ques différents de ceux observés pour les amines nitroaromatiques hydrogénées.

En conséquence, les différences observées entre les deux dérivés, au niveau des longueurs des liaisons relatives à l'atome d'azote du groupement amine d'une part, des déformations du cycle aromatique (en particulier l'angle C(6)–C(1)–C(2) dans le dérivé TFT) d'autre part, sembleraient dues pour une grande part aux contraintes engendrées par la substitution d'un atome de fluor par un groupement de fort encombrement stérique.

Il faut toutefois remarquer que, au contraire du dérivé TFT, il existe dans le composé étudié ici des interactions intermoléculaires non négligeables. Par conséquent, l'empilement dans le solide cristallin pourrait être un des facteurs important des différences observées. D'autres structures en cours donneront, en particulier, des indications de l'influence sur le groupement amine de substitutions sur le cycle aromatique.

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Medium Ring Compounds. XXVIII. 1,5-Diaza-6,10-cyclodecadione

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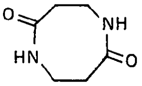
Crystals of the title compound are monoclinic, $a=6\cdot902$, $b=5\cdot017$, $c=12\cdot368$ Å, $\beta=99\cdot28°$, space group Pn, $Z=2$. Analysis of X-ray data in terms of an ordered crystal structure gave an R of 0·084. This could be reduced to 0·030 by introducing additional parameters to account for disorder. The ten-membered ring skeleton adopts an approximately centrosymmetric crown conformation and does not show the mirror symmetry that would be more in line with the structural formula. The two amide groups are thus structurally non-equivalent; they are both *transoid* and non-planar.

Introduction

In parallel with our studies of the conformations of medium-ring lactams (Winkler & Dunitz, 1971, 1975; Dunitz & Winkler, 1975) we have been interested in medium-ring compounds containing two amide groups. A previous publication (White & Dunitz, 1972) described the crystal and molecular structure of cyclodi-β-alanyl (1), where the ring adopts the 'flexible' chair conformation, with the two *cis*-amide groups related by a crystallographic dyad axis. Crystals of the higher homologue 1,6-diaza-2,7-cyclodecadione (2) are long, thin, fibrous needles that yield poor X-ray photographs. Preliminary investigation shows the crystals to be monoclinic, space group $P2_1/a$, $a=8\cdot03$, $b=11\cdot71$, $c=4\cdot84$ Å, $\beta=107\cdot2°$, $Z=2$ (Koyama & Dunitz, 1972). The molecule thus possesses a crystallographic centre of symmetry, and the translational repeat of 4·84 Å is strong evidence that the amide groups are *trans* (Winkler & Dunitz, 1971). However, the poor quality of the crystals discouraged us from carrying out a detailed analysis.



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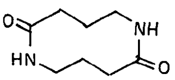

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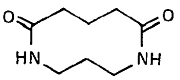

3

In this paper we describe an analysis of crystals of the isomeric diamide 1,5-diaza-6,10-cyclodecadione (3) formally derivable by condensation of 1,3-diaminopropane with glutaric acid. This compound is the only product obtained by Beckman rearrangement of cyclooctane-1,5-dioxime (Glover & Rapoport, 1964); in contrast to the corresponding rearrangement of analogous dioximes of other even-membered rings the

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centrosymmetric isomer 2 does not seem to be formed in this reaction.

Crystal data

$C_8H_{14}N_2O_2$; monoclinic, $a=6{\cdot}902$, $b=5{\cdot}017$, $c=12{\cdot}368$ Å, $\beta=99{\cdot}28°$, $U=422{\cdot}7$ Å^3, $Z=2$, $D_x=1{\cdot}34$ g cm^{-3}. Space group, *Pn* from systematic absences and structure analysis.

Experimental

Crystals suitable for X-ray analysis were obtained by slow evaporation of an isopropanol/acetic acid solution.

Intensities from a crystal of dimensions $0{\cdot}35\times0{\cdot}32\times0{\cdot}23$ mm were collected on an automated Hilger–Watts Y290 diffractometer with Mo $K\alpha$ radiation ($\lambda=0{\cdot}71069$ Å) monochromatized by reflexion from graphite. 726 reflexions were measured in the range $\theta\le25°$ and converted to $|F_o|$ and $|E|$ values in the usual way. Absorption corrections were neglected ($\mu=1{\cdot}04$ cm^{-1}).

Structure analysis

From the systematic absences ($h0l$, $h+l$ odd) the space group could be *Pn* or *P*2/*n*. Intensity statistics tests were inconclusive. The higher space group would require the molecules to be centrosymmetric, but this can be ruled out by the molecular constitution. However, since the possibility of a disordered structure had to be considered, the higher space group could not be rejected outright.

Application of the *MULTAN* program (Germain, Main & Woolfson, 1970) using 106 reflexions with $|E|\ge1{\cdot}4$ failed to yield a reasonable trial structure in the centrosymmetric space group *P*2/*n*. (We would have done better in $P2_1/n$!)

When we turned to the alternative space group *Pn*, the set of phases with the best figure of merit yielded an *E* map in which the 12 highest peaks corresponded to a well-defined 10-membered ring skeleton with two substituents in 1,5 positions, as expected from the structural formula. The distribution of peaks showed several interesting features:

(1) The 10-membered ring skeleton was approximately centrosymmetric.

(2) The two peaks corresponding to the O atoms (the strongest scatterers) were 11th and 12th in order of decreasing strength.

(3) The next strongest peaks in the *E* map (numbers 13 and 14) corresponded to two alternative positions for the O atoms, related to the first two positions by inversion across the approximate symmetry centre of the ring.

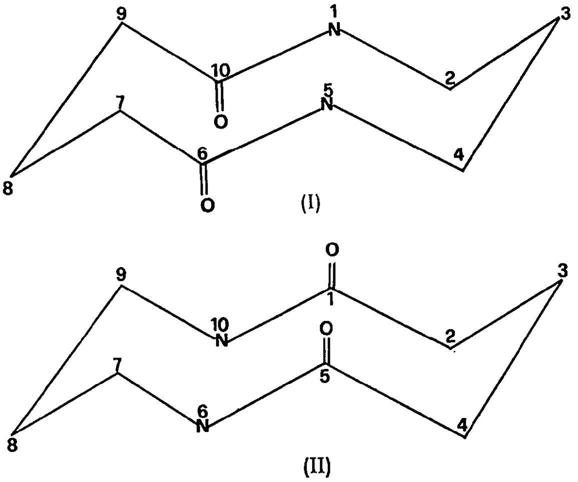

Fig. 1. The atom numbering in the two alternative structures I (above) and II (below) that may occupy a given molecular site.

The direct phasing procedure had thus led to two possible structures [depicted schematically as (I) and (II) in Fig. 1], differing only in the positions of the O atoms and in the identification of the C′ and N atoms of the amide groups. The failure to discriminate between these structures could be attributed to the exclusive use of reflexions with large *E* values in the analysis up to this stage and to the approximately centrosymmetric form of the ring skeleton.

Structure I was then refined by least-squares analysis. Three cycles of isotropic refinement were followed by two cycles of anisotropic refinement, converging at $R=0{\cdot}125$ without inclusion of H atoms. At this stage, an (F_o-F_c) synthesis of reflexions with $\sin\theta/\lambda<0{\cdot}4$ showed peaks corresponding to all H atoms in the molecule at positions close to those expected from stereochemical considerations. The two amide H atoms of structure I are, of course, very close to the O positions in the alternative structure II; the corresponding peaks in the (F_o-F_c) synthesis were stronger (about 0·7 e Å^{-3}) than those associated with the other 12 H atoms of the molecule (0·35 – 0·45 e Å^{-3}). Three further refinement cycles (experimental weights, H atoms fixed at positions estimated from stereochemical considerations, anisotropic temperature factors for C, N, O) led to convergence at $R=0{\cdot}084$. A final cycle with a modified weighting scheme (Dunitz & Seiler, 1973) did not lead to any significant changes in parameters.

At this stage we might have terminated the analysis, were it not for the suspicion that the alternative structure II or some statistical mixture of structures I and II might fit the data at least as well as structure I alone.

If the ring skeleton were exactly centrosymmetric (impossible according to the structural formula 3) then structures I and II would be equivalent, related by a change of origin. Moreover, if the ring centre were located at $y=\frac{1}{4}$, then the statistically disordered superposition of equal proportions of structures I and II would have space group $P2_1/n$. The final outcome of the refinement described above gave the ring centre at $y=0{\cdot}2509$, close enough to $y=\frac{1}{4}$ to call for careful consideration of the possibility that the structure might be disordered in $P2_1/n$. Indeed, well exposed precession photographs show weak diffuse intensity streaks along reciprocal lattices lines parallel to **a*** and **c***. On the other hand, the $0k0$ reflexions with k odd, which should be systematically extinguished in $P2_1/n$, were undoubtedly present, although weak. It was clear that various models would have to be examined before structure I could be accepted as correct.

Structure II is nearly but not exactly equivalent to structure I. We therefore refined structure II alone, starting from the coordinates obtained from the E map. After five cycles (three isotropic, two anisotropic, H atoms excluded) the results were virtually identical with those obtained at the same stage of refinement of structure I.

We then examined the possibility of a disordered structure in more detail. Since the ring skeleton is almost centrosymmetric, its transform is everywhere real when the origin of coordinates is taken at its centre. With this origin, the B part of the structure factor ($F=A+iB$) arises almost entirely from the contributions of the O atoms. (The small contribution arising from the difference between the scattering factors of C and N can be neglected in first approximation.) For either structure I or II alone, 12 non-hydrogen atoms contribute to A, only two to B, so that for most reflexions $A \gg B$ and consequently $F=(A^2+B^2)^{1/2}$ is relatively insensitive to the magnitude of B. If we are concerned with the degree to which a nearly centrosymmetric structure deviates from a strictly centrosymmetric one, we have to look at the reflexions for which A is small – only these reflexions will be sensitive to the magnitude of B.

Table 1 shows that for these reflexions, $|F_o|$ is systematically smaller than $|F_c|$ for structure I (a similar result is obtained for structure II). This means that $|B_c|$ is systematically too large, or, in other words, that the actual structure is more nearly centrosymmetric than structure I alone. On the other hand, $|F_o|$ is systematically larger than $|A_c|$, confirming that the actual structure cannot be exactly centrosymmetric (1:1 superposition of structures I and II). Table 1 suggests that the ratio might be more like 4:1.

Table 1. *Sample list of structure factors at* $R=0{\cdot}084$ (*structure* I) *for which* $|B_c| \gg |A_c|$ (*sin* $\alpha > 0{\cdot}95$)

h	k	l	$\|F_o\|$	$\|F_c\|$	ΔF	$\|A_c\|$	$\|B_c\|$
0	1	0	10·04	14·35	−4·31	0·00	14·35
0	4	4	1·55	2·24	−0·69	0·62	2·15
0	5	0	5·30	6·00	−0·70	0·00	6·00
1	1	−3	9·33	12·65	−3·32	1·19	12·59
1	1	4	3·80	5·44	−1·64	0·89	5·37
1	2	−4	4·27	6·19	−1·92	1·26	6·06
1	2	−2	3·82	6·01	−2·19	1·04	5·92
1	2	1	11·31	18·29	−6·98	2·59	18·11
1	2	7	3·63	4·56	−0·93	0·02	4·56
2	0	12	0·75	1·34	−0·59	0·38	1·28
2	1	−7	3·38	4·45	−1·07	0·87	4·36
2	1	−4	3·75	4·54	−0·79	1·03	4·42
2	1	2	6·39	8·85	−2·46	0·51	8·83
2	3	−10	0·79	1·38	−0·59	0·23	1·36
2	4	4	1·66	2·83	−1·17	0·88	2·69
3	1	1	2·07	2·72	−0·65	0·24	2·71
3	2	0	2·31	3·34	−1·03	0·93	3·21
3	2	3	5·70	9·27	−3·57	2·45	8·94
3	2	9	1·85	2·06	−0·21	0·41	2·02
3	3	−3	0·81	0·95	−0·14	0·06	0·95
3	4	−4	2·19	2·58	−0·39	0·39	2·55
3	4	7	1·40	1·58	−0·18	0·49	1·50
4	0	−10	0·93	1·63	−0·70	0·12	0·92
4	0	−2	1·39	1·96	−0·57	0·10	1·96
4	1	−11	0·70	1·75	−1·05	0·49	1·68
4	1	−4	3·79	5·16	−1·37	1·60	4·91
4	1	2	4·05	5·48	−1·43	1·49	5·27
4	1	8	0·94	1·21	−0·27	0·14	1·20
4	4	−9	1·98	2·13	−0·14	0·65	2·03
4	5	−1	1·17	1·28	−0·11	0·01	1·28
5	1	−9	1·56	2·40	−0·84	0·38	2·37
5	1	−6	1·44	2·29	−0·85	0·68	2·19
6	0	−6	1·37	1·56	−0·19	0·33	1·53
6	4	−2	0·90	1·32	−0·42	0·02	1·32
7	1	−5	0·82	0·94	−0·12	0·03	0·94
7	2	3	1·10	1·38	−0·28	0·30	1·35

Table 3. 1,5-*Diaza*-6,10-*cyclodecadione*: *atomic coordinates* ($\times 10^4$) *and vibrational tensor components* ($\times 10^3$) *for* C, N, *and* O *atoms* (*e.s.d.'s in parentheses*)

The U_{ij} values (in Å^2) correspond to the temperature factor expression $T=\exp[-2\pi^2(U_{11}h^2a^{*2}+\ldots+2U_{12}hka^*b^*\ldots)]$. The coordinates are referred to an origin midway between the glide planes, at the approximate symmetry centre of the ring skeleton, *i.e.* the equivalent positions are to be taken as x,y,z; $\frac{1}{2}+x,\frac{1}{2}-y,\frac{1}{2}+z$.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)[a]	1668 (4)	38 (5)	853 (2)	36 (2)	34 (2)	43 (2)	−2 (1)	3 (1)	3 (1)
C(2)	576 (6)	−504 (7)	1755 (4)	57 (2)	39 (2)	36 (2)	−5 (2)	5 (2)	2 (2)
C(3)	−1078 (4)	1429 (7)	1789 (4)	62 (2)	33 (2)	42 (2)	−5 (2)	17 (2)	−6 (2)
C(4)	−2816 (7)	1198 (7)	848 (4)	47 (2)	38 (2)	57 (2)	1 (2)	19 (2)	−4 (2)
N(5)[a]	−2248 (6)	1749 (6)	−218 (4)	42 (2)	34 (2)	44 (2)	1 (1)	2 (1)	1 (1)
C(6)[b]	−1747[e]	−168 (6)	−844[e]	37 (2)	35 (2)	40 (2)	−1 (1)	−2 (1)	−3 (2)
C(7)	−665 (6)	492 (6)	−1763 (4)	54 (2)	31 (1)	36 (2)	−2 (2)	4 (2)	5 (2)
C(8)	1080 (7)	−1405 (6)	−1796 (4)	52 (2)	34 (1)	41 (2)	−8 (2)	18 (2)	−2 (1)
C(9)	2776 (7)	−1112 (7)	−844 (4)	41 (2)	46 (2)	51 (2)	−1 (2)	15 (2)	0 (2)
C(10)[b]	2197 (7)	−1936 (6)	218 (4)	37 (1)	25 (1)	49 (2)	2 (1)	6 (1)	2 (1)
O(6)[c]	−2124 (7)	−2639 (6)	−648 (4)	46 (1)	25 (1)	55 (2)	−4 (1)	8 (1)	1 (1)
O(10)[c]	2130 (8)	−4393 (7)	470 (4)	67 (2)	29 (1)	54 (2)	2 (1)	9 (1)	5 (1)
O(5)[d]	−2163 (21)	4341 (22)	−387 (11)						
O(1)[d]	2087 (18)	2605 (21)	673 (10)						

(*a*) Scattering factor f taken as $n_{\rm I}f_{\rm N}+(1-n_{\rm I})f_{\rm C}$. (*b*) Scattering factor f taken as $n_{\rm I}f_{\rm C}+(1-n_{\rm I})f_{\rm N}$. (*c*) Occupancy factor $n_{\rm I}=0{\cdot}755$ (7). (*d*) Occupancy factor $n_{\rm II}=0{\cdot}245$. (*e*) These parameters were fixed to define the origin along x and z.

A new refinement was therefore undertaken with *both* pairs of O atoms included in the structure model with variable occupancy factor n under the constraint $n_{\text{I}}+n_{\text{II}}=1$. To keep down the number of parameters and to avoid excessive parameter correlation, the thermal parameters of only one pair of O atoms were refined, those of the other pair being set equal to those of the first pair across the approximate symmetry centre. The scattering factors of the ring atoms 1, 5, 6 and 10 were taken as averages of C and N, appropriately weighted by the occupancy factor n – and similarly for the contributions of the amide H atoms.

In the first cycles only the occupancy factor was refined. From an initial value of 0·9 ($R=0{\cdot}079$) it reduced to 0·76 ($R=0{\cdot}044$). Subsequent cycles (experimental weights, positional parameters of all atoms except amide H atoms, anisotropic thermal parameters for non-hydrogen atoms, isotropic for H except amide H, extinction parameter) converged at a final R of 0·030 for 715 reflexions with $|F_o|>1{\cdot}5\sigma(F_o)$. Although full-matrix refinement of a nearly centrosymmetric structure can lead to ill-conditioning of the normal equations (Ermer & Dunitz, 1970) no serious difficulties were encountered in this case; the largest correlation coefficients between parameters amount to 0·7.

Scattering factors were taken from *International Tables for X-ray Crystallography* (1962). The $|F_o|$ values are given in Table 2.*

Results

Final positional and thermal parameters are given in Table 3 for non-hydrogen and in Table 4 for H atoms. A stereoscopic view of the molecule (structure I) is shown in Fig. 2, and selected structural parameters in Fig. 3. These parameters ostensibly refer to structure I but they actually represent weighted averages over structures I and II in ratio approximately 3:1. Thus, for example, from the distances cited for N(1)–C(2)= 1·469 and C(9)–C(10)=1·491 Å the actual distances in the diamide molecule can be estimated to be 1·458 and 1·502 Å, respectively, and similarly for other pairs of related parameters.

Table 4. 1,5-*Diaza*-6,10-*cyclodecadione*: *atomic coordinates* (×10³) *and isotropic vibrational parameters* (*e.s.d.'s in parentheses*)

The amide hydrogen atoms [H(1), H(5), H(6), H(10)] were included at calculated positions with appropriate weights in the structure factors but were not refined.

	x	y	z	U (Å² × 10²)
H(1)	202	204	69	6·3
H(2*A*)	145 (6)	−32 (7)	242 (4)	5·1 (1·2)
H(2*B*)	9 (5)	−232 (7)	167 (3)	3·4 (1·0)
H(3*A*)	−164 (6)	89 (9)	252 (3)	6·5 (1·4)
H(3*B*)	−69 (7)	341 (9)	177 (4)	7·0 (1·5)
H(4*A*)	−389 (7)	238 (9)	98 (3)	5·4 (1·2)
H(4*B*)	−341 (6)	−67 (8)	84 (3)	4·7 (1·3)
H(5)	−219	368	−47	6·3
H(6)	221	−380	46	6·3
H(7*A*)	−158 (6)	47 (8)	−246 (3)	5·5 (1·4)
H(7*B*)	−15 (7)	236 (9)	−171 (4)	7·4 (1·4)
H(8*A*)	54 (5)	−319 (7)	−184 (3)	3·6 (1·1)
H(8*B*)	162 (5)	−120 (7)	248 (3)	4·1 (1·2)
H(9*A*)	386 (8)	−216 (9)	−97 (4)	6·6 (1·3)
H(9*B*)	318 (7)	77 (8)	−80 (4)	6·6 (1·4)
H(10)	−211	−202	−71	6·3

In general, the bond lengths and angles shown in Fig. 3 express the mirror symmetry required by the molecular constitution 3 and appear to be quite reasonable, except that the C=O bond lengths (1·274 (5) and 1·297 (4) Å for structure I; 1·320 (12) and 1·346 (11) Å for structure II) are much too long compared with the expected value of about 1·24 Å (Marsh & Donohue, 1967). We do not know the reason for this discrepancy. In one series of least-squares calculations the starting model was adjusted to have C=O distances of 1·24 Å, but after two cycles both distances had returned to their anomalously long values.

The amide groups are markedly non-planar, as expressed by the deviations of the $C_\alpha C'NC_\alpha$ torsion angles (±163°) from 180°, but the uncertainty in the amide H position does not allow the relative contributions of χ_N and ω (see Winkler & Dunitz (1971) for

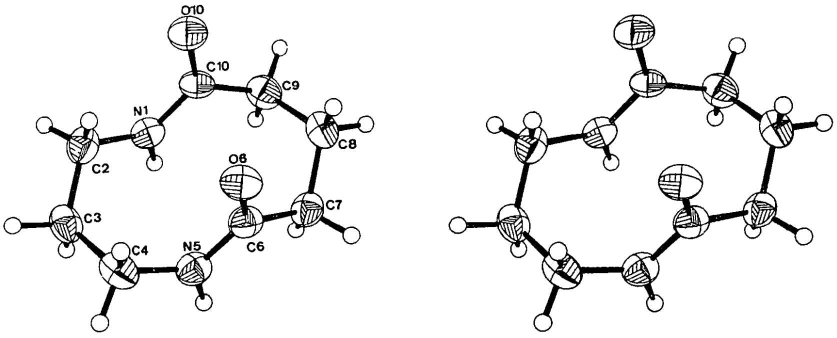

Fig. 2. Stereoscopic view of the molecule (structure I) in the conformation adopted in the crystal.

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* This table has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30874 (2 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

explanation of terms) to be reliably estimated. The value of χ_C is very small, as is usually the case in non-planar amides (Dunitz & Winkler, 1975).

Discussion

Our analysis has led to the following results:

(*a*) The ring skeleton is approximately centrosymmetric although the only symmetry elements compatible with the structural formula 3 would be a mirror plane or a dyad axis through atoms 3 and 8.

(*b*) The crystal structure is disordered.

(*c*) The vibrational parameters of the atoms (Table 3) are remarkably small, considering that each atomic position listed actually represents a weighted mean over the two components (structures I and II) of the disordered structure.

The approximately centrosymmetric nature of the ring skeleton is apparent from Fig. 2 or from the torsion angle pattern of Fig. 3. The conformation adopted is similar to that found in crystals of a centrosymmetric *trans,trans*-cyclodeca-1,6-diene derivative (White, 1973) and evidently belongs to the crown family of conformations of the ten-membered ring. In

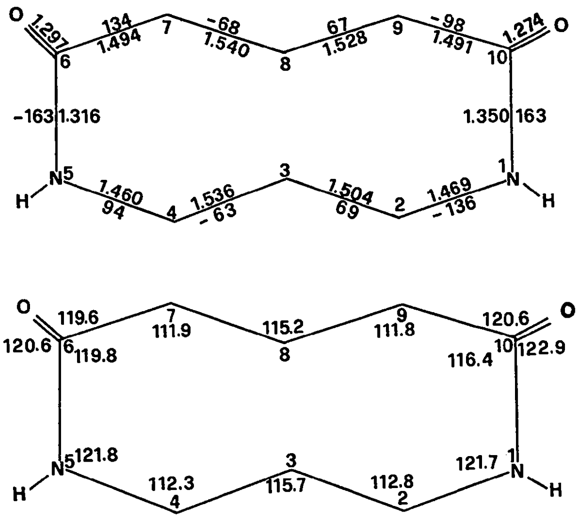

Fig. 3. Bond distances, bond angles and torsion angles. The atom numbering corresponds to structure I, but the parameters reported are derived from the atomic positions of Table 3 and hence correspond to a weighted average over structures I and II.

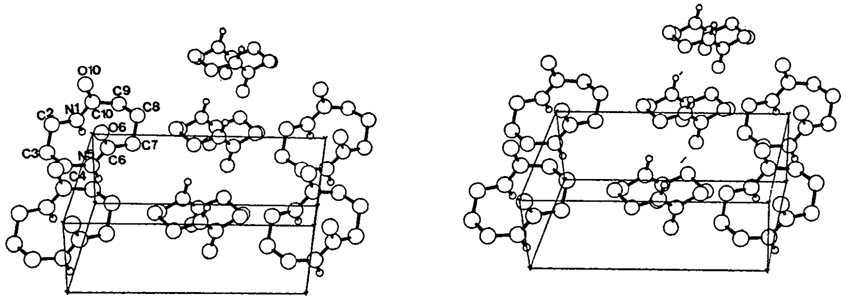

Fig. 4. Stereoscopic view of the packing. Each molecule shown forms part of a hydrogen-bonded column along the *b* axis.

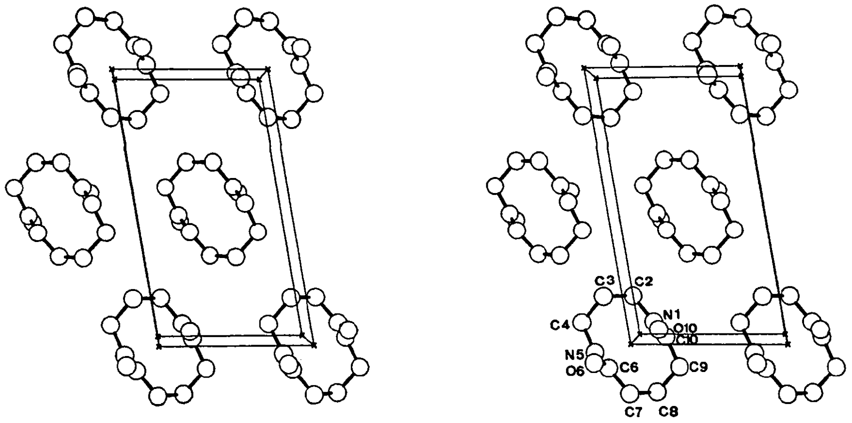

Fig. 5. Stereoscopic view of the packing, looking down the *b* axis.

the mirror-symmetric version of this conformation, obtained by interchanging the values of the $CC_{\alpha}C'N$ and $C'NC_{\alpha}C$ torsion angles (Ψ and φ) of one of the two amide groups, N(1) would be exactly opposite N(5) and C(10) opposite C(6). In the observed conformation (Fig. 3) the two groups are displaced so that N(1) sits opposite C(6) at a distance of 2·894 Å. Presumably, there is a weak attractive interaction between these two atoms involving donation from the filled π-orbital of the N atom to the empty π^*-orbital of the carbonyl group, although any resulting pyramidalization at C(6) is too small to be detected in this disordered structure.

In our previous studies of cyclic mono-amides (Dunitz & Winkler, 1975) we noted that Ψ tends to adopt the value closer to 120°, φ the value closer to 60° but pointed out that since conformations with these values interchanged occur in solution in comparable amounts, they must have very nearly the same free energy. Here one amide group has $\Psi\sim135°$, $\varphi\sim95°$, the other has these values interchanged. Presumably the small energy cost is more than offset by the more favourable interactions between the amide groups in the observed conformation.

The *b* translation of about 5·0 Å is characteristic for association of *transoid* amide groups by NH···O hydrogen bonds.

The crystal structure consists of columns of molecules running parallel to **b**, each molecule being engaged in four hydrogen bonds (Table 5). In the ordered structure, each column is polar, that is, all the carbonyl groups in a given column point along the same direction (Fig. 4). Columns related by the *a* and *c* translations have the same polarity, those related by the glide plane operation have opposite polarity. The polarity of individual columns is almost certainly preserved in the disordered structure since introduction of a type II molecule in a column composed of type I molecules would not only interrupt the hydrogen-bond arrangement but would also lead to intolerable intermolecular contacts. On the other hand, there are no very strong interactions *between* columns, and the packing arrangement normal to the **b** direction (Fig. 5) is hardly altered by changing the polarity sense of a column. Such changes must occur quite frequently, *i.e.* the extent of perfectly ordered domains in the **a** and **c** directions must be quite small, because the observed diffraction pattern is well reproduced by adding diffraction amplitudes ($F=F_{\rm I}+F_{\rm II}$) rather than by adding intensities ($F^2=F_{\rm I}^2+F_{\rm II}^2$).

Table 5. *Hydrogen-bond distances* (Å) *and angles* (°)

N(1)—H(1)—O($10^{\rm i}$)	168	N(1)—O($10^{\rm i}$)	2·860 (4)
N(5)—H(5)—O($6^{\rm i}$)	169	N(5)—O($6^{\rm i}$)	2·870 (5)
N(10)–H(10)–O($1^{\rm ii}$)	170	N(10)–O($1^{\rm ii}$)	2·800 (11)
N(6)—H(6)—O($5^{\rm ii}$)	166	N(6)—O($5^{\rm ii}$)	2·836 (12)

Superscripts refer to the following symmetry-related positions:
(i) $x,1+y,z$ (ii) $x,-1+y,z$

The change from a structure I column to a structure II column is equivalent to introducing a set of local centres of symmetry into the structure. As pointed out earlier, the ring skeleton is approximately centrosymmetric so that the ring atoms of the inverted molecule are nearly coincident with those of the original one. The relatively low values of the averaged vibrational parameters suggest that the superposition must be almost exact.

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