



transform a cyclopropyl carbon atom into a bridgehead atom and the bridgehead and two adjacent carbon atoms into cyclopropyl atoms. Doering and Roth furthermore predicted that the NMR spectra of bullvalene would contain only one single sharp band since successive rapid Cope-rearrangements would lead to a fluxional structure where the carbon atoms change their places constantly.

The synthesis of the molecule was published by Schröder.<sup>3</sup> He has also taken temperature dependent NMR spectra of the molecule. At about 100°C only one single sharp band is observed. At lower temperatures the band broadens and at about -25°C two separate bands can be observed. At even lower temperatures down to -85°C, the bands do not change.

The experimental data for the electron diffraction studies of bullvalene were recorded at the Oslo apparatus at a nozzle temperature of about 100°C. The molecular parameters were determined by least squares refinement on the experimental molecular intensity function in the  $s$ -range 1.25 to 41.75 Å<sup>-1</sup>. The molecule is refined under the assumption of  $C_{3v}$  symmetry.

The parameters used in the refinement of the carbon skeleton were the interatomic distances  $C_1-C_2$ ,  $C_3-C_3$ ,  $C_3-C_4$ ,  $C_4-C_5$ , and two angular parameters, the angle between the  $C_1-C_2$  bond and the threefold axis ( $v_1$ ) and the angle  $C_1C_2C_3$  ( $v_2$ ). An attempt to refine the C-H bond distances proved not successful. The  $u$ -values for the distances  $C_1-C_2$ ,  $C_3-C_4$ , and  $C_4-C_5$  were put equal and refined together.

Experimentally determined bond distances and angle parameters.

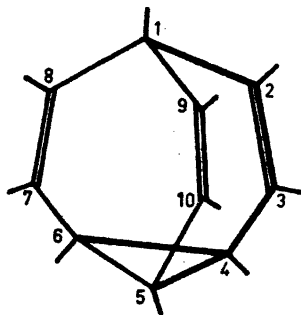
Distance or angle	$r_{ij}$ or $v_i$	stand. dev.
$C_1-C_2$	1.527 <sub>4</sub> Å	0.0045 Å
$C_2-C_3$	1.347 <sub>8</sub> Å	0.0028 Å
$C_3-C_4$	1.466 <sub>7</sub> Å	0.0037 Å
$C_4-C_5$	1.544 <sub>4</sub> Å	0.0054 Å
$v_1$	72.5 <sub>7</sub> °	0.54°
$v_2$	122.6 <sub>9</sub> °	0.80°

Experimentally determined  $u_{ij}$ -values.

Distance	$u_{ij}$ -value (in Å)	stand. dev. (in Å)
$C_1-C_2$ } $C_3-C_4$ }	0.048 <sub>2</sub>	0.0030
$C_4-C_5$ } $C_2-C_3$ }	0.045 <sub>5</sub>	0.0023

The angle  $C_2C_3C_4$  turned out to be 125.9°. There is no doubt that the molecule has normal double bonds, significantly different from the other bond lengths. Of these the values determined for the  $C_1-C_2$  and  $C_3-C_4$  bond distances are what could be expected while the bond-distances in the cyclopropane ring are longer than we had expected. The refinements will be continued to check the accuracy of the value determined for the  $C_4-C_5$  distance.

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1. Doering, W. v. E. and Roth, W. R. *Angew. Chem.* **75** (1963) 27.
2. Doering, W. v. E. and Roth, W. R. *Tetrahedron* **19** (1963) 715.
3. Schröder, G. *Angew. Chem.* **75** (1963) 722.

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