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Crystal Structure of 3,6-Spiro-dicyclooctylidene-1,2,4,5-tetraoxacyclohexane ("Dimeric Cyclooctanone Peroxide")

P. GROTH

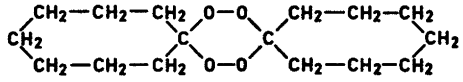
Universitetets kjemiske institutt, Blindern, Oslo 3, Norway

The crystals belong to the monoclinic system and the space group is $P2_1/c$. The unit cell, containing two molecules, has the following parameters:

$a = 9.79_1$ Å
$b = 7.33_4$ » $\beta = 114.2°$
$c = 11.77_9$ »

The phase problem was solved by a computer procedure based on direct methods. The R-value arrived at by full-matrix least squares refinement was 5.7 % when the 1282 observed reflections were included. The O—O distance is found to be 1.47_4 Å while the two C—O bond lengths are 1.44_6 Å and 1.44_1 Å, respectively. The asymmetric environment of the spiro carbon atom is probably caused by *intra*molecular hydrogen-oxygen repulsions. The conformation of the cyclooctylidene ring corresponds to the "boat-chair". Average value of C—C—C angles is 116.5°. Some short *intra*molecular H—H contacts are observed.

Dimeric cyclooctanone peroxide has been synthesized by T. Ledaal at this university.[1] The crystal structure analysis of this compound was carried out as part of a study of the stereochemical features of cyclic organic peroxides,[2,3] and also with the intention of obtaining precise information about the conformation of the cyclooctylidene ring.



CRYSTAL DATA

Dimeric cyclooctanone peroxide belongs to the monoclinic system, the systematic absences leading to the space group $P2_1/c$.

The cell parameters were found by means of $h0l$ and $0kl$ Weissenberg photographs taken with unfiltered Cu-radiation. Powder diagrams of BaF_2

(a = 6.2001 Å) were superimposed on the films for calibration purpose. 90 2θ-values were measured and used in least squares refinement of the lattice parameters. With the wavelengths for CuK-radiation taken as $\bar{\alpha}$ = 1.54178, α_1 = 1.54051, α_2 = 1.54433 and β = 1.39217 Å the following cell parameters with their estimated standard deviations resulted:

a = 9.791 Å	σ(Å) = 0.002
b = 7.334 »	σ(Å) = 0.001
c = 11.779 »	σ(Å) = 0.003
β = 114.19°	σ(°) = 0.03

The unit cell contains two molecules (ϱ_{calc}= 1.22 g.cm^{-3}, ϱ_{obs}= 1.21 g.cm^{-3}).

The intensity material was obtained from photometric measurements of integrated Weissenberg diagrams corresponding to $h0l$, $h1l$, ..., $h6l$ (Cu$K\alpha$-radiation), and of $0kl$-precession diagrams (Mo$K\alpha$-radiation). The number of reflections accessible from these diagrams is 1502, 1282 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 = 3.71 Å^2, was used in the calculation of unitary structure factors, as well as a starting parameter in the isotropic least squares refinement.

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

DETERMINATION OF THE STRUCTURE

Solution of the phase problem has been obtained by means of a computer procedure based on direct methods.[4-6] 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 $h0l$-projection (plane group $p2$). The corresponding $|U|$-distribution, given in Table 1, suggests that inequalities should be able to give some sign relationships.

Table 1. $|U|$-distribution of the $h0l$-projection.

Range of $\|U\|$	Number of reflections
0.00—0.01	2
0.01—0.02	10
0.02—0.03	13
0.03—0.04	3
0.04—0.05	7
0.05—0.10	27
0.10—0.15	24
0.15—0.20	25
0.20—0.25	10
0.25—0.30	5
0.30—0.35	2
0.35—0.40	3
0.40—0.50	6
0.50—1.00	1

The programmed systematic application of Harker-Kasper inequalities (based upon Grison's scheme [7]) gave 10 signs in terms of 3. Two of these, had been chosen positive in order to specify the origin while the third, $S(\bar{1}06)$, could be either + or −.

14 additional large and moderate unitary structure factors were selected for permutation, a process which was carried out twice, with $S(\bar{1}06) = +$ and $S(\bar{1}06) = -$.

Among the 27 reflections used, 71 relationships could be found for the sums:

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

43 "small" U's ($|U| \leqq 0.06$) gave 133 products in the zero-check expression:

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

χ_e given by

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

where N is the number of equal atoms in the cell, was calculated, and found to be $\chi_e = 3.927$.

Among the $2^{14} = 16\,384$ sign combinations tested, only *one* satisfied $\chi > 0.9.\chi_e$ for $S(\bar{1}06) = +$, while *two* satisfied this condition for $S(\bar{1}06) = -$.

The resulting sets of 27 signs were used in calculations of three electron-density maps. The one corresponding to $S(\bar{1}06) = +$ showed clearly the projection of the molecule,[15] and the trial parameters derived from this map gave $R_{h0l} = 47.0$ %. Applying a programme (written in FORTRAN IV for UNIVAC 1107 by the author) based upon the "minimum residual method" [8] the R-value was reduced to 8.5 %.

Comparison with the signs corresponding to $R_{h0l} = 8.5$ % showed that 26 had been determined correctly.

Using the z-parameters obtained, approximate values for the y-coordinates in the $0kl$-projection were determined from a model. The R-value arrived at after least squares refinement was $R_{0kl} = 9.3$ %.

REFINEMENT OF THE STRUCTURE

The coordinates arrived at by the two-dimensional analysis and the statistically determined overall B-value were used as starting parameters in a three-dimensional full-matrix least squares refinement, applying a programme written by Gantzel, Sparks, and Trueblood [9] (revised for UNIVAC 1107 by cand. real. Chr. Rømming at this university). The weighting scheme No. 1 was adopted by taking A1 = 10.0, A2 = 14.0, B1 = 0.0, B2 = −0.5, and FB = 2.0, and the form factors calculated by Hanson, Herman, Lea and Skillman [10] were used.

Anisotropic thermal vibrational parameters were introduced for the oxygen and carbon atoms after 3 cycles of isotropic refinement. The hydrogen atom positions were calculated by assuming tetrahedral C—H bonds of length 1.05 Å, and reflections corresponding to $\sin \theta/\lambda < 0.45$ were included in the

refinement of these atoms. The R-value arrived at for the 1282 observed reflections was 5.7 %.

The 220 accidentally absent reflections had previously been given the value $(1/4)I_{min}$ and had been scaled along with the rest of the data although 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 $(1282/(1282 + 220))^{\frac{1}{2}} = 0.92$. The actual improvement in the average coordinate E.S.D.'s corresponded to a factor 0.96. The R-value arrived at for all data was 7.0 %.

A final difference Fourier map, calculated with the observed data and with phases determined by the parameters corresponding to $R = 5.7$ % contained no positive or negative areas greater than 0.20 e·Å^{-3}, the E.S.D. of the electron density [11] being $\sigma(\varrho) = 0.05$ e·Å^{-3}.

The fractional atomic coordinates and the thermal vibration parameters arrived at are given in Tables 2, 3, and 4; 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

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

A comparison between calculated and osberved structure factors is presented in Table 5, while accidental absences (with F_u-values corresponding to I_{min}) are compared with the calculated values in Table 6.

Table 2. Fractional atomic coordinates for oxygen and carbon atoms.[a] (Estimated standard deviations in parantheses).

	x	y	z
O_1	0.0426	0.1545	−0.0407
	(0.0002)	(0.0002)	(0.0001)
O_2	−0.0265	0.0204	0.1059
	(0.0002)	(0.0002)	(0.0001)
C_1	0.0911	0.1204	0.0907
	(0.0002)	(0.0003)	(0.0002)
C_2	0.0845	0.3064	0.1437
	(0.0002)	(0.0004)	(0.0002)
C_3	0.2241	0.4283	0.1785
	(0.0003)	(0.0004)	(0.0002)
C_4	0.3317	0.4284	0.3169
	(0.0003)	(0.0004)	(0.0002)
C_5	0.4152	0.2534	0.3742
	(0.0002)	(0.0004)	(0.0002)
C_6	0.3175	0.0922	0.3772
	(0.0003)	(0.0004)	(0.0002)
C_7	0.2910	−0.0496	0.2764
	(0.0003)	(0.0004)	(0.0002)
C_8	0.2401	0.0191	0.1425
	(0.0002)	(0.0003)	(0.0002)

[a] For numbering of atoms, see Fig. 1.

Table 3. Anisotropic thermal vibration parameters. (Estimated standard deviations in parantheses).

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
O_1	0.0131	0.0177	0.0061	−0.0035	0.0040	−0.0008
	(0.0002)	(0.0004)	(0.0001)	(0.0004)	(0.0002)	(0.0003)
O_2	0.0119	0.0199	0.0065	−0.0031	0.0058	−0.0031
	(0.0002)	(0.0004)	(0.0001)	(0.0004)	(0.0002)	(0.0003)
C_1	0.0108	0.0186	0.0059	−0.0013	0.0037	−0.0002
	(0.0002)	(0.0005)	(0.0002)	(0.0005)	(0.0003)	(0.0004)
C_2	0.0124	0.0202	0.0079	0.0030	0.0017	−0.0029
	(0.0003)	(0.0006)	(0.0002)	(0.0006)	(0.0004)	(0.0005)
C_3	0.0166	0.0194	0.0084	−0.0031	0.0044	−0.0016
	(0.0003)	(0.0006)	(0.0002)	(0.0007)	(0.0004)	(0.0005)
C_4	0.0142	0.0218	0.0089	−0.0051	0.0041	−0.0074
	(0.0003)	(0.0006)	(0.0002)	(0.0007)	(0.0004)	(0.0005)
C_5	0.0128	0.0252	0.0080	−0.0012	0.0009	−0.0050
	(0.0003)	(0.0007)	(0.0002)	(0.0007)	(0.0004)	(0.0005)
C_6	0.0147	0.0248	0.0071	0.0060	0.0051	0.0027
	(0.0003)	(0.0007)	(0.0002)	(0.0007)	(0.0004)	(0.0005)
C_7	0.0133	0.0211	0.0086	0.0034	0.0052	0.0026
	(0.0003)	(0.0006)	(0.0002)	(0.0006)	(0.0004)	(0.0005)
C_8	0.0114	0.0207	0.0073	0.0015	0.0038	−0.0031
	(0.0003)	(0.0006)	(0.0002)	(0.0006)	(0.0003)	(0.0005)

Table 4. Fractional coordinates and isotropic thermal vibration parameters for hydrogen atoms.[a] (Estimated standard for deviations in parantheses).

	x	y	z	B (Å^2)
H_2	0.059	0.282	0.222	2.8
	(0.002)	(0.002)	(0.002)	(0.4)
H_2'	0.000	0.373	0.076	4.9
	(0.002)	(0.003)	(0.002)	(0.5)
H_3	0.183	0.560	0.158	2.1
	(0.002)	(0.002)	(0.002)	(0.4)
H_3'	0.293	0.380	0.128	3.6
	(0.002)	(0.002)	(0.002)	(0.5)
H_4	0.272	0.460	0.367	5.1
	(0.003)	(0.003)	(0.002)	(0.5)
H_4'	0.408	0.527	0.332	1.6
	(0.002)	(0.002)	(0.001)	(0.4)
H_5	0.491	0.292	0.458	3.1
	(0.002)	(0.003)	(0.002)	(0.4)
H_5'	0.485	0.210	0.324	3.0
	(0.002)	(0.003)	(0.002)	(0.4)
H_6	0.217	0.142	0.366	2.9
	(0.002)	(0.003)	(0.002)	(0.4)
H_6'	0.368	0.018	0.456	2.1
	(0.002)	(0.002)	(0.002)	(0.4)
H_7	0.224	−0.144	0.277	4.7
	(0.002)	(0.003)	(0.002)	(0.5)
H_7'	0.386	−0.123	0.295	2.2
	(0.002)	(0.002)	(0.002)	(0.4)
H_8	0.235	−0.088	0.087	1.4
	(0.002)	(0.002)	(0.001)	(0.4)
H_8'	0.318	0.120	0.137	2.9
	(0.002)	(0.003)	(0.002)	(0.4)

[a] Referring to Fig. 1 the hydrogen atoms H_n and $H_{n'}$ are bonded to C_n.

Table 5. Observed and calculated structure factors.

h	k	l	F_o	F_c
1	0	0	50.1	49.3
2	0	0	5.9	-4.6
3	0	0	62.2	62.9
4	0	0	27.0	26.4
5	0	0	5.9	-4.2
6	0	0	11.7	-11.0
7	0	0	5.7	-5.8
8	0	0	6.6	-6.5
9	0	0	4.5	-3.6
10	0	0	1.7	1.8
11	0	0	2.7	-3.6
-11	0	2	3.7	-3.6
-10	0	2	9.9	-9.9
-9	0	2	5.9	-6.1
-8	0	2	19.5	-19.4
-7	0	2	28.7	-28.1
-6	0	2	4.8	-5.2
-5	0	2	14.5	13.2
-4	0	2	17.7	-17.2
-3	0	2	23.0	-21.4
-2	0	2	137.0	135.9
-1	0	2	41.1	41.5
0	0	2	21.3	-19.8
1	0	2	38.1	38.9
2	0	2	5.0	-5.3
3	0	2	23.3	24.2
4	0	2	13.0	14.0
5	0	2	1.3	2.5
6	0	2	4.0	4.2
7	0	2	18.1	18.5
8	0	2	14.9	15.2
9	0	2	1.1	-.2
10	0	2	4.3	4.3
-12	0	4	3.7	-4.0
-10	0	4	6.1	-5.9
-9	0	4	16.2	-15.8
-8	0	4	6.7	-6.1
-7	0	4	2.1	-3.7
-6	0	4	13.1	-12.8
-5	0	4	30.5	-30.2
-4	0	4	1.3	-.8
-3	0	4	14.9	-15.3
-2	0	4	32.7	-32.0
-1	0	4	14.9	-13.5
0	0	4	69.0	-67.4
1	0	4	8.9	-8.0
2	0	4	21.1	19.6
3	0	4	4.9	-4.1
4	0	4	1.4	-.8
5	0	4	13.0	13.5
6	0	4	20.0	21.0
7	0	4	4.2	4.3
8	0	4	3.7	2.9
-12	0	6	1.9	2.1
-11	0	6	1.1	-1.1
-10	0	6	1.5	2.0
-9	0	6	1.8	2.0
-8	0	6	3.9	4.3
-7	0	6	3.9	-5.0
-6	0	6	1.7	-2.6
-5	0	6	2.4	-3.8
-4	0	6	19.1	-19.6
-3	0	6	11.0	-11.3
-2	0	6	39.7	-35.8
-1	0	6	31.2	-27.5
0	0	6	4.3	3.8
1	0	6	3.7	-3.4
2	0	6	3.7	-3.5
3	0	6	6.0	-5.6
4	0	6	11.5	12.1
5	0	6	7.0	7.0
6	0	6	1.5	-1.0
7	0	6	5.6	-5.5
8	0	6	2.4	-2.4
-12	0	8	1.4	1.1
-11	0	8	1.0	1.2
-10	0	8	4.0	4.0
-9	0	8	2.2	2.0
-8	0	8	2.2	2.1
-7	0	8	7.4	7.2
-6	0	8	4.6	-4.7
-5	0	8	2.4	2.1
-4	0	8	9.3	-9.6
-3	0	8	3.7	-2.8
-2	0	8	6.6	7.2
-1	0	8	9.4	8.5
0	0	8	8.6	-8.7
1	0	8	4.5	4.5
2	0	8	12.6	12.3
3	0	8	1.7	1.1
4	0	8	3.2	-2.2
5	0	8	2.8	-2.3
-11	0	10	1.9	-1.2
-9	0	10	4.7	4.7
-8	0	10	4.4	-3.7
-6	0	10	3.9	3.6
-5	0	10	6.6	-7.2
-4	0	10	5.7	-6.1
-3	0	10	1.6	1.5
-2	0	10	3.5	2.4
-1	0	10	8.2	-9.4
1	0	10	7.7	7.9
3	0	10	4.1	4.1
5	0	10	3.4	3.2
-10	0	12	1.8	-1.6
-9	0	12	2.0	-1.4
-8	0	12	2.3	1.6
-7	0	12	6.5	-6.4
-6	0	12	5.8	-6.5
-5	0	12	3.9	-3.9
-4	0	12	6.7	-6.8
-3	0	12	8.9	-10.0
-2	0	12	3.2	-3.3
-1	0	12	1.4	-1.2
0	0	12	5.8	-6.0
1	0	12	2.7	2.3
2	0	12	1.5	-1.3
-7	0	14	.8	.4
-6	0	14	3.5	-3.5
-5	0	14	3.0	-2.7
-2	0	14	3.2	-4.0
1	1	0	57.1	-57.9
2	1	0	9.4	-10.4
3	1	0	41.3	-41.3
4	1	0	21.8	-22.5
5	1	0	15.3	-15.4
6	1	0	5.7	-6.3
8	1	0	3.7	3.6
9	1	0	5.4	5.0
10	1	0	1.8	-1.9
11	1	0	1.9	1.8
-11	1	1	.7	-1.0
-10	1	1	5.7	-5.7
-9	1	1	3.5	-3.2
-8	1	1	2.6	2.1
-7	1	1	9.3	-9.5
-6	1	1	17.1	-17.7
-5	1	1	5.4	5.5
-4	1	1	15.8	16.9
-3	1	1	3.9	-3.5
-2	1	1	21.6	21.8
-1	1	1	52.4	54.7
0	1	1	38.9	40.8
1	1	1	28.5	29.4
2	1	1	40.3	41.8
3	1	1	32.7	33.7
4	1	1	10.5	11.5
5	1	1	6.1	7.1
6	1	1	10.4	10.4
7	1	1	5.1	5.5
8	1	1	1.9	2.1
9	1	1	3.1	2.9
-11	1	2	3.4	-3.4
-10	1	2	2.2	-2.2
-8	1	2	2.0	1.9
-7	1	2	11.3	10.8
-6	1	2	13.0	13.2
-5	1	2	21.4	21.4
-4	1	2	11.0	11.8
-3	1	2	21.4	21.6
-2	1	2	8.9	9.4
-1	1	2	40.3	40.9
0	1	2	4.2	3.9
1	1	2	42.5	41.3
2	1	2	4.6	-4.7
3	1	2	.7	.1
4	1	2	6.9	-7.7
5	1	2	3.8	3.5
6	1	2	4.0	4.5
7	1	2	4.4	3.8
8	1	2	6.0	-6.2
9	1	2	1.1	-1.5
10	1	2	1.0	-1.1
-12	1	3	3.9	-3.9
-11	1	3	3.9	-3.7
-10	1	3	1.9	2.0
-9	1	3	8.4	-8.0
-8	1	3	20.4	-20.5
-7	1	3	12.3	-12.6
-6	1	3	10.6	-10.1
-5	1	3	19.4	-20.4
-4	1	3	9.7	-10.3
-3	1	3	19.7	-19.3
-2	1	3	10.9	-10.7
-1	1	3	19.0	17.9
0	1	3	5.3	5.6
1	1	3	1.2	-.9
2	1	3	1.8	1.9
3	1	3	1.8	1.4
4	1	3	8.1	8.8
5	1	3	5.9	6.0
6	1	3	2.5	3.1
7	1	3	6.7	6.6
8	1	3	6.0	5.7
9	1	3	.8	.8
10	1	3	1.6	2.2
-12	1	4	1.2	-1.3
-10	1	4	1.6	-1.5
-9	1	4	5.3	5.1
-8	1	4	3.2	3.1
-7	1	4	5.9	5.7
-6	1	4	4.0	4.2
-5	1	4	5.6	5.5
-4	1	4	9.4	9.2
-3	1	4	5.2	-4.4
-2	1	4	14.6	15.5
-1	1	4	16.0	-16.0
0	1	4	18.5	17.2
1	1	4	36.9	36.0
2	1	4	4.4	-4.0
3	1	4	9.7	10.4
4	1	4	8.1	8.0
5	1	4	3.8	3.5
6	1	4	7.2	-7.5
7	1	4	5.8	-6.1
8	1	4	3.3	-3.3
9	1	4	6.9	-7.4
-12	1	5	2.4	2.8
-10	1	5	4.3	-4.2
-9	1	5	3.8	-3.7
-8	1	5	6.2	-6.1
-7	1	5	3.5	-3.2
-6	1	5	3.5	3.4
-5	1	5	15.4	-15.5
-4	1	5	18.6	-20.0
-3	1	5	3.6	3.0
-2	1	5	9.5	-10.4
-1	1	5	19.3	-19.8
0	1	5	24.6	-24.1
1	1	5	25.2	-25.3
2	1	5	3.2	-3.8
3	1	5	.9	-1.2
4	1	5	10.6	-10.7
6	1	5	8.3	8.1
8	1	5	1.5	-1.9
-12	1	6	1.2	-1.2
-11	1	6	1.1	.7
-10	1	6	1.9	-1.4
-9	1	6	1.0	-.7
-7	1	6	5.1	-5.4
-6	1	6	2.9	3.0
-5	1	6	4.0	-3.7
-4	1	6	5.7	6.4
-3	1	6	13.9	14.1
-2	1	6	34.2	33.2
-1	1	6	5.2	6.1
0	1	6	14.7	15.1
1	1	6	13.4	13.3
3	1	6	5.0	5.8
4	1	6	1.9	1.6
5	1	6	5.1	-5.5
6	1	6	1.1	-1.2
7	1	6	6.0	-5.8
-12	1	7	1.3	1.4
-11	1	7	1.2	1.2
-10	1	7	1.7	-1.4
-9	1	7	4.8	4.5
-8	1	7	12.4	12.2
-7	1	7	4.8	4.9
-5	1	7	7.6	6.5
-4	1	7	4.3	4.0
-3	1	7	4.7	5.1
-2	1	7	8.8	-8.3
-1	1	7	25.5	-25.7
0	1	7	1.1	.6
1	1	7	10.8	10.5
2	1	7	10.9	-10.9
3	1	7	3.7	-4.0
4	1	7	11.4	11.0
5	1	7	1.5	1.8
6	1	7	3.1	-3.2
7	1	7	2.1	2.0
-12	1	8	1.4	-1.5
-11	1	8	1.5	-1.3
-10	1	8	.9	-.4
-9	1	8	6.4	-6.1
-8	1	8	1.2	-1.1
-7	1	8	3.8	-3.8
-6	1	8	5.8	-5.0
-5	1	8	1.6	-1.5
-3	1	8	10.5	10.5
-2	1	8	2.3	2.4
-1	1	8	9.9	9.8
0	1	8	9.3	9.6
1	1	8	2.7	3.4
2	1	8	9.5	9.6
3	1	8	3.7	3.6
4	1	8	2.3	2.4
-12	1	9	1.2	-1.1
-10	1	9	4.9	4.6
-9	1	9	5.1	4.7
-8	1	9	3.3	3.3
-7	1	9	1.0	.1
-6	1	9	8.9	8.8
-4	1	9	1.8	2.0
-3	1	9	17.0	-16.9
-2	1	9	1.3	1.3
-1	1	9	16.9	17.4
0	1	9	1.1	-.9
1	1	9	1.4	1.2
2	1	9	12.5	12.9
3	1	9	5.1	5.5
4	1	9	3.1	3.0
5	1	9	5.1	5.0
6	1	9	1.9	-1.6
-11	1	10	1.7	-1.6
-9	1	10	1.5	-1.4
-8	1	10	4.7	-4.2
-7	1	10	2.5	-2.4
-6	1	10	6.7	-6.6
-5	1	10	2.4	-2.1
-4	1	10	4.4	-4.2
-3	1	10	2.9	-2.8
-1	1	10	1.1	-.7
0	1	10	6.2	6.2
1	1	10	1.0	1.0
2	1	10	4.9	4.8
3	1	10	3.0	3.3
4	1	10	1.5	1.7
5	1	10	3.9	4.7
-11	1	11	1.2	1.2
-10	1	11	1.6	1.6
-9	1	11	4.0	-3.7
-8	1	11	6.3	-5.6
-7	1	11	1.2	.8
-6	1	11	1.1	-1.1
-5	1	11	12.9	-12.7
-4	1	11	6.5	-6.8
-3	1	11	4.1	4.4
-2	1	11	1.0	-1.0
-1	1	11	1.4	1.0
0	1	11	3.9	4.1
1	1	11	1.3	-.9
2	1	11	3.6	3.7
3	1	11	5.4	5.4
-10	1	12	1.7	-1.9
-9	1	12	.8	-.9
-8	1	12	4.9	-4.4
-7	1	12	3.6	-3.5
-6	1	12	2.6	-2.4
-5	1	12	5.3	-5.2
-4	1	12	2.1	-2.0
-3	1	12	1.4	-1.2
-2	1	12	1.6	1.6
-1	1	12	1.7	-1.6
0	1	12	1.7	1.7
1	1	12	1.3	1.4
2	1	12	2.1	-2.0
-10	1	13	2.1	-2.4
-9	1	13	.6	-.5
-7	1	13	4.1	-4.3
-6	1	13	4.5	-4.8
-5	1	13	2.6	-2.7
-4	1	13	2.4	-2.7
-3	1	13	.8	.4
-2	1	13	1.5	-1.8
-1	1	13	7.1	-7.4
0	1	13	2.0	-2.1
1	1	13	1.2	1.2
-8	1	14	.6	.6
-7	1	14	1.7	-1.8
-5	1	14	1.3	-1.3
-4	1	14	1.3	-1.7
-5	1	15	1.2	1.0
1	2	0	8.1	9.0
2	2	0	45.4	-44.5
3	2	0	25.3	24.9
4	2	0	26.8	26.0
5	2	0	1.8	2.0
6	2	0	10.5	10.0
7	2	0	2.4	1.9
8	2	0	4.5	3.9
9	2	0	4.3	3.9
10	2	0	1.4	-1.2
11	2	0	1.2	-1.4
-11	2	1	.8	-.7
-10	2	1	1.6	-1.5
-8	2	1	1.1	.8
-6	2	1	2.5	2.8
-5	2	1	9.0	8.8
-4	2	1	17.9	17.5
-3	2	1	25.6	26.4
-2	2	1	2.4	2.7
-1	2	1	1.4	1.3
0	2	1	22.3	24.9
1	2	1	1.2	-.7
2	2	1	23.4	-23.9
3	2	1	12.4	-12.1
4	2	1	5.8	-6.1
5	2	1	5.5	-6.1
6	2	1	5.5	-5.8
7	2	1	2.7	-2.7
8	2	1	1.8	-1.8
9	2	1	1.2	-1.3
-11	2	2	1.5	-1.5
-10	2	2	1.6	.8

Table 5. Continued.

h	k	l	F_o	F_c
-9	2	2	.9	.2
-8	2	2	1.0	-.7
-7	2	2	14.5	-13.7
-6	2	2	1.5	.6
-5	2	2	10.9	9.6
-4	2	2	43.1	-42.6
-3	2	2	23.3	-23.3
-2	2	2	13.7	13.5
-1	2	2	14.1	12.6
0	2	2	24.1	-22.6
1	2	2	1.8	1.4
2	2	2	21.9	21.7
3	2	2	6.9	6.8
4	2	2	21.3	21.1
5	2	2	2.6	-1.9
6	2	2	2.4	-1.9
7	2	2	9.5	9.1
8	2	2	2.4	2.5
10	2	2	1.1	-1.2
-11	2	3	.8	-.5
-10	2	3	2.7	-2.3
-9	2	3	3.5	-3.3
-8	2	3	2.0	1.8
-7	2	3	3.4	3.2
-6	2	3	8.9	9.0
-5	2	3	27.1	26.0
-4	2	3	13.8	13.9
-3	2	3	3.4	3.8
-2	2	3	41.7	40.7
-1	2	3	15.5	14.8
0	2	3	23.3	-21.7
1	2	3	.5	-.1
2	2	3	.6	-.2
3	2	3	10.9	-10.5
4	2	3	1.5	-1.4
5	2	3	1.8	-2.1
6	2	3	5.3	-5.4
7	2	3	1.9	-2.2
8	2	3	.8	.4
9	2	3	2.2	-2.3
12	2	3	.6	.6
-11	2	4	1.7	1.6
-10	2	4	2.1	1.9
-9	2	4	7.7	-6.9
-8	2	4	5.3	-4.7
-7	2	4	5.8	5.1
-6	2	4	16.2	-15.5
-5	2	4	19.8	-19.6
-4	2	4	9.4	-9.4
-3	2	4	1.2	-2.0
-2	2	4	5.6	-7.0
-1	2	4	20.8	-21.4
0	2	4	14.2	-13.6
1	2	4	22.6	-22.0
2	2	4	5.9	6.4
3	2	4	14.4	-14.0
4	2	4	25.3	-24.7
5	2	4	2.3	-1.9
6	2	4	2.2	-1.6
7	2	4	4.2	-3.8
11	2	5	2.4	-2.6
-12	2	5	2.5	-2.4
-11	2	5	2.2	-2.1
-10	2	5	3.0	3.1
-9	2	5	15.6	14.6
-8	2	5	6.7	6.1
-7	2	5	1.1	-.2
-6	2	5	25.2	25.3
-5	2	5	9.6	9.9
-4	2	5	12.7	-11.1
-3	2	5	13.9	14.2
-2	2	5	11.9	12.0
-1	2	5	3.4	-2.0
0	2	5	16.0	15.9
1	2	5	11.1	10.3
2	2	5	4.2	4.0
3	2	5	3.9	3.3
4	2	5	3.9	-4.0
5	2	5	4.1	-4.3
12	2	6	2.1	2.4
11	2	6	.8	.8
10	2	6	1.7	-1.1
9	2	6	6.7	6.0
8	2	6	3.0	2.5
7	2	6	4.9	4.8
6	2	6	5.1	5.0
5	2	6	8.4	8.2
4	2	6	11.9	11.2
3	2	6	1.8	1.6
2	2	6	.6	-.1
1	2	6	18.8	-18.4
0	2	6	2.6	-2.7
-1	2	6	2.1	-2.4
-2	2	6	25.6	-25.5
-3	2	6	10.6	-10.9
-4	2	6	7.5	-7.5
-5	2	6	1.3	.4
-6	2	6	4.8	-4.8
-7	2	6	6.7	-6.5
-8	2	6	1.2	1.3
-9	2	6	3.0	-3.1
-10	2	6	7.2	6.7
-11	2	7	2.0	-1.8
-10	2	7	7.4	-6.8
-9	2	7	4.5	3.9
-8	2	7	3.4	-3.2
-7	2	7	10.6	-9.8
-6	2	7	6.0	6.2
-5	2	7	1.9	2.2
-4	2	7	2.9	3.4
-3	2	7	26.8	26.3
-2	2	7	19.2	18.8
-1	2	7	6.3	6.3
0	2	7	12.9	13.2
1	2	7	9.3	9.0
2	2	7	1.8	-1.8
3	2	7	2.9	-3.2
4	2	7	.7	-.9
-12	2	8	1.8	1.8
-11	2	8	2.6	2.0
-10	2	8	4.1	3.6
-9	2	8	8.8	8.3
-8	2	8	7.3	7.3
-7	2	8	10.1	9.7
-6	2	8	9.1	9.3
-5	2	8	15.3	15.6
-4	2	8	1.2	.2
-3	2	8	2.5	3.0
-2	2	8	16.5	16.4
-1	2	8	.9	-.0
0	2	8	.9	.1
1	2	8	.9	.7
2	2	8	7.9	7.0
3	2	8	4.8	4.2
4	2	8	5.7	-5.5
-11	2	9	2.3	2.4
-10	2	9	1.9	-1.7
-9	2	9	5.3	-5.1
-8	2	9	4.0	-4.0
-7	2	9	9.5	-9.5
-6	2	9	10.6	-10.8
-5	2	9	3.5	-3.4
-4	2	9	9.1	-8.7
-3	2	9	4.2	-4.0
-2	2	9	16.5	16.0
-1	2	9	12.0	11.7
0	2	9	4.3	4.6
1	2	9	10.4	10.6
2	2	9	7.4	7.8
3	2	9	2.3	2.4
4	2	9	1.9	1.8
5	2	9	2.9	-2.7
-11	2	10	2.0	-2.1
-10	2	10	1.6	1.6
-8	2	10	2.7	-2.2
-7	2	10	2.0	-1.7
-6	2	10	4.7	4.8
-4	2	10	3.7	-3.5
-3	2	10	10.9	11.2
-2	2	10	6.2	6.3
-1	2	10	6.3	6.7
0	2	10	7.3	7.1
1	2	10	7.8	7.8
2	2	10	9.7	9.5
3	2	10	1.3	1.3
4	2	10	2.7	2.9
-11	2	11	.5	-.8
-10	2	11	2.3	-2.1
-9	2	11	6.0	-5.8
-8	2	11	5.3	-5.1
-7	2	11	2.6	-2.7
-6	2	11	6.9	-6.5
-5	2	11	5.7	-5.5
-4	2	11	3.0	2.9
-3	2	11	2.8	2.5
-2	2	11	1.1	1.0
-1	2	11	.9	.7
0	2	11	2.6	-1.9
2	2	11	1.0	1.1
3	2	11	4.1	-3.9
-10	2	12	3.3	-3.3
-7	2	12	2.8	-2.8
-6	2	12	5.9	-5.9
-5	2	12	.8	.9
-4	2	12	1.8	-1.4
-3	2	12	.9	-.9
-2	2	12	3.4	3.2
-1	2	12	.9	-.7
0	2	12	1.8	1.9
1	2	12	.6	.4
2	2	12	1.1	1.3
-8	2	13	.8	-.9
-7	2	13	1.0	-1.3
-4	2	13	1.1	.9
-3	2	13	2.5	-2.6
-2	2	13	6.2	-6.2
-1	2	13	1.8	-2.0
0	2	13	1.0	-1.2
-8	2	14	1.0	-1.1
-7	2	14	.7	.6
-6	2	14	1.9	-1.9
-5	2	14	2.3	-2.8
-4	2	14	1.2	1.2
-3	2	14	2.3	-2.9
-2	2	14	2.1	-2.6
1	3	0	18.6	-20.6
2	3	0	1.4	-1.4
3	3	0	16.1	15.4
4	3	0	12.0	-11.6
5	3	0	7.8	7.1
6	3	0	1.4	-1.0
7	3	0	2.4	-2.2
8	3	0	10.5	10.7
10	3	0	1.8	-2.2
-10	3	1	1.9	-1.6
-9	3	1	2.0	2.0
-8	3	1	3.7	2.8
-7	3	1	2.9	-2.4
-6	3	1	2.5	-2.4
-5	3	1	9.0	8.5
-4	3	1	10.5	9.7
-3	3	1	3.5	-4.2
-2	3	1	5.0	-5.2
-1	3	1	4.6	4.2
0	3	1	3.4	3.1
1	3	1	7.0	-8.2
2	3	1	9.2	-9.5
3	3	1	5.6	5.4
4	3	1	2.4	2.4
5	3	1	1.0	-1.0
6	3	1	4.4	4.1
7	3	1	5.9	5.1
8	3	1	3.7	3.8
9	3	1	3.3	3.5
10	3	1	1.5	1.8
-10	3	2	4.1	-4.5
-9	3	2	2.0	-1.9
-8	3	2	1.2	-.9
-7	3	2	4.5	-3.9
-6	3	2	8.7	8.4
-5	3	2	8.8	-8.5
-4	3	2	1.0	-.4
-3	3	2	20.2	18.7
-2	3	2	5.8	-5.3
-1	3	2	3.4	-3.3
0	3	2	12.2	-14.5
1	3	2	2.9	-3.0
2	3	2	11.5	-12.2
3	3	2	3.1	2.8
4	3	2	11.3	-11.7
5	3	2	13.0	-13.1
6	3	2	14.2	14.5
7	3	2	1.5	-1.3
8	3	2	5.8	-6.1
9	3	2	2.3	2.3
10	3	2	.8	1.1
-11	3	3	1.1	-.7
-10	3	3	2.3	2.0
-9	3	3	1.4	.8
-8	3	3	2.8	-2.6
-7	3	3	2.7	2.4
-6	3	3	8.0	7.6
-5	3	3	15.8	-14.6
-4	3	3	13.4	-12.5
-3	3	3	13.3	-13.5
-2	3	3	20.7	-20.7
-1	3	3	31.2	-34.3
0	3	3	15.9	-17.8
1	3	3	10.7	-11.3
2	3	3	7.8	-7.9
3	3	3	3.0	-3.1
4	3	3	2.3	-2.3
5	3	3	4.5	4.8
6	3	3	1.1	-1.1
9	3	3	1.1	-1.1
-11	3	4	1.8	-2.1
-8	3	4	6.1	6.3
-6	3	4	4.2	3.8
-5	3	4	13.2	12.2
-4	3	4	1.0	-.8
-3	3	4	12.9	12.2
-2	3	4	9.7	-8.2
-1	3	4	20.9	-19.5
0	3	4	1.1	-.8
1	3	4	8.5	8.3
2	3	4	12.8	-12.4
3	3	4	19.2	-19.3
4	3	4	16.0	16.4
5	3	4	1.8	1.3
6	3	4	7.7	-8.0
7	3	4	2.5	2.1
8	3	4	1.7	-1.9
9	3	4	1.2	-1.5
-11	3	5	3.5	3.9
-10	3	5	2.9	2.8
-9	3	5	3.3	3.0
-8	3	5	7.0	7.1
-7	3	5	8.4	7.9
-5	3	5	5.6	-4.7
-4	3	5	7.2	-6.7
-3	3	5	7.9	-7.2
-2	3	5	10.3	-9.7
-1	3	5	12.9	-13.3
0	3	5	13.7	-15.0
1	3	5	8.4	-9.3
2	3	5	2.2	-3.2
3	3	5	10.9	-11.8
4	3	5	12.8	-13.3
5	3	5	4.9	-5.1
6	3	5	4.8	-5.4
7	3	5	8.8	-8.8
8	3	5	4.3	-4.8
10	3	6	1.3	1.3
-9	3	6	3.0	3.3
-8	3	6	2.7	2.6
-7	3	6	4.4	4.2
-5	3	6	9.3	10.4
-4	3	6	3.3	-2.4
-3	3	6	11.8	-11.8
-2	3	6	4.6	4.9
-1	3	6	11.2	11.3
0	3	6	2.3	2.2
1	3	6	7.5	-7.2
2	3	6	15.1	15.4
3	3	6	8.9	9.1
4	3	6	2.5	-2.1
5	3	6	5.5	4.8
6	3	6	4.4	-4.6
7	3	6	2.1	-2.1
-11	3	7	1.7	2.1
-10	3	7	2.7	2.9
-9	3	7	6.5	6.7
-8	3	7	5.1	5.0
-7	3	7	3.6	4.1
-6	3	7	2.5	2.5
-5	3	7	4.7	5.3
-4	3	7	15.0	15.9
-3	3	7	13.5	14.0
-2	3	7	6.6	6.6
-1	3	7	10.5	10.0
0	3	7	13.9	13.4
1	3	7	3.4	3.5
2	3	7	8.3	-8.8
3	3	7	7.8	-8.2
4	3	7	5.1	-5.4
5	3	7	9.3	-9.1
6	3	7	8.6	-9.0
7	3	7	4.4	-4.8
-11	3	8	1.6	2.0
-8	3	8	2.2	-2.4
-7	3	8	1.4	1.3
-6	3	8	5.4	-5.1
-5	3	8	8.0	-8.9
-2	3	8	5.0	5.2
-1	3	8	3.2	2.6
0	3	8	9.9	9.1
1	3	8	10.9	10.7
2	3	8	3.4	3.4
3	3	8	8.4	7.6
4	3	8	3.6	-3.2
5	3	8	2.3	-1.9
6	3	8	1.8	2.0
-11	3	9	1.4	-.9
-9	3	9	8.3	8.8
-5	3	9	14.7	15.5
-4	3	9	12.3	12.8
-3	3	9	12.6	12.9
-2	3	9	15.0	15.4
-1	3	9	15.8	15.9
0	3	9	6.3	6.1
1	3	9	1.2	-1.1
2	3	9	1.2	1.2
3	3	9	2.0	-1.9
5	3	9	.8	-1.1
-9	3	10	1.5	-1.8
-8	3	10	3.9	-4.1
-7	3	10	5.5	-5.7
-6	3	10	2.6	-2.8
-5	3	10	3.6	-3.7
-4	3	10	2.6	2.0
-1	3	10	7.0	6.8
1	3	10	2.8	2.6
2	3	10	1.7	-1.3
3	3	10	3.5	-3.1
4	3	10	.8	-.5
-10	3	11	2.3	-2.5
-9	3	11	3.7	-4.3
-8	3	11	2.1	-2.0
-7	3	11	2.6	2.6
-6	3	11	3.7	3.7
-4	3	11	1.3	1.3
-3	3	11	1.6	1.3
-2	3	11	5.3	5.4
-1	3	11	4.4	4.4
0	3	11	1.3	-.9
1	3	11	1.1	.9
2	3	11	2.1	2.0
3	3	11	3.2	3.6
4	3	11	4.2	4.5
-9	3	12	1.2	-1.4
-8	3	12	1.1	-1.3
-7	3	12	1.0	-1.0
-6	3	12	2.0	2.1
-5	3	12	1.1	-.8
-4	3	12	2.6	-2.4
-3	3	12	4.5	4.7
-2	3	12	3.0	-2.7
-1	3	12	2.8	-2.9
0	3	12	4.1	-4.4
1	3	12	1.4	-1.6
-8	3	13	3.4	-3.6
-5	3	13	2.8	-3.2
-4	3	13	1.7	-2.0
-3	3	13	2.7	-2.8
-2	3	13	1.8	-1.9
-1	3	13	1.7	-1.9
0	3	13	.7	-.8
-8	3	14	3.1	3.9
-5	3	14	1.0	-1.1
-4	3	14	1.4	-2.0
-1	4	0	7.8	-8.8
2	4	0	2.9	3.1
3	4	0	1.5	-1.1
4	4	0	9.2	8.7
5	4	0	11.1	11.8
6	4	0	2.2	-1.7
7	4	0	6.9	6.9
8	4	0	3.4	3.5
-10	4	1	1.8	-2.1

Table 5. Continued.

h	k	l	F_o	F_c
-9	4	1	1.7	-1.6
-8	4	1	4.3	-4.7
-7	4	1	4.3	-4.7
-6	4	1	3.3	-3.0
-5	4	1	7.0	-7.2
-4	4	1	1.0	-.1
-3	4	1	1.7	-2.3
-2	4	1	19.7	-19.8
-1	4	1	15.2	-15.9
0	4	1	.4	-.2
1	4	1	11.7	-12.9
2	4	1	16.6	-16.5
3	4	1	2.5	2.7
4	4	1	6.0	5.7
5	4	1	4.3	4.4
6	4	1	9.3	9.5
7	4	1	6.4	6.5
8	4	1	3.7	4.0
9	4	1	2.6	3.1
10	4	1	1.6	2.4
-9	4	2	3.6	3.6
-8	4	2	1.8	-1.4
-7	4	2	2.4	2.4
-6	4	2	4.3	3.8
-5	4	2	.9	-.9
-4	4	2	20.0	-19.3
-3	4	2	7.5	-7.3
-2	4	2	8.5	-9.2
-1	4	2	9.3	-10.7
0	4	2	9.9	-10.2
1	4	2	2.6	-2.6
2	4	2	11.0	10.9
3	4	2	2.7	-2.5
4	4	2	6.3	6.6
5	4	2	2.5	2.4
6	4	2	1.1	-.5
7	4	2	4.3	4.9
8	4	2	1.3	-1.1
9	4	2	1.5	2.1
-11	4	3	1.3	1.0
-10	4	3	1.3	1.0
-9	4	3	1.5	.9
-8	4	3	3.2	3.1
-7	4	3	2.8	-2.3
-6	4	3	3.0	2.7
-5	4	3	19.4	-18.8
-4	4	3	24.0	-22.3
-3	4	3	4.0	-4.7
-2	4	3	17.0	-15.8
-1	4	3	27.9	-27.4
0	4	3	2.1	-2.5
1	4	3	1.6	.8
2	4	3	4.1	-4.4
3	4	3	5.6	5.4
4	4	3	5.0	4.7
5	4	3	1.8	-1.4
6	4	3	1.4	1.1
7	4	3	.8	-.8
8	4	3	1.9	2.5
9	4	3	2.3	2.6
-11	4	4	1.7	1.6
-10	4	4	1.3	1.0
-9	4	4	1.3	1.2
-8	4	4	4.3	4.2
-7	4	4	13.8	-13.8
-6	4	4	10.7	-10.4
-5	4	4	5.5	-5.5
-4	4	4	15.6	-15.0
-3	4	4	10.2	-10.3
-2	4	4	11.3	-11.3
-1	4	4	2.8	-3.3
0	4	4	7.3	-8.0
1	4	4	1.1	-.6
2	4	4	6.9	-7.0
3	4	4	11.3	-11.3
4	4	4	3.1	2.9
5	4	4	4.5	-4.7
6	4	4	3.6	-4.1
7	4	4	1.5	2.4
8	4	4	2.9	3.3
9	4	4	2.4	2.4
-10	4	5	8.9	9.2
-9	4	5	8.1	-7.8
-8	4	5	11.4	-10.6
-7	4	5	7.8	6.9
-6	4	5	15.8	-14.6
-5	4	5	2.4	2.0
-4	4	5	2.6	1.6
-3	4	5	5.9	-6.2
-2	4	5	3.2	2.9
-1	4	5	9.9	-9.5
0	4	5	2.9	-2.3
1	4	5	2.8	-2.9
2	4	5	1.5	2.4
3	4	5	2.1	2.2
4	4	5	1.9	1.9
5	4	5	5.8	6.4
6	4	5	2.1	2.5
7	4	5	1.4	-.1
8	4	5	10.1	10.1
-9	4	6	1.7	1.6
-8	4	6	1.0	-1.2
-7	4	6	3.0	2.1
-6	4	6	3.1	2.3
-5	4	6	5.0	-5.2
-4	4	6	16.8	-16.3
-3	4	6	3.6	-3.5
-2	4	6	7.7	-7.5
6	4	6	8.1	-8.1
7	4	6	2.8	-3.2
-11	4	7	1.4	-2.1
-8	4	7	5.4	5.7
-7	4	7	4.9	-4.8
-6	4	7	7.5	7.5
-5	4	7	4.7	4.8
-4	4	7	1.1	-.9
-3	4	7	5.7	6.1
-2	4	7	2.3	2.0
-1	4	7	3.4	-3.3
0	4	7	6.5	6.0
1	4	7	1.2	-.8
2	4	7	12.8	-12.0
3	4	7	2.8	-2.0
4	4	7	1.2	.8
5	4	7	2.8	-2.8
-11	4	8	1.0	-.3
-9	4	8	1.0	1.1
-8	4	8	1.0	1.8
-7	4	8	1.0	2.0
-6	4	8	13.8	14.4
-5	4	8	11.9	12.6
-4	4	8	7.3	7.3
-3	4	8	12.6	12.5
-2	4	8	10.7	11.0
-1	4	8	9.1	8.5
0	4	8	3.2	-3.1
1	4	8	1.8	-1.7
2	4	8	3.3	-2.7
3	4	8	3.7	-3.7
4	4	8	1.0	-.9
5	4	8	1.5	-1.5
6	4	8	2.0	2.5
7	4	9	1.7	1.4
-6	4	9	2.6	1.6
-5	4	9	4.7	5.2
-4	4	9	2.2	-2.2
-3	4	9	5.4	-5.1
-2	4	9	4.1	3.5
0	4	9	10.6	-10.0
1	4	9	3.6	-3.0
2	4	9	1.1	.7
3	4	9	.9	-.2
4	4	9	.9	.9
-10	4	10	1.1	-1.3
-9	4	10	1.7	-1.7
-8	4	10	3.4	-4.0
-7	4	10	2.2	2.2
-6	4	10	4.8	5.8
-5	4	10	4.2	4.7
-4	4	10	5.7	6.1
-3	4	10	3.4	4.2
-2	4	10	9.3	9.4
-1	4	10	4.7	4.6
0	4	10	1.0	1.0
1	4	10	1.2	1.0
2	4	10	4.1	4.2
-8	4	11	1.1	.6
-7	4	11	2.7	3.1
-6	4	11	1.5	-1.4
-5	4	11	4.2	-4.3
-4	4	11	1.7	1.4
-3	4	11	3.6	-3.5
-2	4	11	3.3	-3.1
-1	4	11	1.5	-1.2
0	4	11	1.3	1.1
1	4	11	1.6	2.0
2	4	11	2.0	-2.6
-8	4	12	1.0	-.9
-7	4	12	3.4	-3.5
-6	4	12	2.6	-2.7
-5	4	12	1.2	-1.0
-4	4	12	1.4	-1.5
-3	4	13	2.1	2.4
-2	4	13	2.2	2.7
-1	5	0	2.9	-2.5
0	5	0	12.6	13.8
1	5	0	10.0	10.2
2	5	0	14.3	15.0
3	5	0	5.8	6.3
4	5	0	1.9	1.8
5	5	0	4.1	4.9
6	5	0	3.7	4.1
7	5	0	1.2	1.1
-9	5	1	2.7	2.7
-7	5	1	1.5	1.8
-6	5	1	4.9	5.4
-5	5	1	6.0	5.8
-4	5	1	2.7	2.3
-3	5	1	6.7	-6.3
-2	5	1	6.6	-6.1
-1	5	1	.9	1.2
0	5	1	4.3	6.2
1	5	1	1.3	-.3
2	5	1	3.1	3.8
3	5	1	17.1	17.1
4	5	1	17.1	17.2
5	5	1	7.6	7.9
6	5	1	3.2	3.2
7	5	1	4.4	4.3
8	5	1	2.7	2.6
9	5	1	1.6	-1.8
-7	5	2	1.8	-1.6
-6	5	2	8.6	-9.1
-5	5	2	10.5	-10.8
-4	5	2	16.1	-17.1
-3	5	2	8.4	-9.3
-2	5	2	6.0	-7.1
-1	5	2	14.2	-15.6
0	5	2	2.5	-3.2
1	5	2	6.2	-7.2
2	5	2	6.4	5.7
3	5	2	3.9	4.1
4	5	2	7.5	-7.8
5	5	2	1.5	-.5
6	5	2	5.3	5.2
7	5	2	3.3	3.7
8	5	2	2.9	-2.9
-10	5	3	1.4	-.5
-9	5	3	2.0	-1.5
-8	5	3	8.4	-9.1
-7	5	3	17.3	-17.0
-6	5	3	7.1	-7.8
-5	5	3	3.9	3.0
-4	5	3	4.8	-5.0
-3	5	3	4.5	-4.2
-2	5	3	8.2	7.8
-1	5	3	11.1	10.7
0	5	3	7.0	6.5
1	5	3	3.8	-3.5
2	5	3	1.8	1.2
3	5	3	2.0	-2.5
-9	5	4	1.8	1.9
-8	5	4	1.5	-1.1
-7	5	4	3.7	-3.8
-6	5	4	5.2	-5.6
-5	5	4	1.8	.8
-4	5	4	4.5	-4.9
-3	5	4	3.4	-3.9
-2	5	4	12.7	-12.1
-1	5	4	2.9	-3.3
0	5	4	2.8	1.7
1	5	4	15.0	-14.7
2	5	4	10.1	-9.8
3	5	4	2.5	2.0
4	5	4	4.7	-4.6
-9	5	5	2.9	-3.2
-8	5	5	1.5	-1.5
-7	5	5	1.5	-1.3
-6	5	5	9.4	-9.6
-5	5	5	4.4	-4.4
-4	5	5	2.8	-2.5
-3	5	5	2.4	-2.2
-2	5	5	1.8	-2.0
-1	5	5	4.0	-3.8
0	5	5	3.5	2.8
1	5	5	2.1	-1.8
2	5	5	13.6	-13.1
3	5	5	5.7	-5.4
4	5	5	3.5	-3.8
5	5	5	2.4	-2.7
-9	5	6	2.2	2.6
-8	5	6	2.4	2.7
-7	5	6	4.4	4.6
-6	5	6	4.8	5.0
-5	5	6	4.1	4.3
-4	5	6	4.3	4.2
-3	5	6	2.9	-2.5
-2	5	6	6.6	5.7
-1	5	6	9.0	8.7
0	5	6	10.1	9.3
1	5	6	3.8	-3.8
2	5	6	3.5	-3.0
3	5	6	1.9	-1.6
4	5	6	2.1	2.2
-9	5	7	1.5	1.7
-7	5	7	2.3	2.4
-6	5	7	1.5	1.0
-5	5	7	1.5	1.6
-4	5	7	7.4	7.5
-3	5	7	4.5	-4.2
-2	5	7	6.1	5.6
-1	5	7	3.9	3.4
0	5	7	9.2	-8.6
1	5	7	4.1	-3.7
2	5	7	2.7	-2.4
-8	5	8	2.1	2.2
-7	5	8	2.3	2.6
-6	5	8	3.1	3.7
-5	5	8	1.8	1.4
-4	5	8	1.9	-1.3
-3	5	8	5.0	5.0
-2	5	8	2.7	-2.5
-1	5	8	4.9	-4.7
0	5	8	2.4	-2.5
1	5	8	2.8	-2.4
2	5	8	1.8	-1.3
3	5	8	1.9	-1.5
4	5	8	1.7	1.6
-9	5	9	1.1	1.6
-8	5	9	1.2	-.9
-6	5	9	4.0	4.7
-4	5	9	1.8	-1.4
-3	5	9	4.7	4.6
-2	5	9	4.2	4.0
0	5	9	3.4	2.9
1	5	9	1.9	1.4
2	5	9	1.2	-.3
3	5	9	4.1	4.5
-8	5	10	1.1	.8
-7	5	10	3.1	2.0
-5	5	10	3.0	2.3
-3	5	10	2.9	-2.7
-1	5	10	2.9	-3.0
0	5	10	2.6	-2.2
1	5	10	2.4	-2.5
-7	5	11	1.7	-1.7
-2	5	11	2.6	2.5
-1	5	11	1.1	-.5
0	5	11	2.2	-2.2
1	5	11	2.4	2.7
-7	5	12	2.1	2.4
-6	5	12	1.4	1.2
-5	5	12	1.0	-1.2
-2	5	12	1.1	-.9
-1	5	12	1.8	-1.8
1	6	0	12.4	15.7
2	6	0	11.7	12.6
3	6	0	2.1	1.9
4	6	0	5.0	5.9
5	6	0	3.5	2.9
7	6	0	1.9	-.9
8	6	0	2.0	2.1
-6	6	1	2.3	-2.4
-5	6	1	1.7	-1.4
-4	6	1	4.4	-4.3
-3	6	1	10.6	-11.0
-2	6	1	8.6	-9.2
-1	6	1	7.2	-8.3
0	6	1	11.2	-12.2
1	6	1	1.8	-1.8
2	6	1	4.2	6.2
3	6	1	1.1	1.0
4	6	1	3.8	3.9
5	6	1	10.5	10.5
6	6	1	2.6	2.0
7	6	1	3.1	-2.3
8	6	1	1.4	1.4
-8	6	2	2.6	-2.7
-7	6	2	2.2	-3.3
-6	6	2	2.8	-3.1
-5	6	2	3.0	-2.9
-4	6	2	3.3	-3.5
-3	6	2	2.6	-2.7
-2	6	2	3.6	3.2
-1	6	2	4.9	5.0
0	6	2	6.5	-6.5
1	6	2	8.0	8.9
2	6	2	8.2	9.4
3	6	2	1.9	-.8
4	6	2	6.9	9.3
5	6	2	5.1	5.1
6	6	2	3.1	2.5
7	6	2	4.0	4.3
8	6	2	5.0	5.3
-7	6	3	1.7	1.2
-6	6	3	1.9	-1.3
-5	6	3	3.8	-4.1
-4	6	3	1.8	-1.7
-3	6	3	2.7	-2.8
-2	6	3	8.0	-8.4
-1	6	3	2.1	-1.5
0	6	3	5.6	5.2
1	6	3	4.0	-3.9
2	6	3	4.7	-4.4
3	6	3	2.9	1.5
4	6	3	3.4	-3.2
5	6	3	8.8	-8.7
6	6	3	5.4	-5.1
7	6	3	3.4	-2.9
8	6	3	3.6	-3.2
-7	6	4	1.4	-1.4
-6	6	4	1.3	-1.7
-5	6	4	3.7	-4.3
-4	6	4	2.7	-2.5
-3	6	4	2.3	-2.2
-2	6	4	11.4	-11.3
-1	6	4	3.8	-3.6
0	6	4	8.4	-8.2
1	6	4	2.3	1.7
2	6	4	2.8	-2.3
3	6	4	6.1	5.4
4	6	4	3.4	2.7
5	6	4	1.9	1.1
6	6	4	1.8	1.7
-9	6	5	1.3	-1.0
-8	6	5	2.7	2.5
-7	6	5	3.2	3.4
-6	6	5	8.6	8.5
-5	6	5	3.5	3.0
-4	6	5	2.8	-1.8
-3	6	5	1.7	-1.4
-2	6	5	5.7	-5.3
-1	6	5	6.0	-5.4
0	6	5	4.1	-3.7
1	6	5	5.5	-5.8
2	6	5	1.7	-1.3
3	6	5	1.6	1.6
4	6	5	4.6	-4.9
5	6	5	6.2	-6.1
-8	6	6	2.4	-1.9
-7	6	6	4.3	-3.5
-6	6	6	5.1	-5.3
-5	6	6	2.4	-2.2
-4	6	6	6.1	4.9
-3	6	6	1.6	1.4
-2	6	6	4.6	-4.9
-1	6	6	5.0	4.9
0	6	6	2.2	1.1
1	6	6	1.9	-1.5
-7	6	7	1.7	2.1
-6	6	7	3.6	4.2
-5	6	7	5.0	4.9
-4	6	7	2.2	1.1
-2	6	7	1.9	-1.5

Table 5. Continued.

h	k	l	F_o	F_c
4	6	7	3.5	3.7
-7	6	8	3.7	-3.9
-6	6	8	1.8	1.8
-5	6	8	1.8	-1.4
-4	6	8	1.8	-1.9
-3	6	8	3.9	3.1
-2	6	8	1.9	1.2
-1	6	8		
0	6	8		
1	6	8	2.4	1.7
2	6	8	2.2	-1.7
3	6	8	1.2	-.5
-7	6	9	1.7	1.3
-6	6	9	1.9	-2.0
-4	6	9	1.8	1.3
0	6	9	2.6	-2.2
1	6	9	3.0	-2.7
2	6	9	2.5	2.7
-5	6	10	1.5	1.3
-4	6	10	1.7	-2.3
-3	6	10	1.5	-1.5
-1	6	10	1.6	1.6
0	6	10	3.2	-3.0
1	6	10	3.1	-3.3
-5	6	11	1.6	+2.4
-4	6	11	1.2	-.7
-3	6	11	1.2	.9
-2	6	11	2.3	-2.6

Table 6. Unobserved and calculated structure factors. (The F_u's have been obtained by taking unobserved intensities as equal to the smallest observable).

h	k	l	F_u	F_c
-12	0	2	.6	-1.5
-11	0	4	1.1	.5
-9	0	4	.9	-.4
-8	0	4	1.2	-1.3
-7	0	4	.5	1.0
-12	0	10	.5	-.1
-10	0	10	1.1	.8
-7	0	10	1.3	.0
-6	0	10	1.3	-.3
-5	0	10	1.3	.7
-4	0	10	1.0	.4
-11	0	12	.6	3.2
-3	0	12	.4	-2.4
-9	0	14	.6	-1.9
-8	0	14	.8	.8
-4	0	14	.9	.4
-3	0	14	.9	-.1
-2	0	14	.7	.1
7	1	0	1.0	-.8
10	1	1	.8	.5
11	1	1	.4	-.5
-12	1	2	.4	.2
-9	1	2	1.0	-.2
-11	1	4	.8	.7
-11	1	5	.9	-.3
5	1	5	1.0	-.7
7	1	5	.9	.1
9	1	5	.4	2.9
-8	1	6	1.0	.4
-5	1	6	.7	-.3
8	1	6	.6	.2
-4	1	8	.9	.2
5	1	8	.9	.2
3	1	8	1.0	.2
5	1	8	.9	.0
7	1	8	.3	2.3
-11	1	9	.8	.5
-8	1	9	1.0	.5
-10	1	10	.8	-.1
-2	1	10	1.0	.4
4	1	11	.4	-3.1
-11	1	12	.4	.1
-8	1	13	.8	.5
-9	1	14	.4	-1.0
-8	1	14	.7	.1
-5	1	14	.7	.5
-4	1	14	.7	.0
-2	1	14	.6	.8
-8	1	15	.4	.6
-4	1	15	.4	1.8
-9	2	1	.9	.4
-7	2	1	.9	-.6
10	2	1	.7	.3
9	2	2	.8	-.1
7	2	4	.9	-.1
9	2	4	.6	.5
-10	2	5	.9	.3
4	2	5	.9	-.6
-12	2	7	.5	.1
-10	2	7	.9	.2
5	2	7	.9	-.7
6	2	8	.6	.4
-9	2	10	.8	-.1
-5	2	10	.9	.5
5	2	10	.2	-.5
1	2	11	.8	.4
-9	2	13	.5	-.4
-6	2	13	.7	.4
-5	2	13	.8	.2
1	2	13	.3	-4.6
-1	2	14	.3	2.3
9	3	0	1.2	-.7
-11	3	1	.7	-1.1
-11	3	2	.8	1.0
-5	3	3	1.0	-.9
6	3	3	1.3	.4
8	3	3	1.1	.8
-10	3	4	1.2	.1
-9	3	4	1.3	-.5
-7	3	4	1.3	.6
-6	3	5	1.2	.9
-11	3	6	.9	-.6
-10	3	6	1.2	.1
-10	3	8	1.1	.6
-9	3	8	1.2	-.7
-4	3	8	1.3	.1
-3	3	8	1.2	.9
6	3	8	.7	.8
-10	3	9	1.0	-1.2
-9	3	9	1.2	.5
-7	3	9	1.3	-.9
3	3	9	1.2	.1
-11	3	10	.6	-.6
-10	3	10	.9	.8
-2	3	10	1.3	.2
-2	3	10	1.3	.9
0	3	10	1.3	-.1
-10	3	12	.5	-.9
0	3	12	1.0	-.6
2	3	12	.4	-2.7
-9	3	13	.5	1.2
-8	3	13	.8	.2
0	3	13	.6	.6
-2	3	14	.3	1.2
9	4	0	1.1	-.5
10	4	0	.8	-.0
-11	4	2	.5	-1.8
-10	4	2	1.0	-.2
-5	4	2	1.1	.0
-6	4	3	1.2	-.1
7	4	3	1.2	.6
-9	4	5	1.2	-.7
-3	4	5	1.0	.6
3	4	5	1.3	-.3
6	4	5	1.2	-.8
-7	4	6	1.3	.3
-6	4	6	1.3	-.5
-10	4	7	1.0	.0
6	4	7	.8	-1.0
-10	4	8	1.0	.4
-6	4	8	1.3	.3
-11	4	9	.4	1.7
-10	4	9	.9	-.3
-1	4	9	1.3	.4
0	4	10	1.2	.0
-10	4	11	.5	1.1
-9	4	11	.8	.0
-4	4	11	1.2	-.1
-7	4	12	.9	.4
-6	4	12	1.0	.1
-4	4	12	1.1	.6
-3	4	12	1.0	.8
1	4	12	.5	2.7
-7	4	13	.7	-.7
-6	4	13	.8	.2
-3	4	13	.8	.3
-2	4	13	.7	-.3
-1	4	13	.5	1.9
-8	5	1	1.5	-.0
-10	5	2	1.0	.6
-9	5	2	1.4	-.8
-8	5	2	1.6	-1.1
-10	5	1	.7	-.2
-9	5	3	1.4	.1
-6	5	3	1.6	-.4
4	5	3	1.6	1.0
7	5	3	1.4	.5
-10	5	4	1.1	1.9
-7	5	4	1.7	.7
4	5	4	1.7	-.8
7	5	4	1.2	-.4
-10	5	5	1.1	-.0
-4	5	5	1.5	.1
5	5	5	1.5	.0
-10	5	6	1.1	-.2
-7	5	6	1.7	-1.3
3	5	6	1.7	.7
-10	5	7	1.0	1.6
-8	5	7	1.5	-.8
-3	5	7	1.6	1.2
3	5	7	1.6	.1
5	5	7	1.1	-.0
-10	5	8	.9	1.1
-9	5	8	1.3	-.7
5	5	8	.5	-2.7
-7	5	9	1.5	-1.2
-5	5	9	1.6	1.2
-1	5	9	1.6	-.3
4	5	9	.5	1.0
-9	5	10	1.0	.7
-8	5	10	1.5	-.4
-4	5	10	1.6	-1.0
-2	5	10	1.5	-.9
2	5	10	1.0	.6
-8	5	11	1.0	-.9
-6	5	11	1.3	-1.1
-5	5	11	1.4	1.1
-4	5	11	1.4	-.9
-3	5	11	1.4	.1
-4	5	12	1.1	1.1
-3	5	12	1.1	-.1
6	6	0	2.0	-.8
-9	6	1	1.0	1.1
7	6	1	1.7	-.3
-9	6	3	1.3	.7
-8	6	4	1.9	-1.0
-2	6	4	1.6	-.6
2	6	4	1.9	.1
7	6	4	1.8	2.7
-6	6	5	1.9	-1.3
-5	6	5	1.9	.7
-1	6	5	1.8	-.6
-9	6	6	1.3	.1
-6	6	6	1.6	.5
-7	6	6	1.8	.9
-2	6	6	1.9	-.4
2	6	6	1.9	.1
3	6	6	1.8	.1
4	6	6	1.6	-.2
-8	6	7	1.6	.3
-7	6	7	1.8	-.6
-4	6	7	1.9	.9
-1	6	7	1.9	1.0
0	6	7	1.9	-.6
1	6	7	1.9	-.1
3	6	7	1.6	-.3
-9	6	8	1.0	2.0
-8	6	8	1.4	.4
-6	6	8	1.8	-.7
-2	6	8	1.9	.4
-8	6	9	1.3	-.4
-6	6	9	1.7	.1
-5	6	9	1.7	.8
-4	6	9	1.8	.4
-2	6	9	1.8	.3
-7	6	10	1.3	-.4
-6	6	10	1.5	.4
-2	6	10	1.6	.0
-8	6	11	1.1	1.3

THERMAL MOTIONS

The principal axes of the thermal vibration ellipsoids for oxygen and carbon atoms were calculated from the temperature parameters given in Table 3. Root mean square amplitudes and the corresponding B-values for the atomic anisotropic thermal vibration along the principal axes together with the components of these axes along the crystal axes are given in Table 7.

The atomic vibration tensors referred to crystal axes were calculated according to Scheringer [12] and applied in a rigid-body analysis of translational and librational motion of the molecule about the centre of symmetry.[13,*]

* The programme has been modified by cand.real. F. Gram at this university.

The **T** and ω tensors were calculated in an orthogonal coordinate system defined by the principal inertial axes of the molecule. The results are given in Tables 8 and 9.

Table 7. 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	$(\bar{u}^2)^{\frac{1}{2}}$ (Å)	B (Å^2)
	0.089	−0.044	−0.014	0.253	5.06
O_1	0.016	0.126	−0.024	0.216	3.71
	0.067	0.027	0.089	0.184	2.68
	0.049	−0.122	0.024	0.239	4.51
O_2	0.082	0.041	−0.023	0.226	4.03
	0.058	0.045	0.087	0.188	2.79
	0.070	−0.080	−0.020	0.231	4.22
C_1	0.045	0.110	−0.021	0.222	3.90
	0.075	0.009	0.088	0.181	2.58
	0.060	0.053	−0.043	0.281	6.21
C_2	0.032	−0.125	−0.013	0.226	4.03
	0.089	0.009	0.081	0.192	2.92
	0.085	−0.014	−0.026	0.289	6.61
C_3	0.024	−0.121	0.042	0.233	4.30
	0.069	0.060	0.079	0.209	3.45
	0.051	0.046	0.072	0.279	6.15
C_4	0.072	−0.105	−0.053	0.259	5.29
	0.070	0.074	0.027	0.191	2.88
	0.053	0.061	−0.047	0.289	6.58
C_5	0.049	−0.118	−0.003	0.259	5.29
	0.086	0.030	0.080	0.186	2.73
	0.072	0.097	0.000	0.276	6.01
C_6	0.052	−0.090	−0.032	0.249	4.89
	0.068	−0.034	0.087	0.197	3.07
	0.072	0.020	−0.039	0.259	5.30
C_7	0.025	0.123	0.039	0.245	4.75
	0.082	−0.054	0.075	0.207	3.39
	0.046	0.088	−0.039	0.257	5.20
C_8	0.059	−0.101	−0.015	0.225	3.99
	0.083	0.023	0.083	0.193	2.95

Table 8. Components of atomic vibration tensors $\mathbf{U}_e$ ($\times 10^4$ Å^2) referred to crystal axes; as calculated from the thermal parameters arrived at by least squares refinement (EXP.), their estimated standard deviations (E.S.D.), and as calculated from rigid-body parameters (R.B.).

		U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
	EXP.	637	481	426	−64	117	−18
O_1	E.S.D.	6	11	9	7	7	7
	R.B.	617	505	474	−32	143	14
	EXP.	576	542	454	−55	168	−68
O_2	E.S.D.	9	11	8	7	7	7
	R.B.	573	559	463	−14	158	−20
	EXP.	523	507	414	−24	109	− 5
C_1	E.S.D.	12	15	11	9	9	9
	R.B.	560	484	465	3	137	− 8
	EXP.	600	551	556	55	50	−63
C_2	E.S.D.	13	16	13	11	10	11
	R.B.	640	509	540	22	109	−55
	EXP.	808	527	588	−57	127	−34
C_3	E.S.D.	17	17	14	12	12	11
	R.B.	760	534	619	−75	103	−51
	EXP.	689	593	628	−93	119	−162
C_4	E.S.D.	15	17	15	12	12	12
	R.B.	718	614	627	−58	97	−120
	EXP.	620	686	563	−22	26	−108
C_5	E.S.D.	14	19	13	12	11	12
	R.B.	626	686	561	−17	90	−109
	EXP.	713	676	500	108	147	59
C_6	E.S.D.	15	18	13	13	11	11
	R.B.	671	660	480	14	89	−38
	EXP.	646	576	604	62	151	55
C_7	E.S.D.	14	17	14	11	11	12
	R.B.	652	581	500	49	71	−11
	EXP.	551	565	511	26	111	−67
C_8	E.S.D.	12	16	12	10	10	10
	R.B.	553	560	498	18	123	−39

Table 9. Principal moments of inertia of the molecule (a.m.u. Å^2), unit vectors along principal axes, L, M, N, defining the molecular coordinate system, and the rigid-body tensors referred to this coordinate system.

Axis	Moment	x	y	z
L	536.9	0.0764	0.0632	0.0740
M	2441.2	−0.0333	0.1203	−0.0423
N	3119.2	−0.0747	0.0111	0.0374

$$\mathbf{T} = \begin{pmatrix} 454 & -18 & 31 \\ & 462 & -5 \\ & & 617 \end{pmatrix} \times 10^{-4} \text{ Å}^2$$

$$\omega = \begin{pmatrix} 66 & 6 & -3 \\ & 10 & 3 \\ & & 10 \end{pmatrix} \times 10^{-4} \text{ rad}^2$$

Table 9. Continued.

	Eigenvalues	r.m.s. values	Direction cosines L	M	N
T	0.0622 Å²	0.249 Å	−0.1865	0.0506	−0.9811
	0.0472 »	0.217 »	−0.5388	0.8297	0.1453
	0.0437 »	0.209 »	−0.8214	−0.5558	0.1274
ω	0.0067 rad²	4.69°	0.9941	0.1001	−0.0414
	0.0012 »	2.05°	0.0335	−0.6483	−0.7606
	0.0006 »	1.45°	0.1030	−0.7547	0.6478

Table 10. Corrections in fractional atomic coordinates due to rigid-body motion compared with the estimated standard deviations of the coordinates. (The σ's and Δ's have been multiplied by 10^4).

	Δx	σx	Δy	σy	Δz	σz
O_1	1	2	6	2	−2	1
O_2	−3	2	−1	2	2	1
C_1	1	2	3	3	0	2
C_2	−1	2	8	4	0	2
C_3	2	3	12	4	−1	2
C_4	3	3	10	4	1	2
C_5	5	2	3	4	3	2
C_6	3	3	−3	4	5	2
C_7	5	3	−6	4	4	2
C_8	5	2	−2	3	1	2
H_2	−3	20	7	20	3	20
H_2'	−3	20	12	30	−1	20
H_3	0	20	17	20	−2	20
H_3'	5	20	10	20	−2	20
H_4	0	30	10	30	3	20
H_4'	4	20	13	20	0	10
H_5	6	20	3	30	4	20
H_5'	8	20	1	30	2	20
H_6	0	20	0	30	5	20
H_6'	4	20	−6	20	7	20
H_7	3	20	−9	30	5	20
H_7'	7	20	−9	20	4	20
H_8	6	20	−5	20	1	10
H_8'	7	20	1	30	0	20

The good agreement between atomic vibration tensor components derived from the least squares refinement, and those calculated from the rigid-body parameters (Table 8), supports the assumption of regarding the molecule as an oscillating rigid body. While the translational motion is almost isotropic (Table 9), the ω tensor implies anisotropic angular oscillation. The largest r.m.s. amplitude is obtained about an axis approximately parallel to the molecular L-axis, which is the principal axis corresponding to the smallest moment of inertia.

The corrections in fractional coordinates for librational effects may be found in Table 10.

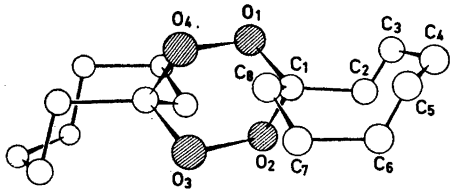


Fig. 1. Schematical drawing of the molecule.

DISCUSSION

Referring to Fig. 1, the interatomic distances and angles, before and after correcting for librational effects, are listed in Table 11. Standard deviations in bond lengths and angles were calculated from the standard deviations given in Tables 2 and 4 without taking into account the E.S.D.'s of the cell parameters.

Table 11. Interatomic distances and angles before and after correcting for librational effects, and their estimated standard deviations (Fig. 1).

Bond	Uncorr. (Å)	Corr. (Å)	σ (Å)
O_2-O_3	1.470	1.474	0.002
O_1-C_1	1.442	1.446	0.002
O_2-C_1	1.436	1.441	0.002
C_1-C_2	1.513	1.517	0.003
C_2-C_3	1.541	1.545	0.003
C_3-C_4	1.537	1.539	0.003
C_4-C_5	1.522	1.528	0.004
C_5-C_6	1.530	1.535	0.003
C_6-C_7	1.518	1.521	0.003
C_7-C_8	1.531	1.536	0.003
C_8-C_1	1.524	1.529	0.002
C_2-H_2		1.07	0.02
C_2-H_2'		1.01	0.02
C_3-H_3		1.04	0.02
C_3-H_3'		1.13	0.02
C_4-H_4		1.02	0.01
C_4-H_4'		1.01	0.02
C_5-H_5		1.00	0.01
C_5-H_5'		1.12	0.02
C_6-H_6		1.02	0.01
C_6-H_6'		1.01	0.01
C_7-H_7		0.96	0.02
C_7-H_7'		1.02	0.01
C_8-H_8		1.01	0.02
C_8-H_8'		1.09	0.02
Angle	**Uncorr. (°)**	**Corr. (°)**	**σ (°)**
$O_3-O_2-C_1$	109.1	108.9	0.1
$O_4-O_1-C_1$	107.5	107.4	0.1
$O_1-C_1-O_2$	107.1	107.3	0.1
$O_1-C_1-C_2$	103.6	103.5	0.2
$O_2-C_1-C_2$	103.7	103.7	0.1
$O_1-C_1-C_8$	110.7	110.7	0.1

Angle	Uncorr. (°)	Corr. (°)	σ (°)
$O_2-C_1-C_8$	112.7	112.6	0.2
$C_8-C_1-C_2$	118.0	118.1	0.2
$C_1-C_2-C_3$	116.7	116.7	0.1
$C_2-C_3-C_4$	115.5	115.3	0.2
$C_3-C_4-C_5$	118.7	118.8	0.2
$C_4-C_5-C_6$	115.8	115.8	0.1
$C_5-C_6-C_7$	114.8	114.8	0.2
$C_6-C_7-C_8$	117.4	117.3	0.2
$C_7-C_8-C_1$	115.2	115.2	0.1

Angle	Corr. (°)	σ (°)
$C_1-C_2-H_2$	106	1
$C_1-C_2-H_2'$	106	1
$H_2-C_2-H_2'$	111	1
$H_2-C_2-C_3$	111	1
$H_2'-C_2-C_3$	106	1
$C_2-C_3-H_3$	105	1
$C_2-C_3-H_3'$	110	1
$H_3-C_3-H_3'$	115	1
$H_3-C_3-C_4$	106	1
$H_3'-C_3-C_4$	105	1
$C_3-C_4-H_4$	108	1
$C_3-C_4-H_4'$	109	1
$H_4-C_4-H_4'$	107	1
$H_4-C_4-C_5$	106	1
$H_4'-C_4-C_5$	108	1
$C_4-C_5-H_5$	104	1
$C_4-C_5-H_5'$	110	1
$H_5-C_5-H_5'$	104	1
$H_5-C_5-C_6$	114	1
$H_5'-C_5-C_6$	108	1
$C_5-C_6-H_6$	108	1
$C_5-C_6-H_6'$	111	1
$H_6-C_6-H_6'$	113	1
$H_6-C_6-C_7$	108	1
$H_6'-C_6-C_7$	102	1
$C_6-C_7-H_7$	113	1
$C_6-C_7-H_7'$	110	1
$H_7-C_7-H_7'$	100	1
$H_7-C_7-C_8$	108	1
$H_7'-C_7-C_8$	106	1
$C_7-C_8-H_8$	109	1
$C_7-C_8-H_8'$	111	1
$H_8-C_8-H_8'$	110	1
$H_8-C_8-C_1$	110	1
$H_8'-C_8-C_1$	103	1

The O—O distance and the C—O distances agree closely with those of dimeric cyclohexanone peroxide [2] and dimeric cycloheptanone peroxide.[3] The angle $O_3-O_2-C_1$ (108.9°) is *possibly significantly* [14] larger than the corresponding angle in the cyclohexanone (107.5°) and cycloheptanone peroxide (107.6°), while the other two angles of the tetraoxacyclohexane ring are equal within probable limits of error.

The asymmetric environment of the spiro carbon atom, may, as for the peroxides mentioned above, roughly be described as a "rotation" of the

cyclooctylidene ring (*ca.* 4°) about an axis through C_1 approximately normal to the plane defined by C_1, C_2, and C_8. Following the argument given for dimeric cyclohexanone peroxide, it appears probable that the distortion is caused by *intra*molecular hydrogen-oxygen repulsions.

The preliminary results of the present investigation [15] showed that the conformation of the cyclooctylidene ring corresponds to what Hendrickson [16] for cyclooctane has called the "boat-chair". It has since been reported that this conformation is preferred also in the heterocyclic compound 5-methyl-1-thia-5-azacyclooctane-1-oxide perchlorate [17] and in cyclooctane-1,2-*trans*-dicarboxylic acid.[18] Electron diffraction investigations of gaseous cyclooctane show,[19] however, that the free molecule cannot be described with the assumption of any single conformation.

From Table 11 it may be seen that the C—C—C angles are greater than in paraffin-chain molecules. Comparing with the peroxides of cyclohexanone and cycloheptanone, the average values of the C—C—C angles are:

dimeric cyclohexanone peroxide	111.3°
dimeric cycloheptanone peroxide	115.3°
dimeric cyclooctanone peroxide	116.5°

116.5° agrees with the value reported by Dunitz for cyclooctane-1,2-*trans*-dicarboxylic acid.[18]

Some of the *intra*molecular contacts of hydrogen atoms not bonded to the same carbon atom are very short. The distance $H_2—H_6$ is 2.04 Å and $H_3'—H_8'$ is 1.93 Å, with estimated standard deviations of 0.02 Å. Four other H—H contacts are ranging from 2.15 Å to 2.24 Å. The shortest *inter*molecular H—H distance is 1.38 Å (calculated with uncorrected coordinates).

The following fit was obtained for two least squares planes:

Atoms defining the plane	Deviations (Å)
C_1	−0.040
C_2	0.039
C_6	0.033
C_7	−0.032
C_2	0.033
C_3	−0.011
C_5	0.011
C_6	−0.013

The dihedral angles of the cyclooctylidene ring are given in Table 12.

Table 12. Dihedral angles of the cyclooctylidene ring.

Angles	ω [a]	(°)
$C_7-C_8-C_1-C_2$	ω_1	69.7
$C_8-C_1-C_2-C_3$	ω_2	36.5
$C_1-C_2-C_3-C_4$	$-\omega_3$	−98.4
$C_2-C_3-C_4-C_5$	ω_4	66.1
$C_3-C_4-C_5-C_6$	$-\omega_4$	−64.0
$C_4-C_5-C_6-C_7$	ω_3	101.3
$C_5-C_6-C_7-C_8$	$-\omega_2$	−49.0
$C_6-C_7-C_8-C_1$	$-\omega_1$	−61.1

[a] Notation of Hendrickson.

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