Eine ähnliche Konformation zeigt auch die Acetylgruppe im 3'-*O*-Acetyladenosin (Rao, Sundaralingam, Arora & Hall, 1970). Bindungswinkel und -abstände in der Acetylgruppe stimmen recht gut mit den Werten überein, die für Acetylgruppen und allgemein Estergruppierungen in vergleichbarer Position gefunden wurden (Camerman & Trotter, 1965; Mathieson & Welsh, 1965; Mathieson, 1965).

Packung der Moleküle

Fig. 5 zeigt eine Projektion der Kristallstruktur entlang der *b*-Achse. Die Heterocyclen sind im Abstand von 3,68 Å übereinandergestapelt, zeigen aber wenig Überlappung, da die Ringebenen einen Winkel von 45,3° mit der Stack-Achse (=*b*-Achse) bilden. Die Sauerstoffatome O(2) und O(A) liegen in der Nähe von zweizähligen Schraubenachsen und bilden daher gewinkelte Ketten in Richtung der *b*-Achse. Wie bereits erwähnt, zeigt die Kristallstruktur nur eine Wasserstoffbrückenbindung von 3,227 Å Länge zwischen Schwefelatom und Sauerstoffatom O(5'). Der Abstand zwischen dem an Atom O(5') gebunden Wasserstoffatom H(13) und dem Schwefelatom beträgt 2,31 Å, die Abweichung von der Linearität, d.h. der Winkel H(13)–O(5')–S, 17°. Diese Werte erfüllen die Kriterien einer S–O-Wasserstoffbrückenbindung (Srinivasan & Chacko, 1967; Saenger & Suck, 1970, 1971).

Die Strukturamplituden sind in Tabelle 5 enthalten.

Das 3'-*O*-Acetyl-4-thiothymidin stellte uns Dr K. H. Scheit freundlicherweise zur Verfügung. Professor D. Mootz erlaubte uns, auf dem Siemens-Diffraktometer im Institut für molekulare Biologie, Biochemie und Biophysik, Stöckheim die Reflexintensitäten zu messen, die Rechnungen wurden auf der IBM 7040 im Göttinger Rechenzentrum ausgeführt. Wir danken Professor F. Cramer für grosszügige Unterstützung und Förderung dieser Arbeit.

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Determination of the Molecular Packing in the Crystal of *N,N'*-Dicyclohexylurea by means of Potential Energy Calculations

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Crystals of *N,N'*-dicyclohexylurea are monoclinic, space group $P2/c$, with two formula units in a cell with dimensions: $a=11\cdot54$, $b=4\cdot69$, $c=12\cdot03$ Å, $\beta=95°28'$. The structure was solved by crystal packing energy calculations, using van der Waals and hydrogen bonding potentials, and then refined by least-squares methods to a conventional R value of 0·107. The molecular packing is characterized by rows of hydrogen bonds along the b axis, while adjacent rows are tied together by van der Waals interactions. The non-planar distortion of the OC=NH group is about 8°.

Introduction

The determination of the crystal structure of *N,N'*-dicyclohexylurea (DCHU) was undertaken as a part of a research programme developed in this laboratory to solve the phase problem by potential energy calculations [among the most recent papers: Giacomello & Giglio (1970), Giglio (1970), Capaccio, Giacomello &

Giglio (1971)]. From a knowledge of the lattice parameters, crystal symmetry and approximate bond lengths and angles, the potential energy can be computed for all the rotational and translational degrees of freedom which define the asymmetric unit position, as in the case of 5α-androstan-3,17-dione (Damiani, Giglio, Liquori & Mazzarella, 1967). One of the deepest minima of the energy surface corresponds to the real situation in the crystal when the vibrational energy and entropy can be neglected and suitable potentials, describing all the possible interactions, are available.

To verify further the validity of the potentials previously tested (Di Nola & Giglio, 1970; Giglio, 1969; and references cited therein) and to establish the, at present, unknown molecular geometry of an important biological model compound we tried to determine the molecular arrangement of this urea derivative.

Experimental

Crystals of DCHU ($C_{13}H_{24}N_2O$), in the form of thin colourless needles (m.p. 229–230°C), were obtained from toluene by evaporating a saturated solution at room temperature. Aggregates along the *b* axis crystallized more frequently from other solvents such as ethanol, pyridine and *N,N'*-dimethylformamide. The crystals are monoclinic and the unit-cell dimensions, as measured on a Siemens diffractometer with Cu *Kα* radiation ($\lambda = 1\cdot5418$ Å), are: $a = 11.54 \pm 0\cdot01$, $b = 4\cdot69 \pm 0\cdot005$, $c = 12\cdot03 \pm 0\cdot01$ Å, $\beta = 95°28' \pm 5'$.

The space groups *Pc* or *P*2/*c* were indicated by the systematic absence of reflexions *h*0*l* with *l* odd. A density of 1·158 g.cm^{-3}, calculated for two formula units per unit cell, agrees well with the value of $1\cdot15_2 \pm 0\cdot01$ g.cm^{-3} measured by flotation in a carbon tetrachloride–ligroin mixture.

The intensity data over the complete sphere of reflexion were collected employing an approximately needle-shaped crystal of dimensions 0·2 × 0·3 × 0·9 mm mounted with the *b* axis parallel to the recording instrument axis. Diffraction intensities were measured using nickel-filtered copper radiation with an on-line Siemens automatic single-crystal diffractometer, operated as a three-circle instrument. The five-value measurement and the moving-crystal moving-counter method (Troughton, 1970) were adopted to collect the 989 reflexions with intensities above significance level. A check reflexion was monitored every twenty reflexions. The intensities were corrected for Lorentz and polarization factors but no absorption corrections were applied.

Structure determination

The molecule of DCHU studied in this paper is shown in Fig. 1 together with the atomic numbering system. By assuming the cyclohexane rings to be rigid and both in the chair conformation there are three possibilities for the hydrogen atoms linked to C(2) and C(8): (1) both equatorial; (2) both axial; (3) one equatorial and the other axial. The molecule presents four rotational degrees of freedom θ_1, θ_1', θ_2 and θ_2' around the C(1)–N(1), C(1)–N(2), N(1)–C(2) and N(2)–C(8) bonds respectively in spite of the considerable double-bond character of the first two.

Table 1. *Bond lengths and bond angles assumed for DCHU*

C–H	1·08 Å	N–C–O	120°
C–C	1·54	C–N–C	120
C–N	1·48	N–C–N	120
C=N	1·32	C–N–H	120
C–O	1·24	C–C–C	109·5
N–H	1·00	H–C–H	109·5
		C–C–N	109·5
		N–C–H	109·5

Table 2. *The coefficients of the potential functions*

The energy is in kcal per atom pair if the interatomic distance is in Å.

Interaction	$A \times 10^{-3}$	B	C	D
H–H	6·6	4·080	49·2	0
H–C	44·8	2·040	125·0	6
H–N	52·1	2·040	132·0	6
H–O	42·0	2·040	132·7	6
C–C	301·2	0·000	327·2	12
C–N	340·0	0·000	340·0	12
C–O	278·7	0·000	342·3	12
N–N	387·0	0·000	354·0	12
N–O	316·2	0·000	356·0	12
O–O	259·0	0·000	358·5	12

It was decided to start the analysis of the crystal packing with the most energetically stable conformation. The DCHU intramolecular potential energy was computed as a function of the four rotational degrees of freedom by fixing bond lengths and angles at the values reported in Table 1 and by using the coefficients of Table 2 for the atom–atom potentials in the generalized form:

$$U(r) = \frac{A \exp(-Br)}{r^D} - \frac{C}{r^6}.$$

In addition we introduced the following torsional potentials:

$$V(\theta_1) = \frac{V_0(\theta_1)}{2}(1 - \cos 2\theta_1)$$

$$V(\theta_2) = \frac{V_0(\theta_2)}{2}(1 - \cos 3\theta_2)$$

putting $V_0(\theta_1) = 20$ kcal (Ramachandran & Sasisekharan, 1968) and varying $V_0(\theta_2)$ within the range 0–2 kcal with the same results.

The cases 1 and 3 were soon discarded as they correspond to higher energy values as can be proved by simple inspection of models. The more promising case 2 gave rise to two broad minima of equal depth (angular increment 10°) defined by:

I $\theta_1=0;\ \theta_1'=0;\ \theta_2=20;\ \theta_2'=-20°$,
II $\theta_1=0;\ \theta_1'=0;\ \theta_2=20;\ \theta_2'=20°$.

Since the two energy minimum zones can be superimposed, only a section of I is shown in Fig. 2. The DCHU molecules so obtained, characterized by the presence of a twofold axis (I) or a mirror plane (II), were considered as starting models in the crystal analysis. The molecular parameters of Table 1, which are not the best available from the literature, were deliberately adopted so as to ascertain the validity of the energy calculations under unfavourable conditions.

The packing energy in the crystal of DCHU depends on three rotations (ψ_1, ψ_2, ψ_3) and one translation along $\mathbf{b}(t_y)$. The DCHU was rotated in a right-handed orthogonal framework *Oxyz* orientated with respect to the monoclinic crystallographic system *Oabc* in such a way that *Ox* and *Oy* coincided with *Oa* and *Ob* respectively and the positive *Oz* and *Oc* semi-axes lay on the same side of the *xy* plane. Then ψ_1, ψ_2 and ψ_3 stand for counterclockwise rotations about *Oy*, *Oz* and *Oy*, provided that they are performed in the order given, moving *Oxyz* and keeping the molecule fixed. Furthermore, θ_2 was permitted to vary within a short angular range because the minima I and II are shallow.

The potential proposed by Stockmayer (1941) to describe the interaction between two polar gas molecules and successfully employed in the analysis of dimethylglyoxime (Giglio, 1969) and diketopiperazine (Giacomello & Giglio, 1970) crystals was used to take into account the formation of possible intermolecular hydrogen bonds between C=O and N–H groups. This function can be written:

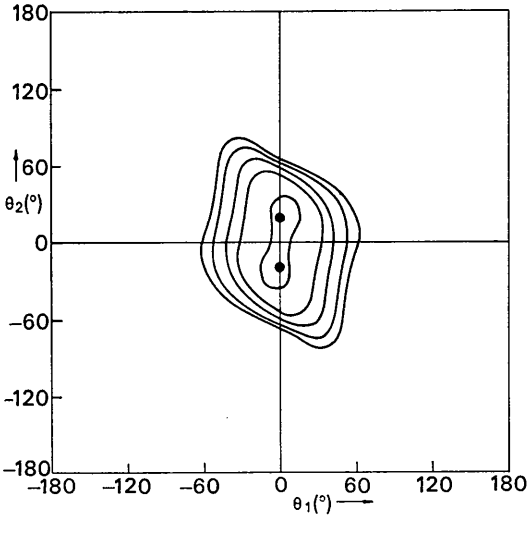

Fig. 2. Section $\theta_1=\theta_1'$ and $\theta_2=\theta_2'$ of the potential energy of DCHU. The minimum I is indicated by a black spot. Contours drawn at intervals of 5 kcal starting from 35 kcal.

$$V(r)=4\varepsilon[(\sigma/r)^{12}-(\sigma/r)^6]-\mu_a\mu_b r^{-3}[2\cos\vartheta_a\cos\vartheta_b-\sin\vartheta_a\sin\vartheta_b\cos(\varphi_b-\varphi_a)]\,.$$

The Lennard-Jones term refers in this case to the non-bonded interaction between H and O atoms. The remaining term represents the electrostatic energy between two point dipoles of magnitude μ_a and μ_b centred on H and O atoms, distance r apart. ϑ_a and ϑ_b are the angles formed by C=O and N–H bonds with

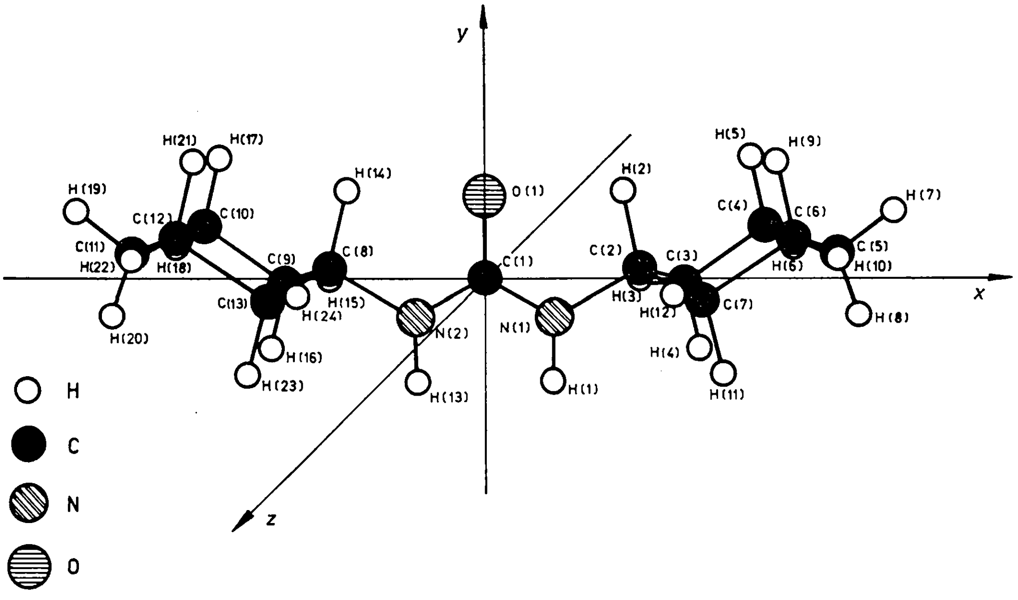

Fig. 1. Atomic numbering of the DCHU molecule. The atoms C(1), C(2), C(5), C(8), C(11), N(1), N(2), O(1) and the hydrogen atoms linked to them lie in the *xy* plane and C(1) is at the origin. All the θ and ψ angles are 0°.

the O...H segment and $(\varphi_b-\varphi_a)$ is the dihedral angle between the planes passing through C, O, H and N, H, O atoms. The value of $\sigma(2{\cdot}83\ \text{Å})$ was determined on the basis of the van der Waals radii of the best potentials found for H–H (Liquori, Giglio & Mazzarella, 1968) and O–O (Giglio, Liquori & Mazzarella, 1969) interactions. The parameters ε and $\mu_a\mu_b$ were deduced by imposing: $V(r)=-5$ kcal and $\delta V(r)/\delta r=0$ for $r=1{\cdot}85$ Å, when, according to experimental data, the C, O, N and H atoms are collinear. Thus $\varepsilon=0{\cdot}0026$ kcal and $\mu_a\mu_b=20{\cdot}8254$ kcal × Å^3 per atom pair. The variations of V with r and ϑ_a are illustrated in Figs. 3 and 4.

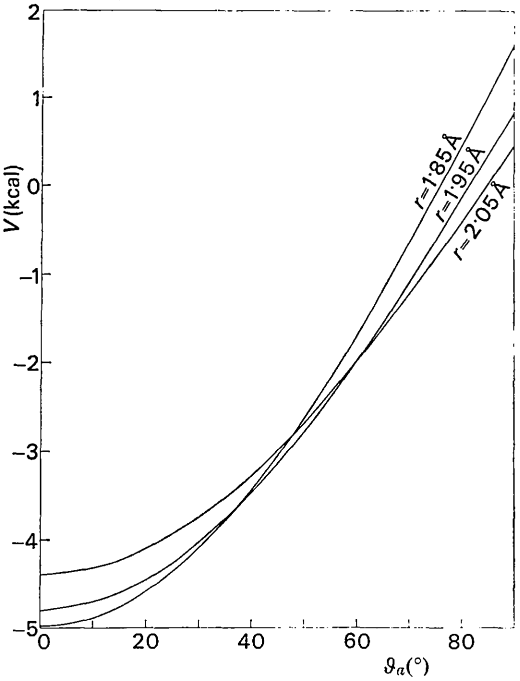

Fig. 3. Section of V vs. ϑ_a with $\vartheta_b=\varphi_b-\varphi_a=0°$. The value of r is constant for every curve.

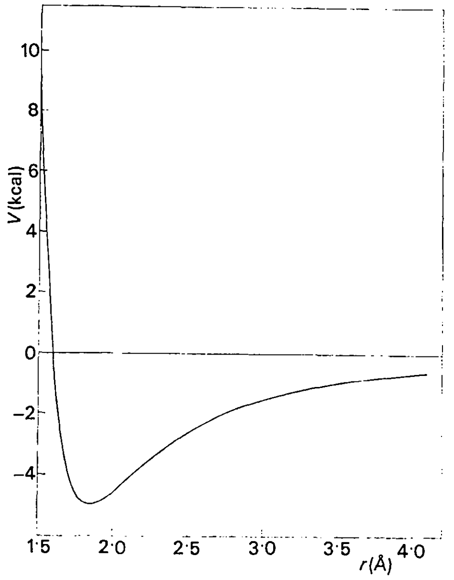

Fig. 4. Section of V vs. r with $\vartheta_a=\vartheta_b=\varphi_b-\varphi_a=0°$.

The packing energy was estimated by considering all the intermolecular non-bonded interactions between forty atoms of one molecule and the atoms of the nearest twenty-two molecules. An H···O cutoff distance of 2·4 Å was assumed in evaluating the hydrogen bonding energy, since V is a slowly increasing function of r (see Fig. 4). Angular and translational increments of 20° and 0·3 Å were given in the first run. Subsequently, the regions of minimum V were explored by progressively reducing the increments to 1° and 0·1 Å.

A noticeably better packing energy was achieved with the twofold axis molecule and the corresponding parameters are: $\theta_2=9$; $\psi_1=40$; $\psi_2=180$; $\psi_3=2°$; $t_y=1{\cdot}3$ Å. The starting model is shown in Fig. 1.

The coordinates of the heavy atoms produced by the packing analysis yielded an R value of 0·34 for the 226 low angle reflexions with $\sin\theta/\lambda\le0{\cdot}35\ \text{Å}^{-1}$, using an overall B of 4·5 Å^2. Only 35 phases belonging to very weak reflexions were found to be wrong when the crystal structure was solved. The best value of R $(\theta_2,\ \psi_1,\ \psi_2,\ \psi_3,\ t_y)$ using the molecular geometry of Table 1 was calculated as 0·30, using angular and translational increments of 1° and 0·05 Å, and assuming for the variables the values $\theta_2=9$; $\psi_1=40$; $\psi_2=180$; $\psi_3=2°$; $t_y=1{\cdot}17$ Å. Initially, the space group $P2/c$ was considered by putting the asymmetric units in the special positions 2(e) or 2(f).

Refinement of the structure

A set of programs, written for a UNIVAC 1108 computer by Domenicano, Spagna & Vaciago (1969), were used to refine the structure. The atomic scattering factors used for the heavy atoms were those given by Cromer & Mann (1968) and for the hydrogen atoms the values were taken from the scattering factor table of Hanson, Herman, Lea & Skillman (1964).

Five cycles of isotropic block-diagonal (4 × 4) least-squares refinement excluding the hydrogen atoms gave $R=0{\cdot}21$ for the 989 observed reflexions. The weight w was taken as $w=(a+F_o+bF_o^2+cF_o^3)^{-1}$ with $a=0{\cdot}70$, $b=0{\cdot}0105$ and $c=0{\cdot}0001$. The refinement was continued with anisotropic thermal factors until convergence was reached. Four cycles of block-diagonal (9 × 9) least-squares refinement reduced R to 0·146. A difference Fourier synthesis showed all the hydrogen atoms at reasonable positions, on peaks between 0·35 and 0·46 e.Å^{-3}, with the exception of H(9) (0·21 e.Å^{-3}). At this stage the hydrogen atoms were included with an isotropic B of 4 Å^2 and the refinement was considered to be complete when the parameter shifts were less than 10% of the estimated standard deviations. The final R value and the weighted discrepancy index were 0·107 and 0·034 respectively for the observed reflexions.

The final atomic coordinates and anisotropic thermal parameters are given in Tables 3 and 4 together with their standard deviations. The observed and calculated structure factors are listed in Table 5.

Table 3. *Final values of the fractional atomic coordinates and their standard deviations* ($\times 10^4$)

	x	y	z	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$
C(1)	5000	1849	2500	0	12	0
C(2)	3295	2197	3597	3	8	3
C(3)	2094	3241	3162	4	12	4
C(4)	1141	2158	3845	4	12	4
C(5)	1411	2752	5075	4	10	4
C(6)	2602	1659	5506	4	14	4
C(7)	3544	2786	4832	3	10	4
N(1)	4176	3357	2952	3	8	3
O(1)	5000	−820	2500	0	8	0
H(1)	4172	5480	2827			
H(2)	3278	−136	3482			
H(3)	2073	5666	3313			
H(4)	1872	2266	2384			
H(5)	1112	−35	3681			
H(6)	340	3159	3584			
H(7)	723	1706	5500			
H(8)	1425	5116	5178			
H(9)	2799	2257	6382			
H(10)	2545	−1035	5415			
H(11)	4308	2168	5164			
H(12)	3548	4947	4884			

Attempts were also made to refine the structure using the space group *Pc* by relaxing the molecular symmetry of *P*2/c. In every case, however, the atomic coordinates of Table 3 were obtained and it was concluded that the space group was not *Pc*.

Discussion

Geometry of the molecule and planarity of the peptide group

The intramolecular distances and angles are reported in Table 6. The bond lengths involving hydrogen atoms all lie between 1·01 and 1·15 Å except for C(7)–H(11) and C(6)–H(10) which are respectively 0·98 and 1·27 Å. The H–C–H bond angles values lie between 106 and 113° with the exception of H(3)–C(3)–H(4) (124°). The angles involving C, N and H atoms are both 117°.

The peptide bond lengths and angles are in good agreement with those of urea (Pryor & Sanger, 1970), of α-glycylglycine (Biswas, Hughes, Sharma & Wilson, 1968; Hughes, 1968) and of perdeutero-α-glycylglycine (Freeman, Paul & Sabine, 1970) and the double-bond character for C(1)–N(1) appears to be retained. This statement is also supported by the usual value of about 1·45 Å for the C(2)–N(1) single bond which is adjacent to a double bond.

The C–C distances in the chair shaped cyclohexane ring (average: 1·514 Å) are systematically somewhat below normal and this shortening may be due to thermal libration of DCHU.

An important feature of this structure with respect to the conformational analysis is the possible rotation about the C(1)–N(1) double-bond. A non-planar distortion of the peptide group has been pointed out in some linear and cyclic polypeptides (Ramachandran, 1968; Lakshminarayanan, 1970) as well as in perdeutero-α-glycylglycine and in *N*,*N*′-diethyl-*N*,*N*′-diphenylurea (Ganis, Avitabile, Benedetti, Pedone & Goodman, 1970), where the hybridization of the *N* (peptide) atoms is flattened tetrahedral. The dihedral angle between the planes O(1)C(1)N(1) and C(1)N(1)C(2) is 8·0° in DCHU and C(2) is at 0·16 Å from the first plane. This distortion from planarity and the angle C(1)–N(1)–C(2) having a value greater than 120° can be explained by steric hindrance between O(1) and H(2). Since the hydrogen atoms cannot be located with sufficient accuracy by X-ray diffraction methods it is impossible to establish if the coordination of N(1) is pyramidal. Nevertheless the torsion of 8° around C(1)–N(1) in a molecule with no strong tendency to deformation suggests that this torsional parameter must be taken into account in working out the conformation of acyclic polypeptides.

Intermolecular contacts and hydrogen bonding

All the intermolecular contacts involving heavy atoms are longer than the sum of their van der Waals radii. The two shortest distances (2·22 and 2·33 Å) involve H atoms and so the reliability of these figures is low. The oxygen atom of the molecule at (x, $1+y$, z) is 2·96 Å from N(1) and N(2) and forms hydrogen bonds to both nitrogen atoms. The approximately estimated values for the H(1)···O distance and the N(1)–H(1)···O angle of 2·0 Å and 152° respectively are comparable with the corresponding ones in urea.

The structure consists of hydrogen-bonded rows of molecules translated along the *b* axis (4·69 Å), whereas

Table 4. *Thermal parameters and their standard deviations* ($\times 10^5$) *for atoms other than hydrogen*

The form of the temperature factor is: $\exp\left[-(b_{11}h^2+b_{12}hk+b_{13}hl+b_{22}k^2+b_{23}kl+b_{33}l^2)\right]$

	b_{11}	b_{12}	b_{13}	b_{22}	b_{23}	b_{33}	$\sigma(b_{11})$	$\sigma(b_{12})$	$\sigma(b_{13})$	$\sigma(b_{22})$	$\sigma(b_{23})$	$\sigma(b_{33})$
C(1)	427	0	308	4959	0	585	32	0	51	263	0	33
C(2)	563	−283	622	4591	−194	697	27	113	43	197	110	27
C(3)	548	68	378	8973	−325	667	28	158	45	304	158	28
C(4)	496	−320	377	9200	−530	815	29	155	49	342	167	35
C(5)	585	−363	758	7393	−454	781	31	137	49	279	143	32
C(6)	833	−326	522	9209	571	687	37	185	55	347	171	31
C(7)	515	63	292	7101	−15	724	28	130	43	262	137	29
N(1)	697	−53	1012	5023	134	1017	27	115	48	186	122	30
O(1)	645	0	652	4366	0	871	28	0	48	196	0	31

Table 5. *Observed and calculated structure amplitudes* (× 10)

H	L	FO	FC
**** K = 0 ****			
0	2	54	12
0	4	307	309
0	6	177	163
0	8	128	127
0	14	12	-12
1	0	425	-364
1	2	889	831
1	-2	51	-17
1	4	776	-698
1	-4	108	92
1	6	28	-17
1	-6	176	186
1	8	35	-32
1	-8	18	-16
1	10	63	72
1	-10	39	36
1	12	23	26
1	-12	50	-52
2	0	248	-244
2	2	458	-413
2	-2	397	-371
2	4	211	206
2	-4	112	112
2	6	345	-344
2	-6	25	-35
2	8	46	39
2	-8	179	185
2	10	56	-54
2	-10	65	73
2	12	32	28
2	-12	29	26
2	14	34	-33
2	-14	15	21
3	0	239	-219
3	2	609	523
3	-2	48	-3
3	4	114	-111
3	-4	333	-320
3	6	157	164
3	-6	47	41
3	8	51	-54
3	-8	12	9
3	10	37	33
3	-10	96	-105
3	12	50	-44
3	-12	36	-42
3	-14	69	73
4	0	180	-175
4	2	324	-293
4	-2	364	-315
4	4	204	198
4	-4	114	-112
4	6	306	-300
4	-6	11	-22
4	8	41	-41
4	-8	219	-226
4	10	42	-40
4	-10	218	-229
4	-12	48	46
4	-14	77	-80
5	0	147	-134
5	2	236	-223
5	-2	54	67
5	4	116	-103
5	-4	292	247
5	6	144	131
5	-6	94	99
5	8	192	-191
5	-8	117	-116
5	10	23	20
5	-10	168	159
5	12	11	5
5	-12	60	-53
5	-14	10	0
6	0	85	86
6	-2	24	-22
6	4	54	50
6	-4	110	122
6	6	137	141
6	-6	116	-120
6	8	168	166
6	-8	143	134
6	10	39	-37
6	-10	40	-40
6	12	59	58
6	-12	51	53
7	0	64	-63
7	2	143	126
7	-2	72	82
7	4	23	-17
7	-4	72	-71
7	6	28	30
7	-6	19	12
7	8	34	36
7	-8	151	-149
7	-10	98	98
8	0	136	139
8	2	22	-23
8	-2	249	239
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5	7	12	9
5	-7	25	-19
5	-9	11	13
6	0	50	47
6	1	83	81
6	-1	50	-53
6	-2	26	27
6	3	39	-41
6	-5	25	-23
6	6	33	31
6	-6	27	22
7	1	23	-31
7	-1	42	40
7	3	43	40
7	-3	11	-19
7	-4	27	26
7	5	27	-24
7	-5	11	15
7	-6	9	-2
7	-7	34	-38
8	1	29	34
8	-1	30	-29
8	-2	37	37
8	3	47	-43
8	-3	17	18
8	-5	17	-20
8	-6	8	12
8	-7	21	25
9	0	25	28
9	-1	8	15
9	3	49	53
9	-5	22	24
**** K = 5 ****			
0	0	9	-6
0	1	40	23
0	3	62	-36
0	4	49	-38
0	5	20	14
0	6	15	-9
0	7	10	-2
1	-1	18	31
1	3	43	29
1	-3	27	-17
1	-4	11	10
1	5	34	-19
1	6	36	-28
1	-6	20	15
1	-7	14	14
2	0	12	-12
2	-1	11	-24
2	3	11	-10
2	4	13	13
2	5	36	21
2	6	23	16
3	1	10	-16
3	-1	14	29
3	2	12	-5
3	-2	12	-13
3	3	18	19
3	-4	15	-17
3	5	16	-11
3	6	10	2
4	2	23	34
4	-2	12	-4
4	3	22	-27
4	-3	8	-22
4	-4	38	40
4	5	11	8
4	-5	20	16
4	-6	13	12
5	0	10	-21
5	1	8	-8
5	-1	30	-39
5	-2	17	27
5	-3	25	-3
5	4	14	17
6	2	18	-24
6	-3	43	6

Table 6. *Intramolecular distances and angles and their estimated standard deviations*

C(1)–O(1)	1·252 (7) Å	N(1)–C(1)–N(2)	116·3 (3)°
C(1)–N(1)	1·341 (5)	O(1)–C(1)–N(1)	121·8 (3)
C(2)–N(1)	1·444 (5)	C(1)–N(1)–C(2)	125·6 (4)
C(2)–C(3)	1·515 (6)	N(1)–C(2)–C(3)	111·1 (3)
C(2)–C(7)	1·511 (6)	N(1)–C(2)–C(7)	112·5 (3)
C(3)–C(4)	1·522 (6)	C(2)–C(3)–C(4)	113·0 (4)
C(4)–C(5)	1·509 (6)	C(3)–C(4)–C(5)	112·0 (4)
C(5)–C(6)	1·511 (6)	C(4)–C(5)–C(6)	111·7 (4)
C(6)–C(7)	1·513 (7)	C(5)–C(6)–C(7)	112·0 (4)
		C(2)–C(7)–C(6)	112·6 (3)
		C(3)–C(2)–C(7)	111·2 (3)

a similar molecular arrangement occurs along the *c* axis (4·72 Å) of the urea crystal. The packing can be represented by layers perpendicular to the *b* axis related by a glide plane, and the cohesion is mainly determined by van der Waals intermolecular forces within a layer and between layers. The structure is shown in Fig. 5.

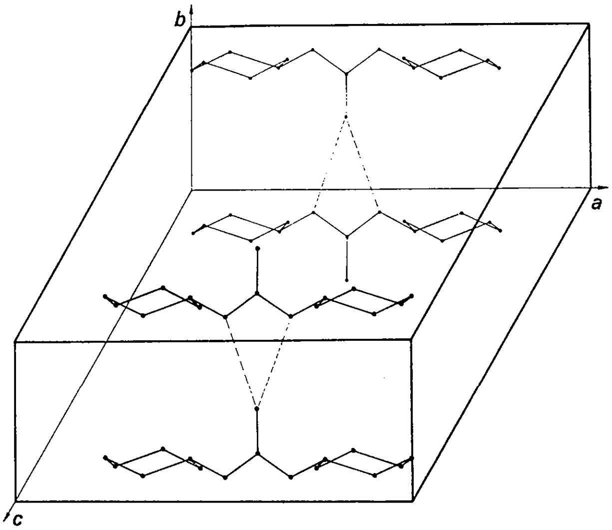

Fig. 5. View of the packing of the DCHU molecules in the crystal. The dashed lines indicate hydrogen bonding.

Packing energy

H···H interactions predominate in the calculation of the position of minimum *V*, since the carbon and nitrogen atoms are screened by hydrogen atoms. Although the H–H potential is soft compared with the others of Table 2, the energy minimum can be located with reasonable accuracy. The corresponding atomic fractional coordinates are reported in Table 7, together with the atomic deviations from the observed structure (column *d*). However, the largest departures from the experimental positions of the atoms C(1), N(1) and O(1) are probably due to the decrease of the C(1)–N(1)–C(2) angle and to the increase of the N(1)–C(1)–N(2) angle in the model of Table 1. To demonstrate this the packing analysis was repeated by using the molecular geometry as derived from the positional parameters of Table 3 and the procedure described above except that the last translational increment was reduced to 0·02 Å. The agreement was surprisingly good. The *x* and *z* atomic coordinates, corresponding to the energy minimum, were identical with the experimentally measured values and the *y* coordinates differed only by 0·05 Å. In addition, the depth of the minimum was much greater than the previous one for both the van der Waals and the hydrogen bonding energies. Thus these results point out again the validity of the potentials, for H–H interactions and hydrogen bond formation, in solving the phase problem. However, it must be stressed that while the potentials used by us are so far qualitatively adequate for determining crystal structures, they are not quantitatively reliable enough to give correct energy values (Giglio, 1970).

The presence of hydrogen bonds does not usually increase the difficulties in the packing analysis, since they provide strong specific linkages which partially freeze some molecular degrees of freedom. As a consequence the hydrogen bonding energy is almost the best possible in many cases, playing a dominating role with respect to the van der Waals energy in the determination of the minimum. Such a situation suggests that only seldom may a competitive effect between hydrogen bonding and van der Waals energies be present.

Table 7. *Atomic fractional coordinates* ($\times 10^4$) *of the molecule used in the packing analysis*

$\theta_2=9$, $\psi_1=40$, $\psi_2=180$, $\psi_3=2°$, $t_y=1·3$ Å. Column *d* refers to the atomic deviations from the experimental structure.

	x	*y*	*z*	*d*
C(1)	5000	2772	2500	0·43 Å
C(2)	3337	2601	3589	0·20
C(3)	2114	3741	3199	0·24
C(4)	1199	2099	3798	0·10
C(5)	1442	2513	5069	0·12
C(6)	2665	1373	5459	0·16
C(7)	3580	3015	4860	0·12
N(1)	4216	4179	3013	0·39
O(1)	5000	128	2500	0·44

In order to evaluate all the local and absolute minima we explored completely the independent part of the energy surface by fixing the size of the angular and translational increments. We have also studied the problem of characterizing minima by means of a multi-parameter search procedure and the results of this study were unsatisfactory mainly for the following reasons: (1) the difficulty of calculating all the minima; (2) the arbitrary choice of the starting position influencing the convergence towards the experimental structure; (3) the dubious possibility of jumping out of a valley after its position has been determined. Furthermore, in our opinion, a false minimum cannot always be rejected on the basis of short intermolecular contacts or by comparing the observed and calculated structure factors for some low angle reflexions as Williams (1969) states. For example, the analysis of the packing of 5-α-androstan-3,17-dione (Damiani, Giglio, Liquori & Mazzarella, 1967) and of dimethylglyoxime (Giglio, 1969) showed the existence of false minima almost as deep as the lowest ones without giving unreasonable intermolecular distances. On the other hand, structure factor calculations, not followed by refinement techniques, may be inadequate as a test especially when the molecular geometry is approximately known. Moreover, it is hardly credible that the observed heat of sublimation will quantitatively agree with the lattice energy of the correct minimum (Williams, 1969), computed by means of crude potentials, except when this condition is imposed in deriving the coefficients of some of the potential functions. In this case, of course, the above relationship cannot be applied for most structures.

In conclusion we believe that the determination of the DCHU crystal structure, in spite of an unfavourable molecular geometry, is a satisfactory test of the reliability of the packing analysis. We hope that this method, coupled with the 'minimum residual' analysis (Damiani, Giglio, Liquori & Ripamonti, 1967) can be used as a powerful tool in solving more complicated crystal structures.

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Structure Cristalline du Solvate du Cyanure Mercurique avec le Tétrahydrofuranne

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The crystal structure of mercury cyanide solvate with tetrahydrofuran, $5Hg(CN)_2.4C_4H_8O$, was solved by the heavy atom method from 439 intensities *hk*0 to *hk*10 registered with a retigraph. Final $R=0.068$ with isotropic agitation of the atoms. The mercury atoms have octahedral surroundings, two summits being occupied by carbon atoms of the same $Hg(CN)_2$ molecule, while the four others are occupied either by four oxygen atoms of the tetrahydrofuran rings, or by four nitrogen atoms of the neighbouring $Hg(CN)_2$ molecules, or by two oxygen and two nitrogen atoms respectively belonging to the tetrahydrofuran rings and neighbouring $Hg(CN)_2$ molecules. The $Hg\cdots N$ bonds between mineral molecules ($Hg\cdots N\simeq 2.85$ Å) are shorter than van der Waals bond lengths.

Introduction

Lors de la décomposition ménagée des cristaux du solvate $5Hg(CN)_2.4C_4H_8O$, effectuée à l'air libre, les cristaux de cyanure mercurique se développent en prenant par rapport au solvate une orientation bien déterminée (Ledésert, Frey & Monier, 1967). En fonction de cette orientation, on a constaté l'existence de