

Table 3. *Parameters and angles (°) describing the geometry of the amide group*

Parameter definition	Values for the title lactam	
	Molecule A	Molecule B
$\omega_1 = \omega C(6)-C(5)-N(4)-C(3) $	-3.5 (4)	-1.8 (4)
$\omega_2 = \omega O(1)-C(5)-N(4)-H(5) $	5 (2)	0 (2)
$\omega_3 = \omega O(1)-C(5)-N(4)-C(3) $	175.6 (2)	178.7 (3)
$\omega_4 = \omega C(6)-C(5)-N(4)-H(5) $	-174 (2)	179 (2)
$\tau' = 2\tau = \omega_1 + \omega_2$	2 (2)	-2 (2)
$\chi_C = \omega_1 - \omega_3 + \pi$	0.9 (4)	-0.5 (5)
$\chi_N = \omega_2 - \omega_3 + \pi$	9 (2)	1 (2)

These parameters along with the statistical χ^2 value for the mean plane through the amide group of molecule A (345.6) indicate much more significant deviation from planarity than for B (38.9). An especially significant contribution to the non-planarity is represented by the value of χ_N for molecule A. It corresponds to out-of-plane bending at the N(4A) atom. Values of χ_C of both molecules A and B represent the small contribution of bending at the C(5A) and C(5B) atoms respectively. The contribution of twisting about the C(5)-N(4) bond is also small as can be seen from values of the parameter τ' . Comparison of coordinates of the atomic subsets A and B shows significant differences in these molecules in any projection.

Thus, the A molecule contains a *cis*-amide group with a significant non-planarity contribution from pyramidal bond arrangement at N; on the other hand, this group of the B molecule is nearly planar. Molecules A are connected by inter-amide hydrogen bonds into cyclic

dimers as are molecules B. No hydrogen bonds of the type $A \cdots B$ were found.

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Structure of 5,5,7,7,8,8-Hexachloro-4-methoxy-2-methyl-5,6,7,8-tetrahydro-6-quinolinone*

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Abstract. $C_{11}H_7Cl_6NO_2$, $M_r = 397.9$, triclinic, $P\bar{1}$, $a = 8.801$ (1), $b = 9.285$ (1), $c = 10.099$ (1) Å, $\alpha = 92.69$ (1), $\beta = 102.70$ (1), $\gamma = 111.38$ (1)°, $V = 742.3$ Å³, $Z = 2$, $D_m = 1.774$ (2), $D_x = 1.780$ Mg m⁻³, $\lambda(\text{Mo K}\alpha) = 0.7107$ Å, $\mu = 1.15$ mm⁻¹, $F(000) = 396$, $T = 293$ K. $R = 0.048$ for 1981 observed reflections. The pyridine moiety is planar. The cyclohexene ring has five atoms coplanar and C(7) deviating by 0.67 (1) Å

from this plane. The presence of Cl atoms on vicinal C atoms induces strong van der Waals interactions which results in a staggered conformation and unusual bond lengths.

Introduction. The title compound was prepared by reacting 4-methoxy-2-methyl-6-acetamidoquinoline with sulfonyl chloride at 298 K for 48 h and the subsequent removal of sulfonyl chloride at 323-333 K (Moghe, Pol & Mhaskar, 1985). The structure was established by the X-ray studies.

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Experimental. Crystal $\sim 0.45 \times 0.30 \times 0.85$ mm; D_m by flotation; Nonius CAD-4F-11M diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan mode, scan speed 1° min^{-1} , $\theta \leq 23.5^\circ$, h 0 to 9, k -10 to 10, l -11 to 11. 2431 reflections collected, 1981 judged significant ($|F_o| > 3\sigma|F_c|$), lattice parameters from 23 reflections ($22 \leq 2\theta \leq 34^\circ$), three standard reflections (344, 454 and 156) every 2000 s, 2.5%

Table 1. Fractional atomic coordinates ($\times 10^4$) with e.s.d.'s in parentheses and equivalent isotropic temperature-factor coefficients (\AA^2)

$$B_{\text{eq}} = \frac{1}{3}(B_{11}a^2 + B_{22}b^2 + B_{33}c^2 + B_{12}ab + B_{13}ac + B_{23}bc).$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
N	1140 (5)	4882 (5)	8046 (4)	3.03
C(2)	1550 (6)	3653 (6)	8330 (5)	3.17
C(3)	3097 (6)	3623 (6)	8292 (5)	3.00
C(4)	4263 (5)	4883 (5)	7915 (4)	2.44
C(5)	5037 (5)	7537 (5)	7118 (4)	2.20
C(6)	4373 (6)	8805 (5)	6649 (5)	2.38
C(7)	2444 (6)	8284 (5)	6259 (5)	2.71
C(8)	1761 (5)	7464 (5)	7414 (4)	2.71
C(9)	2250 (5)	6075 (4)	7663 (4)	2.30
C(10)	3827 (5)	6154 (4)	7559 (3)	2.11
C(11)	233 (6)	2294 (6)	8703 (6)	4.91
C(12)	6334 (6)	3747 (6)	8244 (5)	4.07
O(1)	5809 (4)	4986 (4)	7863 (3)	3.31
O(2)	5290 (4)	10077 (4)	6582 (3)	3.73
Cl(1)	5642 (2)	6967 (1)	5663 (1)	3.27
Cl(2)	6881 (1)	8491 (1)	8486 (1)	3.26
Cl(3)	1905 (2)	9902 (2)	5951 (2)	4.39
Cl(4)	1601 (1)	6906 (1)	4744 (1)	3.66
Cl(5)	-418 (1)	6974 (1)	7093 (1)	4.08
Cl(6)	2795 (2)	8867 (1)	8961 (1)	3.75

Table 2. Bond lengths (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses

N—C(2)	1.341 (7)	N—C(9)	1.322 (6)
C(2)—C(3)	1.381 (8)	C(2)—C(11)	1.499 (8)
C(3)—C(4)	1.380 (7)	C(4)—O(1)	1.342 (6)
C(4)—C(10)	1.407 (6)	C(5)—C(6)	1.544 (7)
C(5)—C(10)	1.499 (6)	C(6)—C(7)	1.537 (8)
C(6)—O(2)	1.178 (6)	C(7)—C(8)	1.536 (6)
O(1)—C(12)	1.428 (7)	C(8)—C(9)	1.517 (6)
C(9)—C(10)	1.391 (7)	C(5)—Cl(1)	1.788 (4)
C(5)—Cl(2)	1.783 (4)	C(7)—Cl(3)	1.754 (5)
C(7)—Cl(4)	1.776 (5)	C(8)—Cl(5)	1.751 (5)
C(8)—Cl(6)	1.812 (4)		
C(2)—N—C(9)	117.9 (4)	N—C(2)—C(3)	122.6 (5)
N—C(2)—C(11)	116.4 (5)	C(3)—C(2)—C(11)	121.0 (5)
C(2)—C(3)—C(4)	119.3 (5)	C(3)—C(4)—O(1)	124.3 (4)
C(3)—C(4)—C(10)	118.8 (4)	O(1)—C(4)—C(10)	116.9 (4)
C(4)—O(1)—C(12)	119.3 (4)	Cl(1)—C(5)—Cl(2)	108.9 (2)
Cl(1)—C(5)—C(6)	103.5 (3)	Cl(1)—C(5)—C(10)	111.2 (3)
Cl(2)—C(5)—C(6)	106.4 (3)	Cl(2)—C(5)—C(10)	110.1 (3)
C(6)—C(5)—C(10)	116.3 (4)	C(5)—C(6)—C(7)	115.4 (4)
C(5)—C(6)—O(2)	122.0 (4)	C(7)—C(6)—O(2)	122.6 (5)
Cl(3)—C(7)—C(8)	109.7 (3)	Cl(3)—C(7)—Cl(4)	109.3 (3)
Cl(3)—C(7)—C(8)	112.6 (3)	C(6)—C(7)—Cl(4)	108.5 (3)
C(6)—C(7)—C(8)	108.5 (4)	Cl(4)—C(7)—C(8)	108.1 (3)
C(4)—O(1)—C(12)	119.3 (4)	C(7)—C(8)—Cl(5)	110.7 (3)
C(7)—C(8)—Cl(6)	106.3 (3)	C(7)—C(8)—C(9)	111.5 (4)
Cl(5)—C(8)—Cl(6)	108.5 (2)	Cl(5)—C(8)—C(9)	112.8 (3)
Cl(6)—C(8)—C(9)	106.7 (3)	N—C(9)—C(8)	115.9 (4)
N—C(9)—C(10)	124.4 (4)	C(8)—C(9)—C(10)	119.6 (4)
C(4)—C(10)—C(5)	120.8 (4)	C(4)—C(10)—C(9)	117.0 (4)
C(5)—C(10)—C(9)	122.2 (4)		

variation in intensity. No corrections for absorption. Structure solved by direct methods using MULTAN78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least-squares refinement of scale factor, positional and anisotropic thermal parameters (isotropic thermal parameters for H atoms, initial H positions calculated) converged to $R = 0.048$ and $wR = 0.047$, $S = 1.25$, $\sum w(|F_o| - |F_c|)^2$ minimized and $w = (4.0 + 1.0|F_o| + 0.03|F_o|^2)^{-1}$. $(\Delta/\sigma)_{\text{max}} = 0.1$. Final $\Delta\rho$ excursions $< 10.2 \text{ e \AA}^{-3}$. No correction for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Program LALS (Gantzel, Sparks & Trueblood, 1961) used for refinement.

Discussion. The atomic coordinates with their e.s.d.'s and equivalent isotropic temperature factors are given in Table 1.* Bond lengths and bond angles involving the non-H atoms are in Table 2. Fig. 1 shows the numbering scheme of the atoms and Fig. 2 shows a perspective view of the molecule.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and selected torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42446 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

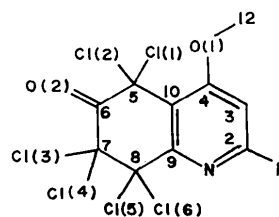


Fig. 1. Atomic numbering.

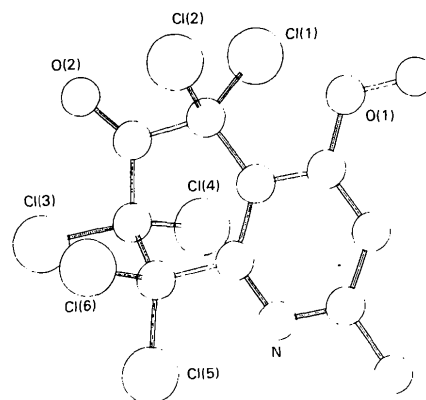


Fig. 2. Perspective view of the molecule.

The pyridine moiety is essentially planar (within 0.01 Å), while in the cyclohexene moiety C(7) is 0.67 (1) Å away from the plane formed by C(5), C(6), C(8), C(9) and C(10). The dihedral angle between the mean planes of the two rings is 9.7 (5)°. The presence of *gem*-dichloro substituents at C(5), C(7) and C(8) induces a considerable amount of strain in the molecule. This strain is mainly distributed in the vicinity of the bulky Cl substituents. The distances between Cl atoms attached to the same C atom are 2.906 (1), 2.880 (2) and 2.892 (2) Å for Cl(1)···Cl(2), Cl(3)···Cl(4) and Cl(5)···Cl(6), respectively, similar to the value in hexachlorocyclohexa-2,5-dienone of 2.897 (1) Å (Gali, Miravittles & Font-Altaba, 1975). The distances Cl(3)···Cl(5), Cl(3)···Cl(6) and Cl(4)···Cl(5) are 3.191 (2), 3.248 (2) and 3.273 (1) Å, which minimize van der Waals repulsive interactions between neighbouring Cl atoms. This effect also results in a considerable shortening of the bonds C(7)—Cl(3) = 1.754 (5) Å and C(8)—Cl(5) = 1.751 (5) Å as compared to the standard C(sp³)—Cl distance of 1.781 (1) Å (Lide, 1962), and a lengthening of the C(8)—Cl(6) distance to 1.812 (4) Å as in perchlorocyclopentadiene (Chang & Bauer, 1971) and *trans*-2,2,3,4,5,6-hexachloro-6-methylcyclohex-3-enone (Hartshorn, Martyn & Vaughan, 1984). Due to the presence of the highly negative Cl substituents at vicinal

C atoms, the C=O distance [1.178 (6) Å] is slightly shortened (Hartshorn *et al.*, 1984).

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Structure and Disorder in Two Isomeric Racemic Cyclooctatriene Compounds

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Abstract. The crystal structures of *trans*-5a,11a-dihydro-2,3-dimethylcycloocta[*b*][1,4]benzodithiin (I) and 8,9-dihydro-2,3-dimethylcycloocta[*b*][1,4]benzodithiin (II) are reported. {These IUPAC names show the relationship between (I) and (II); (I) was called 10,11-(4,5-dimethylbenzo)-9,12-dithia-*trans*-

bicyclo[6.4.0]dodeca-2,4,6,10-tetraene in a previous structure determination [Kaiser, Richter, Moegel & Schroth (1979). *Tetrahedron*, **35**, 505–509].} (I) C₁₆H₁₆S₂, *M_r* = 272.4, monoclinic, *P*2₁/*c*, *a* = 10.724 (2), *b* = 6.542 (4), *c* = 20.435 (3) Å, β = 101.28 (1)°, *V* = 1405.95 Å³, *Z* = 4, *D_x* = 1.29 g cm⁻³, λ(Mo Kα) = 0.71069 Å, μ = 3.09 cm⁻¹, *F*(000) = 576, *T* = 293 K, *R* = 0.048 for 2779 unique

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