

The First X-ray Crystal Structures of 5,5,10,10-Tetrahalotricyclo[7.1.0.04,6]decanes

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While a number of bicyclo and tricyclo compounds have been extensively studied (the norbornanes come to mind), other tricyclic compounds remain relatively uncharacterized. To this end, tetrahalogenated tricyclo[7.1.0.04,6]decanes have been synthesized to be characterized by x-ray crystallographic methods. Halogenation raises the melting point and provides for a convenient synthetic method of cyclopropanation of 1,5-cyclooctadiene to produce crystals suitable for x-ray diffraction. Our laboratory has now synthesized six bromine and chlorine substituted tricyclo[5.1.0.04,6]decanes and we are currently analyzing these compounds structurally.

While one of these compounds was synthesized by Louis Fieser and the space group determined by Jean Hartsuck and William N. Lipscomb in the 1960s, a high resolution structure was never produced. Among the challenges of solving these structures is that the tricyclodecane rings have at times exhibited significant ring disorder, and perhaps this accounts for the lack of a structure from the Harvard investigation of years ago. The cyclooctane rings are highly puckered, providing a number of opportunities for disordered arrangement of the fairly flexible rings. Of these compounds, 5,5,10,10-tetrabromotricyclo[7.1.0.0]decane, while puckered, was not disordered. 5,5,10,10-tetrachlorotricyclo[7.1.0.0]decane, on the other hand, exhibited at least two puckered structures superimposed on one another. The average structure resembles a boat form cyclohexane, consistent with Hartsuck and Lipscomb's original work.

To our knowledge, these structures represent the first x-ray crystal structures of any tricyclo[7.1.0.0]decanes.