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CRYSTAL AND MOLECULAR STRUCTURE OF 5.8.10-TRIMETHYLBENZO[c] ACEHEPTYLENE

H. J. Lindner

Institut für Organische Chemie der Technischen Hochschule Darmstadt

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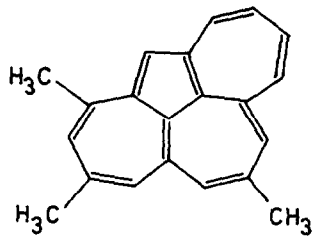
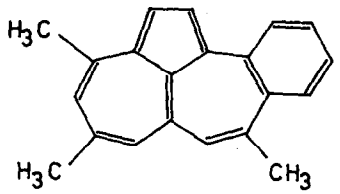
During the studies leading to the preparation of 5.8.10-trimethylazuleno-[8.8a.1.2-def]heptalene (I) Hafner and coworkers (1) isolated an isomer of (I), which could be identified as 5.8.10-trimethylbenzo[c] aceheptylene (II) (2). The structure of (II) has now been established by X-ray crystal structure analysis.



I



II

(II) crystallizes from light petrol (b.p. 40-60°) in green prisms, m.p. 103 - 104°. Crystal data: $C_{21}H_{18}$, M = 270.4, orthorhombic, a = 15.23, b = 12.40, c = 7.83 Å, Z = 4, space group $P2_12_12_1$ (No. 19). X-ray intensity data were collected on an automatic Weissenberg diffractometer using monochromated CuK_α radiation. The structure analysis was based on 991 symmetry independent reflections. The structure has been determined by the symbolic addition procedure using the tangent formula (3) and Fourier methods. The final R-factor after full-matrix least squares refinement including the positions of the hydrogen was 0.097. The molecular geometry found is given in the figure. The standard deviations of the C-C bond lenghts are 0.015 Å, those of the angles less than

1°. The molecule shows small but significant deviations from planar structure. The two seven-membered rings are slightly distorted and the two other rings are bent out of the plane. The bond lengths found by structure analysis agree satisfactorily with those calculated by the SCF-LCAO-MO method (4).

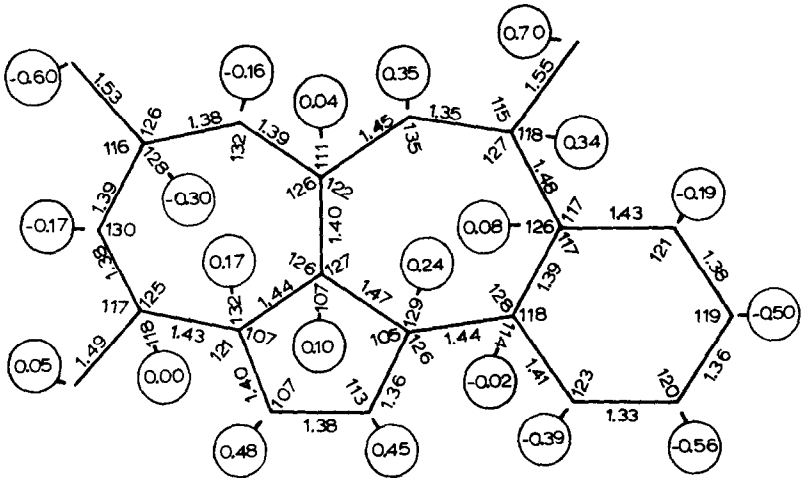


fig. Bond lengths and bond angles of the C-C bonds and deviations of the C-atom positions from the least-squares plane.

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