

## Crystal Structure of Cyclohexane-1,4-dioxime

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The dioxime crystals belong to the orthorhombic system and the space group is *Pbca*. The unit cell, containing 8 molecules, has the following parameters:

$$a = 7.14_1 \text{ \AA}, \quad b = 13.25_1 \text{ \AA}, \quad c = 14.72_4 \text{ \AA}.$$

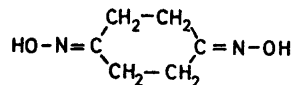
The phase problem was solved by a computer procedure based on direct methods. Full-matrix least squares refinement gave the *R*-value 5.6 % when the 990 observed reflections were included. The six-membered ring has the "twisted boat" form and the dioxime molecule has nearly twofold symmetry. The angle between the two C=N directions is 129.4°. Average C=N bond lengths and N—O bond lengths are 1.27<sub>4</sub> Å and 1.41<sub>1</sub> Å, respectively. Each dioxime molecule is linked to four other molecules by hydrogen bonds from O to N of lengths 2.83<sub>6</sub> Å and 2.77<sub>6</sub> Å. The infinite double-chains thus formed are parallel to [010].

The crystal structure of cyclohexane-1,4-dione has been determined both at room temperature<sup>1</sup> and at low temperature<sup>2</sup> using three-dimensional X-ray methods. The results of the two investigations are, within the probable limits of error, identical, and they show that the molecular conformation may be described in terms of a "twisted boat" form with an angle between the two C=O bonds of 154°, an angle which is in good agreement with that to be expected from the dipole moment found in solution.

This "movable" form of the ring has also been observed in two addition compounds of the dione. In the (1:1) addition compound formed with diiodoacetylene,<sup>3</sup> which has a statistically disordered crystal structure, the charge transfer bond is relatively weak with an I—O distance of 2.95 Å, and the angle between the two C=O directions (155.6°) is practically the same as that of the dione itself. In the corresponding compound (1:1) formed with HgCl<sub>2</sub>,<sup>4</sup> however, the bonds between oxygen and mercury are apparently much stronger and here the angle between the two C=O bonds is found equal to 176°.

An X-ray crystallographic investigation of cyclohexane-1,4-dioxime was carried out with the chief aim of determining the hydrogen bonding system, and to establish the configuration of the six-membered ring in this compound.

## CRYSTAL DATA



Cyclohexane-1,4-dioxime belongs to the orthorhombic system, the systematically absent reflections leading to the space group *Pbca*. The cell parameters were determined by means of *h0l*- and *0kl*-Weissenberg photographs taken with unfiltered Cu-radiation. Powder diagrams of BaF<sub>2</sub> ( $a = 6.2001 \text{ \AA}$ ) were superimposed on the films for calibration purpose. The  $2\theta$ -values of 69 reflections were used in a least squares refinement of the lattice parameters. The programme used has been written by R.A. Sparks at UCLA. With the wavelengths for CuK $\alpha$ -radiation taken as  $\alpha = 1.54178$ ,  $\alpha_1 = 1.54051$ ,  $\alpha_2 = 1.54433$ , and  $\beta = 1.39217 \text{ \AA}$ , the following cell parameters with their estimated standard deviations resulted:

$$\begin{array}{ll} a = 7.141 \text{ \AA} & \sigma(\text{\AA}) = 0.001 \\ b = 13.251 \text{ \AA} & \sigma(\text{\AA}) = 0.001 \\ c = 14.724 \text{ \AA} & \sigma(\text{\AA}) = 0.003 \end{array}$$

The number of molecules in the unit cell is  $Z = 8$  ( $\rho_{\text{calc}} = 1.35 \text{ gcm}^{-3}$ ,  $\rho_{\text{obs}} = 1.35 \text{ gcm}^{-3}$ ).

The intensity material was obtained from photometric measurements of integrated Weissenberg diagrams corresponding to *h0l*, *h1l*, .... *h10l* (CuK $\alpha$ -radiation) and of *0kl*-precession diagrams (MoK $\alpha$ -radiation).

The number of reflections accessible from these diagrams is 1272, 990 of which were strong enough to be measured.

The intensities were statistically put on an absolute scale, and the overall temperature factor thus obtained,  $B = 2.719 \text{ \AA}^2$ , was used in the calculation of the unitary structure factors, and also as starting parameter in the isotropic least squares refinement.

No corrections have been made for absorption or secondary extinction effects.

## DETERMINATION OF THE STRUCTURE

The phase problem has been solved by a computer procedure based on direct methods.<sup>5-7</sup> The programmes are written in FORTRAN IV for UNIVAC 1107 by the author and may handle two- and three-dimensional data.

The sign determination process was carried out for the *0kl*-projection (plane group *pgm*), the  $|U|$ -distribution of which is listed in Table 1.

In order to specify the origin, positive signs were given to  $U(014) = 0.30$  and  $U(031) = 0.23$ . With 15 additional large  $U$ 's the number of terms in the sums

$$\chi = \sum_{\mathbf{h}} \sum_{\mathbf{h}'} U_{\mathbf{h}} \cdot U_{\mathbf{h}'} \cdot U_{\mathbf{h}+\mathbf{h}'} \quad (1)$$

Table 1.  $|U|$ -distribution of the  $0kl$ -projection.

Range of $ U $	Number of reflections
0.00–0.01	1
0.01–0.02	3
0.02–0.03	3
0.03–0.04	5
0.04–0.05	10
0.05–0.10	22
0.10–0.15	17
0.15–0.20	18
0.20–0.25	17
0.25–0.30	7
0.30–0.35	9
0.35–0.40	1
0.40–0.50	1
0.50–1.00	0

was 31. 22 “small”  $U$ 's ( $|U| \leq 0.05$ ) gave 51 products for the zero-check expression:

$$\psi_0 = \sum_{\mathbf{k}} \left| \sum_{\mathbf{h}} U_{\mathbf{h}} \cdot U_{\mathbf{h} + \mathbf{k}} \right| \quad (2)$$

$\chi_e$  given by

$$\chi_e = \sum_{\mathbf{h}} \sum_{\mathbf{h}'} |U_{\mathbf{h}} \cdot U_{\mathbf{h}'} \cdot U_{\mathbf{h} + \mathbf{h}'}| \cdot \tanh(N \cdot |U_{\mathbf{h}} \cdot U_{\mathbf{h}'} \cdot U_{\mathbf{h} + \mathbf{h}'}|) \quad (3)$$

where  $N$  is the number of equal atoms in the cell, was calculated, and found to be  $\chi_e = 0.472$ .

Table 2.  $\chi$ - and  $\psi_0$ -values of the sign sets satisfying  $\chi > \chi_e$ .

Signset number	$\chi$	$\psi_0$
1	0.486	1.729
2	0.512	1.872
3	0.481	1.656
4	0.606	1.842
5	0.487	1.471
6	0.487	1.467
7	0.585	1.362
8	0.487	1.351
9	0.585	1.366
10	0.489	1.259

Among the  $2^{15} = 32\,768$  sign combinations tested, 10 satisfied  $\chi > \chi_e$ . Table 2 gives the corresponding  $\chi$ - and  $\psi_0$ -values, which point out the sets 7, 8, 9, and 10 as the most probable.

However, as may be seen from Table 3, the exclusion of  $S(0\,7\,3)$  from sets 7 and 10 makes these sets identical, while the same effect is obtained for 8 and 9 when excluding  $S(0\,1\,12)$  from these sets. Therefore, with the two resulting sign sets, A and B, as basis, signs of 20 additional unitary structure factors were derived using the relationship

$$S(U_{\mathbf{k}}) = S\left(\sum_{\mathbf{h}} U_{\mathbf{h}} \cdot U_{\mathbf{h} + \mathbf{k}}\right) \quad (4)$$

Table 3. The four "most probable" sets of signs.

<i>h k l</i>	<i>A</i>		<i>B</i>	
	7	10	8	9
0 1 4	+	+	+	+
0 3 1	+	+	+	+
0 2 8	+	+	+	+
0 2 12	+	+	-	-
0 0 4	+	+	-	-
0 0 8	+	+	+	+
0 2 4	+	+	-	-
0 2 0	+	+	+	+
0 1 8	+	+	-	-
0 1 12	+	+	* +	-
0 6 0	-	-	-	-
0 9 3	-	-	+	+
0 6 1	+	+	-	-
0 8 7	-	-	+	+
0 3 3	+	+	-	-
0 7 3	* -	+	+	+
0 5 1	-	-	-	-

in a somewhat special way, the signs being accepted in the order of decreasing probabilities given by

$$P_+(\mathbf{k}) = \frac{1}{2} + \frac{1}{2} \tanh(N \cdot |U_{\mathbf{k}}| \cdot \sum_{\mathbf{h}} U_{\mathbf{h}} \cdot U_{\mathbf{h}+\mathbf{k}}) \quad (5)$$

and the  $\chi$ - and  $\psi_0$ -values for the total set, in this case containing 36 signs, being calculated after the last cycle of iteration. The resulting  $\chi$ - and  $\psi_0$ -values were:

$$\begin{array}{ll} \text{A:} & \chi = 1.861 \quad \psi_0 = 4.665 \\ \text{B:} & \chi = 2.067 \quad \psi_0 = 2.415 \end{array}$$

According to this strong indication, the set obtained on basis of B was used in evaluation of 31 more signs by a combined application of (4) and (5), signs now being derived in the same order as the reflections were read by the machine, and accepted when the probability exceeded a certain limit.

The 67 signs thus established were used in evaluating a Fourier map which, it turned out, could easily be interpreted.<sup>8</sup> The trial parameters derived from the map gave  $R_{0kl} = 42.2\%$ . Applying a programme (written in FORTRAN IV for UNIVAC 1107) based upon the "minimum residual method"<sup>9</sup> the  $R$ -value was reduced to 7.4%.

Comparison with the signs corresponding to  $R_{0kl} = 7.4\%$  showed no discrepancies for the 36 signs determined by the first two steps of the process, while two signs had been obtained incorrectly by the last stage.

Using the obtained  $z$ -parameters, approximate values for the  $x$ -coordinates were found by allowing the  $h0l$ -projection of the molecule (determined from a model) to move in small steps parallel to the  $x$ -axis. The  $R$ -factor was calculated for each step, and the parameters corresponding to the minimum  $R$ -value ( $R_{h0l} = 17.8\%$ ) were chosen as starting coordinates for the threedimensional refinement.

## REFINEMENT OF THE STRUCTURE

With the final coordinates from the two-dimensional analysis and the statistically determined overall  $B$ -value as starting parameters, a three-dimensional full-matrix least squares refinement was carried out using a programme written by Gantzel, Sparks and Trueblood<sup>10</sup> (revised for UNIVAC 1107 by cand. real Christian Rømming at this university). The weighting scheme No. 1 was adapted by taking  $A1 = 10.0$ ,  $A2 = 25.0$ ,  $B1 = 0.0$ ,  $B2 = -0.5$ , and  $FB = 6.0$ , and the atomic form factors used were those of Hanson, Herman, Lea and Skillman.<sup>11</sup>

After 3 cycles of isotropic refinement, anisotropic thermal vibrational parameters were introduced for the oxygen, nitrogen, and carbon atoms. All hydrogen atom positions, except for the two bonding hydrogen atoms, were calculated assuming tetrahedral C—H bonds of lengths 1.05 Å. The two bonding hydrogen atoms were localized from a difference Fourier map, and reflections corresponding to  $\sin \theta/\lambda < 0.45$  were included in the refinement of hydrogen atoms. The  $R$ -value arrived at for the 990 observed reflections was 5.6 %.

The 282 accidentally absent reflections had previously been given the value  $(\frac{1}{2})I_{\min}$  and had been scaled along with the rest of the data although

Table 4. Fractional coordinates of oxygen, nitrogen, and carbon atoms.\* (Estimated standard deviations in parantheses).

	$x$	$y$	$z$
O <sub>1</sub>	0.2177 (0.0003)	0.1996 (0.0001)	0.2453 (0.0002)
O <sub>2</sub>	-0.4357 (0.0003)	0.5086 (0.0002)	0.1236 (0.0001)
N <sub>1</sub>	0.1019 (0.0003)	0.1916 (0.0002)	0.1682 (0.0001)
N <sub>2</sub>	-0.2442 (0.0003)	0.5285 (0.0002)	0.1393 (0.0001)
C <sub>1</sub>	0.0739 (0.0003)	0.2781 (0.0002)	0.1328 (0.0002)
C <sub>2</sub>	-0.0511 (0.0004)	0.2854 (0.0002)	0.0514 (0.0002)
C <sub>3</sub>	-0.2109 (0.0003)	0.3603 (0.0002)	0.0708 (0.0002)
C <sub>4</sub>	-0.1396 (0.0003)	0.4564 (0.0002)	0.1122 (0.0002)
C <sub>5</sub>	0.0686 (0.0004)	0.4665 (0.0002)	0.1247 (0.0002)
C <sub>6</sub>	0.1472 (0.0004)	0.3741 (0.0002)	0.1731 (0.0002)

\* For numbering of atoms see Fig. 1.

not used in any calculations of the scale factors. Allowance for the inclusion of these reflections in the least squares refinement was made by applying weighting scheme No. 3 ( $W = 1/\sigma$ ). Satisfactory weight analysis was obtained by giving all unobserved reflections the same constant weight  $W = A1/3$ .

With the inclusion of the accidental absences it would be roughly expected that the standard deviations would decrease by a factor  $(990/(990 + 282))^{1/2} = 0.88$ . The actual improvement in the average coordinate E.S.D.'s corresponded to a factor 0.89. The  $R$ -value arrived at for all data was 7.1 %.

A final difference Fourier map, calculated with the observed data and with phases determined by the parameters corresponding to  $R = 5.6$  % contained no positive or negative areas greater than  $0.30 \text{ e} \cdot \text{\AA}^{-3}$ , the E.S.D. of the electron density <sup>12</sup> being  $\sigma(\rho) = 0.07 \text{ e} \cdot \text{\AA}^{-3}$ .

The fractional atomic coordinates and the thermal vibration parameters arrived at are given in Tables 4, 5, 6, the expressions for the anisotropic and isotropic vibrations, respectively, being

$$\exp[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{12}hk + B_{13}hl + B_{23}kl)]$$

and  $\exp[-B \sin^2\theta/\lambda^2]$

A comparison between calculated and observed structure factors is presented in Table 7. The accidental absences (with  $F_u$ -values corresponding to  $I_{\min}$ ) and the calculated values, are given in Table 8.

Table 5. Anisotropic thermal vibration parameters. (Estimated standard deviations in parantheses).

	$B_{11}$	$B_{22}$	$B_{33}$	$B_{12}$	$B_{13}$	$B_{23}$
O <sub>1</sub>	0.0209 (0.0005)	0.0047 (0.0001)	0.0050 (0.0001)	-0.0017 (0.0004)	-0.0095 (0.0004)	0.0020 (0.0002)
O <sub>2</sub>	0.0132 (0.0004)	0.0046 (0.0002)	0.0054 (0.0001)	0.0012 (0.0004)	-0.0017 (0.0003)	-0.0017 (0.0002)
N <sub>1</sub>	0.0124 (0.0004)	0.0045 (0.0002)	0.0039 (0.0001)	0.0001 (0.0004)	-0.0026 (0.0003)	-0.0006 (0.0002)
N <sub>2</sub>	0.0147 (0.0004)	0.0037 (0.0002)	0.0037 (0.0001)	0.0001 (0.0004)	-0.0018 (0.0003)	-0.0002 (0.0002)
C <sub>1</sub>	0.0111 (0.0004)	0.0042 (0.0002)	0.0033 (0.0001)	0.0006 (0.0005)	0.0003 (0.0004)	-0.0002 (0.0002)
C <sub>2</sub>	0.0157 (0.0005)	0.0043 (0.0002)	0.0031 (0.0001)	0.0021 (0.0005)	-0.0020 (0.0004)	-0.0017 (0.0002)
C <sub>3</sub>	0.0140 (0.0005)	0.0040 (0.0002)	0.0031 (0.0001)	0.0004 (0.0004)	-0.0021 (0.0004)	-0.0006 (0.0002)
C <sub>4</sub>	0.0149 (0.0005)	0.0034 (0.0002)	0.0027 (0.0001)	-0.0007 (0.0005)	-0.0011 (0.0004)	0.0002 (0.0002)
C <sub>5</sub>	0.0150 (0.0005)	0.0042 (0.0002)	0.0041 (0.0001)	-0.0034 (0.0005)	-0.0017 (0.0004)	0.0008 (0.0002)
C <sub>6</sub>	0.0149 (0.0005)	0.0039 (0.0002)	0.0043 (0.0001)	-0.0014 (0.0005)	-0.0039 (0.0004)	-0.0001 (0.0002)

Table 6. Fractional coordinates and isotropic thermal vibration parameters for hydrogen atoms.\* (Estimated standard deviations in parantheses).

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (Å <sup>2</sup> )
H <sub>2c</sub>	−0.109 (0.003)	0.217 (0.002)	0.035 (0.001)	0.7 (0.5)
H <sub>2a</sub>	0.027 (0.003)	0.316 (0.002)	−0.004 (0.002)	1.6 (0.5)
H <sub>3c</sub>	−0.301 (0.003)	0.326 (0.002)	0.116 (0.007)	1.4 (0.5)
H <sub>3a</sub>	−0.275 (0.003)	0.376 (0.002)	0.015 (0.002)	0.6 (0.5)
H <sub>5c</sub>	0.099 (0.003)	0.535 (0.002)	0.158 (0.002)	1.3 (0.5)
H <sub>5a</sub>	0.131 (0.003)	0.469 (0.002)	0.061 (0.002)	2.1 (0.6)
H <sub>6c</sub>	0.103 (0.003)	0.375 (0.002)	0.238 (0.002)	1.5 (0.5)
H <sub>6a</sub>	0.292 (0.003)	0.371 (0.002)	0.171 (0.002)	2.5 (0.6)
H <sub>01</sub>	0.210 (0.004)	0.141 (0.002)	0.277 (0.002)	3.8 (0.7)
H <sub>0a</sub>	−0.500 (0.006)	0.571 (0.003)	0.138 (0.002)	6.2 (1.0)

\* Referring to Fig. 1 the hydrogen atoms H<sub>nc</sub> and H<sub>na</sub> are, respectively, equatorially and axially bonded to C<sub>n</sub>. H<sub>01</sub> and H<sub>0a</sub> are bonded to O<sub>1</sub> and O<sub>2</sub>, respectively.

#### THERMAL MOTIONS

The principal axes of the thermal vibration ellipsoids for the oxygen, nitrogen, and carbon atoms were calculated from the temperature parameters given in Table 5. Root mean square amplitudes for the atomic anisotropic thermal vibrations along the principal axes as well as the components of these axes along the crystal axes are given in Table 9.

Since the oscillations of the whole molecule are dependent on the hydrogen bonding system, a rigid-body analysis of translational and librational motion has not been carried out.

#### DISCUSSION

Interatomic distances and angles of cyclohexane-1,4-dioxime, except those involving hydrogen atoms which are presented in Table 10, may be found in Fig. 1. Standard deviations in bond lengths and angles were calculated

Table 7. Observed and calculated structure factors.

<i>h</i>	<i>k</i>	<i>l</i>	<i>F</i> <sub>o</sub>	<i>F</i> <sub>c</sub>	<i>h</i>	<i>k</i>	<i>l</i>	<i>F</i> <sub>o</sub>	<i>F</i> <sub>c</sub>	<i>h</i>	<i>k</i>	<i>l</i>	<i>F</i> <sub>o</sub>	<i>F</i> <sub>c</sub>	<i>h</i>	<i>k</i>	<i>l</i>	<i>F</i> <sub>o</sub>	<i>F</i> <sub>c</sub>
0	0	2	44.9	-49.2	3	1	14	7.3	7.0	3	2	8	5.1	-4.3	4	3	5	38.6	-40.7
0	0	4	152.7	-102.1	3	1	17	5.6	5.8	3	2	9	15.6	12.6	4	3	6	26.2	27.0
0	0	6	3.5	3.6	4	1	1	41.1	41.0	3	2	10	15.5	-13.3	4	3	7	4.7	4.4
0	0	8	65.1	65.6	4	1	2	6.3	-6.5	3	2	11	3.0	-2.3	4	3	8	5.2	4.2
0	0	10	51.8	-51.3	4	1	3	36.4	35.6	3	2	12	14.5	14.5	4	3	9	8.3	7.7
0	0	12	10.7	-10.1	4	1	4	5.1	37.7	3	2	13	5.1	3.6	4	3	10	5.2	5.6
0	0	14	7.2	-7.2	4	1	5	15.5	-16.0	4	2	1	21.5	-27.9	4	3	11	15.3	16.2
0	0	16	26.0	25.2	4	1	6	16.6	-17.7	4	2	2	6.0	-4.5	4	3	12	3.0	1.0
0	0	18	5.7	5.3	4	1	7	35.9	-36.7	4	2	3	11.2	11.3	4	3	13	2.6	-3.5
1	0	2	47.4	47.6	4	1	9	15.7	17.4	4	2	4	23.8	-23.9	4	3	14	2.9	3.0
1	0	4	86.4	93.5	4	1	10	8.6	-9.7	4	2	5	8.6	7.1	4	3	15	8.3	7.5
1	0	6	28.2	-29.8	4	1	11	12.2	11.6	4	2	6	34.3	-34.8	4	3	16	4.0	-4.4
1	0	8	18.2	-16.4	4	1	13	11.9	-11.6	4	2	7	13.4	14.3	5	3	1	10.7	10.5
1	0	10	17.7	18.2	4	1	14	11.0	-10.8	4	2	8	5.0	-5.1	5	3	2	7.5	-7.9
1	0	12	7.9	-7.7	4	1	15	6.0	-6.3	4	2	9	10.8	-10.0	5	3	3	4.1	4.5
1	0	14	15.5	-14.9	5	1	1	6.2	-6.8	4	2	10	11.5	-10.5	5	3	4	3.0	-2.9
1	0	16	10.6	10.7	5	1	2	21.7	21.1	4	2	11	6.7	-5.9	5	3	5	6.6	-7.1
2	0	0	22.0	-20.6	5	1	3	21.3	-20.8	4	2	12	11.4	11.0	5	3	6	7.0	-6.7
2	0	2	52.3	51.6	5	1	4	10.2	-9.8	4	2	13	6.3	5.9	5	3	7	19.1	20.2
2	0	4	49.5	-50.2	5	1	6	11.9	11.6	4	2	14	2.8	-3.4	5	3	8	13.1	-12.7
2	0	6	33.8	32.3	5	1	9	7.7	7.5	5	2	2	15.0	15.8	5	3	9	6.9	6.7
2	0	10	12.8	12.7	5	1	10	4.5	3.9	5	2	3	5.6	-5.3	5	3	10	7.0	7.0
2	0	12	26.6	-28.9	5	1	13	3.9	-5.1	5	2	4	19.1	-19.0	5	3	11	5.2	5.5
2	0	14	6.1	-6.0	5	1	14	10.0	8.3	5	2	5	18.0	-17.1	5	3	12	2.9	2.9
2	0	16	2.8	2.0	6	1	1	3.7	4.6	5	2	6	18.5	-18.3	6	3	1	9.1	-9.2
2	0	18	7.5	7.0	6	1	2	12.9	12.7	5	2	7	15.3	15.9	6	3	2	11.5	11.4
3	0	0	63.9	-62.5	6	1	3	11.4	11.4	5	2	8	4.1	4.2	6	3	3	7.0	7.7
3	0	2	85.4	43.8	6	1	4	7.3	-7.5	5	2	9	14.9	14.6	6	3	4	12.6	12.7
3	0	4	29.0	26.8	6	1	5	17.8	-18.0	5	2	10	10.7	11.5	6	3	5	3.5	3.4
3	0	6	31.0	-30.4	6	1	7	8.6	8.6	5	2	11	6.6	-6.2	6	3	6	11.1	-11.7
3	0	10	14.1	14.7	6	1	7	12.5	-12.6	5	2	12	9.4	-9.3	6	3	7	7.2	7.4
3	0	12	11.6	10.4	6	1	8	26.1	-25.8	5	2	13	5.8	-5.5	6	3	8	2.0	-1.8
3	0	16	15.4	-14.9	6	1	9	7.2	7.3	5	2	14	3.2	2.8	6	3	9	10.9	-11.5
4	0	0	5.6	-4.4	6	1	10	3.3	-3.1	5	2	15	4.7	5.2	7	3	1	11.6	-10.9
4	0	2	5.3	4.3	6	1	11	12.9	13.4	6	2	1	12.9	12.7	7	3	2	2.9	2.9
4	0	4	29.8	28.6	7	1	2	11.2	11.2	6	2	2	3.0	-2.7	7	3	3	5.6	5.2
4	0	6	25.0	-25.7	7	1	3	8.1	8.7	6	2	3	3.0	-3.7	7	3	4	14.0	15.2
4	0	8	12.4	12.5	7	1	4	9.1	8.4	6	2	4	11.4	12.6	7	3	5	3.0	2.5
4	0	10	16.7	-16.7	7	1	9	4.8	-4.6	6	2	5	7.9	-8.8	7	3	6	2.9	2.8
4	0	14	17.1	16.7	7	1	11	4.6	-4.6	6	2	6	8.0	7.6	7	3	7	4.8	4.9
4	0	18	2.0	2.7	7	1	12	7.9	8.4	6	2	8	11.2	-11.8	7	3	8	3.2	-3.8
5	0	0	9.5	7.8	8	1	0	9.4	10.3	6	2	11	4.7	-4.6	7	3	10	2.0	-2.1
5	0	2	21.8	21.5	8	1	1	3.2	4.2	6	2	12	6.2	5.7	7	3	11	4.3	4.7
5	0	4	15.9	-16.1	8	1	2	5.7	-6.2	6	2	13	6.3	6.2	8	3	0	9.5	-10.5
5	0	6	7.5	-7.3	8	1	3	3.4	-3.0	6	2	14	3.0	-3.3	8	3	0	11.2	-12.1
5	0	8	44.8	45.9	8	1	4	5.9	5.1	6	2	15	13.8	-13.1	8	3	0	3.7	3.7
5	0	10	6.0	5.8	8	1	8	7.3	7.0	7	2	3	7.3	-6.9	0	4	0	102.3	103.4
5	0	14	26.6	27.1	0	2	0	8.5	10.0	7	2	4	4.7	4.5	0	4	1	51.7	53.3
5	0	16	5.3	-4.7	0	2	1	27.9	19.6	7	2	5	5.2	4.9	0	4	2	64.7	64.0
5	0	18	18.5	-18.7	0	2	2	22.7	25.4	7	2	8	7.0	-6.3	0	4	4	86.4	-86.0
5	0	20	11.3	11.6	0	2	3	86.4	96.8	7	2	11	3.0	3.0	0	4	6	10.5	10.5
5	0	22	9.7	9.7	0	2	5	141.5	-145.1	8	2	0	3.9	4.0	0	4	8	4.8	-5.1
5	0	24	7.4	-6.8	0	2	5	5.8	4.2	8	2	1	3.7	-3.1	0	4	6	26.2	-24.5
5	0	26	7.8	6.0	0	2	6	13.0	14.0	8	2	3	4.9	-5.1	0	4	8	76.3	74.1
5	0	28	10.7	11.1	0	2	7	11.9	12.3	1	3	1	26.4	27.3	0	4	10	17.5	-17.8
5	0	30	9.0	-9.2	0	2	8	53.8	54.2	1	3	3	15.0	13.7	0	4	12	39.8	-38.5
5	0	32	7.7	-7.3	0	2	9	23.0	-22.9	1	3	4	6.5	6.3	0	4	14	31.8	-30.7
5	0	34	5.1	-5.4	0	2	10	32.5	31.4	1	3	5	9.9	-11.9	0	4	15	9.8	-10.7
5	0	36	6.2	6.9	0	2	11	6.3	6.5	1	3	6	20.3	20.8	0	4	16	9.5	9.5
5	0	38	5.7	-5.3	0	2	12	32.5	-31.9	1	3	7	4.0	-3.1	0	4	17	5.1	-5.3
5	0	40	9.3	8.9	1	2	13	6.2	-6.6	1	3	8	21.5	21.6	0	4	18	2.7	-2.5
5	1	1	36.1	-31.1	0	2	16	5.1	5.2	1	3	10	4.0	-4.4	1	4	1	34.6	32.9
5	1	2	119.8	-122.2	0	2	15	6.4	-5.8	1	3	11	13.4	-14.2	1	4	2	35.9	35.6
5	1	3	1.8	.5	1	2	1	28.2	-26.9	1	3	12	12.3	-12.8	1	4	3	30.5	-30.3
5	1	4	4.2	4.3	1	2	2	78.9	84.8	1	3	14	2.7	-2.6	1	4	4	40.7	-38.0
5	1	5	6.2	4.8	1	2	3	39.5	-38.2	1	3	15	8.2	8.4	1	4	5	7.1	7.0
5	1	7	9.0	9.3	1	2	4	42.6	-43.5	1	3	16	2.0	-3.0	1	4	6	23.6	-24.0
5	1	8	30.7	-29.5	1	2	5	21.1	20.6	2	3	0	24.4	25.6	1	4	8	17.6	17.2
5	1	10	4.5	4.2	1	2	6	52.8	-54.2	2	3	1	41.6	43.9	1	4	9	16.3	-15.0
5	1	12	8.5	7.5	1	2	7	32.1	30.4	2	3	2	13.3	-14.3	1	4	10	9.3	9.2
5	1	13	4.7	3.9	1	2	8	9.7	9.7	2	3	3	16.4	16.6	1	4	11	9.0	-9.4
5	1	14	9.4	8.8	1	2	10	22.6	21.1	2	3	4	26.4	-26.2	1	4	12	5.8	6.2
5	1	16	8.0	7.5	1	2	11	4.0	-3.0	2	3	5	4.9	2.7	1	4	13	12.2	12.0
5	1	17	5.3	-4.5	1	2	12	4.4	-4.4	2	3	6	8.8	-7.4	1	4	14	7.1	-7.3
5	1	18	3.1	-3.4	1	2	17	3.2	-3.8	2	3	8	23.1	22.8	1	4	16	2.7	-3.3
5	1	20	67.1	-72.0	2	2	0	2.9	3.5	2	3	9	8.8	9.7	2	4	0	62.5	61.1
5	1	22	26.5	26.2	2	2	1	13.8	15.2	2	3	10	3.5	2.2	2	4	1	15.7	-17.0
5	1	24	72.6	76.4	2	2	2	38.5	-39.2	2	3	11	13.5	-13.5	2	4	2	31.6	30.4
5	1	26	10.6	11.9	2	2	3	33.2	-35.0	2	3	13	3.1	-3.3	2	4	3	31.9	31.1
5	1	28	21.3	21.6	2	2	4	20.4	22.5	2	3	16	2.5	1.3	2	4	4	30.8	-29.9
5	1	30	32.9	-33.6	2	2	5	9.7	10.0	3	3	1	19.3	19.3	2	4	5	47.8	-47.1
5	1	32	38.9	-38.8	2	2	6	39.6	-39.4	3	3								



Table 7. Continued.

k	h	l	F <sub>0</sub>	F <sub>c</sub>	k	h	l	F <sub>0</sub>	F <sub>c</sub>	k	h	l	F <sub>0</sub>	F <sub>c</sub>	k	h	l	F <sub>0</sub>	F <sub>c</sub>
3	4	12	5.5	-5.4	5	5	3	10.7	11.0	5	6	2	19.0	19.0	6	7	1	3.0	2.8
3	4	13	6.7	-7.2	5	5	4	13.7	14.0	5	6	3	36.3	-35.4	6	7	2	3.3	-4.3
3	4	15	6.0	-5.0	5	5	5	9.5	-9.4	5	6	5	18.6	-18.2	6	7	3	8.1	9.9
3	4	16	8.0	-7.8	5	5	6	8.9	-9.2	5	6	6	16.7	-16.3	6	7	4	3.2	-4.3
3	4	17	3.4	4.6	5	5	7	5.5	-5.1	5	6	7	27.3	26.8	6	8	0	3.2	2.1
4	4	0	7.3	7.6	5	5	8	5.7	-5.2	5	6	8	3.1	3.7	6	8	1	2.7	2.9
4	4	1	19.8	-18.3	5	5	9	6.0	5.4	5	6	9	9.0	9.3	6	8	2	16.3	19.7
4	4	2	18.0	-18.8	5	5	10	5.2	4.8	5	6	10	5.6	-4.0	6	8	3	14.5	-16.2
4	4	3	34.2	-34.2	5	5	11	6.8	-6.0	5	6	13	4.0	-4.0	6	8	4	26.9	-28.1
4	4	5	27.7	27.0	5	5	12	4.9	-5.1	6	6	0	16.8	-13.8	6	8	5	23.1	-22.6
4	4	6	15.5	15.1	5	5	14	4.0	-5.3	6	6	1	3.2	4.1	6	8	6	7.6	7.9
4	4	7	8.5	-10.0	5	5	15	20.7	-20.8	6	6	2	3.7	4.3	6	8	7	17.6	-17.8
4	4	10	9.4	-9.7	6	5	0	11.9	8.5	6	6	4	9.9	9.1	6	8	8	8.4	-8.7
4	4	11	22.4	23.6	6	5	3	12.1	12.4	6	6	5	3.5	-2.5	6	8	9	12.7	-14.4
4	4	13	11.1	-11.2	6	5	4	14.5	14.2	6	6	6	4.9	-4.5	6	8	10	5.3	3.7
4	4	14	9.3	-9.2	6	5	5	25.0	-25.8	7	6	1	3.8	-3.7	6	8	12	5.7	-6.0
4	4	15	10.1	-10.8	6	5	7	2.5	2.2	7	6	3	3.1	-3.5	6	8	13	16.1	16.1
4	4	16	3.3	-3.9	6	5	9	4.1	-4.1	7	6	5	2.2	-2.6	6	8	14	10.5	12.6
4	4	17	7.2	7.3	6	5	11	3.0	4.0	7	6	6	4.0	4.4	6	8	15	3.2	-4.3
4	4	18	6.7	6.6	7	5	1	2.4	1.9	7	6	7	3.0	-3.4	6	8	16	5.2	4.1
4	4	19	10.0	-10.1	7	5	2	3.9	4.3	7	6	9	1.9	2.2	1	0	1	3.9	4.6
4	4	20	6.3	-6.6	7	5	3	2.6	-1.8	6	6	1	8.4	-8.6	1	8	2	9.6	11.6
4	4	21	5.6	-4.8	7	5	4	8.9	-8.6	6	6	3	3.8	-3.7	1	8	3	14.3	-14.8
4	4	22	4.3	-4.8	6	5	6	3.6	-4.2	6	6	4	1.9	2.4	1	8	4	6.8	5.4
4	4	23	33.6	-33.9	7	5	7	4.3	3.7	6	6	5	7.7	7.7	1	8	5	2.2	2.1
6	4	0	3.6	4.8	7	5	8	7.3	7.3	6	6	6	2.3	2.8	1	8	6	4.4	3.4
6	4	3	12.1	12.4	7	5	9	4.4	-4.9	1	7	1	25.4	30.4	1	8	7	14.6	15.1
6	4	4	23.4	24.5	7	5	10	5.4	4.1	1	7	2	28.8	-33.9	1	8	9	3.5	-3.4
6	4	5	3.4	-3.9	8	5	0	12.1	12.5	1	7	3	5.5	-3.1	1	8	11	9.4	-10.4
6	4	6	9.5	-9.9	8	5	1	11.9	2.7	1	7	4	26.2	-28.2	1	8	12	12.7	-11.1
6	4	7	6.9	-6.9	8	5	2	2.5	2.5	1	7	6	19.9	20.3	1	8	15	2.3	1.2
6	4	8	11.4	-11.3	8	5	3	7.8	-8.8	1	7	7	9.4	10.1	1	8	16	2.9	3.9
6	4	9	5.0	5.7	8	5	4	6.7	-7.7	1	7	9	14.5	14.3	2	8	0	5.2	5.9
6	4	10	9.3	8.9	8	5	5	2.5	-2.5	1	7	10	10.4	-9.9	2	8	1	9.4	-8.7
6	4	11	3.9	3.7	8	5	6	79.4	84.1	1	7	11	20.7	-21.5	2	8	2	21.6	-19.3
6	4	12	5.6	6.5	0	6	1	60.6	-64.3	1	7	12	7.1	-7.1	2	8	3	4.5	-2.7
6	4	13	6.5	-8.1	0	6	2	16.3	-17.4	1	7	13	10.3	-10.6	2	8	4	4.1	3.3
7	4	3	13.3	13.2	0	6	3	48.9	54.5	1	7	14	4.5	4.0	2	8	5	32.0	-3.3
7	4	4	4.2	-5.5	0	6	4	35.3	-33.5	1	7	15	7.0	8.0	2	8	6	13.5	-14.7
7	4	5	2.9	-3.2	0	6	5	3.3	2.6	1	7	16	5.4	6.4	2	8	8	23.8	25.3
7	4	7	4.8	-5.1	0	6	7	22.2	-19.2	1	7	17	11.4	2.7	2	8	9	4.2	4.4
8	4	0	10.8	-10.4	0	6	8	16.9	17.0	2	7	1	30.7	33.3	2	8	11	7.0	7.0
8	4	1	3.3	3.7	0	6	10	6.5	-6.5	2	7	2	6.2	6.2	2	8	12	4.1	-4.9
8	4	3	4.2	5.3	0	6	11	42.2	40.4	2	7	4	11.6	-10.6	2	8	14	3.3	-4.1
8	4	4	5.5	6.5	0	6	13	22.2	-22.8	2	7	5	7.2	-7.1	2	8	15	5.5	7.0
8	4	5	3.5	-3.5	0	6	15	31.5	-10.4	2	7	6	4.4	-4.2	2	8	16	1.8	2.7
1	5	2	47.1	-46.4	0	6	16	3.7	3.9	2	7	7	3.6	3.6	2	8	17	6.3	6.3
1	5	3	41.2	-41.9	0	6	17	5.2	-5.7	2	7	8	6.4	5.5	3	8	1	11.3	11.1
1	5	4	26.4	-24.0	1	6	1	15.5	15.8	2	7	9	3.7	4.5	3	8	2	17.3	17.0
1	5	5	10.3	9.3	1	6	2	22.4	24.3	2	7	10	10.6	10.9	3	8	3	4.0	-11.5
1	5	6	34.8	-34.2	1	6	3	14.8	13.8	2	7	12	6.1	-6.0	3	8	4	3.1	-3.4
1	5	7	9.1	10.5	1	6	4	13.6	12.1	2	7	13	4.8	-4.3	3	8	5	14.9	-15.4
1	5	8	5.6	6.1	1	6	5	30.4	-29.5	2	7	14	6.8	-6.4	3	8	6	18.4	-18.5
1	5	9	5.3	-3.7	1	6	6	19.4	-19.6	-7	0	5	3.8	-4.0	3	8	7	8.2	8.2
1	5	10	28.0	-27.9	1	6	7	11.8	11.1	2	7	15	9.1	-10.0	3	8	8	6.8	-7.4
1	5	11	17.5	17.2	1	6	9	4.8	4.8	3	7	16	1.9	-7.7	3	8	9	6.1	5.7
1	5	13	2.9	3.6	1	6	10	7.0	6.0	3	7	1	23.9	23.2	3	8	11	4.6	-5.0
1	5	14	16.2	14.9	1	6	11	16.4	-15.7	3	7	2	2.7	-2.6	3	8	12	4.6	-5.0
1	5	15	8.1	-8.6	1	6	12	6.1	-5.2	3	7	3	7.7	-7.4	3	8	13	3.5	-3.5
1	5	16	2.0	1.9	1	6	13	7.0	6.3	3	7	4	5.0	5.3	4	8	0	5.4	5.2
2	5	0	25.6	24.9	1	6	14	6.2	-6.0	3	7	6	4.8	-3.9	4	8	1	9.1	-8.3
2	5	1	18.8	-20.0	1	6	15	19.4	-20.7	3	7	7	4.4	-3.1	4	8	2	9.9	9.4
2	5	2	15.2	-10.3	1	6	16	7.4	7.9	3	7	8	5.8	-5.6	4	8	3	14.2	-13.8
2	5	3	30.3	37.0	1	6	17	6.9	-8.7	3	7	9	15.4	-15.4	4	8	4	16.7	-16.4
2	5	4	4.0	-3.4	2	6	0	13.1	-12.7	3	7	10	3.7	3.6	4	8	5	14.7	-14.0
2	5	5	15.9	-15.2	2	6	1	59.5	57.5	3	7	12	3.0	3.1	4	8	6	16.0	16.0
2	5	6	8.9	9.0	2	6	2	4.5	4.1	3	7	13	2.8	2.2	4	8	7	6.0	-6.9
2	5	7	31.4	-31.0	2	6	3	15.8	15.8	3	7	15	2.4	2.6	4	8	8	9.9	9.3
2	5	8	5.7	-5.1	2	6	4	10.5	-10.2	3	7	16	4.8	-4.8	4	8	9	3.0	3.5
2	5	9	8.4	9.2	2	6	5	13.5	14.0	4	7	0	4.4	-3.9	4	8	11	20.0	19.0
2	5	10	7.7	7.5	2	6	6	11.2	11.0	4	7	1	3.6	3.0	4	8	12	22.8	20.9
2	5	11	20.6	21.9	2	6	7	10.9	-10.1	4	7	3	27.5	27.7	4	8	13	6.4	6.3
2	5	12	4.8	5.2	2	6	8	4.4	5.3	4	7	4	11.3	9.9	4	8	14	9.1	-9.6
2	5	13	11.5	-19.2	2	6	9	25.8	25.2	4	7	5	6.9	5.4	4	8	15	7.4	-7.0
2	5	14	15.1	-15.5	2	6	10	9.9	10.1	4	7	6	12.0	-11.5	4	8	16	5.1	5.4
2	5	15	5.7	4.7	2	6	11	16.2	-16.7	4	7	7	8.5	8.1	4	8	17	2.9	-3.2
2	5	16	3.0	-3.1	2	6	12	4.4	-4.6	4	7	8	5.2	5.4	4	8	18	3.6	4.8
2	5	17	7.0	8.5	2	6	13	13.3	-11.8	4	7	9	8.3	-8.0	4	8	19	9.3	-10.0
3	5	2	15.2	-13.3	2	6	14	6.4	-6.3	4	7	10	4.9	-4.6	4	8	20	2.0	1.7
3	5	3	15.0	-14.9	2	6	15	3.8	-3.3	4	7	11	2.6	-3.1	4	8	21	4.8	-6.0
3	5	4	4.5	4.0	2	6	17	6.2	8.9	4	7	13	8.2	-8.6	4	8	22	3.0	2.6
3	5	5	4.1	4.1	3	6	1	20.5	-19.9	4	7	14	2.4	2.4	6	8	1	10.3	9.7
3	5	6	11.0	-11.4	3	6	3	3.3	3.8	4	7	15	1.8	-1.9	6	8			

Table 7. Continued.

<i>h k l</i>	$F_o$	$F_c$	<i>h k l</i>	$F_o$	$F_c$	<i>h k l</i>	$F_o$	$F_c$	<i>h k l</i>	$F_o$	$F_c$
1 9 9	18.5	19.0	4 9 10	6.2	5.9	1 10 5	4.2	3.2	5 10 2	10.4	11.0
1 9 10	12.1	-11.3	4 9 11	3.7	3.8	1 10 7	21.4	21.7	5 10 3	13.3	-13.9
1 9 11	4.9	4.1	4 9 12	3.1	2.5	1 10 8	6.8	4.8	5 10 4	7.0	5.3
1 9 12	3.6	-3.3	4 9 13	2.5	-2.4	1 10 10	6.1	4.0	5 10 5	13.0	-14.1
1 9 13	5.0	-4.8	4 9 14	4.5	-3.6	1 10 11	7.0	-7.0	5 10 6	8.5	-9.3
1 9 14	5.9	6.4	4 9 15	5.5	4.2	1 10 12	3.3	2.9	5 10 7	15.5	15.3
1 9 15	1.8	-1.6	5 9 6	8.5	-8.0	1 10 14	3.7	-3.8	5 10 8	2.4	1.6
2 9 0	16.0	19.1	5 9 8	5.3	-4.5	2 10 0	12.6	14.7	5 10 9	13.0	15.0
2 9 1	17.4	22.8	5 9 9	5.0	-4.7	2 10 1	14.4	12.7	5 10 10	5.2	6.1
2 9 2	5.4	-5.8	5 9 10	12.4	13.2	2 10 2	19.2	16.8	5 10 11	8.1	-10.0
2 9 3	15.1	13.8	5 9 12	3.0	3.8	2 10 3	9.7	8.7	6 10 0	9.1	-10.1
2 9 4	4.9	-5.2	6 9 1	4.0	-4.0	2 10 4	22.0	-22.0	6 10 1	4.3	3.2
2 9 5	26.3	-25.8	6 9 2	2.7	-2.9	2 10 5	13.4	-13.2	6 10 2	4.5	3.3
2 9 6	5.6	-6.3	6 9 4	8.5	9.5	2 10 6	7.0	7.0	6 10 3	4.4	-3.6
2 9 7	8.0	-8.2	6 9 5	5.7	-6.7	2 10 8	10.2	-9.6	7 10 1	4.7	-5.3
2 9 8	3.9	3.2	6 9 8	9.4	-9.6	2 10 9	16.5	15.5	7 10 2	6.7	7.7
2 9 9	5.8	-5.3	6 9 7	4.3	-3.8	2 10 12	6.6	-6.1	7 10 3	3.1	2.4
2 9 11	8.6	8.3	6 9 8	3.9	-3.4	2 10 13	8.9	-10.0	7 10 4	5.0	-3.4
2 9 12	7.4	7.3	6 9 9	2.4	2.3	2 10 14	4.0	4.7	7 10 5	97.0	-99.2
2 9 13	2.0	1.8	6 9 10	1.1	-1.4	3 10 1	9.5	9.5	7 10 6	7.8	-7.2
2 9 14	2.7	-2.6	7 9 1	2.4	2.4	3 10 2	10.2	11.3	7 10 7	6.7	6.1
2 9 15	3.7	-2.7	7 9 2	1.4	1.4	3 10 3	12.1	-12.7	7 10 8	12.4	-13.7
3 9 1	7.7	-4.9	7 9 3	1.7	2.2	3 10 4	11.8	12.0	7 10 9	7.7	7.9
3 9 2	7.5	-8.2	7 9 4	6.4	7.5	3 10 5	6.1	-5.3	7 10 10	6.4	6.4
3 9 3	6.6	-8.0	7 9 5	3.8	-3.2	3 10 6	2.9	-1.1	7 10 11	6.8	7.4
3 9 4	7.3	8.2	7 9 6	2.4	-2.9	3 10 7	5.3	6.0	7 10 12	10.5	10.8
3 9 5	7.4	8.3	8 9 0	19.4	22.0	3 10 8	6.5	-4.5	7 10 13	11.4	10.5
3 9 6	5.8	6.9	8 9 1	22.3	23.0	3 10 9	5.7	3.9	8 10 1	7.8	6.7
3 9 7	2.4	3.3	8 9 2	16.9	-21.6	3 10 10	8.1	8.9	8 10 3	13.1	-13.5
3 9 8	3.2	-2.9	8 9 3	13.1	13.3	3 10 11	3.9	3.9	8 10 4	10.2	9.5
3 9 9	3.6	-3.2	8 9 4	16.4	17.2	3 10 12	7.2	-7.9	8 10 5	8.1	7.9
3 9 10	5.7	-6.2	8 9 5	6.7	6.0	4 10 1	3.4	-3.5	8 10 6	10.1	-10.9
3 9 11	4.4	3.6	8 9 6	12.3	12.5	4 10 2	3.0	4.2	8 10 7	10.4	-9.6
3 9 13	4.0	4.5	8 9 8	5.3	6.5	4 10 3	15.4	-17.0	8 10 8	9.1	-7.6
4 9 0	9.2	-10.8	8 9 9	5.5	-4.8	4 10 4	3.3	-3.2	8 10 9	11.1	7.7
4 9 1	9.2	10.1	8 9 10	16.3	-17.1	4 10 5	6.5	5.0	8 10 10	13.2	-11.9
4 9 2	4.3	-4.0	8 9 11	6.5	-6.2	4 10 6	17.7	19.2	8 10 11	10.0	-8.0
4 9 3	4.4	-7.1	8 9 12	5.0	4.6	4 10 7	10.8	-10.9	8 10 12	10.4	8.5
4 9 4	12.4	-13.3	8 9 14	5.8	-6.2	4 10 8	10.7	10.9	8 10 13	10.2	8.2
4 9 5	23.4	19.5	8 9 15	6.5	-8.5	4 10 9	6.0	-5.9	8 10 14	7.4	4.9
4 9 7	13.0	-12.2	1 10 1	12.0	13.1	4 10 10	3.2	2.7			
4 9 8	5.1	-4.6	1 10 2	14.9	-15.1	4 10 12	5.4	-5.0			
4 9 9	9.8	9.5	1 10 3	31.8	-34.3	5 10 1	6.2	6.5			

Table 8. Unobserved and calculated structure factors (The  $F_u$ 's have been obtained by taking unobserved intensities as equal to the smallest observable).

<i>h k l</i>	$F_u$	$F_c$	<i>h k l</i>	$F_u$	$F_c$	<i>h k l</i>	$F_u$	$F_c$	<i>h k l</i>	$F_u$	$F_c$
1 0 16	2.0	-0.6	7 1 5	3.8	-0.3	6 2 5	2.2	1.0	4 4 14	3.3	0.7
2 0 8	2.6	-2.1	7 1 1	3.9	-3.5	6 2 6	2.0	-1.4	1 4 7	2.4	2.6
3 0 4	3.1	-2.1	7 1 6	3.7	-0.9	8 2 7	1.8	-1.2	1 4 14	3.2	-1.5
4 0 2	3.2	-2.1	7 1 7	3.5	-4.2	6 2 8	1.5	-1.6	1 4 15	3.0	1.2
4 0 16	2.0	2.1	7 1 8	3.3	-0.0	9 2 1	1.3	1.2	1 4 17	2.3	0.9
5 0 4	3.0	1.6	7 1 10	2.7	2.4	9 2 2	1.2	1.2	2 4 10	3.2	0.6
5 0 14	2.4	-0.9	8 1 4	3.1	.2	1 3 2	1.3	-2.3	2 4 11	3.3	4.0
7 0 4	3.1	-1.1	8 1 5	2.9	0.3	1 3 9	2.6	3.4	2 4 14	3.2	3.1
7 0 8	2.7	-0.7	8 1 6	2.7	1.1	1 3 13	3.1	-1.8	2 4 17	2.1	2.4
7 0 10	2.2	-3.2	8 1 9	1.5	-2.0	1 3 16	2.6	2.5	3 4 3	2.3	0.5
7 0 12	1.4	-1.9	8 1 16	2.7	1.8	1 3 17	2.6	3.3	3 4 14	3.0	-0.1
1 1 6	2.5	.8	0 2 17	2.3	2.4	2 3 7	2.4	-0.2	4 4 4	2.8	1.0
1 1 9	3.3	.5	0 2 18	1.6	.1	2 3 12	3.1	.9	4 4 8	3.2	0.6
1 1 11	3.7	.5	1 2 9	2.5	-0.2	2 3 14	3.0	-0.3	4 4 9	3.3	0.8
1 1 15	3.8	1.6	1 2 14	3.0	.1	2 3 15	2.6	-2.7	4 4 11	3.3	-2.7
2 1 7	3.0	-1.9	1 2 15	2.9	-0.7	2 3 16	2.0	2.1	4 4 12	3.2	2.3
2 1 15	3.7	-0.6	1 2 16	2.6	-0.4	2 3 18	1.4	-0.9	4 4 13	3.0	1.2
2 1 16	3.3	-1.2	1 2 18	1.8	2.2	3 3 12	3.1	.7	4 4 14	2.7	-1.6
3 1 6	3.1	-0.4	2 2 17	2.1	-0.0	3 3 15	2.6	-1.2	4 4 15	2.3	1.8
3 1 7	3.3	1.0	2 2 18	1.5	-0.7	4 3 1	1.7	1.3	4 4 16	1.7	-0.3
3 1 8	3.4	2.4	3 2 14	2.8	1.0	5 3 3	2.9	-3.2	5 4 2	3.1	0.6
3 1 12	4.0	3.4	3 2 16	2.2	-1.3	5 3 4	2.9	1.5	5 4 6	3.3	-3.5
3 1 13	4.0	-3.9	3 2 17	1.6	2.0	5 3 14	2.1	-1.7	5 4 7	3.3	-1.3
3 1 15	3.5	-0.4	4 2 0	1.4	1.2	5 3 15	1.6	-3.1	5 4 15	1.5	-1.4
3 1 16	3.5	-0.6	4 2 12	3.0	1.3	6 3 0	3.1	.9	6 4 1	3.1	3.3
4 1 0	3.0	-1.2	4 2 15	2.6	-2.1	6 3 8	2.9	-0.1	6 4 1	3.2	-1.9
4 1 8	3.8	-0.4	4 2 17	0.9	-1.1	6 3 9	2.8	3.3	7 4 2	3.2	.2
4 1 12	3.9	3.0	5 2 1	2.7	-0.3	6 3 10	2.7	-1.9	7 4 4	3.1	0.9
4 1 16	2.5	-1.1	6 2 0	3.0	-2.4	6 3 11	2.4	-1.0	7 4 8	2.5	0.3
5 1 3	3.7	1.0	6 2 2	6.5	-0.9	6 3 14	.9	1.6	7 4 9	2.3	0.9
5 1 5	3.4	3.0	6 2 5	2.8	-0.7	6 3 16	2.4	3.4	7 4 10	2.0	-0.1
5 1 7	4.0	3.1	6 2 12	2.7	-1.4	8 3 3	2.3	.1	7 4 11	1.5	-0.9
5 1 11	3.8	-0.7	7 2 1	3.0	-0.4	8 3 4	2.2	-0.7	8 4 2	1.7	0.8
5 1 12	3.6	3.3	7 2 6	2.7	-1.0	8 3 5	2.1	.9	8 4 5	2.2	1.4
5 1 14	2.9	-0.2	7 2 7	2.6	1.9	8 3 6	2.0	-1.9	8 4 6	2.0	1.0
5 1 15	2.9	-1.8	7 2 9	2.3	-0.5	8 3 7	1.8	-0.8	8 4 7	1.7	1.3
6 1 12	3.0	2.4	7 2 10	2.0	-0.8	0 4 2	2.0	2.5	1 5 12	2.6	-0.7
6 1 13	2.5	-1.4	8 2 2	2.4	2.4	0 4 9	2.0	2.4	1 5 17	1.7	1.4
6 1 14	1.8	2.8	8 2 4	2.3	.7	0 4 11	3.2	-2.4	1 5 18	1.0	-1.8

Table 8. Continued.

3 5 11	2.6	-1.8	5 6 4	2.8	-1.7	5 7 13	1.7	-0.9	4 9 3	2.5	-0.0
3 5 13	2.5	-2.1	5 6 11	2.9	.8	5 7 14	1.7	-0.8	5 9 1	2.5	-1.0
3 5 14	2.3	-.6	5 6 12	2.2	-1.2	6 7 1	2.9	.5	5 9 3	2.5	-0.9
3 5 17	1.0	-2.5	5 6 14	1.4	-2.8	6 7 10	2.0	-1.2	5 9 4	2.5	1.3
4 5 3	2.2	1.8	6 6 3	2.8	-2.7	6 7 12	1.1	1.4	5 9 7	2.4	.8
4 5 13	2.2	-1.8	6 6 5	2.8	.1	7 7 2	2.5	2.2	5 9 11	1.7	-2
4 5 16	1.1	-.1	6 6 7	2.0	-2.6	7 7 5	2.3	.6	6 9 0	2.4	1.8
5 5 13	1.9	-.9	6 6 9	2.4	-1.1	7 7 6	2.1	1.8	6 9 3	2.3	-0.3
6 5 2	2.6	.9	6 6 10	2.2	.3	7 7 9	1.3	-0.7	0 10 5	2.3	1.7
6 5 6	2.5	1.7	6 6 11	1.9	-.8	0 8 11	3.1	.1	0 10 13	2.5	-0.5
6 5 8	2.4	-1.3	6 6 12	1.5	.2	1 8 8	2.8	-2.4	1 10 4	2.2	-2
6 5 10	2.1	-1.7	7 6 2	2.6	2.1	1 8 10	2.8	.3	1 10 6	2.6	-0.5
6 5 12	1.6	-1.1	7 6 6	2.3	1.5	1 8 13	2.9	1.1	1 10 9	3.0	.4
6 5 13	1.1	-.8	7 6 8	1.9	.1	1 6 14	2.6	2.3	1 10 13	2.4	.7
7 5 5	2.3	2.8	7 6 10	1.2	-1.5	2 8 7	2.8	2.9	1 10 15	1.5	-0.0
7 5 11	.8	-3.0	8 6 0	1.9	-1.6	2 8 13	2.8	-1.6	2 10 7	2.6	-1.4
8 5 5	1.6	-.4	8 6 2	1.9	-1.1	3 8 10	1.8	-.4	2 10 10	2.8	.5
8 5 7	1.1	-1.4	1 7 4	1.5	-.3	3 8 14	2.2	-1.5	2 10 11	2.9	.8
0 6 5	1.8	2.0	1 7 6	2.6	2.4	3 8 15	1.7	-1.1	4 10 2	2.9	-2.2
0 6 9	2.6	-1.0	2 7 0	1.7	-1.0	4 8 6	3.1	3.3	4 10 7	2.9	-1.9
0 6 12	2.9	2.5	2 7 3	1.9	-.1	4 8 7	3.1	-.3	4 10 11	2.2	-.9
0 6 14	2.7	.4	2 7 11	2.9	-2.5	4 8 8	3.1	-1.3	4 10 13	1.1	-1.0
1 6 6	1.7	-.3	3 7 5	2.5	.1	4 8 10	2.9	2.6	6 10 1	2.6	-.9
2 6 16	1.9	-.8	3 7 14	1.9	-.3	4 8 12	2.5	-1.0	6 10 3	2.6	-.5
3 6 2	2.1	-1.5	3 7 11	2.9	-2.1	4 8 14	1.7	2.5	6 10 6	2.2	-1.4
3 6 4	2.3	.7	4 7 2	2.6	-2.2	5 8 10	2.0	2.6	6 10 7	2.0	1.1
3 6 12	2.8	2.6	4 7 12	1.9	-.1	7 8 1	2.5	.7	6 10 8	1.7	-1.2
3 6 16	1.6	-.5	5 7 3	2.9	1.9	7 8 5	2.2	-.4	6 10 9	1.2	-.2
4 6 2	2.5	-.7	5 7 4	2.9	-1.0	2 9 10	2.5	-.6	7 10 5	1.2	-.5
4 6 10	2.8	-2.8	5 7 7	2.9	1.4	3 9 12	2.2	.7			
4 6 15	1.6	-.2	5 7 8	2.8	2.1	3 9 14	1.0	-1.2			

Table 9. The principal axes of the thermal vibration ellipsoids given by the components of a unit vector in fractional coordinates  $e_x$ ,  $e_y$ ,  $e_z$ ; the corresponding r.m.s. amplitudes, and the  $B$ -values.

Atom	$e_x$	$e_y$	$e_z$	$(\bar{u}^2)^{\dagger}$ (Å)	$B$ (Å <sup>2</sup> )
O <sub>1</sub>	0.093	-0.018	-0.048	0.287	6.50
	0.056	0.069	0.004	0.202	3.21
	0.088	-0.024	0.048	0.167	2.19
O <sub>2</sub>	0.025	0.027	-0.062	0.252	5.01
	0.027	0.068	0.026	0.195	3.02
	0.135	-0.018	0.006	0.181	2.59
N <sub>1</sub>	0.056	0.027	-0.057	0.217	3.71
	0.035	-0.070	-0.019	0.197	3.07
	0.124	0.008	0.031	0.168	2.24
N <sub>2</sub>	0.084	0.006	-0.054	0.211	3.52
	0.109	-0.021	0.038	0.186	2.72
	0.030	0.071	0.018	0.182	2.61
C <sub>1</sub>	0.015	0.066	-0.031	0.194	2.97
	0.027	0.033	0.060	0.189	2.81
	0.137	-0.014	-0.009	0.168	2.24
C <sub>2</sub>	0.084	0.046	-0.036	0.224	3.97
	0.112	-0.039	0.021	0.188	2.79
	0.011	0.046	0.054	0.165	2.16
C <sub>3</sub>	0.097	0.029	-0.042	0.204	3.30
	0.064	-0.067	-0.003	0.187	2.76
	0.079	0.019	0.054	0.170	2.28
C <sub>4</sub>	0.131	-0.015	-0.020	0.199	3.14
	0.049	0.056	0.038	0.173	2.36
	0.015	-0.046	0.054	0.169	2.27

Table 9. Continued.

C <sub>5</sub>	0.077	-0.038	-0.045	0.228	4.11
	0.067	-0.035	0.051	0.199	3.13
	0.096	0.055	0.001	0.173	2.37
C <sub>6</sub>	0.078	-0.006	-0.056	0.233	4.28
	0.055	-0.064	0.023	0.192	2.91
	0.103	0.039	0.030	0.173	2.36

from the standard deviations given in Tables 4 and 6 without taking into account the cell dimension E.S.D.'s. Distances and angles concerning the hydrogen bonding system are presented in Table 11, where the numbering of atoms are referred to Fig. 3, which is a schematical drawing of the structure viewed along [100].

Fig. 2 shows the interatomic distances and angles of cyclohexane-1,4-dione as found by crystal structure analysis at room temperature.<sup>1</sup> This molecule has a symmetry corresponding nearly to that of a twofold axis. Except for the C<sub>5</sub>-C<sub>6</sub> bond, no significant differences in corresponding angles or distances of the six-membered rings are found by comparing Figs. 1 and 2. Thus, according to the values of the angles C-C=N and C=N-O, and the N-O directions, the cyclohexane-1,4-dioxime molecules possess approximately twofold symmetry and the conformation of the six-membered ring may be described in terms of a "twisted boat" form. However, the angle between the

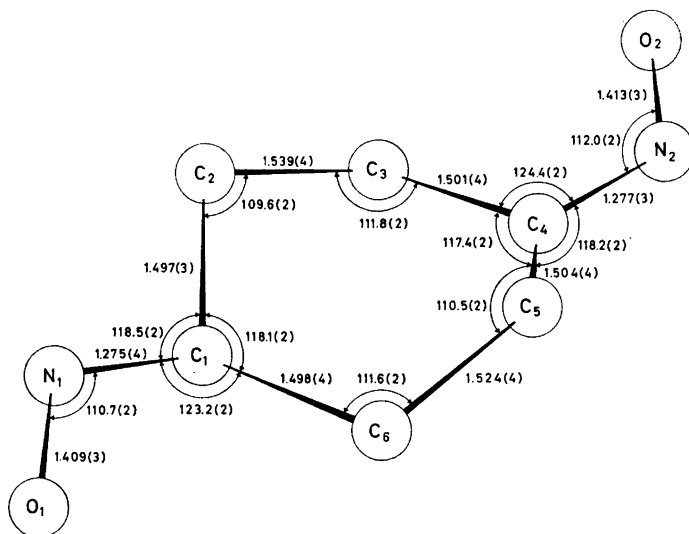


Fig. 1. Schematical drawing of the cyclohexane-1,4-dioxime molecule. The estimated standard deviations in parantheses are multiplied by 10<sup>3</sup> and 10 for distances and angles, respectively.

Table 10. Interatomic distances and angles for the hydrogen atoms of the six-membered ring, and their estimated standard deviations (Fig. 1).

Bond	(Å)	$\sigma$ (Å)
C <sub>2</sub> -H <sub>2e</sub>	1.03	0.02
C <sub>2</sub> -H <sub>2a</sub>	1.06	0.02
C <sub>3</sub> -H <sub>3e</sub>	1.04	0.02
C <sub>3</sub> -H <sub>3a</sub>	0.97	0.02
C <sub>5</sub> -H <sub>5e</sub>	1.06	0.02
C <sub>5</sub> -H <sub>5a</sub>	1.04	0.03
C <sub>6</sub> -H <sub>6e</sub>	1.01	0.02
C <sub>6</sub> -H <sub>6a</sub>	1.04	0.03
Angle	(°)	$\sigma$ (°)
C <sub>1</sub> -C <sub>2</sub> -H <sub>2e</sub>	112	1
C <sub>1</sub> -C <sub>2</sub> -H <sub>2a</sub>	109	1
H <sub>2e</sub> -C <sub>2</sub> -H <sub>2a</sub>	112	2
H <sub>2e</sub> -C <sub>2</sub> -C <sub>3</sub>	108	1
H <sub>2a</sub> -C <sub>2</sub> -C <sub>3</sub>	107	1
C <sub>1</sub> -C <sub>3</sub> -H <sub>3e</sub>	107	1
C <sub>2</sub> -C <sub>3</sub> -H <sub>3a</sub>	110	1
H <sub>3e</sub> -C <sub>3</sub> -H <sub>3a</sub>	111	2
H <sub>3e</sub> -C <sub>3</sub> -C <sub>4</sub>	109	1
H <sub>3a</sub> -C <sub>3</sub> -C <sub>4</sub>	109	1
C <sub>4</sub> -C <sub>5</sub> -H <sub>5e</sub>	110	1
C <sub>4</sub> -C <sub>5</sub> -H <sub>5a</sub>	108	1
H <sub>5e</sub> -C <sub>5</sub> -H <sub>5a</sub>	107	2
H <sub>5e</sub> -C <sub>5</sub> -C <sub>6</sub>	114	1
H <sub>5a</sub> -C <sub>5</sub> -C <sub>6</sub>	107	1
C <sub>5</sub> -C <sub>6</sub> -H <sub>6e</sub>	109	1
C <sub>5</sub> -C <sub>6</sub> -H <sub>6a</sub>	113	1
H <sub>6e</sub> -C <sub>6</sub> -H <sub>6a</sub>	110	2
H <sub>6e</sub> -C <sub>6</sub> -C <sub>1</sub>	106	1
H <sub>6a</sub> -C <sub>6</sub> -C <sub>1</sub>	107	1

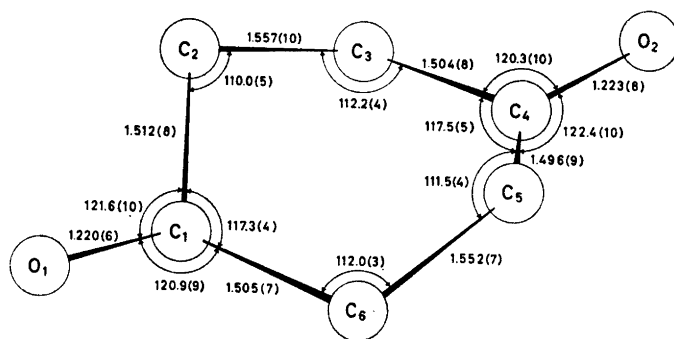


Fig. 2. Schematical drawing of the cyclohexane-1,4-dione molecule. The estimated standard deviations in parantheses are multiplied by  $10^3$  and 10 for distances and angles, respectively.

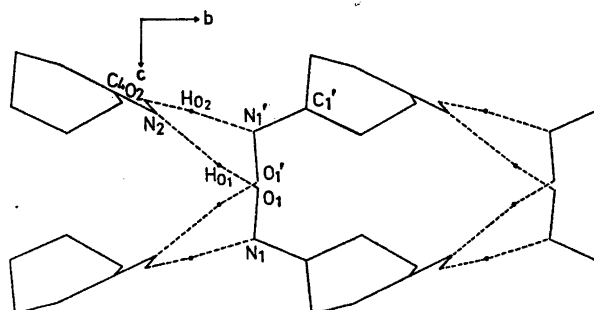


Fig. 3. Schematical drawing of the structure viewed along [100].

two C=N directions of the dioxime is found to be  $129.4^\circ$ , which is about  $25^\circ$  smaller than the angle between the C=O directions of the dione.

From Fig. 1 it may be seen that the two independent C=N bond lengths as well as the N—O bond lengths are equal within the probable limits of error. Their mean values,  $1.27_6 \text{ \AA}$  and  $1.41_1 \text{ \AA}$ , are in close agreement with the results obtained by microwave investigation of formaldoxime ( $1.276 \text{ \AA}$  and  $1.408 \text{ \AA}$ ).<sup>13</sup> The average C=N—O angle ( $111.4^\circ$ ) is somewhat larger than the corresponding angle of formaldoxime ( $110^\circ 12'$ ).

Table 11. Interatomic distances and angles of the hydrogen bonding system and their estimated standard deviations (Fig. 3).

Distance	( $\text{\AA}$ )	$\sigma$ ( $\text{\AA}$ )
$O_1-N_2$	2.839	0.003
$O_2-N_1'$	2.778	0.003
$O_1-H_{01}$	0.91	0.03
$O_2-H_{02}$	0.97	0.04
$N_2-H_{01}$	1.95	0.03
$N_1'-H_{02}$	1.81	0.04
Angle	( $^\circ$ )	$\sigma$ ( $^\circ$ )
$O_1-N_2-O_2$	108.2	0.1
$O_1-N_2-C_4$	138.3	0.2
$N_1-O_1-N_2$	117.4	0.1
$N_1'-O_2-N_2$	102.2	0.1
$O_1'-N_1'-O_2$	120.5	0.2
$C_1'=N_1'-O_2$	128.4	0.2
$C_4=N_2-H_{01}$	134	1
$O_2-N_2-H_{01}$	112	1
$N_1-O_1-H_{01}$	108	2
$O_1-H_{01}-N_2$	166	3
$C_1'=N_1'-H_{02}$	129	1
$O_1'-N_1'-H_{02}$	120	1
$N_2-O_2-H_{02}$	105	2
$O_2-H_{02}-N_1'$	175	4

Each dioxime molecule is linked to four other molecules by hydrogen bonds from O to N (Fig. 3), and the infinite double-chains thus formed are parallel to [010]. The hydrogen bond lengths,  $2.83_9$  Å and  $2.77_8$  Å, are significantly different and both agree with earlier findings for oximes.<sup>14-16</sup> The expected inverse relationship between O—H and O—N distance ( $0.91$  Å,  $0.97$  Å) as well as the correlation between larger O—H----N angle and shorter O—N distance ( $166^\circ$ ,  $175^\circ$ ) seems to hold in this compound. According to the large standard deviations, this cannot, however, be stated with confidence.

## REFERENCES

1. Groth, P. and Hassel, O. *Acta Chem. Scand.* **18** (1964) 923.
2. Mossel, A., Diss., Leiden 1963; Mossel, A. and Romers, C. *Acta Cryst.* **17** (1964) 1217.
3. Groth, P. and Hassel, O. *Acta Chem. Scand.* **19** (1965) 1733.
4. Groth, P. and Hassel, O. *Acta Chem. Scand.* **18** (1964) 1327.
5. Cochran, W. and Douglas, A. S. *Proc. Roy. Soc. (London)* **227** (1954) 486.
6. Cochran, W. and Douglas, A. S. *Proc. Roy. Soc. (London)* **243** (1958) 281.
7. Hughes, E. W. *Acta Cryst.* **6** (1953) 871.
8. Groth, P. *Acta Chem. Scand.* **20** (1966) 579.
9. Stanley, E. *Acta Cryst.* **17** (1964) 1028.
10. Gantzel, P. K., Sparks, R. A. and Trueblood, K. N. *IUCr World List of Crystallographic Computer Programs*, No. 384.
11. Hanson, H. P., Herman, F., Lea, J. D. and Skillman, S. *Acta Cryst.* **17** (1964) 1040.
12. Cruickshank, D. W. J. *Acta Cryst.* **2** (1949) 65.
13. Levine, I. *J. Chem. Phys.* **38** (1963) 2326.
14. Bierlein, T. K. and Lingafelter, E. C. *Acta Cryst.* **4** (1951) 450.
15. Merritt, L. L. and Lanterman, E. *Acta Cryst.* **5** (1952) 811.
16. Folting, K., Lipscomb, W. N. and Jerslev, B. *Acta Cryst.* **17** (1964) 1263.

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