The Crystal and Molecular Structure of
3-Carbethoxy-4-oxo-6-methyl-6,7,8,9-tetrahydrohomopyrimidazole, $C_{12}H_{16}N_2O_3$

By K. Sasvári

Central Research Institute for Chemistry of the Hungarian Academy of Sciences, 1525-Budapest POB 17, Hungary

and K. Simon

Chinoin Factory for Pharmaceutical and Chemical Products, Budapest, Hungary

3-Carbethoxy-4-oxo-6-methyl-6,7,8,9-tetrahydrohomopyrimidazole (COMHHP) crystallizes in the monoclinic space group $A2/a$ with 8 molecules in the unit cell of dimensions $a = 10·04$, $b = 16·73$, $c = 14·66$ Å and $\beta = 90·1°$. The phase problem was solved by the direct method, and the final atomic parameters were obtained by three-dimensional least-squares refinement with the final conventional $R = 0·113$ for the observed reflexions. The molecule is planar to a good approximation. Only three atoms deviate from the plane by more than 0·5 Å. The molecule shows a higher stability than the unsaturated form ($C_{12}H_{12}N_2O_3$), in accordance with the shortened bond length $N-C(=O) = 1·411$ Å, which in the unsaturated form was found to be 1·472 Å.

Introduction

The title compound (COMHHP) has been obtained by catalytic hydrogenation of $C_{12}H_{12}N_2O_3$ (Mészáros *et al.*, 1972). According to spectroscopic investigations the formation of the new molecule takes place according to the following scheme:
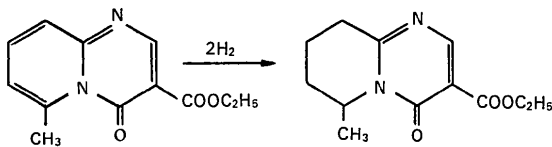
After hydrogenation the molecule showed a considerably increased stability compared with the original molecule. Whereas before hydrogenation the molecule in aqueous solution suffered ring scission at the N–CO bond no such effect was observed after hydrogenation. The purpose of the present crystal structure determination is to follow the alteration of molecular conformation and find an explanation of the increased stability.

Experimental

The crystals used for X-ray diffraction were crystallized from petrol solution. The colourless transparent crystals are stubby shaped with well developed planes and edges.

The cell dimensions were determined from Buerger precession photographs taken around the crystallo-

Table 1. *Final fractional coordinates* ($\times 10^4$) *and anisotropic thermal parameters* ($\times 10^4$) *for the non-hydrogen atoms*

Estimated standard deviations are given in parentheses. The b_{ij} are defined by:

$$T = \exp(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{13}hl + b_{23}kl) .$$

	x	y	z	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
O(12)	5382 (5)	2316 (3)	1197 (5)	123 (5)	68 (2)	203 (5)	1 (6)	12 (9)	−67 (6)
O(13)	4694 (5)	3558 (3)	1287 (4)	150 (5)	56 (2)	169 (4)	−25 (5)	−60 (8)	−22 (5)
O(17)	3333 (4)	1177 (2)	1073 (3)	134 (4)	47 (1)	92 (2)	27 (4)	18 (5)	−33 (3)
N(1)	790 (4)	3047 (3)	1305 (3)	119 (5)	45 (2)	90 (3)	24 (5)	−1 (7)	−23 (4)
N(5)	1222 (4)	1671 (2)	1157 (3)	114 (4)	41 (1)	44 (2)	3 (4)	6 (5)	−15 (3)
C(2)	2108 (6)	3170 (3)	1293 (4)	133 (6)	48 (2)	74 (3)	2 (6)	10 (8)	−21 (5)
C(3)	3060 (5)	2583 (3)	1239 (4)	116 (5)	46 (2)	60 (3)	12 (6)	16 (6)	−24 (4)
C(4)	2618 (5)	1766 (3)	1151 (3)	106 (5)	47 (2)	52 (2)	19 (5)	6 (6)	−24 (4)
C(6)	725 (6)	824 (3)	1067 (4)	154 (7)	41 (2)	55 (3)	−19 (6)	17 (7)	−15 (4)
C(7)	−667 (6)	801 (4)	721 (4)	154 (7)	63 (3)	61 (3)	−49 (7)	0 (8)	−27 (5)
C(8)	−1584 (6)	1329 (4)	1283 (5)	134 (7)	71 (3)	85 (4)	−38 (8)	15 (9)	−29 (6)
C(9)	−1096 (6)	2186 (4)	1212 (5)	114 (6)	64 (3)	85 (4)	10 (7)	−2 (8)	−11 (6)
C(10)	374 (6)	2300 (3)	1235 (4)	116 (5)	56 (2)	58 (3)	30 (6)	1 (7)	−18 (4)
C(11)	4480 (5)	2784 (4)	1241 (4)	117 (6)	56 (2)	79 (3)	4 (6)	−9 (8)	−31 (5)
C(14)	6106 (8)	3826 (5)	1346 (9)	148 (8)	74 (4)	245 (10)	−45 (10)	−91 (16)	−20 (11)
C(15)	6203 (10)	4595 (6)	952 (7)	264 (12)	110 (5)	140 (7)	−130 (13)	46 (16)	24 (11)
C(16)	913 (7)	395 (4)	1988 (5)	206 (9)	54 (3)	79 (4)	−7 (8)	8 (10)	20 (5)

graphic axes with Cu $K\alpha$ radiation. The summarized crystallographic data are as follows:

$a = 10\cdot04$ (1) Å   M.W.236·276

$b = 16\cdot73$ (1) $D_m = 1\cdot287$ g cm^{-3}

$c = 14\cdot66$ (2) $D_x = 1\cdot274$ g cm^{-3}

$\beta = 90\cdot1$ (5)° $Z = 8$

$F(000) = 1008$ $\mu(\text{Cu } K\alpha) = 7\cdot78$ cm^{-1}

The space group $A2/a$ has been determined from the systematic absences, hkl and $0kl$ if $k + l$ odd, $h0l$ and

Table 2. *Final fractional coordinates* ($\times 10^3$) *and isotropic thermal parameters* (Å^2) *of the hydrogen atoms*

Estimated standard deviations are given in parentheses.

	x	y	z	B
H(2)	236 (19)	372 (12)	125 (14)	12·96
H(6)	147 (6)	65 (4)	54 (5)	3·81
H(71)	−94 (8)	27 (5)	82 (6)	5·52
H(72)	−72 (8)	98 (5)	14 (6)	5·09
H(81)	−153 (9)	115 (5)	202 (6)	6·13
H(82)	−249 (10)	130 (6)	107 (7)	6·99
H(91)	−151 (6)	255 (4)	187 (5)	3·56
H(92)	−140 (8)	249 (5)	64 (6)	5·26
H(161)	178 (8)	32 (5)	210 (6)	5·83
H(162)	77 (9)	−15 (6)	192 (6)	6·00
H(163)	15 (9)	63 (6)	252 (6)	6·45

Table 3. *Observed and calculated structure factors* ($\times 10$)

Unobserved reflexions are marked with an asterisk.

Table 3 (*cont.*)

$h00$ if h odd (l = even) and $0k0$ if k odd. On the basis of intensity statistics the Aa space group was excluded. Intensity data were recorded on an equi-inclination Weissenberg camera for the layers 0 to 9, 0 to 9 and 0 to 2 around the a, b and c crystal axes, respectively. The intensities were estimated visually using calibrated intensity scales. In total 1900 reflexions were collected of which 304 were unobserved. For reflexions with zero intensity the value of $I_o = \frac{1}{4}I_{min}$ was taken. $\mu R \le 0\cdot3$ was satisfied for all samples and no absorption correction was made. To bring the intensities to common scale, multiple film and (after Lorentz–polarization correction) interlayer scaling were applied. The absolute scale and overall temperature factor $B = 4\cdot3$ Å^2 were determined by a Wilson plot from all the three-dimensional data.

Determination of the structure

The phase problem was solved by the symbolic addition method using E value magnitudes calculated from the observed structure-factor values. To perform the calculations the program set LSAM of Main, Woolfson & Germain (1968) was used; this is composed of three programs and operates completely automatically for centrosymmetric structures. For sign determination 479 reflexions with $E \ge 1\cdot04$ were chosen, taking the restriction of $P_{min} \ge 0\cdot80$.

At the first attempt to run the second program of LSAM with an allocation of four sign symbols, it was found, in contrast with earlier experience, that the frequencies of the symbols were very low and only 33 signs could be determined. Inspection of the origin-determining reflexions and of those to which the program had allocated sign symbols revealed that of the four reflexions with symbols only two were independent. To avoid such difficulties in the automatic operation, all the reflexions with highest weight which become candidates for symbol allocation are rejected if they are related in a triple product to two reflexions which have already been given signs or symbols. The program is flexible and this rejection test can easily be incorporated at the point where reflexions are saved for allocating signs or symbols to them.

After these alterations the program produced 10 probable sign combinations. Of these, the one with the highest figures of merit gave the right solution for the signs of 231 E values, only one of which later proved to be incorrect.

The calculated E map revealed electron density peaks for 13 of the 17 non-hydrogen atoms of the asymmetric unit. The Fourier function calculated from all structure factors, obtained from the previous 13 atoms, revealed the positions of all the remaining non-hydrogen atoms, resulting in a structure-factor agree-

ment of $R = 30.8\%$. For calculating the Fourier function the program of Domenicano & Vaciago (1966) was used.

The atomic parameters obtained from the Fourier function were refined directly by the block-diagonal least-squares method using the program of Albano, Domenicano & Vaciago (1966), minimizing the function $\varphi = \sum_h w_h (F_{oh} - 1/G|F_{ch}|)^2$ where G is the scaling factor. The weighting factor of Cruickshank (1961) $w_h = 1/(a + b \cdot F + c \cdot F^2)$ has been used with $a = 8.0$, $b = 1$ and $c = 0.004$. Three cycles with isotropic and three further cycles with anisotropic thermal parameters gave $R = 0.136$ for the observed reflexions.

The difference Fourier now revealed 11 hydrogen atoms. The positions of the 5 hydrogens of the ethoxyl group, could, however, not be determined, and even geometric generation was unsuccessful. The parameters of the 11 H atoms were refined by full matrix least-squares methods, applying three cycles with individual isotropic thermal parameters. Then one more cycle of anisotropic refinement was applied for the non-hydrogen atoms, resulting in the final structure-factor agreement $R = 0.113$ and $R = 0.130$ for the observed and all reflexions, respectively. The final atomic parameters are summarized in Tables 1 and 2. Structure factors calculated with the final atomic

Table 4. *Atomic least-squares plane of the molecule*

The plane constants $(Ax + By + Cz = D)$ are referred to the orthogonalized axial system as defined in *International Tables for X-ray Crystallography* (1959) and coordinates are considered in Å.
Plane constants: $A = 0.01030$, $B = -0.09243$, $C = 0.99567$, $D = 1.43427$

Atoms forming the plane:		Deviation from plane (Å)		
N(1), C(2), C(3), C(4),	0.0075,	−0.0154,	0.0064,	−0.0003,
N(5), C(6), C(9), C(10),	0.0088,	0.0032,	−0.0146,	0.0166,
C(11), O(12), O(17)	−0.0071,	0.0104,	−0.0156	

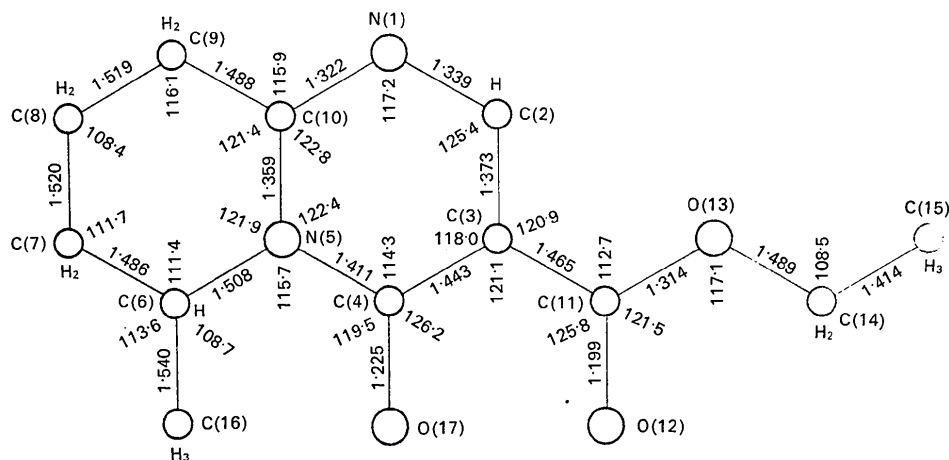| Atoms not forming the plane:				
C(7), C(8), O(13),	−0.5127,	0.2165,	−0.0574	
C(14), C(15), C(16)	0.0019,	−0.6911,	1.4158	

Fig. 1. Atomic numbering with bond lengths and angles of the molecule.

parameters are given in Table 3. The atomic scattering factors of all atoms were taken from *International Tables for X-ray Crystallography* (1962).

The geometry of the molecule

The schematic view of the molecule with atomic numbering can be seen in Fig. 1. The non-hydrogen atoms of the molecule are coplanar, with the exception of those in the ethoxyl and methyl groups and atoms C(7) and C(8) of the double ring. The least-squares plane of the coplanar part of the molecule and the deviations of all atoms from this plane are given in Table 4. The mean deviation of the coplanar atoms from this plane is less than 0·02 Å. The two neighbouring atoms C(7) and C(8) are on opposite sides of the plane. The bond C(6)–C(16) linking the methyl group to the double ring is inclined to the plane at an

Table 5. *Bond lengths and bond angles with their estimated standard deviations in parentheses*

N(1)—C(2)	1·339 (7) Å
N(1)—C(10)	1·322 (7)
N(5)—C(10)	1·359 (7)
N(5)—C(4)	1·411 (6)
N(5)—C(6)	1·508 (7)
C(2)—C(3)	1·373 (8)
C(3)—C(4)	1·443 (7)
C(3)—C(11)	1·465 (8)
C(6)—C(7)	1·486 (8)
C(6)—C(16)	1·540 (9)
C(7)—C(8)	1·520 (9)
C(8)—C(9)	1·519 (9)
C(9)—C(10)	1·488 (8)
C(4)—O(17)	1·225 (6)
C(11)–O(12)	1·199 (8)
C(11)–O(13)	1·314 (8)
C(14)–O(13)	1·489 (11)
C(14)–C(15)	1·414 (14)
C(2)—H(2)	0·953 (20) Å
C(6)—H(6)	1·106 (8)
C(7)—H(71)	0·933 (10)
C(7)—H(72)	0·913 (10)
C(8)—H(81)	1·115 (11)
C(8)—H(82)	0·962 (12)
C(9)—H(91)	1·219 (9)
C(9)—H(92)	1·020 (10)
C(16)–H(161)	0·894 (11)
C(16)–H(162)	0·926 (11)
C(16)–H(163)	1·165 (12)
Mean: C–H = 1·019 Å	
N(1)—C(2)—C(3)	125·4 (5)°
C(2)—C(3)—C(4)	118·0 (5)
C(3)—C(4)—N(5)	114·3 (4)
C(4)—N(5)—C(10)	122·4 (4)
N(5)—C(10)-N(1)	122·8 (5)
C(2)—N(1)—C(10)	117·2 (5)
C(10)-N(5)—C(6)	121·9 (4)
C(7)—C(6)—C(16)	113·6 (5)
N(5)—C(6)—C(16)	108·7 (4)
N(5)—C(4)—O(17)	119·5 (5)
C(3)—C(4)—O(17)	126·2 (5)
C(4)—C(3)—C(11)	121·1 (5)
C(4)——N(5)—C(6)	115·7 (4)

Table 5 (*cont.*)

|	
N(5)——C(6)—C(7)	111·4 (4)
C(6)——C(7)—C(8)	111·7 (5)
C(7)——C(8)—C(9)	108·4 (5)
C(8)——C(9)—C(10)	116·1 (5)
C(9)——C(10)-N(5)	121·4 (5)
C(9)——C(10)-N(1)	115·9 (5)
C(2)——C(3)—C(11)	120·9 (5)
C(3)——C(11)-O(12)	125·8 (6)
C(3)——C(11)-O(13)	112·7 (5)
C(12)—C(11)-O(13)	121·5 (6)
C(11)—O(13)-C(14)	117·1 (6)
O(13)—C(14)-C(15)	108·5 (8)
N(1)——C(2)—H(2)	114·2 (13)
C(3)——C(2)—H(2)	120·0 (13)
N(5)——C(6)—H(6)	94·6 (5)
C(7)——C(6)—H(6)	112·9 (6)
C(6)——C(16)-H(161)	110·5 (8)
C(6)——C(16)-H(162)	110·0 (8)
C(6)——C(16)-H(163)	110·8 (7)
H(161)-C(16)-H(162)	91·7 (10)
H(161)-C(16)-H(163)	124·1 (10)
H(162)-C(16)-H(163)	107·7 (10)
C(16)—C(6)—H(6)	114·0 (6)
C(6)——C(7)—H(71)	104·2 (7)
C(6)——C(7)—H(72)	111·7 (8)
C(8)——C(7)—H(71)	107·0 (7)
C(8)——C(7)—H(72)	106·3 (8)
C(7)——C(8)—H(81)	109·9 (7)
C(7)——C(8)—H(82)	111·2 (8)
C(9)——C(8)—H(81)	107·3 (7)
C(9)——C(8)—H(82)	109·4 (8)
C(8)——C(9)—H(91)	108·2 (6)
C(8)——C(9)—H(92)	115·1 (7)
C(10)—C(9)—H(91)	104·9 (6)
C(10)—C(9)—H(92)	104·7 (7)
C(6)——C(16)-H(161)	110·5 (8)
C(6)——C(16)-H(162)	110·0 (8)
C(6)——C(16)-H(163)	110·8 (7)
H(161)-C(16)-H(162)	91·7 (10)
H(161)-C(16)-H(163)	124·1 (10)
H(162)-C(16)-H(163)	107·7 (10)
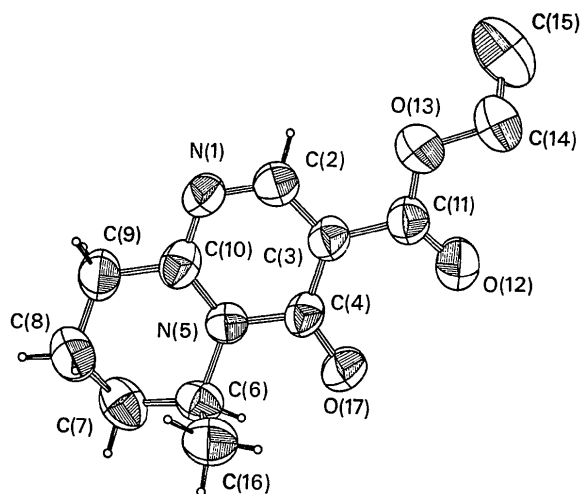
Fig. 2. The perspective view of the COMHHP molecule where the atoms are represented by their 50% probability thermal motion ellipsoids.

angle of 66·7° while the C(6)–H(6) bond has an inclination angle of 36·9° to the plane. The bond lengths and bond angles of the molecule can be seen in Table 5.

Thermal motion analyses

The r.m.s. amplitudes and the vectors of the principal axes of the thermal vibration ellipsoids of the non-hydrogen atoms are listed in Table 6. The perspective

Table 6. *The principal axes of the thermal vibrational ellipsoids for atoms in the asymmetric unit*

The axes are represented by their lengths (Å) and the coordinates of their end points (multiplied by 10^3) are referred to crystal axes. The origin of each vector is considered to be at the site of the corresponding atom.

	i	U_i (Å)	x	y	z
O(12)	1	0·483	537	240	88
	2	0·289	536	215	114
	3	0·250	513	233	120
O(13)	1	0·435	461	353	158
	2	0·298	451	370	128
	3	0·249	451	347	125
O(17)	1	0·331	332	126	87
	2	0·277	310	109	103
	3	0·219	322	128	113
N(1)	1	0·321	82	312	110
	2	0·263	98	314	136
	3	0·224	94	295	127
N(5)	1	0·252	124	180	107
	2	0·242	146	167	118
	3	0·205	125	161	104
C(2)	1	0·298	207	326	112
	2	0·262	186	312	128
	3	0·244	203	328	138
C(3)	1	0·280	306	271	111
	2	0·252	328	263	130
	3	0·209	315	251	114
C(7)	1	0·332	−49	64	79
	2	0·269	−47	84	60
	3	0·229	−56	88	83
C(8)	1	0·347	−148	117	142
	2	0·283	−150	125	113
	3	0·248	−136	139	130
C(9)	1	0·314	−107	231	105
	2	0·292	−107	232	134
	3	0·240	−86	217	121
C(10)	1	0·300	48	245	116
	2	0·248	52	231	137
	3	0·225	54	223	116
C(11)	1	0·321	445	266	141
	2	0·252	443	290	135
	3	0·243	424	277	121
C(14)	1	0·523	602	381	170
	2	0·338	596	401	134
	3	0·244	589	376	132
C(15)	1	0·449	592	480	96
	2	0·399	611	455	69
	3	0·286	599	449	101
C(4)	1	0·279	268	190	105
	2	0·237	282	178	124
	3	0·205	272	170	106
C(6)	1	0·291	97	76	114
	2	0·249	85	91	96
	3	0·223	74	92	117
C(16)	1	0·325	123	38	201
	2	0·308	93	29	182
	3	0·261	94	52	188

view of the molecule is given in Fig. 2, where non-hydrogen atoms are represented by their thermal ellipsoids. Calculations and plotting of these were carried out with the *ORTEP* program of Johnson (1965) adapted for use on the CDC 3300 computer.

The thermal motion of the molecule was also analysed by the rigid-body assumption, using the program of Schomaker & Trueblood (1968). If the whole molecule is assumed to be a rigid body the observed and calculated U tensors of the individual atoms show very great deviations. The whole molecule therefore cannot be treated as a rigid body. But if the ethoxyl group and O(12) atom are omitted the remainder behaves as a rigid body, and one obtains a satisfactory agreement of U tensors (Table 7).

Discussion of the structure

The conformation of the molecule COMHHP deviates to some extent from that of the recently published COMHP (Sasvári, Csonka Horvai & Simon, 1972), from which the former was derived by catalytic hydrogenation. The most important difference between the two molecules is found in the bond lengths and bond angles.

By hydrogenation of the COMHP molecule the left-hand ring, to which the methyl group is linked, becomes saturated; consequently the C–C bond lengths in this ring increase and approximate to the sp^3-sp^3 bond length (Table 5).

Further alterations in the molecule can be found at the N(5)–C bonds. After hydrogenation the N(5)–C(4) and N(5)–C(10) bond lengths, which in molecule COMHP are 1·472 and 1·401 Å, respectively, decrease to 1·411 and 1·359 Å. At the same time the N(5)–C(6) bond length increases to 1·508 Å, so that the average of the three N(5)–C bond lengths (1·426 Å) remains the same as before hydrogenation (1·428 Å).

The decrease in bond length N(5)–C(4) points to a decreased strain in the molecule, which in turn results in increased stability, in consequence of which no ring opening can be found in aqueous solution.

The spatial packing of the molecules in the unit cell conforms to the concept of molecular close packing. The shortest intermolecular distances are given in Table 8.

The shortest intermolecular H–H contact, occurring between the 11 known H positions has a length of 2·57 Å which corresponds to the van der Waals radius, and agrees with values of 1·26 and 1·27 Å found in COMHP and in bicyclohexylidene (Sasvári & Löw, 1965) respectively.

The halved value of the shortest intermolecular C–C distance is 1·69 Å, and compares with the well established van der Waals radius for carbon of 1·70 Å.

The shortest intermolecular C–H distance (2·79 Å) as well as the O–H distance (2·54 Å) is smaller than the sum of the corresponding van der Waals radii, even if the value 1·17 Å (Kitaigorodskii, 1961) is taken for

Table 7. *The observed and calculated components of the* U *tensors of the individual atoms referred to the orthogonalized axial system* (*in* 10^{-4} $Å^2$)

	U_{11}		U_{22}		U_{33}		U_{12}		U_{13}		U_{23}	
	obs	calc	obs	calc	obs	calc	obs	calc	obs	calc	obs	calc
C(2)	677	640	687	617	803	799	10	53	36	9	−129	−147
C(3)	594	582	653	654	609	601	52	43	59	3	−151	−152
C(4)	539	603	663	627	564	559	81	74	20	17	−146	−129
C(6)	784	774	586	666	594	627	−79	−50	63	68	−93	−125
C(7)	788	798	894	893	668	682	−207	−196	0	35	−170	−165
C(8)	684	665	1003	1019	928	868	−161	−129	53	64	−183	−138
C(9)	583	577	902	916	931	999	44	37	−9	−3	−68	−106
C(10)	591	576	797	694	629	637	126	57	3	0	−114	−100
C(11)	597	596	795	822	864	891	19	−5	−36	14	−195	−221
C(16)	1051	1025	773	763	857	864	−29	−61	29	12	125	116
N(1)	609	623	638	659	974	942	104	106	−7	5	−143	−126
N(5)	583	613	583	631	475	453	15	30	20	16	−90	−101
O(17)	683	692	660	674	996	969	115	131	65	55	−205	−168

hydrogen. The same phenomenon was found earlier in the structure of COMHP.

Table 8. *The shortest intermolecular atomic distances* (Å) *approximating van der Waals separations*

From atom in x,y,z	To atom	In position	Distance (Å)
H(2)	H(81)	$-x, \frac{1}{2}-y, \frac{1}{2}-z$	2·68
H(6)	H(71)	$-x, -y, -z$	2·58
H(71)	H(82)	$-\frac{1}{2}-x, y, -z$	2·57
H(82)	H(162)	$-\frac{1}{2}+x, -y, z$	2·89
H(6)	O(17)	$\frac{1}{2}-x, y, -z$	2·54
H(71)	O(17)	$-\frac{1}{2}+x, -y, z$	2·56
H(81)	C(2)	$-x, \frac{1}{2}-y, \frac{1}{2}-z$	2·79
C(4)	C(4)	$\frac{1}{2}-x, y, -z$	3·38

References

ALBANO, V., DOMENICANO, A. & VACIAGO, A. (1966). Block-diagonal least-squares refinement program. Università di Roma.

CRUICKSHANK, D. W. J. (1961). *Computing Methods and the Phase Problem in X-ray Crystal Analysis*. Oxford: Pergamon Press.

DOMENICANO, A. & VACIAGO, A. (1966). Fourier synthesis program.

International Tables for X-ray Crystallography (1959). Vol. II. Birmingham: Kynoch Press.

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

JOHNSON, C. K. (1965). *ORTEP*. ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tennessee, U.S.A.

KITAIGORODSKII, A. I. (1961). *Organic Chemical Crystallography* (Engl. trans.). New York: Consultants Bureau.

MAIN, P., WOOLFSON, M. M. & GERMAIN, G. (1968). LSAM System of Computer Programs for the Automatic Solution of Centrosymmetric Structures by the Logical Symbolic Addition Method, Universities of York, England and Leuven, Belgium.

MÉSZÁROS, Z., KNOLL, J., SZENTMIKLÓSI, P., DÁVID, A., HORVÁTH, G. & HERMETZ, I. (1972). *Arzneimittelforschung*, 5, 815–829.

SASVÁRI, K., LÖW, M. (1965). *Acta Cryst.* 19, 840–848.

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

SASVÁRI, K., CSONKA HORVAI, J. & SIMON, K. (1972). *Acta Cryst.* B28, 2405–2416.