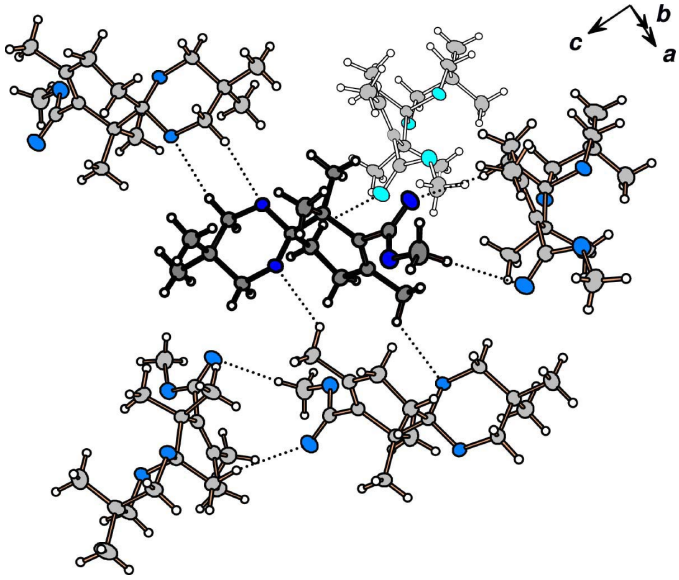


Figure 2
A view of the crystal packing in (I), showing the weak C—H···O contacts (dotted lines) linking the molecules to form a three-dimensional network.

H atoms were placed in calculated positions and treated as riding atoms, with C—H = 0.98–0.99 Å and U_{iso}(H) = 1.5U_{eq}(parent C atom).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Berndt, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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