

## Crystal Structure of 3,6-Diphenyl-1,2,4,5-tetraoxacyclohexane

P. GROTH

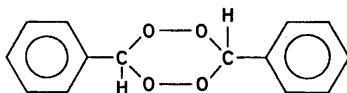
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The crystals belong to the monoclinic system with space group  $P2_1/c$ . The unit cell, containing two molecules has the parameters:

$$\begin{array}{ll} a = 6.09 \text{ \AA} & \\ b = 7.85 \text{ \AA} & \beta = 93.9^\circ \\ c = 12.37 \text{ \AA} & \end{array}$$

The phase problem has been solved by a computer procedure based on direct methods. 350 intensities were measured and the  $R$ -value arrived at by full-matrix least squares refinement was 9.0 %. The O—O distance is found to be 1.48 Å and the two C—O bond lengths 1.41 Å and 1.43 Å, respectively. The benzene ring plane is approximately normal to the plane defined by the four oxygen atoms of the tetraoxacyclohexane ring.

3,6-Diphenyl-1,2,4,5-tetraoxacyclohexane has been synthesized by T. Ledaal at this university.<sup>1</sup> The present structure analysis was carried



out as part of a study of the geometry of cyclic organic peroxides,<sup>2-4</sup> and in order to establish the orientation of the benzene ring planes with respect to the plane defined by the oxygen atoms of the tetraoxacyclohexane ring.

### CRYSTAL DATA

3,6-Diphenyl-1,2,4,5-tetraoxacyclohexane belongs to the monoclinic system, and the systematic absences lead to the space group  $P2_1/c$ . The cell dimensions determined from  $0kl$ -Weissenberg and  $h0l$ -precession photographs are:

$$\begin{array}{ll} a = 6.09 \text{ \AA} & \\ b = 7.85 \text{ \AA} & \\ c = 12.37 \text{ \AA} & \\ \beta = 93.9^\circ & \end{array}$$

With two molecules in the unit cell the calculated density,  $\rho_c = 1.38 \text{ g/cm}^3$ , corresponds closely to the density measured by flotation,  $\rho_o = 1.36 \text{ g/cm}^3$ .

Several crystals obtained from benzene and acetonitrile, respectively, were examined, and all turned out to be twins with common (001)-plane and the angle between the  $c$ -axes equal to  $172.2^\circ$  (Fig. 1).

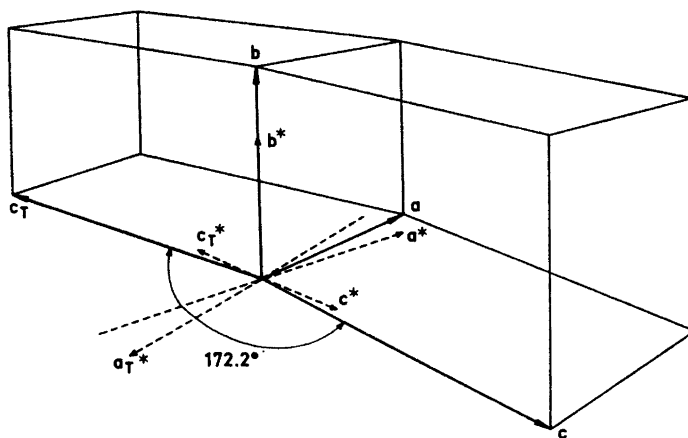


Fig. 1. Schematical drawing showing two unit cells with the twin plane (001).

Thus all  $0kl$ -reflections overlap completely, and intensity data of this projection was obtained by photometric measurements of integrated Weissenberg diagrams ( $\text{CuK}\alpha$ -radiation).

With the exception of the  $0kl$ -reflections, which overlap completely, all  $h0l$ -reflections are resolved. Intensity data of this projection was obtained by visual estimation of the reflections, from each of the twins separately, on precession diagrams ( $\text{MoK}\alpha$ -radiation).

The corresponding intensities from the twins were added in order to obtain consistency with the  $00l$ -reflections.

The reflections on  $1kl$ - and  $2kl$ -Weissenberg diagrams taken with  $\text{CuK}\alpha$ -radiation (not integrated) are resolved, and intensities were estimated visually. A total number of 350 intensities were measured.

#### DETERMINATION OF THE STRUCTURE

The phase problem was solved by a sign determining computer procedure based on direct methods<sup>5-7</sup> which was applied to the  $0kl$ -projection. The  $|U|$ -distribution is given in Table 1. The origin was specified by giving positive signs to  $U(021) = 0.53$  and  $U(055) = 0.31$ , and the programmed systematic application of Harker-Kasper inequalities gave  $S(040) = -$ , and  $S(042) = +$ .

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

Range of $ U $	Number of reflections
0.00–0.01	3
0.01–0.02	5
0.02–0.03	24
0.03–0.04	1
0.04–0.05	4
0.05–0.10	33
0.10–0.15	15
0.15–0.20	13
0.20–0.25	14
0.25–0.30	4
0.30–0.35	2
0.35–0.40	1
0.40–0.45	2
0.45–0.50	0
0.50–1.00	1

15 additional large and moderate unitary structure factors were selected for sign permutation. Among the 19 signs thus involved, 35 relationships could be found for the sums:

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

33 "small"  $U_{\mathbf{k}}$ 's ( $|U_{\mathbf{k}}| \leq 0.04$ ) gave 77 products in the zero-check expression:

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

$\chi_c$  given by

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

where  $N$  is the number of equal atoms in the cell, was calculated, and found to be  $\chi_c = 0.711$ .

Among the  $2^{15}$  sign combinations tested, three satisfied  $\chi > 0.8 \cdot \chi_c$ .

On the basis of these three sets, in turn 42 additional signs were derived using the relationship

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

in a somewhat special way:

The signs were accepted in the order of decreasing probabilities given by:

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

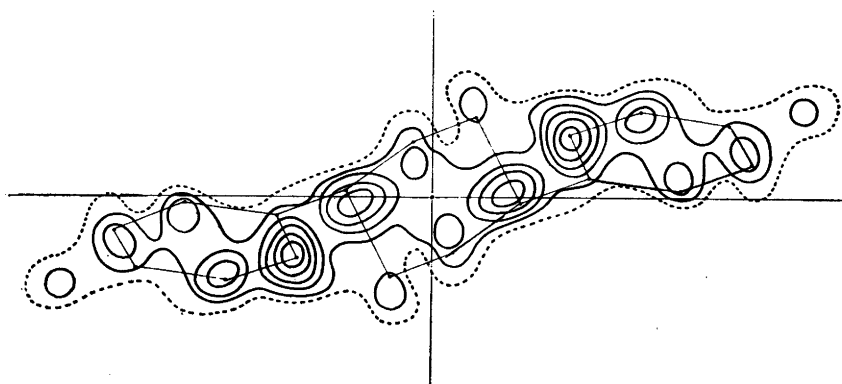
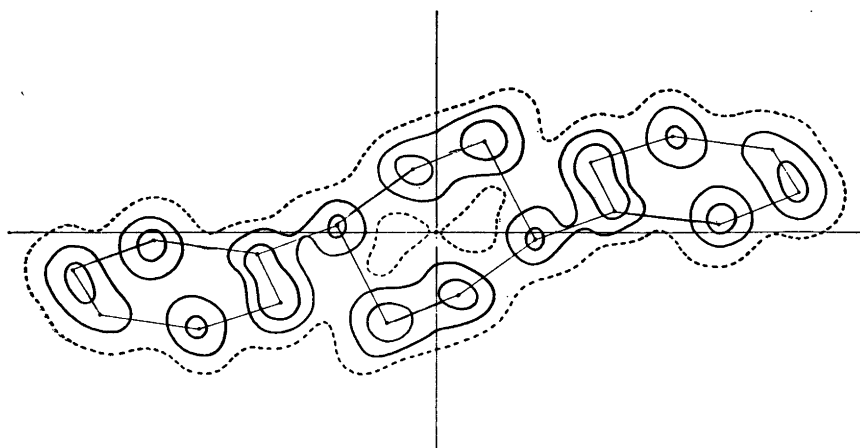
and redetermined until no further signshifts occurred. The  $\chi$ - and  $\psi_0$ -values for the total set, in this case containing 61 signs, were calculated after the last cycle of iteration.

Table 2 gives the results of this process, and Fig. 2 shows the Fourier map calculated with the 61 signs contained in set No. 2 corresponding to the smallest  $\psi_0$  and the medium  $\chi$ .

The parameters derived from Fig. 2 gave  $R_{0kl} = 47.6\%$ . Using a programme based on the "minimum residual method",<sup>8</sup> the  $R$ -value was reduced to 13.0%. The final Fourier map of this projection is reproduced in Fig. 3.

Table 2. Results of the sign determination of  $0kl$ -projection.

Step 1 (permutation)	$\chi$	$\chi_0$
Set No. 1	0.585	3.577
2	0.579	2.863
3	0.579	2.668
Step 2 (application of (4))		
Basic set No. 1	2.268	9.824
2	2.337	7.775
3	2.462	9.053

Fig. 2. Fourier projection along the  $a$ -axis based on the 61 "most probable" signs.Fig. 3. Final electron-density map projecting along the  $a$ -axis.

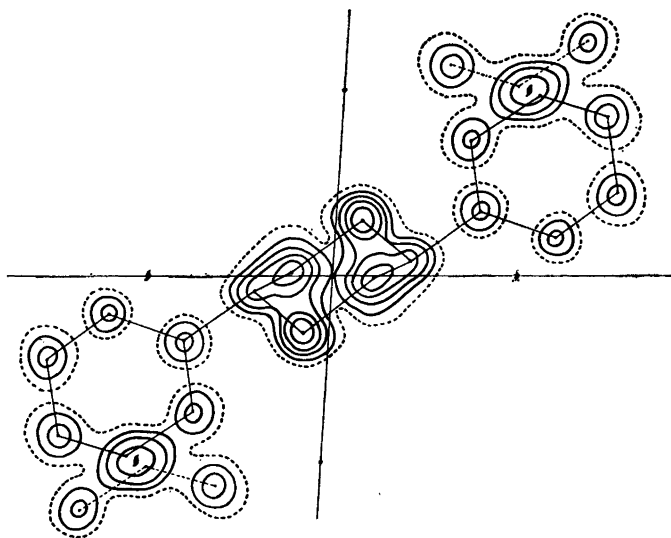


Fig. 4. Final Fourier projection along the *b*-axis.

Comparison of determined signs with those corresponding to  $R_{ohl} = 13.0\%$  revealed as many as 15 discrepancies. The reason for this bad result is probably that  $S(042)$  had been obtained incorrectly from Harker-Kasper inequalities.

The *x*-parameters were determined from a model of the molecule and the final electron density map of the  $h0l$ -projection is shown in Fig. 4.

#### REFINEMENT OF THE STRUCTURE

Starting with the parameters obtained by the two-dimensional analysis, a three-dimensional full-matrix least squares refinement was carried out using a programme written by Gantzel, Sparks and Trueblood<sup>9</sup> (revised for UNIVAC 1107 by cand.real Chr. Rømming at this university). The weighting scheme No. 1:

$$\begin{array}{ll} \text{for } F_o \leq \text{FB}, & W = A1(F_o)^{B1} \\ \text{for } F_o > \text{FB}, & W = A2(F_o)^{B2} \end{array}$$

was adapted by taking  $A1 = 10.0$ ,  $A2 = 14.0$ ,  $B1 = 0.0$ ,  $B2 = -0.5$ , and  $\text{FB} = 2.0$ . The atomic form factors used were those of Hanson, Herman, Lea and Skillman.<sup>10</sup>

After 3 cycles of isotropic refinement, anisotropic thermal vibrational parameters were introduced for the oxygen and carbon atoms. The hydrogen atom positions were calculated assuming  $sp^3$ , respectively  $sp^2$  bonding with C—H bond-lengths equal to 1.05 Å. These parameters were not refined.

The *R*-value arrived at for the 350 observed reflections was 9.0 %.

The fractional atomic coordinates and the thermal vibration parameters

Table 3. Fractional atomic coordinates for oxygen and carbon atoms.<sup>a</sup> (Estimated standard deviations in parentheses).

O <sub>1</sub>	1.0143 (0.0014)	0.3483 (0.0008)	0.0530 (0.0005)
O <sub>2</sub>	0.8415 (0.0015)	0.6047 (0.0008)	0.0343 (0.0005)
C <sub>1</sub>	0.8239 (0.0024)	0.4532 (0.0013)	0.1924 (0.0007)
C <sub>2</sub>	0.6297 (0.0026)	0.3754 (0.0015)	0.1713 (0.0008)
C <sub>3</sub>	0.4945 (0.0031)	0.3352 (0.0015)	0.2543 (0.0010)
C <sub>4</sub>	0.5673 (0.0028)	0.3682 (0.0013)	0.3597 (0.0009)
C <sub>5</sub>	0.7664 (0.0027)	0.4485 (0.0018)	0.3835 (0.0009)
C <sub>6</sub>	0.8982 (0.0023)	0.4882 (0.0013)	0.2998 (0.0007)
C <sub>7</sub>	0.9741 (0.0024)	0.5012 (0.0011)	0.1076 (0.0007)

<sup>a</sup> For numbering of atoms see Fig. 5.

Table 4. Anisotropic thermal vibration parameters. (Estimated standard deviations in parentheses).

	B <sub>11</sub>	B <sub>22</sub>	B <sub>33</sub>	B <sub>12</sub>	B <sub>13</sub>	B <sub>23</sub>
O <sub>1</sub>	0.0307 (0.0036)	0.0208 (0.0012)	0.0081 (0.0004)	-0.0001 (0.0042)	0.0032 (0.0023)	0.0005 (0.0011)
O <sub>2</sub>	0.0397 (0.0039)	0.0223 (0.0012)	0.0093 (0.0005)	0.0033 (0.0049)	0.0098 (0.0024)	0.0032 (0.0014)
C <sub>1</sub>	0.0322 (0.0048)	0.0179 (0.0016)	0.0083 (0.0007)	0.0024 (0.0071)	-0.0047 (0.0034)	-0.0014 (0.0017)
C <sub>2</sub>	0.0400 (0.0064)	0.0249 (0.0023)	0.0095 (0.0009)	-0.0117 (0.0079)	0.0003 (0.0041)	-0.0017 (0.0022)
C <sub>3</sub>	0.0325 (0.0039)	0.0279 (0.0023)	0.0112 (0.0008)	-0.0118 (0.0078)	0.0025 (0.0034)	0.0028 (0.0024)
C <sub>4</sub>	0.0410 (0.0059)	0.0244 (0.0021)	0.0108 (0.0009)	-0.0105 (0.0088)	0.0063 (0.0040)	0.0077 (0.0021)
C <sub>5</sub>	0.0344 (0.0056)	0.0292 (0.0024)	0.0095 (0.0008)	0.0123 (0.0092)	-0.0001 (0.0039)	0.0034 (0.0023)
C <sub>6</sub>	0.0350 (0.0053)	0.0254 (0.0020)	0.0080 (0.0007)	-0.0004 (0.0069)	0.0050 (0.0036)	0.0027 (0.0019)
C <sub>7</sub>	0.0424 (0.0065)	0.0209 (0.0016)	0.0075 (0.0006)	0.0127 (0.0067)	0.0038 (0.0039)	0.0034 (0.0018)

Table 5. Observed and calculated structure factors.

<i>h</i>	<i>k</i>	<i>l</i>	<i>F<sub>o</sub></i>	<i>F<sub>c</sub></i>	<i>h</i>	<i>k</i>	<i>l</i>	<i>F<sub>o</sub></i>	<i>F<sub>c</sub></i>	<i>h</i>	<i>k</i>	<i>l</i>	<i>F<sub>o</sub></i>	<i>F<sub>c</sub></i>	<i>h</i>	<i>k</i>	<i>l</i>	<i>F<sub>o</sub></i>	<i>F<sub>c</sub></i>
0	0	2	10.9	-8.6	1	0	4	2.6	3.3	1	5	-4	6.2	-7.2	2	4	-3	6.2	6.1
0	0	4	7.1	-3.9	1	0	6	17.5	16.7	1	5	-3	1.8	-2.0	2	4	-1	14.3	16.0
0	0	8	5.9	-6.5	1	0	8	6.0	-5.8	1	5	-2	6.2	-7.1	2	4	0	7.2	-7.2
0	0	10	1.9	-1.5	1	0	10	3.0	2.0	1	5	0	8.0	8.4	2	4	1	11.3	-11.6
0	0	12	6.8	-6.1	1	0	12	3.9	-4.5	1	5	1	1.7	1.9	2	4	4	5.8	-6.0
0	0	14	4.7	-4.3	1	0	14	2.5	-2.6	1	5	2	4.6	-4.9	2	4	5	3.5	-3.8
0	1	1	34.5	-34.8	1	1	1	11.7	10.7	1	5	3	6.3	6.9	2	4	7	3.8	-3.9
0	1	3	18.7	-22.8	1	1	10	2.8	2.2	1	5	4	6.0	6.9	2	5	-6	5.4	-4.7
0	1	4	2.5	2.8	1	1	9	1.3	-0.8	1	5	5	6.5	7.1	2	5	-5	3.7	-3.4
0	1	5	6.3	-7.1	1	1	7	9.1	9.2	1	5	7	2.5	2.4	2	5	-4	7.3	-6.9
0	1	6	10.6	-11.1	1	1	6	8.3	7.1	1	5	9	2.8	2.6	2	5	-3	3.5	-3.9
0	1	7	6.2	5.9	1	1	5	15.6	12.4	1	5	10	3.0	3.0	2	5	-2	4.3	-4.4
0	1	9	7.7	9.1	1	1	4	6.8	-8.4	1	5	11	1.2	-2.1	2	5	0	15.4	14.6
0	1	10	7.8	8.1	1	1	2	6.5	-6.5	1	6	-8	1.4	-1.0	2	5	1	13.5	-14.0
0	1	11	2.5	-1.7	1	1	-1	15.0	15.1	1	6	-6	3.2	3.1	2	5	2	3.5	-3.6
0	1	13	2.2	2.4	1	1	0	9.5	8.7	1	6	-4	3.5	-4.7	2	5	3	3.6	1.8
0	1	15	2.6	2.3	1	1	1	91.5	-90.7	1	6	-3	1.4	-1.1	2	5	4	9.3	9.6
0	2	0	52.0	-48.2	1	1	2	37.4	-34.2	1	6	-2	5.5	5.5	2	5	6	10.2	11.2
0	2	1	62.4	63.7	1	1	1	26.8	-26.0	1	6	-1	7.7	-8.3	2	6	-5	5.5	-3.5
0	2	2	8.1	-10.2	1	1	4	11.4	-27.5	1	6	0	5.0	4.8	2	6	-1	3.8	2.4
0	2	3	2.0	2.6	1	1	5	22.9	19.7	1	6	1	3.6	4.4	2	6	0	13.0	13.0
0	2	4	15.8	-18.5	1	1	6	2.3	2.4	1	6	2	3.1	2.4	2	6	1	9.3	-9.6
0	2	5	7.2	-8.1	1	1	7	10.2	-9.8	1	6	3	3.1	-3.2	2	6	2	7.7	-7.5
0	2	6	6.0	6.1	1	1	8	1.3	-1.6	1	6	4	3.2	-4.0	2	6	3	7.8	7.7
0	2	7	2.7	5.9	1	1	10	2.0	1.9	1	6	5	5.6	-6.6	3	0	-6	4.1	4.0
0	2	8	1.8	-1.7	1	1	12	2.2	-2.2	1	6	6	4.3	-5.6	3	0	-4	20.6	-24.8
0	2	9	2.2	-1.6	1	1	13	1.3	1.5	1	6	7	2.9	3.0	3	0	-2	7.1	8.0
0	2	10	11.5	12.0	1	1	11	6.8	-5.9	1	6	8	1.4	-1.4	3	0	2	13.2	-12.3
0	2	11	4.4	-4.6	1	1	10	7.4	8.2	1	6	10	1.2	1.4	3	0	2	12.7	15.8
0	2	12	4.4	-4.3	1	1	9	7.8	9.0	1	7	-6	2.6	3.8	3	0	4	7.0	-7.1
0	2	13	2.2	-1.5	1	1	8	1.3	-1.1	1	7	-5	2.5	3.2	3	0	6	9.0	-10.4
0	2	14	2.1	-1.5	1	1	7	1.2	-0.8	1	7	-4	2.5	2.6	3	0	8	3.5	-3.5
0	2	15	1.9	-1.7	1	1	6	10.9	-8.7	1	7	-3	1.4	-1.4	3	0	8	4.0	2.6
0	3	1	12.6	20.5	1	1	5	18.2	-16.9	1	7	-2	2.0	2.0	4	0	-10	3.6	-3.5
0	3	2	7.9	7.0	1	1	4	9.9	-11.1	1	7	-1	3.8	-3.8	4	0	-8	6.0	7.6
0	3	3	11.0	13.9	1	1	3	9.0	8.0	1	7	0	4.1	6.0	4	0	-4	2.9	-2.8
0	3	4	14.6	15.8	1	1	2	16.2	-17.4	1	7	1	4.6	-5.6	4	0	-2	4.5	4.7
0	3	5	10.9	11.6	1	1	1	48.7	48.1	1	7	2	2.0	-7.7	4	0	2	5.3	5.5
0	3	6	5.4	-5.3	1	1	0	3.9	-3.5	1	7	3	1.4	-1.4	4	0	6	22.7	-24.6
0	3	7	7.4	-8.7	1	1	0	43.8	41.9	1	7	4	4.2	5.3	4	0	8	5.1	5.1
0	3	8	3.9	2.5	1	1	2	25.2	-25.4	1	7	6	2.4	-2.9	5	0	-8	3.6	3.4
0	3	9	8.4	-8.3	1	1	3	30.5	28.3	1	7	8	2.1	-2.4	5	0	-6	7.0	5.5
0	3	10	9.2	8.9	1	1	4	5.6	-6.3	2	0	-10	11.2	-12.1	5	0	-4	6.5	-6.1
0	4	0	18.8	-19.1	1	2	5	3.5	3.7	2	0	-8	6.8	-7.1	5	0	-2	14.7	17.9
0	4	2	13.8	-14.2	1	2	6	4.5	3.8	2	0	-6	16.1	-15.1	5	0	0	7.0	8.6
0	4	3	3.5	-3.5	1	2	7	7.7	8.2	2	0	-4	11.2	-12.6	5	0	4	4.4	-5.1
0	4	4	6.3	-6.2	1	2	8	3.9	4.8	2	0	-2	5.3	-5.6	5	0	8	5.5	-5.2
0	4	5	14.6	-14.8	1	2	9	4.4	-4.4	2	0	0	30.0	33.7	6	0	-4	10.3	10.6
0	4	6	4.2	-3.7	1	2	10	4.4	4.1	2	0	2	3.7	2.6	6	0	0	5.4	5.7
0	4	7	2.7	-2.8	1	2	11	3.8	-3.9	2	0	4	13.6	15.6	6	0	2	4.6	3.2
0	4	8	2.4	2.2	1	2	12	1.4	-1.3	2	0	6	11.2	-12.6	7	0	2	6.0	5.3
0	4	9	6.6	-6.5	1	3	11	2.5	-2.6	2	0	8	3.3	4.1	7	0	8	4.2	-2.1
0	4	10	12.1	11.6	1	3	10	2.5	3.3	2	0	10	2.4	-1.2	8	0	0	3.9	3.6
0	4	11	4.6	4.1	1	3	9	10.4	-11.2	2	1	-11	7.8	7.9					
0	5	1	4.5	4.6	1	3	8	1.9	-2.4	2	1	-10	3.6	4.2					
0	5	2	5.2	4.7	1	3	7	8.3	-9.0	2	1	-9	5.5	-6.4					
0	5	3	3.5	-3.5	1	3	6	8.6	9.2	2	1	-8	4.8	4.8					
0	5	4	9.2	10.1	1	3	5	18.3	-15.7	2	1	-7	7.0	7.1					
0	5	5	12.3	12.3	1	3	4	5.5	4.9	2	1	-6	2.8	1.6					
0	5	7	2.9	-2.6	1	3	2	20.9	-18.1	2	1	-5	38.6	37.5					
0	5	9	4.4	-4.1	1	3	1	27.4	26.3	2	1	-4	27.6	22.6					
0	6	0	11.0	10.6	1	3	0	14.8	15.2	2	1	-3	7.9	-7.5					
0	6	2	3.5	-3.5	1	3	1	7.5	8.1	2	1	-2	4.9	-5.9					
0	6	3	2.5	-2.0	1	3	2	20.0	-21.8	2	1	0	23.5	-25.6					
0	6	4	1.5	-1.0	1	3	3	11.7	11.9	2	1	1	30.4	-30.9					
0	6	5	3.5	3.5	1	3	4	13.3	-12.4	2	1	2	20.2	-17.8					
0	6	7	1.4	-1.5	1	3	5	7.1	7.0	2	1	4	16.3	-15.9					
0	6	9	1.8	-2.1	1	3	6	5.3	4.9	2	1	5	10.4	10.6					
0	6	11	4.4	3.2	1	3	7	2.2	-2.4	2	1	6	13.5	-14.0					
0	7	1	5.3	-4.0	1	3	8	2.4	1.8	2	1	7	4.6	-5.3					
0	7	2	2.0	-1.7	1	3	10	3.2	3.9	2	2	-9	10.6	-11.2					
0	7	3	2.9	-2.3	1	3	11	8.1	-8.8	2	2	-7	8.0	-7.3					
0	7	4	3.3	-2.7	1	4	-12	3.1	2.4	2	2	-5	12.7	-12.1					
0	7	5	5.7	5.2	1	4	-11	1.4	-1.4	2	2	-4	13.5	-13.3					
0	7	6	4.9	-3.8	1	4	-10	5.3	5.8	2	2	-3	3.2	2.7					
0	7	10	1.7	-1.4	1	4	-9	3.5	3.7	2	2	-2	2.1	-2.8					
0	8	0	5.6	4.4	1	4	-8	6.7	8.1	2	2	0	13.6	-14.5					
0	8	1	5.9	4.9	1	4	-7	7.0	7.8	2	2	1	31.4	31.3					
0	8	2	2.3	1.5	1	4	-6	9.4	10.6	2	2	3	17.8	17.9					
0	8	3	1.9	1.4	1	4	-5	2.4	-2.2	2	2	5	24.8	24.3					
0	8	4	2.4	-1.9	1	4	-4	13.2	-11.6	2	3	-10	6.8	6.0					
0	8	5	4.2	3.2	1	4	-3	17.8	18.4	2	3	-9	11.8	-12.4					
0	8	7	2.8	2.6	1	4	-2	18.4	-16.0	2	3	-7	5.0	-4.7					
0	9	1	1.7	-1.0	1	4	-1	1.5	-1.8	2	3	-4	8.5	10.5					
0	9	2	1.3	-0.5	1	4	0	17.3	17.3	2	3	-3	7.6	8.8					
0	9	3	1.1	-1.7	1	4	2	25.1	-27.6	2	3	-2	6.7	-6.8					
0	9	4	1.1	-0.8	1	4	3	7.8	-8.1	2	3	-							

obtained are given in Tables 3 and 4; the expression for the anisotropic vibration being:

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

A comparison between calculated and observed structure factors is presented in Table 5.

#### THERMAL MOTIONS

The principal axes of the thermal vibration ellipsoids for the oxygen atoms and the carbon atoms were calculated from the temperature parameters in Table 4. 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 6.

Table 6. 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 <sup>2</sup> )
O <sub>1</sub>	0.013	0.118	0.029	0.256	5.16
	-0.061	0.046	-0.071	0.248	4.93
	-0.152	-0.008	0.025	0.238	4.47
O <sub>2</sub>	0.111	0.049	0.055	0.299	7.05
	-0.081	0.111	-0.003	0.259	5.31
	-0.091	-0.039	0.060	0.240	4.53
C <sub>1</sub>	0.100	0.027	-0.058	0.279	6.13
	0.022	-0.124	-0.013	0.234	4.33
	0.129	0.000	0.055	0.223	3.92
C <sub>2</sub>	-0.111	0.094	0.000	0.301	7.15
	-0.042	-0.036	0.073	0.276	6.00
	0.114	0.078	0.034	0.244	4.72
C <sub>3</sub>	0.048	-0.100	-0.043	0.313	7.72
	0.052	-0.059	0.069	0.288	6.54
	0.148	0.053	-0.004	0.233	4.27
C <sub>4</sub>	0.028	-0.088	-0.056	0.316	7.86
	0.141	-0.032	0.041	0.290	6.63
	0.080	0.087	-0.041	0.227	4.06
C <sub>5</sub>	0.060	0.116	0.018	0.315	7.81
	0.068	0.004	-0.071	0.275	5.96
	0.137	-0.052	0.034	0.234	4.34
C <sub>6</sub>	0.012	0.120	0.027	0.286	6.45
	-0.144	0.028	-0.040	0.261	5.38
	-0.079	-0.033	0.065	0.237	4.43
C <sub>7</sub>	0.134	0.071	0.018	0.300	7.12
	0.079	-0.061	-0.057	0.248	4.84
	0.054	-0.087	0.055	0.226	4.03



A rigid-body analysis of translational and librational motion has not been carried out; partly by reason of the uncomplete set of observed intensities, but also because of the rotational freedom of the phenyl group about the C<sub>7</sub>-C<sub>1</sub> bond.

## DISCUSSION

The interatomic distances and angles, referring to Fig. 5, are listed in Table 7. Standard deviations in bond lengths and angles were calculated from the standard deviations given in Table 3.

Table 7. Interatomic distances and angles, and their estimated standard deviations.

Bond	(Å)	$\sigma$ (Å)
O <sub>2</sub> -O <sub>3</sub>	1.48	0.01
O <sub>1</sub> -C <sub>7</sub>	1.41	0.01
O <sub>2</sub> -C <sub>7</sub>	1.43	0.01
C <sub>1</sub> -C <sub>7</sub>	1.49	0.02
C <sub>1</sub> -C <sub>2</sub>	1.34	0.02
C <sub>2</sub> -C <sub>3</sub>	1.39	0.02
C <sub>3</sub> -C <sub>4</sub>	1.37	0.02
C <sub>4</sub> -C <sub>5</sub>	1.38	0.02
C <sub>5</sub> -C <sub>6</sub>	1.39	0.02
C <sub>6</sub> -C <sub>1</sub>	1.40	0.01
Angle	(°)	$\sigma$ (°)
O <sub>3</sub> -O <sub>2</sub> -C <sub>7</sub>	105.5	0.8
O <sub>4</sub> -O <sub>1</sub> -C <sub>7</sub>	105.5	0.7
O <sub>1</sub> -C <sub>7</sub> -O <sub>2</sub>	106.9	0.7
O <sub>1</sub> -C <sub>7</sub> -C <sub>1</sub>	104.9	0.8
O <sub>2</sub> -C <sub>7</sub> -C <sub>1</sub>	104.1	1.0
C <sub>7</sub> -C <sub>1</sub> -C <sub>2</sub>	123.8	0.9
C <sub>7</sub> -C <sub>1</sub> -C <sub>6</sub>	116.4	1.1
C <sub>1</sub> -C <sub>2</sub> -C <sub>3</sub>	121.1	1.0
C <sub>2</sub> -C <sub>3</sub> -C <sub>4</sub>	119.2	1.5
C <sub>3</sub> -C <sub>4</sub> -C <sub>5</sub>	120.8	1.3
C <sub>4</sub> -C <sub>5</sub> -C <sub>6</sub>	119.2	1.0
C <sub>5</sub> -C <sub>6</sub> -C <sub>1</sub>	119.8	1.2
C <sub>6</sub> -C <sub>1</sub> -C <sub>2</sub>	119.8	1.0

Within the limit of probable errors the results obtained are in agreement with earlier investigations.<sup>2-4</sup>

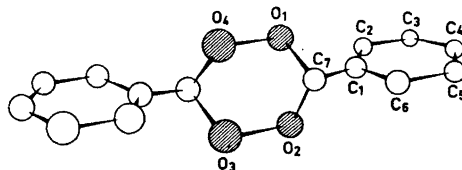


Fig. 5. Schematical drawing of the molecule.

The least squares plane defined by the benzene ring atoms gives the following fit:

Atom	Deviation (Å)
C <sub>1</sub>	0.009
C <sub>2</sub>	-0.011
C <sub>3</sub>	0.013
C <sub>4</sub>	-0.013
C <sub>5</sub>	0.010
C <sub>6</sub>	-0.008

The angle between the plane defined by the oxygen atoms O<sub>1</sub>, O<sub>2</sub>, O<sub>3</sub>, O<sub>4</sub> and the benzene ring least squares plane is 88.2°.

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