about the xy plane at $z=\frac{1}{2}$. The lysine molecules in the former are held together by hydrogen bonds involving the α-amino and the α-carboxylate groups belonging to them whereas the latter are stabilized by hydrogen bonds between the α-amino group and the α- and side-chain carboxylates belonging to the aspartate groups in the layer. The two layers are interconnected primarily by three hydrogen bonds made by the side-chain amino group of lysine with O atoms belonging to the neighbouring aspartate groups. There is also an additional hydrogen bond between the α-amino N atom of lysine and an α-carboxylate O atom of the aspartate group.

References

BHAT, T. N. & VIJAYAN, M. (1974). *Curr. Sci.* **43**, 276–277.

BUGAYONG, R. R., SEQUEIRA, A. & CHIDAMBARAM, R. (1972). *Acta Cryst.* B**28**, 3214–3219.

CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.

CRUICKSHANK, D. W. J. (1949). *Acta Cryst.* **2**, 65–82.

DERISSEN, J. L., ENDEMAN, H. J. & PEERDEMAN, A. F. (1968). *Acta Cryst.* B**24**, 1349–1354.

IUPAC-IUB COMMISION ON BIOCHEMICAL NOMENCLATURE (1970). *J. Mol. Biol.* **52**, 1–17.

KARLE, J. & KARLE, I. L. (1966). *Acta Cryst.* **21**, 849–859.

KOETZLE, T. F., LEHMANN, M. S., VERBIST, J. J. & HAMILTON, W. C. (1972). *Acta Cryst.* B**28**, 3207–3214.

RAO, S. T. (1973). *Acta Cryst.* B**29**, 1718–1720.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

VIJAYAN, M. (1975). In *Handbook of Biochemistry and Molecular Biology*, 3rd ed., edited by G. D. FASMAN. Cleveland, Ohio: CRC Press.

WRIGHT, D. A. & MARSH, R. E. (1962). *Acta Cryst.* **15**, 54–64.

The Crystal Structures of Two Tetracyclic Spirodilactams Containing Non-Planar Amide Bonds

BY S. E. EALICK, D. M. WASHECHECK AND DICK VAN DER HELM*

Department of Chemistry, University of Oklahoma, Norman, Oklahoma 73069, U.S.A.

The crystal structures of 1,4-diaza-5,12-dioxotetracyclo[5,5,1,0^{4,13},0^{10,13}]tridecane, $C_{11}H_{14}N_2O_2$, and 1,5-diaza-6,13-dioxotetracyclo[6,5,1,0^{5,14},0^{11,14}]tetradecane, $C_{12}H_{16}N_2O_2$, have been determined and refined by three-dimensional least-squares techniques. Both molecules crystallize in space group $C2/c$ and have similar unit-cell dimensions. Unit-cell dimensions for the former are $a = 13\cdot6008$ (4), $b = 8\cdot3177$ (2), $c = 10\cdot0596$ (2) Å and $\beta = 120\cdot563$ (2)° while those for the latter are $a = 13\cdot7523$ (3), $b = 8\cdot9985$ (2), $c = 10\cdot2316$ (2) Å and $\beta = 120\cdot527$ (1)°. The final R values are 3·7% for the tridecane derivative and 3·9% for the tetradecane derivative. Each molecule lies on the twofold rotation axis of space group $C2/c$. The amide group in each molecule is non-planar even though the conversion of a five-membered ring to a six-membered ring in the tetradecane derivative releases a certain amount of the strain.

Introduction

The geometry of the non-planar amide group (or the non-planar peptide group in biological systems) has been the subject of several theoretical and experimental studies in the last few years (Warshel, Levitt & Lifson, 1970; Winkler & Dunitz, 1971; Ramachandran, Lakshminarayanan & Kolaskar, 1973; Ramachandran & Kolaskar, 1973; Dunitz & Winkler, 1975). The present study deals with two compounds of a series of polycyclic spirodilactams. The syntheses and infrared

spectra of these compounds have been published (Smolikova, Koblicova & Bláha, 1973). The first compound of this series, a tricyclic spirodilactam, and a preliminary study of the present tetracyclic compounds have been reported (Ealick & van der Helm, 1975; van der Helm, Ealick & Waschecheck, 1975). The occurrence of both *cis* and *trans* non-planar peptide groups has been observed in cyclic and acyclic molecules (*e.g.* Sletten, 1970; Winkler & Dunitz, 1971; Pedone, Benedetti, Immirzi & Allegra, 1970). One must also consider the occurrence of the non-planar peptide group in protein molecules. In the present paper we present the description of the structures of two molecules containing non-planar amide bonds.

Experimental

Crystalline samples of 1,4-diaza-5,12-dioxotetracyclo-[5,5,1,0^{4,13},0^{10,13}]tridecane (I) and 1,5-diaza-6,13-dioxo-tetracyclo[6,5,1,0^{5,14},0^{11,14}]tetradecane (II) were supplied by Professor K. Bláha, Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, Prague. Suitable crystals of (I) were obtained by slow cooling of an ethanolic solution of the compound. The crystal used for the diffraction experiment was in the shape of an octahedron with maximum dimensions 0·55 × 0·45 × 0·30 mm. Crystals of (II) were obtained by equilibrating a saturated benzene solution of the compound with hexane. The crystal selected for data collection had the shape of a rectangular prism with dimensions 0·32 × 0·24 × 0·12 mm. Integrated intensity data and crystallographic data (Table 1) were collected on a Nonius CAD-4 automatic diffractometer. For intensity data Ni-filtered Cu $K\bar{\alpha}$ radiation ($\lambda = 1\cdot5418$ Å) was used and for determination of unit-cell parameters Cu $K\alpha_1$ radiation ($\lambda = 1\cdot54051$ Å) was used. 1011 intensities for (I) and 1129 for (II), comprising all unique data with $2\theta < 150°$, were measured by θ–2θ scans. The θ-scan width was calculated as $(1\cdot0 + 0\cdot1 \tan \theta)°$. A variable receiving aperture was used with a width of $(4\cdot0 + 0\cdot4 \tan \theta)$ mm; the height was constant at 6 mm.
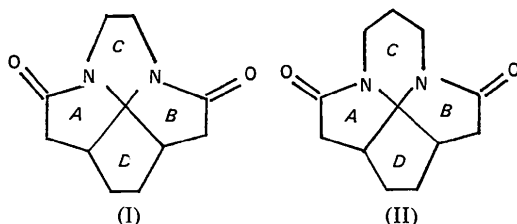

(I) (II)

Table 1. Crystallographic data

	(I)	(II)
Formula	$C_{11}H_{14}N_2O_2$	$C_{12}H_{16}N_2O_2$
F.W.	206·25	220·27
Space group	$C2/c$	$C2/c$
a	13·6008 (4) Å	13·7523 (3) Å
b	8·3177 (2)	8·9985 (2)
c	10·0596 (4)	10·2316 (2)
β	120·563 (2)°	120·527 (1)°
V	979·91 Å^3	1090·66 Å^3
Z	4	4
D_o	1·400 g cm^{-3}	1·336 g cm^{-3}
D_c	1·398	1·341

The cell dimensions were determined from the least-squares fit to $+2\theta$ and -2θ values of 38 reflections for (I) and 48 reflections for (II) at 27°C. The reflections were taken from all quadrants of reciprocal space.

For each structure three data sets were collected with a variable scan speed so that a maximum time of 60 s was spent on each individual intensity with $\frac{2}{3}$ of the time used for scanning the peak and $\frac{1}{6}$ of the time used for scanning each of the high-θ and low-θ backgrounds. An intensity was considered indistinguishable from the background when $I < 1\cdot4\sigma(I)$ and was assigned a value equal to the square root of the total count (peak plus twice the left and right background) for the purpose of least-squares refinement. The three separate data sets for each compound were averaged together, a reflection being excluded if it differed from the average by more than three standard deviations $[\sigma(I)]$. The R values $(= \sum|\Delta I|/\sum I)$ for the data sets were 0·0079, 0·0069 and 0·0095 for compound (I), and 0·0083, 0·0083 and 0·0086 for compound (II). Absorption corrections were applied to the averaged intensities $[\mu = 8\cdot080$ cm^{-1} for (I) and $\mu = 7\cdot598$ cm^{-1} for (II)]. Lorentz and polarization corrections were made and

Table 2. Positional parameters ($\times 10^5$) and thermal parameters (Å$^2 \times 10^4$) for carbon, oxygen and nitrogen atoms of (I) and (II)

Calculated standard deviations for the last digit are listed in parentheses. Thermal parameters are of the form
$$\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)].$$

(I)	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	0	42182 (17)	25000	269 (6)	282 (6)	296 (6)	0	150 (5)	0
C(2)	8072 (9)	53363 (13)	38520 (12)	297 (5)	343 (5)	318 (5)	−39 (4)	144 (4)	−34 (4)
C(3)	19552 (9)	51265 (15)	39303 (14)	270 (5)	425 (6)	450 (6)	−30 (4)	140 (5)	19 (5)
C(4)	18840 (9)	34933 (16)	32305 (14)	305 (6)	476 (6)	404 (6)	69 (5)	190 (5)	57 (5)
C(5)	3062 (11)	14811 (15)	20347 (15)	458 (6)	330 (5)	404 (6)	33 (5)	187 (5)	−31 (4)
C(6)	2764 (11)	70095 (15)	33803 (16)	404 (6)	329 (5)	528 (7)	−28 (4)	215 (6)	−89 (5)
N	7517 (8)	31221 (12)	22877 (10)	315 (4)	338 (4)	347 (5)	51 (4)	170 (4)	7 (4)
O	26793 (8)	26322 (15)	34421 (14)	385 (5)	712 (7)	681 (6)	182 (4)	281 (5)	11 (5)
(II)									
C(1)	0	38972 (14)	25000	391 (6)	300 (6)	422 (7)	0	244 (6)	0
C(2)	8150 (9)	49276 (12)	38181 (11)	475 (6)	378 (5)	428 (5)	−74 (4)	254 (4)	−34 (4)
C(3)	19416 (10)	47461 (14)	38752 (15)	411 (6)	508 (6)	654 (7)	−72 (5)	242 (5)	−22 (5)
C(4)	18420 (9)	33193 (14)	30465 (15)	420 (6)	518 (6)	635 (7)	63 (5)	286 (5)	54 (5)
C(5)	3004 (11)	15151 (13)	15147 (15)	597 (6)	396 (6)	619 (7)	63 (5)	270 (6)	−112 (5)
C(6)	0	5645 (17)	25000	560 (9)	317 (7)	631 (10)	0	97 (8)	0
C(7)	2919 (11)	64727 (12)	33638 (14)	650 (7)	357 (5)	637 (7)	−52 (5)	406 (6)	−90 (5)
N	7374 (7)	29518 (10)	22313 (11)	421 (4)	357 (4)	546 (5)	45 (3)	259 (4)	−38 (4)
O	26111 (8)	26116 (13)	30786 (14)	505 (5)	849 (7)	1079 (9)	176 (5)	430 (6)	−47 (7)

individual structure factor amplitudes were derived. Each amplitude was assigned an experimental weighting scheme, based on counting statistics, that has been described previously (van der Helm, Ealick & Burks, 1975).

Structure determination and refinement

The structure of (II) was determined by the symbolic addition procedure (Zachariasen, 1952; Karle & Karle, 1966). The intensities were placed on an absolute scale using the results of a Wilson plot (Wilson, 1942) and normalized structure factors, $|E|$, were derived. An analysis of the distribution of $|E|$'s indicated a center of symmetry and the space group was taken to be $C2/c$ rather than Cc. The signs of 159 E's with $|E| > 1.5$ were generated leading to an E map which revealed the positions of all nine non-hydrogen atoms.

The structure was refined by block-diagonal (9×9) least-squares methods in which the quantity $\sum w_F(kF_o - F_c)^2$ was minimized. The non-hydrogen atoms were given anisotropic temperature factors and the refinement proceeded to an $R = \sum ||kF_o| - |F_c||/\sum |kF_o|$ of 0.11. At this point a difference Fourier map was calculated in which the positions of the eight H atoms were located. After several further cycles of least-squares refinement a comparison of F_o's and F_c's indicated the presence of minor secondary extinction effects. The observed intensities were corrected for secondary extinction as follows: $I_c = I_o \exp(-CI_o)$, where $I_c =$ corrected intensity, $I_o =$ observed intensity and $C = 8.2 \times 10^{-7}$. The least-squares refinement was terminated when all shifts for non-hydrogen atoms were small fractions of the corresponding estimated standard deviation. The R value based on final parameters (Tables 2 and 3) for all data is 0.039.

The present two structures have very similar unit-cell dimensions (Table 1) and the molecular structures differ by only one $-CH_2-$ group. Therefore the structure of (I) was determined by assuming that the molecule had the same orientation and location as structure (II). Idealized coordinates were calculated using (II) as a template and a structure factor calculation was

Table 3. *Positional parameters* $(\times 10^3)$ *and isotropic thermal parameters for hydrogen atoms in* (I) *and* (II)

(I)	x	y	z	B (Å^2)
H(C2)	87 (1)	498 (2)	482 (1)	3·3 (3)
H(C3)A	262 (1)	509 (2)	502 (2)	4·8 (4)
H(C3)B	208 (1)	596 (2)	331 (2)	3·7 (3)
H(C5)A	95 (1)	70 (2)	245 (2)	4·9 (4)
H(C5)B	−24 (1)	126 (2)	96 (2)	3·8 (3)
H(C6)A	−36 (2)	718 (2)	363 (2)	4·9 (4)
H(C6)B	84 (2)	788 (2)	389 (2)	4·5 (4)
(II)				
H(C2)	89 (1)	460 (2)	481 (2)	5·0 (3)
H(C3)A	208 (1)	556 (2)	336 (2)	5·7 (3)
H(C3)B	259 (1)	463 (2)	495 (2)	6·0 (4)
H(C5)A	−42 (1)	164 (2)	47 (2)	5·6 (3)
H(C5)B	86 (1)	104 (2)	137 (2)	5·8 (4)
H(C6)	−66 (1)	−9 (2)	184 (2)	6·3 (4)
H(C7)A	87 (1)	726 (2)	387 (2)	4·9 (3)
H(C7)B	−28 (1)	664 (2)	368 (2)	5·0 (3)

made. Although the initial R value was 0.65 the refinement proceeded quickly and converged at $R = 0.15$ for the isotropic model. The non-hydrogen atoms were made anisotropic and after several more cycles of refinement a difference Fourier map revealed positions of the seven H atoms. After refinement converged corrections were made for secondary extinction effects $(C = 2.91 \times 10^{-6})$. The refinement was terminated when all shifts for non-hydrogen atoms were small fractions of the corresponding estimated standard deviations. The R value based on final parameters (Tables 2 and 3) is 0.037.

Final difference Fourier maps for structures (I) and (II) showed no peaks larger than 0.2 e Å^{-3} nor less than -0.2 e Å^{-3}. All least-squares refinements were done with the block-diagonal least-squares program of Ahmed (1966). The absorption correction program was that of Coppens, Leiserowitz & Rabinovich (1965). The atomic scattering factors for O, N and C were from *International Tables for X-ray Crystallography* (1962) while scattering factors for H atoms were those of Stewart, Davidson & Simpson (1965).

In the structure factor analysis for both structures (I) and (II) the average values of $w_F \Delta F^2$ did not show a
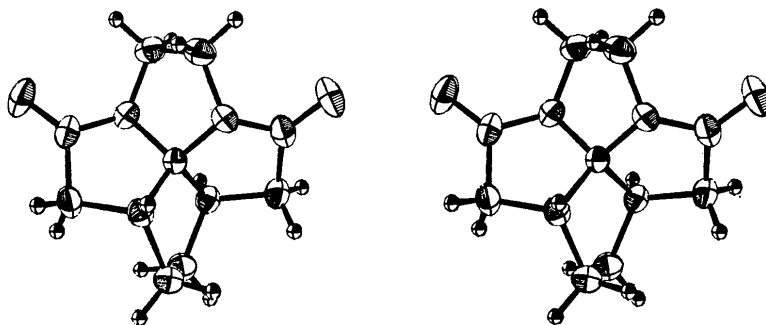

Fig. 1. Stereo view of (I) (Johnson, 1965).

significant variation with either $|F_o|$ or $\sin \theta/\lambda$ indicating that the weighting scheme used was reasonable.*

Description and discussion of the structure

Stereo views of single molecules of (I) and (II) are shown in Figs. 1 and 2 respectively. (I) consists of four five-membered rings fused together at a central quaternary C atom. The molecule contains a twofold axis which is a symmetry element of the space group and which passes through atom C(1) and bisects the bonds C(5)–C(5') and C(6)–C(6'). (II) also has point symmetry $C2$ with the twofold axis passing through atoms C(1) and C(6) and bisecting bond C(7)–C(7').

In (I) rings C and D are in the twist conformation as required by the presence of a twofold axis passing through the rings. The conformation of ring $A(B)$ is intermediate between twist (C_2) and envelope (C_s). This can best be seen through the use of the pseudorotation parameter Δ (Altona, Geise & Romers, 1968) for which $\Delta=0°$ for the C_2 conformation and $\Delta= \pm 36°$ for the C_s conformation. The value of Δ for ring $A(B)$ of (I) is 17·5°. In (II) all five-membered rings are in the C_2 conformation. Ring D lies on the twofold axis while the value of Δ for ring $A(B)$ is 1·6°. The conformation of the six-membered ring (C) is best described as twist-boat. Comparison of the $A(B)$ rings indicates a release of strain in (II). As the C ring is changed from a five-membered to a six-membered ring, ring $A(B)$ adopts a more normal twist conformation and the amide group becomes more planar. Fig. 3 lists the values of the torsion angles for both molecules and Table 4 lists the theoretical values for cyclopentane in the twist and envelope conformations (Ouannes & Jacques, 1965) and cyclohexane in the twist-boat conformation

---

* The final F_o, F_c tables for (I) and (II) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31333 (13 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 4. *Theoretical values for the twist and envelope conformations of cyclopentane (Ouannes & Jacques, 1965) and the twist-boat conformation of cyclohexane (Hendrickson, 1961)*

	Cyclopentane		Cyclohexane
	Twist (C_2)	Envelope (C_s)	Twist-boat
φ_1	15·1	46·1	33·2
φ_2	−39·4	−28·6	−70·6
φ_3	48·1	0	33·2
φ_4	−39·4	28·6	33·2
φ_5	15·1	−46·1	−70·6
φ_6			33·2
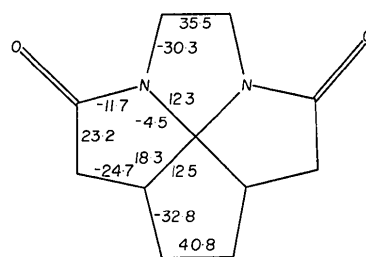
(I)
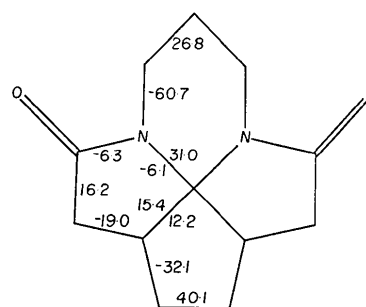
(II)

Fig. 3. Torsion angles for (I) and (II). Standard deviations range from 0·10° for torsion angles involving uncorrelated atoms to 0·24° for angles containing a twofold axis.
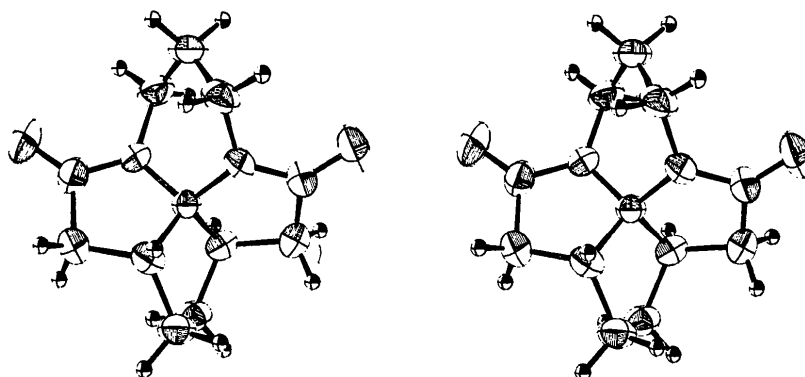

Fig. 2. Stereo view of (II) (Johnson, 1965).

(Hendrickson, 1961). Considerable flattening of all rings is observed compared to the expected conformations of the carbocyclic analogs. The flattening results from changes in bond distances and angles brought about by the inclusion of the amide group in the ring and the tendency for this group to be planar.

Bond distances and angles for (I) and (II) are given in Figs. 4 and 5 respectively. The most interesting distances are those involving atoms of the amide groups. The amide bond distance [C(4)–N] is lengthened while the carbonyl bond distance [C(4)–O] is shortened from the expected values for planar amide groups (Ramachandran, Kolaskar, Ramakrishnan & Sasisekharan, 1974). The C–H bond distances range from 0·97 to 1·03 Å with an average value of 1·00 Å for (I) and from 0·96 to 1·03 Å with an average value of 1·00 Å for (II).

Rigid-body corrections were carried out for each molecule by the method of Schomaker & Trueblood (1968) with the appropriate restrictions imposed for a molecule of point symmetry $C2$. The average ΔU_{ij}

was 0·0016 Å^2 and r.m.s. U_{ij} 0·0005 Å^2 for (I) and the average ΔU_{ij} was 0·0024 Å^2 and r.m.s. U_{ij} 0·0006 Å^2 for (II). The rigid-body model for both molecules is acceptable only at the limits of statistical significance. It should be noted, however, that the carbonyl O atoms show some independent thermal motion in both compounds. The bond distances corrected for rigid-body thermal motion are given in Table 5.

Table 5. *Bond distances corrected for rigid-body thermal motion*

	(I)	(II)
C(1)–C(2)	1·555	1·555
C(1)–N	1·470	1·460
C(2)–C(3)	1·538	1·536
C(2)–C(6)	1·532	–
C(2)–C(7)	–	1·530
C(3)–C(4)	1·516	1·512
C(4)–N	1·375	1·356
C(4)–O	1·225	1·224
C(5)–C(5')	1·543	–
C(5)–C(6)	–	1·537
C(5)–C(N)	1·467	1·462
C(6)–C(6')	1·537	–
C(7)–C(7')	–	1·532

There are three short intermolecular contacts which should be pointed out: O$\cdots$H(C3)B $(\frac{1}{2}-x, -\frac{1}{2}+y, \frac{1}{2}-z)$ of 2·40 Å in (I) and O$\cdots$H(C5)A $(\frac{1}{2}-x, -\frac{1}{2}+y, \frac{1}{2}-z)$ of 2·52 Å and H(C5)$B\cdots$H(C7)B $(\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z)$ of 2·23 Å in (II). The corresponding sums of the van der Waals radii are 2·6, 2·6 and 2·4 Å respectively.
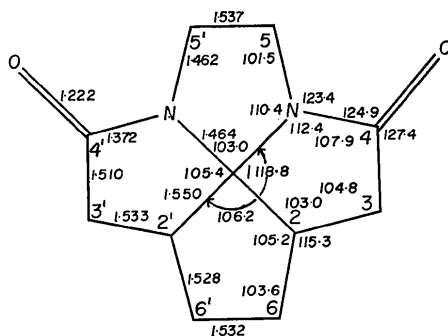

Fig. 4. Bond distances and angles for (I). Standard deviations are 0·0014–0·0018 Å for bond distances involving uncorrelated atoms and 0·0032 Å for the C(5)–C(5') and C(6)–C(6') bonds. Standard deviations for bond angles are 0·1°.
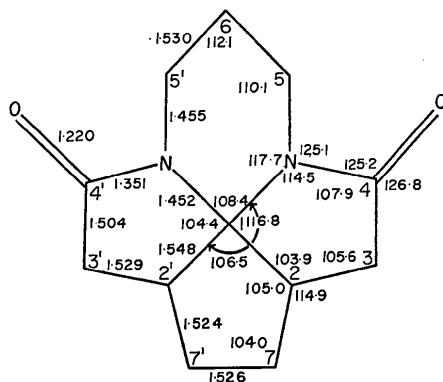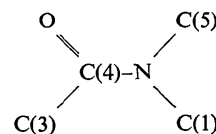

Fig. 5. Bond distances and angles for (II). Standard deviations are 0·0012–0·0019 Å for bond distances involving uncorrelated atoms and 0·0028 Å for the C(7)–C(7') bond. Standard deviations for bond angles are 0·1°.

Table 6. *Conformational parameters for non-planar amide groups of* (I) *and* (II)

For further definition of symbols see Winkler & Dunitz (1971).

Parameter	Definition	(I)	(II)
ω_1	C(3)–C(4)–N–C(5)	−148·0 (1)	−167·1 (1)
ω_2	O——C(4)–N–C(1)	168·5 (1)	174·4 (1)
ω_3	O——C(4)–N–C(5)	32·2 (2)	13·5 (2)
ω_4	C(3)–C(4)–N–C(1)	−11·7 (1)	−6·3 (1)
τ'	$2\tau = \omega_1 + \omega_2$	20·5	7·3
χ_C	$\omega_1 - \omega_3 + \pi$ (mod 2π)	−0·2	−0·6
χ_N	$\omega_2 - \omega_3 + \pi$	−43·7	−19·1

The non-planar amide group

Conformational parameters for the non-planar amide groups are given in Table 6, following the convention outlined by Winkler & Dunitz (1971). By comparison it can be seen that the amide group of (II) is more planar than that of (I). This also appears to be reflected in bond distances as lengthening of the amide bond

occurs to a greater extent in (I), thus substantiating the expected correlation between non-planarity and the amide bond length. In addition the N–C(1) and N–C(5) bonds also become shorter as the amide group becomes more planar. It might also be pointed out that the values of χ_C in each molecule are very small while the major contribution to non-planarity comes from χ_N and τ' which is consistent with the predicted energy requirement for deformation of the amide group (Winkler & Dunitz, 1971).

The primary interest in this series of compounds is the predicted correlation of non-planarity with the wave number $v(C'-N)$ (Smolikova et al., 1973). From the results of these two structures and the first member of the series (Ealick & van der Helm, 1975) it appears that the suggested correlation of ω_3 and $v(C'-N)$ is a reasonable one and as more data become available the quantitative nature of these relationships will be explored.

References

AHMED, F. R. (1966). S. F. L. S. program, NRC-10, Ottawa, National Research Council.

ALTONA, C., GEISE, H. J. & ROMERS, C. (1968). Tetrahedron, 24, 13–32.

COPPENS, P., LEISEROWITZ, L. & RABINOVICH, D. (1965). Acta Cryst. 18, 1305–1308.

DUNITZ, J. D. & WINKLER, F. K. (1975). Acta Cryst. B31, 251–263.

EALICK, S. E. & VAN DER HELM, D. (1975). Acta Cryst. B31, 2676–2680.

HENDRICKSON, J. B. (1961). J. Amer. Chem. Soc. 83, 4537–4547.

International Tables for X-ray Crystallography (1962). Vol. III, p. 202. Birmingham: Kynoch Press.

JOHNSON, C. K. (1965). ORTEP. Oak Ridge National Laboratory Report ORNL-3794.

KARLE, J. & KARLE, I. L. (1966). Acta Cryst. 22, 849–859.

OUANNES, C. & JACQUES, J. (1965). Bull. Soc. Chim. Fr. pp. 3601–3610.

PEDONE, C., BENEDETTI, E., IMMIRZI, A. & ALLEGRA, G. (1970). J. Amer. Chem. Soc. 92, 3549–3552.

RAMACHANDRAN, G. N. & KOLASKAR, A. S. (1973). Biochim. Biophys. Acta, 303, 385–388.

RAMACHANDRAN, G. N., KOLASKAR, A. S., RAMARKISHNAN, C. & SASISEKHARAN, V. (1974). Biochim. Biophys. Acta, 359, 298–302.

RAMACHANDRAN, G. N., LAKSMINARAYANAN, A. V. & KOLASKAR, A. S. (1973). Biochim. Biophys. Acta, 303, 8–13.

SCHOMAKER, V. & TRUEBLOOD, K. N. (1968). Acta Cryst. B24, 63–76.

SLETTEN, E. (1970). J. Amer. Chem. Soc. 92, 172–177.

SMOLIKOVA, J., KOBLICOVA, Z. & BLÁHA, K. (1973). Coll. Czech. Chem. Commun. 38, 532–547.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). J. Chem. Phys. 42, 3175–3187.

VAN DER HELM, D., EALICK, S. E. & BURKS, J. E. (1975). Acta Cryst. B31, 1013–1018.

VAN DER HELM, D., EALICK, S. E. & WASCHECHECK, D. M. (1975). Abstract 6.1-12, Tenth Congress of the International Union of Crystallography. Acta Cryst. A31, S104.

WARSHEL, A., LEVITT, M. & LIFSON, S. (1970). J. Mol. Spectrosc. 33, 84–99.

WILSON, A. J. C. (1942). Nature, Lond. 150, 151–152.

WINKLER, F. K. & DUNITZ, J. D. (1971). J. Mol. Biol. 59, 169–182.

ZACHARIASEN, W. H. (1952). Acta Cryst. 5, 68–73.