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

<table>
<thead>
<tr>
<th>Parameter definition</th>
<th>Values for the title lactam molecule A</th>
<th>Values for the title lactam molecule B</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \omega_1 = \omega(C(6) - C(5) - N(4) - C(3)) )</td>
<td>-3.5 (4)</td>
<td>-1.8 (4)</td>
</tr>
<tr>
<td>( \omega_2 = \omega(O(1) - C(5) - N(4) - H(5)) )</td>
<td>5 (2)</td>
<td>0 (2)</td>
</tr>
<tr>
<td>( \omega_3 = \omega(O(1) - C(5) - N(4) - C(3)) )</td>
<td>175.6 (2)</td>
<td>178.7 (3)</td>
</tr>
<tr>
<td>( \omega_4 = \omega(C(6) - C(5) - N(4) - H(5)) )</td>
<td>-174 (2)</td>
<td>179 (2)</td>
</tr>
</tbody>
</table>

\[
\tau' = 2\tau = \omega_1 + \omega_2 \\
\chi_C = \omega_1 - \omega_3 + \pi \quad \text{(mod 2\pi)} \\
\chi_N = \omega_2 - \omega_3 + \pi
\]

These parameters along with the statistical \( \chi^2 \) 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 \( \chi_N \) for molecule A. It corresponds to out-of-plane bending at the N(4A) atom. Values of \( \chi_C \) 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 \( \tau' \). 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.

The authors thank Drs J. Smoliková and E. Körbllová for kindly providing the crystals and Dr K. Huml for helpful discussion.

**References**


Experimental. Crystal ~0.45 x 0.30 x 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, \( k \) to 10, \( l \) to 11. 2431 reflections collected, 1981 judged significant (\( |F_o| > 3\sigma|F_c| \)), lattice parameters from 23 reflections (22 < 2\( \theta \) < 34°), 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 (\( \AA^2 \))

<table>
<thead>
<tr>
<th>( x )</th>
<th>( y )</th>
<th>( z )</th>
<th>( B_{eq} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>N</td>
<td>1140 (5)</td>
<td>4882 (5)</td>
<td>8046 (4)</td>
</tr>
<tr>
<td>C(2)</td>
<td>1550 (6)</td>
<td>3653 (6)</td>
<td>8330 (5)</td>
</tr>
<tr>
<td>C(3)</td>
<td>3097 (6)</td>
<td>3623 (6)</td>
<td>8292 (5)</td>
</tr>
<tr>
<td>C(4)</td>
<td>4263 (5)</td>
<td>4883 (5)</td>
<td>7915 (4)</td>
</tr>
<tr>
<td>C(5)</td>
<td>5037 (5)</td>
<td>7537 (5)</td>
<td>7118 (4)</td>
</tr>
<tr>
<td>C(6)</td>
<td>4373 (6)</td>
<td>8805 (5)</td>
<td>6649 (5)</td>
</tr>
<tr>
<td>C(7)</td>
<td>2444 (6)</td>
<td>8284 (5)</td>
<td>6259 (5)</td>
</tr>
</tbody>
</table>

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.](image1)

![Fig. 2. Perspective view of the molecule.](image2)
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 C1 substituents. The distances between C1 atoms attached to the same C atom are 2.906 (1), 2.880 (2) and 2.892 (2) Å for C1(1)...C1(2), C1(3)...C1(4) and C1(5)...C1(6), respectively, similar to the value in hexachlorocyclohexa-2,5-dienone of 2.897 (1) Å (Gali, Miravitlles & Font-Altaba, 1975). The distances C1(3)...C1(5), C1(3)...C1(6) and C1(4)...C1(5) are 3.191 (2), 3.248 (2) and 3.273 (1) Å, which minimize van der Waals repulsive interactions between neighbouring C1 atoms. This effect also results in a considerable shortening of the bonds C(7)—C(3) = 1.754 (5) Å and C(8)—C(5) = 1.751 (5) Å as compared to the standard C(sp3)—C distance of 1.781 (1) Å (Lide, 1962), and a lengthening of the C(8)—C(6) distance to 1.812 (4) Å as in perchlorocyclopentadiene (Chang & Bauer, 1971) and trans-2,2,3,4,5,6-hexachloro-6-methylocyclohex-3-enone (Hartshorn, Martyn & Vaughan, 1984). Due to the presence of the highly negative C1 substituents at vicinal C atoms, the C=O distance [1.178 (6) Å] is slightly shortened (Hartshorn et al., 1984).

We thank Drs P. P. Moghe, A. V. Pol and V. V. Mhaskar for crystals, and Drs A. P. B. Sinha, L. M. Pant and V. G. Puranik for discussion and encouragement.

References


Structure and Disorder in Two Isomeric Racemic Cyclooctatriene Compounds

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Abstract. The crystal structures of trans-5a,11a-dihydro-2,3-dimethylcyclooctactyl[b]1,4]benzodithiin (I) and 8,9-dihydro-2,3-dimethylcyclooctactyl[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, Mr = 272.4, monoclinic, P21/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 = 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