Crystal structure of tert-butyl-1κC-2(η⁵-cyclopentadienyl)dichloro-1κ²Cl-
tricarbonyl-2κ³C-tintungsten (Sn—W), tert-Bu[Cp(CO)₃W]SnCl₂

K. Jurkschat*, U. Kaltenbrunner and M. Schürmann

Universität Dortmund, Lehrstuhl für Anorganische Chemie II, Otto-Hahn-Str. 6, D-44227 Dortmund, Germany

Received February 24, 1999, CCDC-No. 1267/136

Abstract

Crystal: C₁₂H₂₅Cl₂O₃SnW, triclinic, P₁̅ (No. 2), a = 7.590(1) Å, b = 9.083(1) Å, c = 12.786(1) Å, α = 75.448(1)°, β = 80.461(1)°, γ = 80.326(1)°, V = 834.0 Å³, Z = 2, R_p(F) = 0.040, wR(F²) = 0.099, T = 170 K.

Source of material

Crystals of the title compound were obtained from the reaction of [Cp(CO)₃W]BuSnPh₂ with gaseous HCl and recrystallisation of the crude product from CH₂Cl₂/n-pentane (1:1); mp 380 K – 382 K [1].

Discussion

The tin atom shows a significantly distorted tetrahedral coordination with angles in the range from 130.7(2)° for C(11)—Sn(1)—W(1) to 98.66(11)° for Cl(2)—Sn(1)—Cl(1). The coordination polyhedron around tungsten can be described as a square pyramid with C(1), C(2), C(3), Sn(1) in the plane. The Sn—W distance amounts to 2.7519(6) Å and fits into the range of Sn—W distances between 2.706(1) Å [2] and 2.897 Å [3] given in the Cambridge Structural Data Base for 28 entries.

Table 1. Data collection and handling.

<table>
<thead>
<tr>
<th>Atom</th>
<th>Site</th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>U₁₁</th>
<th>U₂₂</th>
<th>U₃₃</th>
<th>U₁₂</th>
<th>U₁₃</th>
<th>U₂₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>H(4)</td>
<td>2i</td>
<td>0.7286</td>
<td>0.4798</td>
<td>0.2676</td>
<td>0.11(2)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H(5)</td>
<td>2i</td>
<td>0.6813</td>
<td>0.2779</td>
<td>0.4360</td>
<td>0.11</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H(6)</td>
<td>2i</td>
<td>0.5874</td>
<td>0.0602</td>
<td>0.3859</td>
<td>0.11</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H(7)</td>
<td>2i</td>
<td>0.5683</td>
<td>0.1276</td>
<td>0.1875</td>
<td>0.11</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H(8)</td>
<td>2i</td>
<td>0.6646</td>
<td>0.3848</td>
<td>0.1112</td>
<td>0.11</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H(12A)</td>
<td>2i</td>
<td>0.0449</td>
<td>0.7175</td>
<td>0.1685</td>
<td>0.10(1)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H(12B)</td>
<td>2i</td>
<td>0.2129</td>
<td>1.0135</td>
<td>0.0933</td>
<td>0.10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H(12C)</td>
<td>2i</td>
<td>0.2299</td>
<td>1.0119</td>
<td>0.2141</td>
<td>0.10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H(13A)</td>
<td>2i</td>
<td>0.0644</td>
<td>0.8402</td>
<td>0.3649</td>
<td>0.10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H(13B)</td>
<td>2i</td>
<td>-0.0393</td>
<td>0.7195</td>
<td>0.3409</td>
<td>0.10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H(13C)</td>
<td>2i</td>
<td>-0.1146</td>
<td>0.8944</td>
<td>0.3121</td>
<td>0.10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H(14A)</td>
<td>2i</td>
<td>-0.0120</td>
<td>0.6947</td>
<td>0.1436</td>
<td>0.10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H(14B)</td>
<td>2i</td>
<td>0.0637</td>
<td>0.8277</td>
<td>0.0521</td>
<td>0.10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H(14C)</td>
<td>2i</td>
<td>-0.1179</td>
<td>0.8609</td>
<td>0.1261</td>
<td>0.10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* Correspondence author
(e-mail: kjur@platon.chemie.uni-dortmund.de)
Table 3. Continued.

<table>
<thead>
<tr>
<th>Atom</th>
<th>Site</th>
<th>X</th>
<th>y</th>
<th>ζ</th>
<th>U_{11}</th>
<th>U_{22}</th>
<th>U_{33}</th>
<th>U_{12}</th>
<th>U_{13}</th>
<th>U_{23}</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cl(2)</td>
<td>2i</td>
<td>0.5287(4)</td>
<td>0.7431(3)</td>
<td>0.0527(2)</td>
<td>0.087(2)</td>
<td>0.095(2)</td>
<td>0.062(2)</td>
<td>-0.026(1)</td>
<td>0.025(1)</td>
<td>-0.007(1)</td>
</tr>
<tr>
<td>O(1)</td>
<td>2i</td>
<td>0.217(1)</td>
<td>0.4917(7)</td>
<td>0.5004(5)</td>
<td>0.091(5)</td>
<td>0.072(4)</td>
<td>0.053(4)</td>
<td>-0.013(3)</td>
<td>0.007(3)</td>
<td>-0.019(3)</td>
</tr>
<tr>
<td>O(2)</td>
<td>2i</td>
<td>0.130(1)</td>
<td>0.4143(9)</td>
<td>0.1213(7)</td>
<td>0.080(5)</td>
<td>0.103(6)</td>
<td>0.089(5)</td>
<td>-0.003(4)</td>
<td>-0.029(4)</td>
<td>-0.039(5)</td>
</tr>
<tr>
<td>O(3)</td>
<td>2i</td>
<td>0.105(1)</td>
<td>0.141(1)</td>
<td>0.4125(7)</td>
<td>0.115(7)</td>
<td>0.099(6)</td>
<td>0.094(6)</td>
<td>-0.006(5)</td>
<td>0.028(5)</td>
<td>-0.025(5)</td>
</tr>
<tr>
<td>C(1)</td>
<td>2i</td>
<td>0.278(1)</td>
<td>0.4493(9)</td>
<td>0.4246(7)</td>
<td>0.057(5)</td>
<td>0.050(4)</td>
<td>0.060(5)</td>
<td>-0.010(4)</td>
<td>-0.003(4)</td>
<td>-0.012(4)</td>
</tr>
<tr>
<td>Cl(2)</td>
<td>2i</td>
<td>0.221(1)</td>
<td>0.401(1)</td>
<td>0.1854(8)</td>
<td>0.115(7)</td>
<td>0.099(6)</td>
<td>0.094(6)</td>
<td>-0.006(5)</td>
<td>0.028(5)</td>
<td>-0.025(5)</td>
</tr>
<tr>
<td>C(3)</td>
<td>2i</td>
<td>0.206(1)</td>
<td>0.3704(8)</td>
<td>0.057(5)</td>
<td>0.050(4)</td>
<td>0.060(5)</td>
<td>-0.010(4)</td>
<td>-0.003(4)</td>
<td>-0.012(4)</td>
<td></td>
</tr>
<tr>
<td>Cl(3)</td>
<td>2i</td>
<td>0.693(1)</td>
<td>0.358(1)</td>
<td>0.272(1)</td>
<td>0.034(4)</td>
<td>0.067(6)</td>
<td>0.14(1)</td>
<td>-0.005(4)</td>
<td>-0.011(5)</td>
<td>-0.041(7)</td>
</tr>
<tr>
<td>Cl(4)</td>
<td>2i</td>
<td>0.666(1)</td>
<td>0.273(1)</td>
<td>0.366(1)</td>
<td>0.066(7)</td>
<td>0.086(7)</td>
<td>0.083(8)</td>
<td>0.007(5)</td>
<td>-0.023(5)</td>
<td>-0.026(6)</td>
</tr>
<tr>
<td>C(6)</td>
<td>2i</td>
<td>0.613(2)</td>
<td>0.152(1)</td>
<td>0.338(1)</td>
<td>0.069(7)</td>
<td>0.067(6)</td>
<td>0.104(9)</td>
<td>0.018(5)</td>
<td>-0.006(6)</td>
<td>-0.014(6)</td>
</tr>
<tr>
<td>C(7)</td>
<td>2i</td>
<td>0.604(1)</td>
<td>0.189(1)</td>
<td>0.2265(9)</td>
<td>0.054(5)</td>
<td>0.070(6)</td>
<td>0.079(7)</td>
<td>0.004(6)</td>
<td>-0.008(5)</td>
<td>-0.037(5)</td>
</tr>
<tr>
<td>C(8)</td>
<td>2i</td>
<td>0.656(1)</td>
<td>0.332(1)</td>
<td>0.184(1)</td>
<td>0.056(5)</td>
<td>0.099(8)</td>
<td>0.063(6)</td>
<td>0.010(5)</td>
<td>0.014(5)</td>
<td>-0.009(6)</td>
</tr>
<tr>
<td>C(11)</td>
<td>2i</td>
<td>0.101(1)</td>
<td>0.8377(9)</td>
<td>0.2027(7)</td>
<td>0.049(5)</td>
<td>0.046(4)</td>
<td>0.066(5)</td>
<td>-0.010(3)</td>
<td>-0.006(4)</td>
<td>-0.001(4)</td>
</tr>
<tr>
<td>C(12)</td>
<td>2i</td>
<td>0.152(2)</td>
<td>0.998(1)</td>
<td>0.166(1)</td>
<td>0.083(7)</td>
<td>0.051(5)</td>
<td>0.096(8)</td>
<td>0.002(5)</td>
<td>-0.022(6)</td>
<td>-0.001(5)</td>
</tr>
<tr>
<td>C(13)</td>
<td>2i</td>
<td>-0.007(2)</td>
<td>0.822(1)</td>
<td>0.316(1)</td>
<td>0.059(6)</td>
<td>0.094(8)</td>
<td>0.098(9)</td>
<td>0.006(5)</td>
<td>0.022(6)</td>
<td>0.001(6)</td>
</tr>
<tr>
<td>C(14)</td>
<td>2i</td>
<td>-0.000(2)</td>
<td>0.802(1)</td>
<td>0.124(1)</td>
<td>0.083(9)</td>
<td>0.090(8)</td>
<td>0.13(1)</td>
<td>-0.006(6)</td>
<td>-0.005(8)</td>
<td>-0.008(8)</td>
</tr>
</tbody>
</table>

Acknowledgments. We thank the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support.

References