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Ari M. P. Koskinen, Risto S. Laitinen, Raija Oilunkaniemi, Satu K. Savilampi and Reijo J. Toivola
Methyl 3,3,7,7,9-pentamethyl-1,5-dioxaspiro-[5.5]undec-8-ene-8-carboxylate

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.

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

\[ \text{C}_{16}\text{H}_{26}\text{O}_4 \]

\[ M_e = 282.37 \]

Monoclinic, \( P2_1/n \)

\( a = 10.735 \) (2) Å

\( b = 9.4311 \) (19) Å

\( c = 16.610 \) (3) Å

\( \beta = 106.81 \) (3)°

\( V = 1699.7 \) (6) Å³

\( Z = 4 \)

**Data collection**

Bruker–Nonius KappaCCD diffractometer

\( 2855 \) reflections with \( I > 2\sigma(I) \)

\( wR \) = \( 0.039 \)

\( \theta_{\text{max}} = 26.0° \)

\( k_0 = -13 \rightarrow 13 \)

\( l_0 = -20 \rightarrow 20 \)

**Refinement**

Refinement on \( F^2 \)

\[ R[I] = 0.038 \]

\[ wR[I] = 0.120 \]

\( S = 1.08 \)

3133 reflections

187 parameters

H-atom parameters constrained

**Selected geometric parameters (Å, °).**

<table>
<thead>
<tr>
<th>Bond/Angle</th>
<th>Value</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1—C2</td>
<td>1.3394 (15)</td>
<td>C3—C4</td>
</tr>
<tr>
<td>O1—C1</td>
<td>1.4481 (16)</td>
<td>C4—C5</td>
</tr>
<tr>
<td>O2—C2</td>
<td>1.2094 (16)</td>
<td>C4—C6</td>
</tr>
<tr>
<td>C2—C3</td>
<td>1.4984 (17)</td>
<td>C9—C10</td>
</tr>
<tr>
<td>C3—C10</td>
<td>1.3364 (17)</td>
<td>C10—C11</td>
</tr>
<tr>
<td>O2—C2—C3—C10</td>
<td>107.29 (15)</td>
<td></td>
</tr>
</tbody>
</table>