Crystal and Molecular Structure of a Fifteen-membered-ring Compound: 5,9-Dioxo-1,7,13-trioxa-4,10-diazacyclopentadecane

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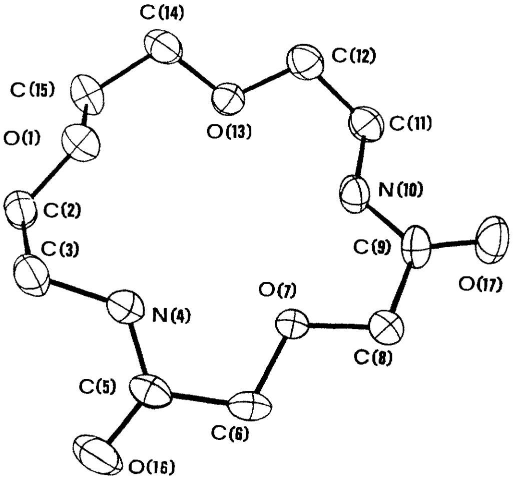
Summary The symmetry of the fifteen-membered cyclic diamide, 5,9-dioxo-1,7,13-trioxa-4,10-diazacyclopentadecane is almost $C2$: the amido-groups are *trans* and lie in the general plane of the molecule.

Interest in the formation of the macrocyclic complexes with diazapolyoxa-macrocyclic ligands[1] and also in the conformations of odd-membered rings[2] stimulated a crystallographic study of the cyclic diamide 5,9-dioxo-1,7,13-trioxa-4,10-diazacyclopentadecane.



Figure

Crystals were grown by slow evaporation of a dilute solution of the compound in $CHCl_3$–C_6H_6. *Crystal data*: $C_{10}H_{18}N_2O_5$, M 246·3, monoclinic, $a = 7{\cdot}951 \pm 0{\cdot}008$, $b = 9{\cdot}252 \pm 0{\cdot}009$, $c = 17{\cdot}92 \pm 0{\cdot}02$ Å, $\beta = 112{\cdot}9 \pm 0{\cdot}1°$, $U = 1214$ Å^3, $Z = 4$, $D_m = 1{\cdot}35$, $D_c = 1{\cdot}346$ g cm^{-3}, space group $P2_1/c$.

The structure analysis was based on 867 independent reflections recorded on a PAILRED diffractometer using Cu-K_α radiation ($\lambda = 1{\cdot}5418$ Å). The structure has been determined by the symbolic addition procedure[3] and Fourier methods. The final values of the conventional and weighted discrepancy factors, after full-matrix least-squares refinement[4] of all non hydrogen atoms were R 0·056 and R_w 0·067. The molecule is shown in the Figure.[5] C–C bond lengths range from 1·487(7)—1·518(7); C–O bond lengths 1·405(6)—1·424(6); C–N bond lengths 1·445(6)—1·468(7) Å. The C–N and C=O bond lengths in the amido-groups range, respectively, from 1·320(7)—1·326(8) and from 1·216(6)—1·224(7) Å.

The fifteen-membered ring is almost planar and the amido-groups lie in the general plane of the molecule and not perpendicular to it as in several other examples.[6] Moreover, as the two amido-groups are *trans*, the hydrogen atoms bonded to N(4) and N(10) are located inside the ring and cannot give rise to N–H · · · O=C intermolecular hydrogen bonds which are often observed in this kind of compound.[6]

On the other hand, the distances between O(7) and the nitrogen atoms show that N(4) and O(7) as well as N(10) and O(7) are linked together by hydrogen bonds. It is possible that these bonds are bifurcated owing to the fact that the

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N(4)–O(1) and N(10)–O(13) distances are also short. However, the last two distances are significantly greater than the N(4)–O(7) and N(10)–O(7) distances, as shown by the values: N(4)–O(7) = 2·576(5), N(4)–O(1) = 2·734(6), N(10)–O(7) = 2·544(5), and N(10)–O(13) = 2·686(5) Å.

The torsional angles about the ring bonds are listed in the Table.

Although the symmetry of the conformation is very near to *C*2, the underlined values in the Table show that the ring deviates significantly from this symmetry.

Torsional angles[a] (°)			
C(6)–O(7)	166·9	O(7)–C(8)	163·7
C(5)–C(6)	−12·1	C(8)–C(9)	2·2
N(4)–C(5)	178·5	C(9)–N(10)	−177·1
C(3)–N(4)	−123·2	N(10)–C(11)	−147·4
C(2)–C(3)	−56·5	C(11)–C(12)	−51·6
O(1)–C(2)	168·2	C(12)–O(13)	173·3
O(15)–O(1)	−165·1	O(13)–C(14)	−170·2
	C(14)–C(15)	73·8	

[a] The signs are given according to the convention of Klyne and Prelog;[7] standard deviations[8] range from 0·3—0·4°.

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