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A New Isomer of Pentachlorocyclohexane

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Abstract. $C_6H_7Cl_5$, monoclinic, $P2_1/c$, $a = 8{\cdot}061\ (1)$, $b = 7{\cdot}992\ (1)$, $c = 16{\cdot}554\ (3)$ Å, $\beta = 110{\cdot}43\ (1)°$, $Z = 4$. The structure was solved by the direct method and refined by the block-diagonal least-squares technique to $R = 0{\cdot}042$ for 1516 reflexions. The Cl atoms adopt the configuration $1\alpha2\beta3\alpha4\alpha5\alpha$, whose conformation is *1e2e3e4a5e*.

Introduction. The systematic absences are $h0l$ when l is odd and $0k0$ when k is odd. Intensities were collected by the θ–2θ scan technique on a Hilger & Watts Y-290 diffractometer with Zr-filtered Mo $K\alpha$ radiation and a crystal of dimensions $0{\cdot}2 \times 0{\cdot}2 \times 0{\cdot}2$ mm. 1758 independent reflexions with $\theta \leq 25°$ were corrected for Lorentz and polarization effects, but not for absorption. The structure was solved by use of the program *MULTAN* (Main, Germain & Woolfson, 1970) with local modifications on a FACOM 270-30 computer. After anisotropic refinement of the non-hydrogen atoms by a block-diagonal least-squares method, the R value converged to 0·042 for 1516 reflexions with $|F_c| > \sigma(F_o)$ and $|\Delta F| < 3\sigma(F_o)$. $\sigma(F_o)$ was estimated as $[\sigma_1^2(F_o) + 0{\cdot}00080|F_o|^2]^{1/2}$, where $\sigma_1(F_o)$ is the e.s.d. depending on counting errors. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974).* Final positional parameters are given in Table 1.

Discussion. Four major decomposition products of γ-irradiation were obtained in a 2-propanol solution of the γ-isomer of 1,2,3,4,5,6-hexachlorocyclohexane. Three of them were identified as γ- and ε-tetrachlorocyclohexenes and *meso*-pentachlorocyclohexane by their IR and NMR spectral data and melting points. The other one was assumed to be an isomer of pentachlorocyclohexane, but not identical to the racemic isomer that was one of the two isomers found so far. The molecular structure of the new isomer was established by X-ray analysis and reported in a preliminary form (Hamada, Kawano, Kawamura & Shiro, 1977).

A perspective view of the molecule is shown in Fig. 1. The configuration and conformation for the Cl atoms in the new isomer (I) are compared with those of the *meso* isomer (II) and racemic isomer (III) of pentachlorocyclohexane:

	Configuration		Conformation
(I)	$1\alpha2\beta3\alpha4\alpha5\alpha$		*1e2e3e4a5e*
(II)	$1\alpha2\alpha3\beta4\alpha5\alpha$	(i)	*1e2a3a4a5e*
		(ii)	*1a2e3e4e5a*
(III)	$1\alpha2\beta3\alpha4\alpha5\beta$		*1e2e3e4a5a*.

Tanaka, Kiso, Yoshida, Kurihara & Nakajima (1975) investigated the molecular structures of (II) and (III) by ^{1}H NMR studies, and indicated that, of the two conformations of (II), (i) is more stable (about 1·6 kJ mol^{-1}) than (ii). The crystal structure analysis showed

Table 1. *Atomic fractional coordinates* ($\times 10^4$; *for* H $\times 10^3$)

	x	*y*	*z*
Cl(1)	10404 (2)	3715 (2)	981 (1)
Cl(2)	10947 (1)	3203 (1)	3003 (1)
Cl(3)	7343 (2)	3859 (1)	3404 (1)
Cl(4)	4928 (1)	5045 (1)	1491 (1)
Cl(5)	3570 (1)	1928 (1)	123 (1)
C(1)	8843 (4)	2675 (4)	1352 (2)
C(2)	8836 (4)	3526 (3)	2172 (2)
C(3)	7391 (4)	2803 (4)	2455 (2)
C(4)	5561 (4)	2904 (3)	1753 (2)
C(5)	5681 (4)	1972 (4)	970 (2)
C(6)	7035 (4)	2737 (4)	640 (2)
H(1)	919	138	147
H(2)	862	481	209
H(3)	754	154	264
H(4)	446	231	200
H(5)	600	62	115
H(6)	677	400	42
H(6′)	715	208	24

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33899 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

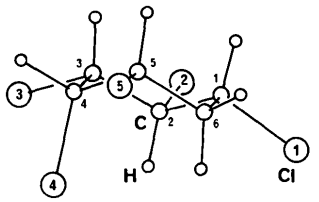

Fig. 1. Perspective view of the molecule.

that the Cl atoms adopt the (i) conformation in the (II) crystal obtained from an *n*-hexane solution (Nakai, Shiro & Hamada, 1978).

Bond lengths, bond angles and torsion angles are given in Fig. 2. The mean e.s.d. values are 0·003 Å for Cl–C, 0·005 Å for C–C, and 0·2° for angles. The mean corrections of bond lengths for the effects of rigid-body libration are 0·010 (ranging from 0·009 to 0·011 Å) for Cl–C and 0·009 (0·008–0·010 Å) for C–C. The shortest intramolecular Cl–Cl distance is 3·228 Å between Cl(3) and Cl(4).

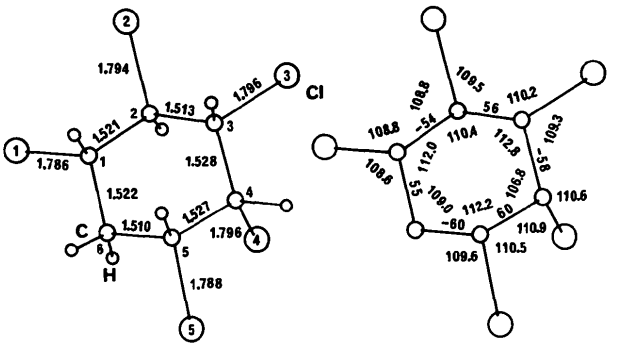

Fig. 2. Bond lengths (Å), bond angles and torsion angles (°).

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Ethyl 3-Methyl-4-oxo-1-phenyl-2-phenylimino-1,3,7,8-tetraazaspiro[4.5]deca-6,9-diene-10-carboxylate–0·5 Benzene

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Abstract. $C_{22}H_{21}N_5O_3 \cdot \frac{1}{2}C_6H_6$, monoclinic, $P2_1/c$, a = 9·388 (2), b = 28·140 (3), c = 9·017 (2) Å, β = 103·4 (1)°, Z = 4, D_x = 1·27 g cm^{-3}, μ(Mo $K\alpha$) = 0·918 cm^{-1}. Counter technique, direct methods, full-matrix least-squares refinement. R = 0·056 for 1304 observed reflexions measured at room temperature (20°C). The main geometrical molecular features and the thermal motion were analysed.

Introduction. The chemical preparation of the title compound and other spiro compounds has been described by Adembri, Chimichi, De Sio, Nesi & Scotton (1976). The reaction between the diethylic ester of 4,5-pyridazinedicarboxylic acid with 1,3-diphenylguanidine and NaOH in the presence of tetrahydrofuran at room temperature gives, with good yield, a product (I) of elementary composition $C_{21}H_{19}N_5O_3$. Spectroscopic data and chemical evidence suggested a spiranic structure for this compound (Nesi, 1977). An early attempt to determine the molecular geometry by crystal-structure analysis failed because the crystals decompose too rapidly to allow the collection of reliable data. However, the unit-cell dimensions were determined to be: a = 17·87 (1), b = 19·08 (1), c = 12·19 (1) Å, and β = 100·7 (5)°; space group $P2_1/a$, Z = 8. Eventually a stable mono-methyl derivative $C_{22}H_{21}N_5O_3$ (II) was prepared by treating (I) with diazomethane. Crystals of (II), obtained from benzene as white needles with m.p. 213–215°C, were kindly supplied by Professor R. Nesi. Intensity data were recorded on a PW 1100 automatic diffractometer (ω–2θ scan, $\theta \leq 20°$, graphite monochromator, Mo $K\alpha$ radiation, λ = 0·7107 Å). The unit-cell dimensions were determined by a