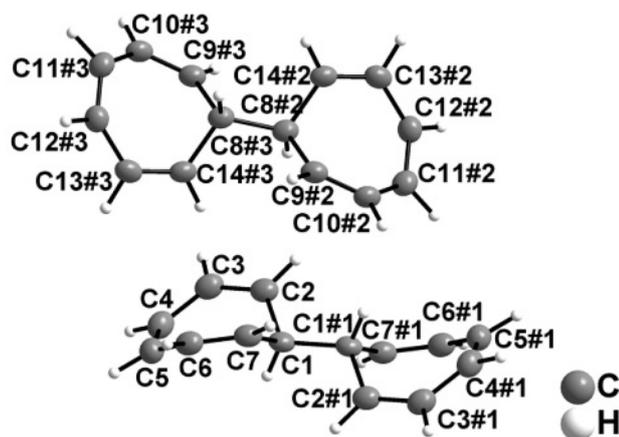


# Crystal structure of 7,7'-bi-1,3,5-cycloheptatrien, C<sub>14</sub>H<sub>14</sub>

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## Abstract

C<sub>14</sub>H<sub>14</sub>, triclinic,  $P\bar{1}$  (no. 2),  $a = 6.450(1)$  Å,  $b = 9.614(2)$  Å,  $c = 9.712(2)$  Å,  $\alpha = 112.11(2)^\circ$ ,  $\beta = 100.55(2)^\circ$ ,  $\gamma = 99.87(2)^\circ$ ,  $V = 529.1$  Å<sup>3</sup>,  $Z = 2$ ,  $R_{\text{gt}}(F) = 0.0536$ ,  $wR_{\text{ref}}(F^2) = 0.1610$ ,  $T = 123$  K.

**Table 1.** Data collection and handling.

Crystal:	yellow plate, size 0.2×0.4×0.4 mm
Wavelength:	Mo $K_{\alpha}$ radiation (0.71073 Å)
$\mu$ :	0.64 cm <sup>-1</sup>
Diffractometer, scan mode:	STOE IPDS I, $\varphi$
$2\theta_{\text{max}}$ :	50.9°
$N(hkl)_{\text{measured}}$ , $N(hkl)_{\text{unique}}$ :	5200, 1829
Criterion for $I_{\text{obs}}$ , $N(hkl)_{\text{gt}}$ :	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$ , 1383
$N(\text{param})_{\text{refined}}$ :	184
Programs:	SHELX [8], DIAMOND [9], OLEX2 [10], XSHAPE [11], publCIF [12]

## Source of material

All manipulations were carried out under argon atmosphere using standard Schlenk techniques. KSnBi was synthesized in a reaction of equimolar amounts of the corresponding elements in a duran ampoule at 723 K. 367 mg (0.5 mmol) KSnBi and 208 mg (0.5 mmol) [(C<sub>7</sub>H<sub>7</sub>)Mo(CO)<sub>3</sub>]PF<sub>6</sub> were dissolved in liquid ammonia together with 48 mg (0.13 mmol) dibenzo-[18]crown6 (1,4,7,14,17,20-Hexaoxa[7.7]orthocyclophan).

## Experimental details

Crystal preparation was done in perfluoretheroil (Galden HT 230, Fa. Ausimont, USA), which was cooled with evaporating liquid nitrogen. An appropriate crystal was mounted on the nylon loop of the Crystal Cap (CrystalCap Copper 14mm with CryoLoop 20 $\mu$  0.3–0.4 mm, Hampton Research, USA) and characterized by X-ray diffraction.

## Discussion

In the course of work on the chemistry of Zintl ions, such as Sn<sub>4</sub><sup>4-</sup>, Si<sub>4</sub><sup>4-</sup>, Pb<sub>9</sub><sup>4-</sup>, Ge<sub>9</sub><sup>4-</sup> or Sn<sub>5</sub><sup>2-</sup>, a variety of reaction pathways could be found. Examples among others are the oligo- or polymerization [1, 2], the functionalization with main-group or transition-metal fragments [3, 4] and the insertion of metal atoms [5, 6]. Utilizing the reductive power of the Zintl anions, the structure on hand was obtained by the reaction of the heteroatomic anion Sn<sub>2</sub>Bi<sub>2</sub><sup>2-</sup> with the tropylium-ligand of the transition metal complex [(C<sub>7</sub>H<sub>7</sub>)Mo(CO)<sub>3</sub>]PF<sub>6</sub>. In the proceeding Birch-reduction, the subsequently formed C<sub>14</sub>H<sub>14</sub> molecule was separated from the transition metal complex, crystallizing without solvent molecules. A similar reaction using sodium amalgam or sodium naphthalide as reductive component was undertaken by M. Winter *et al.*, resulting in the dimerization to the ditropyl complex {Mo(CO)<sub>3</sub>}<sub>2</sub>( $\eta^6, \eta^6$ -C<sub>14</sub>H<sub>14</sub>) [7]. Two crystallographically independent C<sub>14</sub>H<sub>14</sub> molecules are present within the unit cell. Both molecules form layers, which are shifted against each other to form a closest packing. Due to the reduction of the aromaticity and hence the planarity of the tropylium cation is cancelled, resulting in a boat-like conformation for the 1,3,5-cycloheptatrienyl-fragment. Within such a seven-membered ring, single bonds can be found between the carbon atoms C1–C2, C3–C4 and C5–C6 with bond lengths ranging from 1.440(4) Å to 1.515(3) Å. Subsequent double bonds are situated between C2–C3, C4–C5 and C6–C7 with bond lengths ranging from 1.340(4) Å to 1.364(4) Å. The seven-membered rings are connected by a single bond of 1.533(5) Å of length to their symmetry equivalents (see figure). Carbon-hydrogen bond lengths differ from 0.94(3) Å to 1.06 Å. For the second independent C<sub>14</sub>H<sub>14</sub> molecule of the unit cell the bond lengths are similar.

**Table 2.** Atomic coordinates and displacement parameters (in Å<sup>2</sup>).

Atom	Site	$x$	$y$	$z$	$U_{\text{iso}}$
H(1)	2i	0.509(4)	0.975(3)	0.849(3)	0.027(6)
H(2)	2i	0.137(5)	0.879(3)	0.931(3)	0.033(7)
H(3)	2i	-0.019(6)	0.735(4)	0.666(4)	0.051(8)
H(4)	2i	0.196(5)	0.691(3)	0.487(4)	0.042(7)
H(5)	2i	0.522(5)	0.644(3)	0.532(3)	0.040(7)
H(6)	2i	0.683(6)	0.625(4)	0.765(4)	0.055(9)
H(7)	2i	0.654(5)	0.803(3)	1.002(3)	0.040(7)
H(8)	2i	-0.021(5)	0.650(4)	0.018(3)	0.047(8)
H(9)	2i	0.342(6)	0.595(4)	0.149(3)	0.046(8)
H(10)	2i	0.458(5)	0.857(3)	0.318(3)	0.038(7)
H(11)	2i	0.219(5)	1.027(4)	0.338(3)	0.041(7)
H(12)	2i	-0.115(5)	0.940(3)	0.346(3)	0.035(7)
H(13)	2i	-0.256(6)	0.690(4)	0.338(4)	0.058(9)
H(14)	2i	-0.183(5)	0.471(4)	0.165(3)	0.042(7)

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**Table 3.** Atomic coordinates and displacement parameters (in Å<sup>2</sup>).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>11</sub>	<i>U</i> <sub>22</sub>	<i>U</i> <sub>33</sub>	<i>U</i> <sub>12</sub>	<i>U</i> <sub>13</sub>	<i>U</i> <sub>23</sub>
C(1)	2 <i>i</i>	0.4703(4)	0.9351(3)	0.9182(3)	0.030(1)	0.036(1)	0.035(1)	0.007(1)	0.0109(9)	0.021(1)
C(2)	2 <i>i</i>	0.2270(4)	0.8576(3)	0.8553(3)	0.032(1)	0.036(1)	0.043(1)	0.011(1)	0.010(1)	0.022(1)
C(3)	2 <i>i</i>	0.1392(4)	0.7647(3)	0.7037(3)	0.032(1)	0.039(1)	0.044(1)	0.007(1)	0.003(1)	0.019(1)
C(4)	2 <i>i</i>	0.2618(5)	0.7152(3)	0.5925(3)	0.046(2)	0.037(1)	0.035(1)	0.007(1)	0.004(1)	0.016(1)
C(5)	2 <i>i</i>	0.4627(5)	0.6885(3)	0.6219(3)	0.044(2)	0.036(1)	0.035(1)	0.007(1)	0.011(1)	0.013(1)
C(6)	2 <i>i</i>	0.5850(4)	0.7035(3)	0.7692(3)	0.034(1)	0.035(1)	0.045(1)	0.009(1)	0.011(1)	0.019(1)
C(7)	2 <i>i</i>	0.5823(4)	0.8085(3)	0.9062(3)	0.029(1)	0.039(1)	0.039(1)	0.006(1)	0.0080(9)	0.022(1)
C(8)	2 <i>i</i>	0.0110(4)	0.5800(3)	0.0630(3)	0.025(1)	0.034(1)	0.035(1)	0.0069(9)	0.0056(9)	0.018(1)
C(9)	2 <i>i</i>	0.2433(4)	0.6597(3)	0.1622(3)	0.026(1)	0.038(1)	0.037(1)	0.008(1)	0.0087(9)	0.020(1)
C(10)	2 <i>i</i>	0.3034(4)	0.8104(3)	0.2624(3)	0.026(1)	0.039(1)	0.037(1)	0.003(1)	0.0071(9)	0.019(1)
C(11)	2 <i>i</i>	0.1563(4)	0.9081(3)	0.2992(3)	0.039(1)	0.033(1)	0.037(1)	0.006(1)	0.007(1)	0.014(1)
C(12)	2 <i>i</i>	-0.0524(4)	0.8594(3)	0.3036(3)	0.036(1)	0.036(1)	0.034(1)	0.012(1)	0.008(1)	0.013(1)
C(13)	2 <i>i</i>	-0.1597(4)	0.7009(3)	0.2725(3)	0.026(1)	0.046(1)	0.035(1)	0.007(1)	0.0079(9)	0.020(1)
C(14)	2 <i>i</i>	-0.1268(4)	0.5732(3)	0.1714(3)	0.024(1)	0.037(1)	0.036(1)	0.003(1)	0.0045(9)	0.019(1)

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