ACTA CHEMICA SCANDINAVICA 21 (1967) 1206–1216

Hydrogen Bond Studies

12. The Crystal Structure of Hydrazine Bisethanol, $N_2H_4 \cdot 2C_2H_5OH$

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The crystal structure of $N_2H_4 \cdot 2C_2H_5OH$ has been determined from three-dimensional single-crystal X-ray data, recorded at −188°C. The crystals are orthorhombic, space group *Pbcn* with four formula units per unit cell with the dimensions: $a = 18.470$, $b = 4.889$, and $c = 8.755$ Å. The N_2H_4 and C_2H_5OH molecules are hydrogen bonded to each other forming infinite layers running parallel to the yz plane. The hydrogen bond lengths are: N−H···O 3.041 and 3.060 Å, respectively, and O−H···N 2.730 Å. The layers are held together by van der Waals forces.

The present investigation forms part of a series of structure determinations of simple hydrogen-bonded compounds found in the systems N_2H_4-H_2O, N_2H_4-C_2H_5OH, and N_2H_4-CH_3OH. The structure of $N_2H_4 \cdot H_2O$ has been reported earlier.[1] That of $N_2H_4 \cdot 2C_2H_5OH$ will be reported in this paper. Studies based on single crystal X-ray data of the compounds $N_2H_4 \cdot nCH_3OH$, $n = 1$, 2, 4, are in progress by the present author.

The freezing point diagram [2] of the system hydrazine-ethanol indicates only one intermediate compound, $N_2H_4 \cdot 2C_2H_5OH$, melting at −31.2°C. The present paper is concerned with the determination of the crystal structure of this compound based on three-dimensional X-ray data collected at −188°C.

EXPERIMENTAL

Anhydrous hydrazine was prepared by dehydration of commercial hydrazine (KEBO, 95−97 % N_2H_4) in the following way. The hydrazine was mixed with freshly dehydrated barium oxide, and this mixture was allowed to stand for some hours. The hydrazine was then distilled twice in a vacuum system.[3] The product was analyzed at the Central Analytical Laboratory at this Institute and was found to contain 100.0 % N_2H_4.

Anhydrous ethanol was prepared by dehydration of commercial 99.5 % ethanol according to the method of Lund and Bjerrum.[4] Magnesium, activated with iodine, is used as dehydrating agent in this method, which, when carefully performed, gives a product containing less than 0.05 % water.

After the dehydration was completed all subsequent operations with the purified materials were carried out in a dry box in an atmosphere of dry nitrogen.

A sample containing 2.02 moles C_2H_5OH per mole N_2H_4 was prepared by weighing. Capillaries (diameter about 0.3 mm, wall thickness 0.01—0.02 mm) were filled with this solution and were sealed in a flame.

Single crystals were grown in a low-temperature camera described earlier.[5] The melting point was about −32°C; thus in agreement with the earlier reported value.[3] Equi-inclination Weissenberg photographs were taken at −188 ± 2°C with Cu$K\alpha$ radiation using the multiple-film method (five films). Five layers were obtained with the crystal rotating about the [010] axis. The total number of independent reflexions recorded was 794 but 147 of these were too weak to be measured. About 88 % of the reflexions within the copper reflexion sphere were thus collected.

The relative intensities were measured visually by comparison with a calibrated scale. The intensity range was 1 to 13 000. The intensities were corrected for Lorentz and polarization effects. No absorption correction was applied, as the linear absorption coefficient for Cu$K\alpha$ radiation is 6.8 cm^{-1} and the diameter of the crystal was about 0.3 mm.

Some X-ray photographs were also taken at −50°C. No phase transformation was observed between −50 and −188°C.

UNIT CELL AND SPACE GROUP

The diffraction symmetry shown by the data was *mmm*, indicating an orthorhombic space group. The systematic absence of the reflexions $0kl$ for $k \neq 2n$, $h0l$ for $l \neq 2n$ and $hk0$ for $h + k \neq 2n$, indicated uniquely the space group *Pbcn*, assuming that the absences were not accidental. When the structure was refined in this space group good agreement was obtained with the observed data, thus confirming the choice of space group.

The atoms occupy the general eightfold positions in *Pbcn*:[6]

(x,y,z); $(\frac{1}{2}-x,\frac{1}{2}-y,\frac{1}{2}+z)$; $(\frac{1}{2}+x,\frac{1}{2}-y,\bar{z})$; $(\bar{x},y,\frac{1}{2}-z)$
$(\bar{x},\bar{y},\bar{z})$; $(\frac{1}{2}+x,\frac{1}{2}+y,\frac{1}{2}-z)$; $(\frac{1}{2}-x,\frac{1}{2}+y,z)$; $(x,\bar{y},\frac{1}{2}+z)$

The number of independent heavy atoms is four: one nitrogen, one oxygen, and two carbons. In addition there are eight independent hydrogen atoms.

The a and c dimensions of the unit cell were obtained from zero layer oscillation photographs and the b dimension from a rotation photograph. Each of these photographs was calibrated with a quartz single crystal at 24°C, and all exposures were recorded on the same film without removing the film from the cassette. The dimensions a and c, based on 51 observations, were fitted to the measured θ values by the method of least squares. The unit cell dimensions together with their estimated standard deviations are as follows (at −188°C):

$a = 18.470 \pm 0.001$, $b = 4.889 \pm 0.003$, $c = 8.7553 \pm 0.0005$ Å

(λ Cu$K\alpha_1 = 1.54051$ Å, λ Cu$K\alpha_2 = 1.54433$ Å, λ Cu$K\beta = 1.39217$ Å; $a = 4.9130$ and $c = 5.4046$ Å for α quartz at 24°C).

With four units of $N_2H_4 \cdot 2C_2H_5OH$ in the unit cell the calculated density is 1.043 $g \cdot cm^{-3}$. An experimental determination of the density was not made.

DETERMINATION OF THE ATOMIC COORDINATES

From a three-dimensional Patterson function a symmetry minimum function (designated SMF below) was calculated, which was based upon all symmetry relations. This function and the subsequent high order superposition

function were calculated with programmes described in Ref. 7. The programmes were modified by the present author to suit the Fourier programme used in Uppsala.

Two peaks from the SMF were selected as trial atomic positions and two separate high order superposition functions [7] were computed. These were compared with each other and with the SMF. The positions of the nitrogen and oxygen atoms (one independent of each kind) could be determined with confidence from this analysis, but the positions of the two independent carbons could only be approximately inferred. An electron density calculation was made based on the known oxygen and nitrogen positions, and the carbon atoms could then be located from the electron density maps.

The atomic coordinates, together with the inter-layer scale factors and isotropic temperature factors, were refined in a series of least-squares calculations. The total number of parameters varied was 21. After some cycles the agreement factor $R = \sum||F_o| - |F_c||/\sum|F_o|$ was 0.160 and the shifts were no longer significant. A $(F_o - F_c)$ synthesis was calculated, based on the reflexions with sin θ/λ less than 0.5 Å^{-1}. Reasonable positions for the eight independent hydrogen atoms could be deduced from the difference Fourier maps.

Some cycles of least-squares calculations were performed with the eight hydrogen atoms included with fixed parameters. The isotropic temperature factor B used for the hydrogen atoms was 5 Å^2. (The B value for the methyl carbon was 3.6 Å^2.) The R value was immediately reduced to 0.135 (before any refinement) when the hydrogen atoms were included, and dropped further to 0.120 after two cycles. Some errors in the data were noticed at this stage, and after correction of these and two further cycles the R factor was reduced to 0.111, and the shifts were about one tenth of the standard deviations.

Anisotropic temperature factors were now employed for nitrogen, oxygen, and carbon. The atomic coordinates of these atoms and an overall scale factor were also varied, making a total of 37 parameters varied. The inter-layer scale factors used were obtained from the last calculation with isotropic temperature factors. The R value after two cycles was 0.900. A difference Fourier, based on the heavy atoms, was calculated and the hydrogen positions were deduced again. Seven of the eight hydrogens were very well resolved while the peak corresponding to one of the methyl hydrogens was somewhat diffuse. However, all these peaks were at least twice as high as all of the spurious peaks. Two further cycles with the redetermined hydrogen positions included fixed gave a final R factor equal to 0.083. In the last cycle, the shifts of all parameters were less than one tenth of their estimated standard deviations.

The refinement was based on F, minimizing the function $\sum w(|F_o| - |F_c|)^2$. The weighting scheme used was $w = 1/(a + |F_o| + c|F_o|^2)$.[8] The final values used for a and c were 2.50 and 0.037, respectively. These values were suitable, as was shown by the analysis made in the least-squares program. Reflexions too weak to be measured were given zero weight in all calculations. The strongest reflexion (311) was inaccurately measured and was also excluded from all calculations except the Patterson function. The refinement was thus based on 646 observations.

The atomic parameters after the final least-squares cycle are listed in Tables 1 and 2 and the structure factors in Table 5. The root-mean-square

Table 1. Atomic coordinates with estimated standard deviations.

Atom	x	y	z
N	0.0126 ± 0.0001	0.2371 ± 0.0004	0.1709 ± 0.0002
O	0.4111 ± 0.0001	0.1996 ± 0.0004	0.1030 ± 0.0002
C(1)	0.3403 ± 0.0001	0.1850 ± 0.0007	0.1670 ± 0.0003
C(2)	0.3362 ± 0.0001	0.2993 ± 0.0008	0.3241 ± 0.0003
Hydrogen atoms:			
N: H(1)	0.033	0.039	0.152
H(2)	0.031	0.260	0.396
O: H(3)	0.438	0.038	0.131
C(1): H(4)	0.174	0.460	0.168
H(5)	0.301	0.275	0.090
C(2): H(6)	0.283	0.294	0.362
H(7)	0.153	0.017	0.321
H(8)	0.373	0.258	0.390

Table 2. Anisotropic temperature factor parameters with estimated standard deviations, each multiplied by 10^4. The expression used is: exp $[-(h^2\beta_{11} + hk\beta_{12}....)]$.

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
N	18 ± 1	213 ± 12	87 ± 3	6 ± 3	− 5 ± 2	16 ± 8
O	16 ± 1	236 ± 11	103 ± 3	− 1 ± 3	12 ± 2	29 ± 7
C(1)	14 ± 1	495 ± 18	118 ± 4	−13 ± 5	7 ± 2	−54 ± 12
C(2)	19 ± 1	521 ± 20	114 ± 4	− 3 ± 6	13 ± 3	−42 ± 13
H*	37	523	163			

* Isotropic temperature factor in anisotropic form; the B-value used for all hydrogen atoms was 5 Å^2.

Table 3. Root-mean-square components, R_i, of thermal vibration along principal axes of the ellipsoids of vibration, calculated from the β_{ij} values in Table 2 with the programme OR FFE.

Atom	R_1	R_2	R_3
N	0.16 Å	0.17 Å	0.19 Å
O	0.16	0.17	0.21
C(1)	0.16	0.21	0.25
C(2)	0.18	0.21	0.25
H*	0.25	0.25	0.25

* Isotropic temperature factor (See Table 2).

components of thermal displacement along principal axes of the ellipsoids are given in Table 3. Bond distances and angles are listed in Table 4. The standard deviations in these were calculated from the standard deviations of the atomic coordinates. The errors in the cell dimensions were also taken into account. The distances listed in Table 4 are not corrected for thermal motion.

The atomic scattering factors used in the calculations were those for neutral O, N, C, and H as given in the *International Tables*.[9]

Computer programmes: All calculations were performed on the CD 3600 computer in Uppsala, using the following programmes (some of these were briefly described in an earlier paper [10]):

1. *CELSIUS:* Refinement of unit cell dimensions, written by J. Tegenfeldt, Uppsala.
2. *DRF:* Data reduction and Fourier calculations. Local modification of a programme written by A. Zalkin, Berkeley, Calif.
3. *LALS:* Full matrix, least-squares calculations. Local modification of A. Zalkin's version of UCLALS1, originally written by P. K. Gantzel, R. A. Sparks and K. N. Trueblood, Univ. of Calif., Los Angeles, Calif.
4. *DISTAN:* Calculation of distances and angles, written by A. Zalkin.
5. *OR FFE:* Crystallographic function and error programme, written by W. R. Busing, K. O. Martin and H. A. Levy, Oak Ridge, Tennessee.
6. *OR TEP:* A thermal-ellipsoid plot programme for crystal structure illustrations, written by C. K. Johanson, Oak Ridge, Tennessee.

DISCUSSION OF THE STRUCTURE

The structure contains infinite layers of hydrogen-bonded N_2H_4 and C_2H_5OH molecules, running parallel to the *yz* plane (Fig. 1). The *x* coordinate

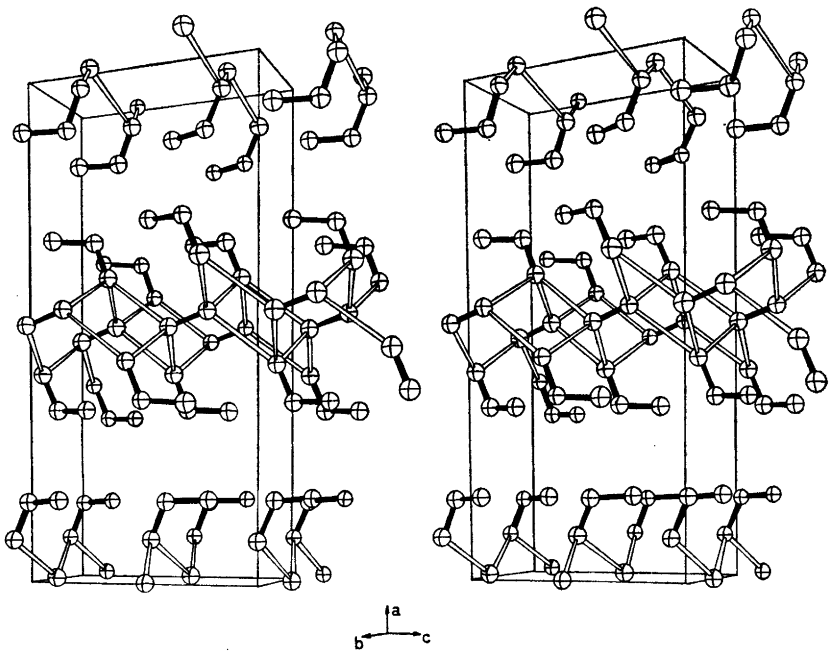

Fig. 1. A stereoscopic pair of drawings of the structure of $N_2H_4 \cdot 2C_2H_5OH$. Covalent bonds are filled, hydrogen bonds are open. Figs. 1, 2, and 4 were drawn with the programme OR TEP.

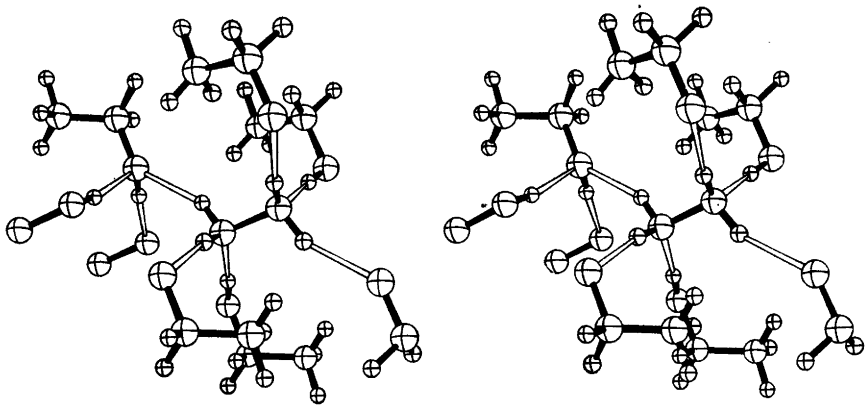

Fig. 2. A stereoscopic pair of drawings showing the location of the hydrogen atoms. The orientation is the same as in Fig. 1.

for the middle of the layers is 0 and 1/2, respectively. In the present structure the relative number of lone pairs and hydrogen atoms, available for hydrogen bonding, is the same. Each nitrogen and oxygen atom is involved in three hydrogen bonds; nitrogen as the acceptor in one bond and donor in two, while oxygen is the acceptor in two bonds and donor in one.

The hydrogen-bonded layers are held together by van der Waals forces. The shortest distance between the carbon atoms (methyl carbons) in two adjacent layers is 4.0 Å. Details of the structure and the hydrogen bonding system will be discussed below.

1. The hydrazine molecule. The N—N distance in the N_2H_4 molecule, 1.460 ± 0.004 Å, is in agreement with values reported earlier: 1.46 ± 0.02 Å in solid N_2H_4,[11] 1.447 ± 0.009 Å in $N_2H_4 \cdot H_2O$,[1] and 1.48 ± 0.02 in $CuCN \cdot N_2H_4$.[12]

The nitrogen atoms within an N_2H_4 molecule are related to each other by a twofold axis parallel to b (at $x = 0$, $z = 1/4$ etc.).

The dihedral angles between planes defined by the nitrogen atoms of an N_2H_4 molecule and the hydrogens and those of nitrogen and the nearest oxygen neighbours, respectively, are illustrated in Fig. 3. The angle φ of internal rotation of the NH_2 groups from the *cis* position is 73° assuming linear hydrogen bonds (Fig. 3). However, the N—H···O bonds are probably

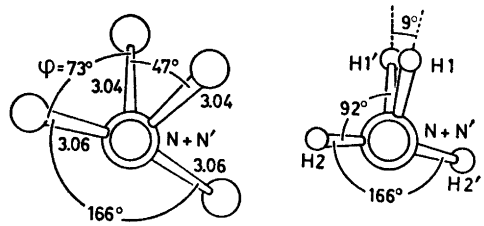

Fig. 3. View down the N—N bond of a N_2H_4 molecule showing dihedral angles between planes defined by N—N···O (left) and N—N—H (right).

more or less bent (see part 4 below); the dihedral angles between the N—N—H planes are also shown in Fig. 3, which indicates that the angle φ is about 90°.

The N_2H_4 molecule was assumed to have the eclipsed configuration (*i.e.* $\varphi = 0$ or 120°) in solid N_2H_4,[11,13] but in $N_2H_4 \cdot H_2O$ [1] the angle φ was found to be 58.5°. Linear hydrogen bonds were assumed in both cases.

2. The ethyl alcohol molecule. The C—O distance in C_2H_5OH, 1.424 ± 0.003 Å, is in agreement with the accepted value, 1.43 Å, for this type of bond,[14] while the C—C distance, 1.487 ± 0.004 Å is shorter than the normally observed C—C single bond, 1.534 Å.[15] (Mean value of 12 measurements made by electron diffraction or spectroscopic methods.) The angle C—C—O in the present case is 113°. The observed dimensions of the C_2H_5OH molecule in the gas phase (electron diffraction) are: C—O = 1.48 ± 0.04 Å, C—C—O angle = 109.5 ± 3°. (The C—C distance was assumed to be 1.54 Å).[16]

Some short C—C bonds of this type have been observed earlier, *e.g.* in crystals of $(TiCl_4 \cdot CH_3COOC_2H_5)_2$,[17] where the C—C bond in the ethyl group

Table 4. Distances and angles with estimated standard deviations (*cf.* Fig. 4). (Note that the errors in the distances and angles involving hydrogen may be large.)

Distances

1) Within N_2H_4

N—N	1.460 ± 0.004 Å
N—H	1.05, 1.00 Å

2) Within C_2H_5OH

O—C(1)	1.424 ± 0.003 Å
C(1)—C(2)	1.487 ± 0.004
C(1)—H	1.13, 1.08 Å
C(2)—H	1.04, 1.08, 0.91
O—H	0.97

3) Hydrogen bonds

N—H(1)···O	3.041 ± 0.003 Å
N—H(2)···O	3.060 ± 0.003
O—H(3)···N	2.730 ± 0.003

Angles

1) Around nitrogen

N—N—O	123.3 ± 0.2°
—O	109.4 ± 0.1
—O	111.8 ± 0.1
O—N—O	102.6 ± 0.1
—O	93.6 ± 0.1
O—N—O	115.7 ± 0.1
N—N—H	105, 107°
H—N—H	107

2) Around oxygen

N—O—N	86.5 ± 0.1°
—N	77.4 ± 0.1
—C(1)	151.1 ± 0.2
N—O—N	115.7 ± 0.1
—C(1)	110.3 ± 0.2
N—O—C(1)	113.1 ± 0.2
H—O—C(1)	109°

3) Around C(1)

O—C(1)—C(2)	113.0 ± 0.2°
O—C(1)—H	105, 111°
H—C(1)—H	104
H—C(1)—C(2)	109, 113

4) Around C(2)

C(1)—C(2)—H	110, 110, 118°
H—C(2)—H	102, 120, 96

was 1.481 Å. The C—O distance, 1.495 Å, was in the latter case on the other hand longer than usual.

3. The hydrogen bonding system. As was mentioned earlier, the number of free electron pairs and the number of hydrogen atoms available for hydrogen bonding is the same per unit of $N_2H_4 \cdot 2C_2H_5OH$. In such cases all lone pairs and hydrogen atoms can be expected to be involved in hydrogen bonding. This is probably the case in the present compound, too.

Each nitrogen atom is surrounded by three oxygen atoms and each oxygen by three nitrogen atoms at possible hydrogen bond distances; two are approximately equal, 3.041 and 3.060 Å, respectively, while the third contact, 2.730 Å, is much shorter (Table 4, Fig. 4). It is reasonable to assume that the two longer ones represent the N—H···O bonds and the shortest one the O—H···N bond.

The N—H···O bonds (3.041, 3.060 Å) can be compared with those in $N_2H_4 \cdot H_2O$ [1] (3.113, 3.149 Å). However, in the latter case, each lone pair of the water molecule accepts two hydrogen atoms, which can be expected to lengthen the N—H···O bonds. (In $N_2H_4 \cdot H_2O$ there are four lone pairs to be divided by six hydrogen atoms available for hydrogen bonding per formula unit.)

The O—H···N bond (2.730 Å) in $N_2H_4 \cdot 2C_2H_5OH$ is shorter than the corresponding bond in $N_2H_4 \cdot H_2O$ (2.790 Å). When these bonds are compared, two of the main differences are: 1) The substitution of hydrogen in water with an ethyl group to give C_2H_5OH; 2) The differences in coordination number of oxygen. As the electronegativities of H and C_2H_5 can be assumed to be comparable, the shortening of the N—H···O bond may be due to the decreased coordination number of oxygen; six in $N_2H_4 \cdot H_2O$ as compared with four in $N_2H_4 \cdot C_2H_5OH$. A shortening of the N—H···X bonds with decreasing coordination number of X in a series of ammonium and alkylammonium halides has been observed by Lindgren and Olovsson.[18]

The bond angles subtended at the oxygen and nitrogen atoms deviate less than about 7° from a tetrahedral angle, except when the N—O bond of 3.060 Å is involved (Table 4, Fig. 4). In the latter case the deviations are in some cases large.

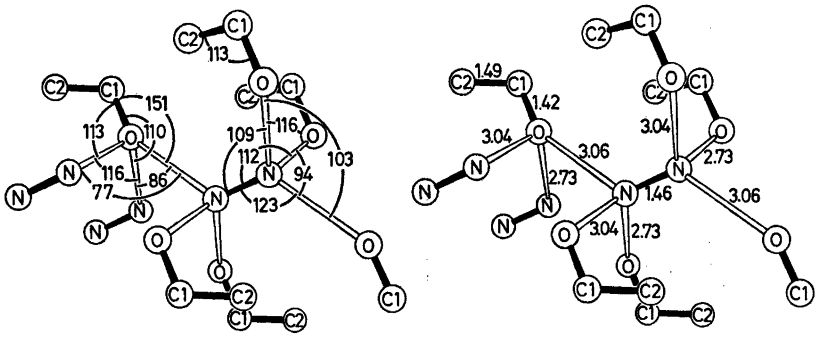

Fig. 4. Bond distances and angles. The orientation of the molecules is the same as in the left part of Fig. 1.

Table 5. Observed and calculated structure factors. Reflexions not included in the refinement are marked with an asterisk: one strong reflexion and those which were too weak to be measured. The F_o values for the latter are given as $\frac{1}{\sqrt{2}} F_{min}$ for the reflexions in question.

h	k	l	$\|F_o\|$	$\|F_c\|$
2	0	0	34.6	38.6
4	0	0	32.4	34.6
6	0	0	52.3	57.8
8	0	0	4.3	1.8
10	0	0	12.4	10.6
12	0	0	51.5	56.8
14	0	0	4.8	5.1
16	0	0	23.7	26.0
18	0	0	5.1	5.0
20	0	0	3.9	2.9
22	0	0	4.3	4.2
0	0	2	47.9	56.3
1	0	2	28.9	30.1
2	0	2	2.7	0.8
3	0	2	52.5	59.6
4	0	2	7.7	6.5
5	0	2	29.9	28.6
6	0	2	54.1	57.5
7	0	2	6.4	6.4
8	0	2	6.7	6.7
9	0	2	20.0	15.6
10	0	2	14.1	12.3
11	0	2	5.2	5.4
12	0	2	15.1	14.5
13	0	2	25.1	25.2
14	0	2	2.9	2.5
15	0	2	20.9	24.1
16	0	2	3.5	3.0
17	0	2	6.1	5.8
18	0	2	10.5	9.8
19	0	2	4.5	3.7
20	0	2	1.3	0.1 *
21	0	2	2.4	2.3
22	0	2	8.0	7.3
23	0	2	4.7	4.7
0	0	4	68.1	72.4
1	0	4	12.0	10.9
2	0	4	18.4	18.1
3	0	4	7.9	7.0
4	0	4	13.7	14.3
5	0	4	7.0	6.0
6	0	4	0.9	1.7 *
7	0	4	20.3	18.4
8	0	4	4.3	3.0
9	0	4	25.7	24.8
10	0	4	10.6	9.0
11	0	4	11.3	10.6
12	0	4	19.5	21.7
13	0	4	1.2	0.3 *
14	0	4	2.8	2.5
15	0	4	5.6	5.5
16	0	4	12.4	12.6
17	0	4	8.5	7.8
18	0	4	3.2	2.1
19	0	4	9.6	7.7
20	0	4	1.6	1.3
21	0	4	9.9	9.2
22	0	4	0.7	0.5 *
0	0	6	25.7	25.4
1	0	6	10.8	9.7
2	0	6	1.1	0.3 *
3	0	6	12.8	12.8
4	0	6	11.0	9.8
5	0	6	2.0	1.2
6	0	6	41.0	43.9
7	0	6	4.7	4.2
8	0	6	7.1	7.0
9	0	6	12.2	10.8
10	0	6	13.8	12.7
11	0	6	6.1	5.5
12	0	6	11.5	11.5
13	0	6	6.8	6.6
14	0	6	3.7	3.1
15	0	6	3.7	3.9
16	0	6	3.1	2.1
17	0	6	3.0	2.0
18	0	6	13.8	11.8
19	0	6	3.1	2.6
20	0	6	0.6	0.2 *
0	0	8	9.3	10.5
1	0	8	3.1	2.9
2	0	8	0.3	0.3 *
3	0	8	6.6	6.5
4	0	8	3.1	3.0
5	0	8	1.8	1.8
6	0	8	17.1	18.0
7	0	8	11.5	10.6
8	0	8	4.3	3.6
9	0	8	13.2	11.8
10	0	8	3.7	3.7
11	0	8	3.6	2.9
12	0	8	7.1	5.6
13	0	8	1.2	0.4 *
14	0	8	0.3	0.3 *
15	0	8	1.0	0.4 *
16	0	8	0.8	1.0 *
17	0	8	0.2	2.7 *
18	0	8	2.5	2.6
0	0	10	1.2	0.6 *
1	0	10	2.1	1.6
2	0	10	1.2	0.3 *
3	0	10	3.4	3.7
4	0	10	3.0	1.7
5	0	10	9.6	7.7
6	0	10	2.5	2.2
7	0	10	2.4	1.9
8	0	10	5.0	4.6
9	0	10	4.9	4.2
10	0	10	3.3	4.2
11	0	10	9.7	8.5
1	0	12	18.8	19.1
3	0	12	11.4	11.7
5	0	12	2.0	2.1
7	0	12	7.8	7.0
9	0	12	5.6	4.9
15	1	0	2.8	2.9
17	1	0	4.5	4.7
19	1	0	1.2	0.2 *
21	1	0	2.2	1.6
1	1	1	17.1	17.3
3	1	1	29.4	32.4
5	1	1	78.6	100.7 *
7	1	1	22.6	23.9
9	1	1	32.8	36.4
11	1	1	11.1	10.5
13	1	1	16.1	15.9
15	1	1	28.3	29.9
17	1	1	33.0	36.9
19	1	1	21.0	20.9
21	1	1	7.7	6.8
23	1	1	1.4	0.3
13	1	1	14.9	14.8
14	1	1	8.4	8.2
15	1	1	20.1	21.2
16	1	1	1.2	0.8 *
17	1	1	8.9	8.8
18	1	1	10.0	10.5
19	1	1	7.6	7.0
20	1	1	9.7	9.0
21	1	1	4.1	3.7
22	1	1	9.6	8.8
23	1	1	2.5	1.9
1	1	2	7.1	6.4
2	1	2	30.6	34.9
3	1	2	2.0	2.2
4	1	2	9.6	6.5
5	1	2	6.3	6.6
6	1	2	0.8	0.9 *
7	1	2	1.2	0.7
8	1	2	2.8	1.9
10	1	2	14.1	13.8
11	1	2	2.6	2.7
12	1	2	9.1	8.8
13	1	2	2.1	0.4 *
14	1	2	9.4	9.8
15	1	2	1.2	0.7 *
16	1	2	1.2	1.1 *
17	1	2	1.2	0.2 *
18	1	2	4.9	4.6
19	1	2	1.2	0.7 *
20	1	2	1.2	0.6 *
21	1	2	1.1	0.4 *
22	1	2	0.9	1.6 *
1	1	3	29.0	31.2
2	1	3	5.1	5.1
3	1	3	7.4	4.5
4	1	3	4.4	4.8
5	1	3	25.7	27.1
6	1	3	16.4	15.8
7	1	3	20.3	19.5
8	1	3	19.8	19.4
9	1	3	13.0	11.9
10	1	3	18.0	16.7
11	1	3	18.0	17.6
12	1	3	11.5	12.2
13	1	3	7.1	7.6
14	1	3	4.3	4.3
15	1	3	2.9	3.5
16	1	3	5.0	4.4
17	1	3	9.0	9.1
18	1	3	11.7	11.6
19	1	3	2.9	2.7
20	1	3	9.3	8.2
21	1	3	6.2	4.9
22	1	3	4.4	4.1
1	1	4	8.0	7.4
2	1	4	9.4	8.6
3	1	4	4.6	3.8
4	1	4	16.0	16.1
5	1	4	9.2	8.0
6	1	4	2.7	2.1
7	1	4	5.7	4.9
8	1	4	12.7	11.9
9	1	4	1.0	1.2 *
10	1	4	5.4	4.5
11	1	4	5.2	4.4
12	1	4	5.3	4.7
13	1	4	3.1	3.0
14	1	4	2.9	2.6
15	1	4	2.0	2.0
16	1	4	3.8	3.3
17	1	4	2.1	2.4
18	1	4	1.7	1.7
19	1	4	1.2	0.2 *
20	1	4	4.2	3.9
21	1	4	0.8	0.6 *
22	1	4	0.4	0.9 *
1	1	5	3.7	3.0
2	1	5	23.7	24.7
3	1	5	33.5	34.5
4	1	5	17.2	17.2
5	1	5	8.6	8.3
6	1	5	4.7	3.5
7	1	5	2.1	1.5
8	1	5	8.9	9.2
9	1	5	20.7	21.4
10	1	5	6.8	6.9
11	1	5	3.3	3.0
12	1	5	12.5	11.9
13	1	5	7.0	6.8
14	1	5	14.8	15.9
15	1	5	9.4	10.2
16	1	5	6.5	5.9
17	1	5	2.9	2.8
18	1	5	1.1	0.8 *
19	1	5	6.4	5.5
20	1	5	1.9	1.8
21	1	5	4.1	4.1
1	1	6	5.3	4.7
3	1	6	1.8	1.8
4	1	6	3.0	2.3
5	1	6	5.0	4.5
6	1	6	3.1	2.7
7	1	6	1.1	0.1 *
8	1	6	2.5	2.3
9	1	6	5.4	4.3
10	1	6	2.8	2.7
11	1	6	1.7	1.1
12	1	6	3.5	3.4
13	1	6	1.2	0.4 *
14	1	6	1.2	0.8 *
15	1	6	1.2	0.6 *
16	1	6	1.7	1.3
17	1	6	1.1	0.9 *
18	1	6	1.0	0.3 *
19	1	6	0.8	1.2 *
1	1	7	14.3	14.6
2	1	7	1.1	0.8 *
3	1	7	19.6	22.2
4	1	7	4.6	4.4
5	1	7	6.2	6.3
6	1	7	1.7	1.5
7	1	7	8.2	7.9
8	1	7	2.6	2.2
9	1	7	18.1	20.2
10	1	7	4.0	3.6
11	1	7	10.9	11.3
12	1	7	3.9	3.9
13	1	7	1.2	0.5 *
14	1	7	1.7	1.5 *
15	1	7	6.8	6.4
16	1	7	2.8	2.7
17	1	7	3.8	4.0
18	1	7	1.2	0.9 *
19	1	7	1.2	0.7 *
1	1	8	2.3	1.7
2	1	8	1.2	0.1 *
3	1	8	1.2	0.1 *
4	1	8	1.2	0.1 *
5	1	8	1.2	0.4 *
6	1	8	1.2	0.3 *
7	1	8	1.2	0.7 *
8	1	8	0.9	0.9 *
9	1	8	1.2	0.1 *
10	1	8	1.2	0.6 *
11	1	8	2.1	1.5
12	1	8	3.1	2.2
13	1	8	1.2	0.6 *
14	1	8	1.1	0.1 *
15	1	8	0.8	0.3 *
16	1	8	1.2	1.6 *
1	1	9	1.5	2.0
2	1	9	6.7	6.5
3	1	9	3.9	4.4
4	1	9	7.1	7.1
5	1	9	3.8	3.4
6	1	9	3.2	2.6
7	1	9	2.7	1.9
8	1	9	2.0	2.0
9	1	9	1.1	1.8
10	1	9	1.1	0.3 *
11	1	9	2.2	2.0
12	1	9	6.2	6.4
13	1	9	0.7	0.6 *
1	1	10	1.6	1.3
2	1	10	1.9	2.1
3	1	10	1.5	0.8
4	1	10	2.9	2.3
5	1	10	1.8	1.7
6	1	10	1.0	0.8 *
7	1	10	0.9	1.3 *
8	1	10	2.1	2.1
9	1	10	1.3	1.3
10	1	10	1.0	1.3
11	1	11	3.5	3.8
12	1	11	0.6	0.9 *
13	1	11	5.3	5.9
14	1	11	1.6	2.1
0	2	0	25.5	26.7
2	2	0	5.5	6.1
4	2	0	20.4	21.6
6	2	0	7.3	7.6
8	2	0	14.6	15.2
10	2	0	29.9	34.0
12	2	0	4.0	4.3
14	2	0	13.4	14.7
16	2	0	2.8	2.5
18	2	0	2.3	1.6
20	2	0	2.3	1.9
22	2	0	0.8	1.2 *
1	2	1	21.9	20.3
3	2	1	23.0	21.4
5	2	1	13.8	12.9
7	2	1	29.6	28.2
9	2	1	7.1	6.4
11	2	1	2.3	2.4
13	2	1	6.8	6.2
15	2	1	1.9	1.7
17	2	1	2.1	1.6
19	2	1	7.6	6.9
21	2	1	4.5	4.2
23	2	1	6.8	6.8
1	2	2	7.4	7.8
3	2	2	9.5	10.1
5	2	2	3.1	3.1
7	2	2	8.0	8.5
9	2	2	5.2	5.2
11	2	2	4.1	3.9
13	2	2	2.9	2.4
15	2	2	2.9	1.9
17	2	2	2.9	2.4
19	2	2	0.7	0.0 *
21	2	2	0.5	0.5 *
0	2	2	21.6	18.9
1	2	2	12.7	12.0
2	2	2	5.9	5.1
3	2	2	28.9	27.5
4	2	2	11.0	10.1
5	2	2	11.9	12.4
6	2	2	26.1	27.1
7	2	2	9.4	7.8
8	2	2	6.9	6.6
9	2	2	7.2	7.3
10	2	2	3.9	3.3
11	2	2	6.1	6.3
12	2	2	8.9	8.4
13	2	2	16.1	17.1
14	2	2	2.8	2.2
15	2	2	15.3	17.3
16	2	2	0.9	0.6 *
17	2	2	5.9	5.8
18	2	2	7.0	6.1
19	2	2	1.7	1.5
20	2	2	0.8	0.3 *
21	2	2	2.2	2.0
22	2	2	3.0	4.5
0	2	3	18.5	17.6
1	2	3	22.2	21.6
2	2	3	8.4	7.4
3	2	3	10.1	9.1
4	2	3	11.2	10.3
5	2	3	11.9	12.0
6	2	3	13.0	12.5
7	2	3	11.8	10.2
8	2	3	3.9	3.7
9	2	3	2.6	2.1
10	2	3	7.2	7.1
11	2	3	11.0	10.9
12	2	3	8.5	8.6
13	2	3	9.0	9.3
14	2	3	0.8	0.6 *
15	2	3	7.9	7.9
16	2	3	5.1	5.2
17	2	3	6.4	6.2
18	2	3	3.2	3.0
19	2	3	2.4	2.1
20	2	3	0.7	0.2 *
21	2	3	3.3	3.4
0	2	4	31.9	34.7
1	2	4	7.0	7.1
2	2	4	10.1	10.1
3	2	4	3.9	4.0
4	2	4	6.9	7.1
5	2	4	4.3	4.1
6	2	4	1.0	1.3
7	2	4	10.2	10.2
8	2	4	0.7	0.7 *
9	2	4	16.3	16.4
10	2	4	8.0	7.8
11	2	4	8.6	8.8
12	2	4	13.1	12.7
13	2	4	1.5	1.3
14	2	4	1.9	1.9
15	2	4	5.1	4.9
16	2	4	6.6	6.0
17	2	4	7.6	7.1
18	2	4	2.2	1.7
19	2	4	7.0	6.8
20	2	4	0.6	0.8 *
0	2	5	6.2	5.5
1	2	5	2.9	1.2
2	2	5	3.3	2.6
3	2	5	9.8	10.2
4	2	5	0.7	0.1 *
5	2	5	10.5	10.7
6	2	5	2.9	3.1
7	2	5	12.1	12.3
8	2	5	0.8	0.9 *
9	2	5	10.2	10.0
10	2	5	0.8	1.0 *
11	2	5	4.3	4.1
12	2	5	2.2	2.6
13	2	5	0.9	1.3 *
14	2	5	1.8	1.7
15	2	5	1.8	1.8
16	2	5	1.2	1.3
17	2	5	3.0	3.0
18	2	5	0.7	0.4 *
19	2	5	4.6	5.0 *
20	2	5	0.9	0.6 *
0	2	6	13.3	14.0
1	2	6	5.1	5.5
2	2	6	3.0	3.0
3	2	6	7.0	7.0
4	2	6	1.7	1.5
5	2	6	9.0	9.7 *
6	2	6	0.8	0.0 *
7	2	6	23.9	27.0
8	2	6	4.4	3.7
9	2	6	6.6	6.7
10	2	6	5.6	5.3
11	2	6	3.9	3.6
12	2	6	7.6	7.5
13	2	6	4.2	4.4
14	2	6	3.4	2.7
15	2	6	2.4	2.7
16	2	6	0.8	0.1 *
17	2	6	2.1	1.6
18	2	6	6.8	7.7
0	2	7	4.7	4.6
1	2	7	2.0	1.2
2	2	7	3.2	2.9
3	2	7	3.3	3.2
4	2	7	3.7	3.8
0	2	8	6.3	6.8
1	2	8	2.5	2.5
2	2	8	2.5	1.7
3	2	8	4.1	4.3
4	2	8	4.1	4.1
5	2	8	0.9	0.4 *
6	2	8	11.0	11.8
7	2	8	6.7	6.9
8	2	8	8.0	8.3
9	2	8	1.5	1.3
10	2	8	3.7	3.4
11	2	8	0.7	0.2 *
12	2	8	0.6	0.5 *
13	2	8	0.4	0.8 *
14	2	8	0.6	0.5 *
15	2	8	0.9	0.9 *
0	2	9	3.1	2.4
1	2	9	1.2	1.4
2	2	9	2.4	2.6
3	2	9	0.9	0.8 *
4	2	9	5.5	5.2
5	2	9	2.8	2.7
6	2	9	5.3	5.5 *
7	2	9	0.4	0.8 *
8	2	9	0.9	0.6 *
9	2	9	4.6	4.6
10	2	9	1.7	1.7
11	2	9	2.5	2.6
12	2	9	0.4	0.3 *
13	2	9	0.9	0.8 *
0	2	10	1.0	1.6
1	2	10	1.0	0.7
2	2	10	1.2	1.0
3	2	10	0.7	0.3 *
4	2	10	2.3	2.7
5	2	10	2.2	1.9
6	2	10	4.8	5.6
7	2	10	1.8	1.9
8	2	10	1.0	1.5
0	3	0	13.2	14.1
1	3	0	7.6	7.4
2	3	0	22.6	22.6
3	3	0	10.0	11.1
4	3	0	1.1	0.3 *
5	3	0	7.0	7.9
6	3	0	6.5	6.7
7	3	0	10.1	9.7
8	3	0	1.1	0.4 *
1	3	1	3.4	3.9
2	3	1	8.3	7.6
3	3	1	30.5	28.7
4	3	1	8.7	6.3
5	3	1	17.7	17.1
6	3	1	3.7	4.2
7	3	1	11.5	11.4
8	3	1	11.4	12.3
9	3	1	14.1	13.6
10	3	1	7.2	8.3
11	3	1	1.2	0.1 *
12	3	1	1.3	0.9 *
13	3	1	2.5	1.9
14	3	1	1.3	0.7
15	3	1	9.3	8.7
16	3	1	1.4	1.1 *
17	3	1	4.3	3.9
18	3	1	6.0	5.2
19	3	1	1.1	1.5 *
20	3	1	4.8	5.5
1	3	2	7.1	6.3
2	3	2	29.5	28.4
3	3	2	2.8	1.7
4	3	2	1.9	0.8
5	3	2	1.6	1.9
6	3	2	8.2	7.0
7	3	2	3.6	2.6
8	3	2	14.2	15.4
9	3	2	5.8	5.5
10	3	2	1.3	1.4 *
11	3	2	1.3	0.9 *
12	3	2	14.3	15.1
13	3	2	1.4	1.5 *
14	3	2	1.4	1.6 *
15	3	2	1.3	0.2 *
16	3	2	8.6	8.2
17	3	2	2.5	1.1 *
18	3	2	0.8	0.6 *
1	3	3	10.1	10.3
2	3	3	0.9	0.7 *
3	3	3	1.0	1.2 *
4	3	3	2.2	0.9
5	3	3	8.7	8.1
6	3	3	7.2	6.5
7	3	3	5.9	5.4
8	3	3	5.1	4.5
9	3	3	15.6	15.6
10	3	3	4.8	4.2
11	3	3	8.1	8.5
12	3	3	7.4	7.0
13	3	3	6.1	6.3
14	3	3	3.6	3.5
15	3	3	5.1	5.7
16	3	3	1.9	1.7
17	3	3	5.1	4.5
18	3	3	4.0	3.9
19	3	3	6.4	6.4
1	3	4	9.5	9.3

Table 5. Continued.

h	k	l	$\lvert F_o\rvert$	$\lvert F_o\rvert$	h	k	l	$\lvert F_o\rvert$	$\lvert F_o\rvert$	h	k	l	$\lvert F_o\rvert$	$\lvert F_o\rvert$	h	k	l	$\lvert F_o\rvert$	$\lvert F_o\rvert$	h	k	l	$\lvert F_o\rvert$	$\lvert F_o\rvert$
2	3	4	4.8	4.3	9	3	6	4.8	4.4	4	4	0	8.2	7.6	17	4	2	3.2	5.0	7	4	5	9.7	10.0
3	3	4	4.1	4.1	10	3	6	1.9	1.8	6	4	0	9.0	9.8	18	4	2	0.3	1.1 *	8	4	5	1.2	1.3
4	3	4	14.9	16.1	11	3	6	6.7	6.9	8	4	0	5.9	6.3	0	4	3	13.2	12.2	9	4	5	8.9	8.7
5	3	4	11.3	10.9	12	3	6	1.9	1.5	10	4	0	7.5	8.0	1	4	3	13.2	12.5	10	4	5	0.8	0.5 *
6	3	4	1.7	1.2	13	3	6	1.3	1.5 *	12	4	0	8.6	8.1	2	4	3	4.8	4.5	11	4	5	2.7	2.3
7	3	4	5.9	6.3	14	3	6	2.9	2.3 *	14	4	0	3.0	2.4	3	4	3	6.6	5.7	12	4	5	2.4	2.5
8	3	4	14.7	14.8	15	3	6	1.0	1.1 *	16	4	0	0.7	0.7 *	4	4	3	8.2	7.7	13	4	5	1.9	1.9
9	3	4	1.3	0.2 *	16	3	6	2.0	2.5	18	4	0	0.5	0.1 *	5	4	3	8.1	7.4	14	4	5	1.7	1.6
10	3	4	4.6	4.5	1	3	7	8.4	9.0	0	4	1	11.8	12.1	6	4	3	9.2	9.3	15	4	5	1.5	1.5
11	3	4	6.4	6.7	2	3	7	1.4	0.6 *	1	4	1	13.8	11.9	7	4	3	5.9	6.2	0	4	6	7.4	6.8
12	3	4	6.6	6.8	3	3	7	9.8	10.4	2	4	1	6.8	7.3	8	4	3	2.8	2.6	1	4	6	2.0	1.5
13	3	4	4.8	4.5	4	3	7	1.4	1.6 *	3	4	1	12.3	12.2	9	4	3	0.8	0.8 *	2	4	6	5.5	5.8
14	3	4	3.2	2.4	5	3	7	1.4	0.4 *	4	4	1	5.0	2.9	10	4	3	6.3	6.2	3	4	6	2.6	1.8
15	3	4	4.1	4.0	6	3	7	1.4	1.0 *	5	4	1	2.5	1.3	11	4	3	7.0	7.0	4	4	6	6.5	6.8
16	3	4	5.7	5.7	7	3	7	1.4	0.1 *	6	4	1	5.2	4.3	12	4	3	6.7	7.6	5	4	6	0.8	0.1 *
17	3	4	4.7	5.1	8	3	7	1.4	0.3 *	7	4	1	3.3	2.3	13	4	3	7.4	8.1	6	4	6	8.6	8.0
18	3	4	2.4	2.5	9	3	7	9.5	9.3	8	4	1	2.4	1.5	14	4	3	0.8	0.4 *	7	4	6	1.6	1.2
19	3	4	0.7	0.8 *	10	3	7	1.9	2.0	9	4	1	6.0	6.0	15	4	3	5.6	6.9	8	4	6	4.8	4.6
1	3	5	4.9	5.0	11	3	7	6.3	6.5	10	4	1	5.3	5.6	16	4	3	4.4	5.3	9	4	6	2.6	2.1
2	3	5	9.4	10.0	12	3	7	2.0	2.1	11	4	1	4.3	4.4	17	4	3	2.5	3.9	10	4	6	2.5	2.4
3	3	5	13.2	14.5	13	3	7	2.5	2.5	12	4	1	6.1	6.8	0	4	4	9.5	9.1	11	4	6	1.6	1.4
4	3	5	6.5	6.4	14	3	7	0.8	1.4 *	13	4	1	7.6	8.9	1	4	4	2.2	1.9	12	4	6	2.8	2.9
5	3	5	6.1	6.6	1	3	8	3.1	3.1	14	4	1	2.3	2.6	2	4	4	5.3	4.8	13	4	6	0.4	0.7 *
6	3	5	2.2	1.8	2	3	8	1.4	0.9 *	15	4	1	6.5	7.6	3	4	4	0.7	0.7 *	0	4	7	4.6	4.5
7	3	5	4.0	4.2	3	3	8	1.4	0.1 *	16	4	1	3.5	3.9	4	4	4	1.0	1.0	1	4	7	1.6	1.0
8	3	5	1.3	1.4 *	4	3	8	7.4	7.7	17	4	1	2.6	2.6	5	4	4	5.1	4.7	2	4	7	0.8	0.7 *
9	3	5	9.2	9.5 *	5	3	8	1.3	0.5 *	18	4	1	1.8	2.6	6	4	4	1.1	1.2	3	4	7	0.8	0.9 *
10	3	5	1.3	1.3 *	6	3	8	1.3	0.3 *	0	4	2	8.4	6.3	7	4	4	4.3	3.7	4	4	7	5.2	5.3
11	3	5	1.9	2.4	7	3	8	1.3	1.0 *	1	4	2	2.0	1.3	8	4	4	2.0	1.4	5	4	7	2.0	1.6
12	3	5	6.5	5.8	8	3	8	7.4	7.6	2	4	2	6.4	5.6	9	4	4	5.5	5.1	6	4	7	6.5	7.0
13	3	5	1.4	0.8 *	9	3	8	1.1	0.2 *	3	4	2	7.1	6.0	10	4	4	3.3	3.1	7	4	7	2.2	2.6
14	3	5	9.2	8.9	10	3	8	1.0	0.4 *	4	4	2	3.2	6.0	11	4	4	6.3	6.2	8	4	7	3.1	3.3
15	3	5	5.3	4.6	11	3	8	2.9	3.1	5	4	2	7.2	6.9	12	4	4	4.0	3.8	9	4	7	1.9	2.1
16	3	5	4.2	4.1	12	3	8	3.2	4.0	6	4	2	8.1	6.5	13	4	4	3.6	3.3	10	4	7	2.3	3.0
17	3	5	0.9	1.2 *	1	3	9	1.1	0.3 *	7	4	2	0.7	1.0 *	14	4	4	0.7	0.8 *	11	4	7	1.2	1.2
18	3	5	0.6	0.9 *	2	3	9	3.9	4.0	8	4	2	4.6	3.9	15	4	4	2.6	3.2	0	4	8	2.5	2.4
1	3	6	6.7	7.5	3	3	9	1.6	1.8	9	4	2	0.7	0.9 *	16	4	4	1.1	1.4	1	4	8	0.6	0.9 *
2	3	6	3.6	3.5	4	3	9	3.2	3.4	10	4	2	3.1	2.5	0	4	5	3.9	4.0	2	4	8	2.6	2.9
3	3	6	2.5	2.7	5	3	9	2.1	2.1	11	4	2	5.6	6.0	1	4	5	0.8	0.2 *	3	4	8	0.6	0.3 *
4	3	6	5.7	6.0	6	3	9	1.4	1.8	12	4	2	3.3	2.5	2	4	5	2.5	2.2	4	4	8	3.4	4.0
5	3	6	3.7	4.0	7	3	9	1.5	1.7	13	4	2	6.2	6.5	3	4	5	8.2	8.4	5	4	8	1.3	1.7
6	3	6	1.3	0.7 *	8	3	9	0.8	0.7 *	14	4	2	1.4	1.4	4	4	5	0.8	0.9 *	6	4	8	3.7	4.1
7	3	6	4.0	3.8	9	3	9	0.5	0.7 *	15	4	2	5.1	5.9	5	4	5	9.8	7.3	7	4	8	1.8	2.3
8	3	6	6.7	6.3	2	4	0	13.6	13.6	16	4	2	1.4	1.2	6	4	5	2.3	2.4					

4. Hydrogen positions. As was described above the hydrogen positions were deduced from difference Fourier maps (Table 1, Fig. 2). Well resolved peaks, except in one case, appeared in the regions, where hydrogen atoms might be expected to lie. Some bond distances and angles involving hydrogen are listed in Table 4. All these have reasonable values, except one of the C(2)—H distances which is too short (0.91 Å) (*cf.* Ref. 19). This hydrogen atom corresponds to the peak which was somewhat diffuse in the difference Fourier maps.

The O—H···N angle is 177°, indicating an approximately linear hydrogen bond. The N—H···O angles are 169° and 157°, respectively. The latter value involves the N—O bond of 3.060 Å (see above).

It should be pointed out that the errors in the hydrogen positions may be rather large.

Acknowledgements. The author wishes to express his gratitude to Dr. I. Olovsson for his great interest in this work and for valuable discussions and suggestions.

The author is indebted to Mr. H. Karlsson for his skilful assistance in the preparation of the crystals and in the preliminary X-ray work, and to Dr. L. Gustafsson for the analysis of the hydrazine.

This work has been supported by grants from the *Swedish Natural Science Research Council* and the *Malmfonden — Swedish Foundation for Scientific Research and Industrial Development* which are here gratefully acknowledged.

REFERENCES

1. Liminga, R. and Olovsson, I. *Acta Cryst.* **17** (1964) 1523.
2. Corcoran, J. M., Kruse, H. W. and Skolnik, S. *J. Phys. Chem.* **57** (1953) 435.
3. Bock, H. *Z. anorg. allgem. Chem.* **311** (1961) 117.
4. Vogel, A. I. *Practical Organic Chemistry*, 3rd Ed., Longmans, London 1956.
5. Olovsson, I. *Arkiv Kemi* **16** (1960) 437.

6. *International Tables for X-ray Crystallography*, Kynoch Press, Birmingham 1952, Vol. I.
7. Simpson, P.G., Dobrott, R.D. and Lipscomb, W.N. *Acta Cryst.* **18** (1965) 169.
8. Cruickshank, D. W. J., Pilling, D. E., Bujosa, A., Lovell, F. M. and Truter, M. R. *Computing Methods and the Phase Problem in X-ray Crystal Analysis*, Pergamon, Oxford 1961, p. 45.
9. *International Tables for X-ray Crystallography*, Kynoch Press, Birmingham 1962, Vol. III.
10. Liminga, R. *Acta Chem. Scand.* **20** (1966) 2483.
11. Collin, R. L. and Lipscomb, W. N. *Acta Cryst.* **4** (1951) 10.
12. Cromer, D. T., Larson, A. C. and Roof, Jr., R. B. *Acta Cryst.* **20** (1966) 279.
13. Busing, W. R., Zocchi, M. and Levy, H. A. *Abstract of American Crystallographic Association*, July 1961, p. 52.
14. Lide, D. *Tetrahedron* **17** (1962) 125.
15. Stoicheff, B. P. *Tetrahedron* **17** (1962) 135.
16. *Tables of Interatomic Distances*, London: The Chemical Society, Burlington House, W. 1, 1958.
17. Brun, L. *Acta Cryst.* **20** (1966) 739.
18. Lindgren, J. and Olovsson, I. *Acta Cryst. In press.*
19. Hamilton, W. C. *Ann. Rev. Phys. Chem.* **13** (1962) 19.

Received February 6, 1967.