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X-RAY STRUCTURE ANALYSIS OF ALL-CIS-1,6-DICHLOROCYCLODECA-1,3,6,8-TETRAENE

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The stereochemistry, molecular dimensions and crystal structure of 1,6-dichlorocyclodeca-1,3,6,8-tetraene were determined by a three-dimensional X-ray diffraction analysis.

The material was prepared by Professor Sondheimer and Dr. Grohmann as described in the previous communication and the crystallographic analysis undertaken to complement the chemical investigations. The structure was solved directly from the X-ray data, with minimal chemical assumptions. The molecule proved to be centrosymmetric, with an all-cis configuration (Fig. 1.) and with dimensions indicated in Table 1.

TABLE 1.

Cl(1) - C(1) = 1.76Å	C($\bar{5}$) - C(1) - Cl(1) = 119°
	C(2) - C(1) - Cl(1) = 114°
C(1) - C(2) = 1.51Å	
C(2) - C(3) = 1.51Å	C($\bar{5}$) - C(1) - C(2) = 128°
	C(1) - C(2) - C(3) = 113°
C(3) - C(4) = 1.33Å	C(2) - C(3) - C(4) = 125°
C(5) - C($\bar{1}$) = 1.31Å	C(3) - C(4) - C(5) = 128°
	C(4) - C(5) - C($\bar{1}$) = 126°
C(4) - C(5) = 1.46Å	
C(2) - H(1) = 1.0Å	C = C - H (av.) = 118°
C(2) - H(2) = 1.1Å	C - C - H (av.) = 112°
C(3) - H(3) = 0.9Å	H(1) - C(2) - H(2) = 109°
C(4) - H(4) = 1.0Å	
C(5) - H(5) = 1.0Å	

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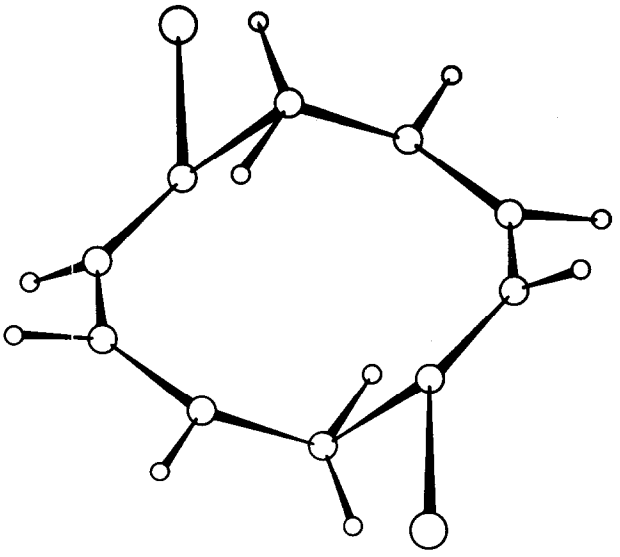
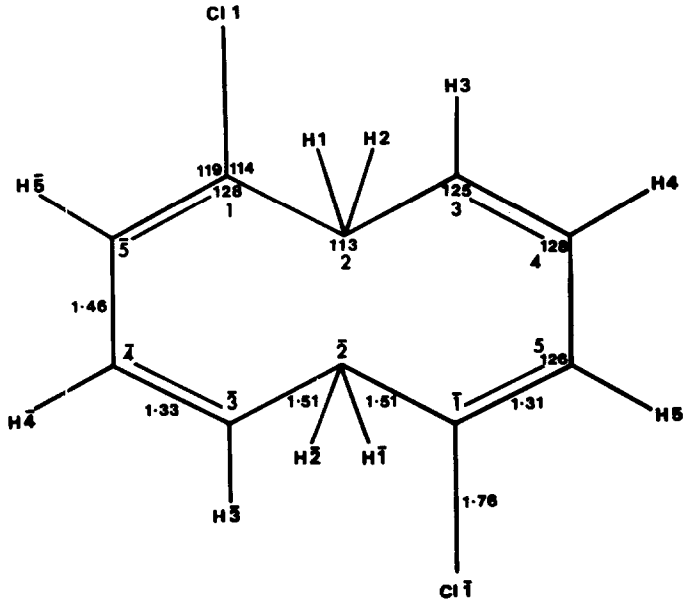
FIGURE 1.




Cl 1
H3
H1
H2
H5̄
119
114
128
1
125
3
H4
5̄
113
2
128
4
1·46
1̄
2̄
5
128
H4̄
1·33
3̄
1·51
1·51
1̄
1·31
H5
H2̄
H1̄
1·76
H3̄
Cl 1̄

Each half of the molecule may be defined by two planes; one through atoms C(2), C(3), C(4), C(5), H(3), H(4) and one through atoms C(4), C(5), C($\bar{1}$), C($\bar{2}$), H(5), Cl($\bar{1}$). The two planes are inclined at an angle of 57.7° to each other. This arrangement of atoms allows for a separation of 2.5Å between the two inner hydrogens H(1) and H($\bar{1}$). The bond lengths and angles found agree closely with expected values.

Crystal Data: $C_{10}H_{10}Cl_2$. M. Wt. = 201.1. Chunky prisms from petroleum ether (b.p. 40-60°). Diagonal extinction on the prominent, diamond-shaped faces.

Monoclinic: a = 7.577Å, b = 7.878Å, c = 8.397Å, av. σ = 0.001Å, β = 109.1°
Z = 2 D(calc) = 1.43 g/cm^3. Space group $P2_1/c$ from absences. Molecular symmetry: $\bar{1}$.

Experimental: The crystals were unstable and had to be enclosed in capillary tubes, filled with nitrogen. After preliminary X-ray photographs, all measurements were carried out on a Picker 4-circle X-ray diffractometer. Of the 863 points measured to a 2θ maximum of 136.5°, 838 were of significant intensity.

Solution and Refinement: The position of the chlorine atom of the asymmetric unit was deduced from a three-dimensional sharpened Patterson map and the choice between alternative locations was made on the basis of the intermolecular chlorine approaches. The coordinates of the carbon atoms were obtained from Fourier maps and the structure refined through three cycles each of isotropic and anisotropic least squares calculations using the X-ray 63 system of programs. The hydrogen contributions were next inserted and a further four cycles of mixed anisotropic/isotropic refinement with 70 variables led to a reliability index of 9.0%(observed terms only). The average values of the standard deviation in atomic coordinates are 0.002Å for Cl, 0.006Å for C and 0.07Å for H. Refinement is continuing.

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