

Fig. 6. Packing arrangement of molecules projected on (010).

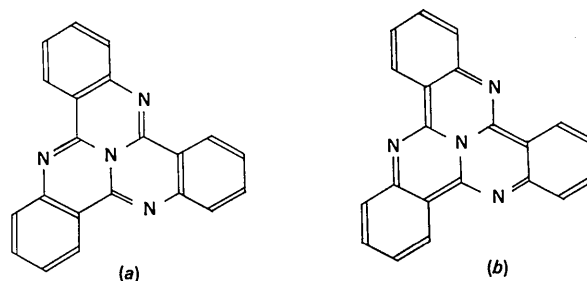


Fig. 7. The two arrangements of double bonds in central rings for covalent Kekulé structures.

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## The Crystal Structure of a Benzocyclopropapyran\*

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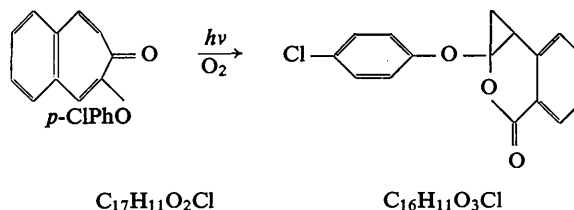
Crystals of 1*a*-(*p*-chlorophenoxy)-1*a*,7*b*-dihydrobenzo[*d*]cyclopropa[*b*]pyran-3(1*H*)one, C<sub>16</sub>H<sub>11</sub>O<sub>3</sub>Cl, belong to the orthorhombic space group *P*2<sub>1</sub>2<sub>1</sub> with four molecules in a unit cell of dimensions *a* = 13.21, *b* = 13.80, and *c* = 7.33, all ± 0.01 Å. The crystal structure was determined by a three-dimensional X-ray analysis of counter data using weighted Patterson superposition techniques and was refined by least-squares methods to a conventional *R* index of 6.9%. The combined benzene and lactone rings are nearly planar and the dihedral angles between this plane and the phenoxy group and cyclopropane ring are 64 and 70°, respectively.

### Introduction

One of the photolysis products found by Stoner (1964) on the irradiation of 2-(*p*-chlorophenoxy)-4,5-benzotropone is 1*a*-(*p*-chlorophenoxy)-1*a*,7*b*-dihydrobenzo[*d*]cyclopropa[*b*]pyran-3(1*H*)one, C<sub>16</sub>H<sub>11</sub>O<sub>3</sub>Cl.

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C<sub>17</sub>H<sub>11</sub>O<sub>2</sub>Cl

C<sub>16</sub>H<sub>11</sub>O<sub>3</sub>Cl

Elemental analysis indicated the loss of one carbon atom and addition of one oxygen atom; this, along with

spectral evidence, suggested that an unusual photochemical rearrangement had taken place. The crystal structure of  $C_{16}H_{11}O_3Cl$  was determined to identify the photolysis product and to characterize its molecular configuration.

### Experimental

Crystals of  $C_{16}H_{11}O_3Cl$  were supplied by Dr O.L. Chapman of this Laboratory. The orthorhombic cell dimensions obtained from an average of values measured on the diffractometer and diffraction photographs recorded using Mo radiation are  $a=13.21 \pm 0.01$ ,  $b=13.80 \pm 0.01$ , and  $c=7.33 \pm 0.01$  Å. The space group as determined from Weissenberg and precession diffraction photographs is  $P2_12_12_1$  with  $Z=4$ . The calculated density is  $1.38 \text{ g.cm}^{-3}$ ; the experimental density was not checked because of the small amount of sample available.

Diffraction intensities were collected on a General Electric XRD-5 diffractometer with Zr-filtered Mo radiation by use of the stationary-crystal stationary-counter technique with a peak count time of 40 seconds and a take-off angle of  $8^\circ$ . Intensity measurements were made with a scintillation counter and a pulse height selector on a crystal fragment of dimensions  $0.30 \times 0.25 \times 0.20$  mm mounted with the  $a$  axis coincident with the  $\varphi$  axis of the diffractometer. A total of 1377 reflections were measured. Background measurements were taken in various regions of reciprocal space so an average background curve could be constructed and used in correcting all of the data.

Four strong reflections were carefully measured along  $2\theta$  to characterize the streaking effect due to noncharacteristic radiation. The assumption was made that the streaking observed on these reflections would be representative of all the data. An average streak curve was constructed so that streaking from reflections  $n_i(hkl)$ , of order  $n_i$ , between the origin of the reciprocal

lattice and some reflection  $n(hkl)$ , of order  $n$ , could be subtracted from the intensity observed for the reflection  $n(hkl)$ . The streak intensity,  $I_s$ , for a given reflection  $n(hkl)$  was calculated by

$$I_s[n(hkl)] = \frac{\sum_{n_i} I[n_i(hkl)] L_p[n(hkl)] \cos \theta[n(hkl)] K(\lambda/\lambda_s)}{L_p[n_i(hkl)] \sin \theta[n(hkl)]}$$

where  $n/2 < n_i < n$ , and  $L_p$  and  $\lambda$  are the Lorentz-polarization factor and wavelength, respectively. The values of  $K$  were obtained from the average streak curve which was conveniently plotted as a function of  $\lambda/\lambda_s$ . This type of correction has been discussed previously by Williams & Rundle (1964).

A correction was applied to the data so that the peak height intensities would correspond to integrated intensities. A correction curve was calculated using the apparent source width, the  $2\theta$  separation of  $K\alpha_1-K\alpha_2$ , and the correction curve given by Alexander & Smith (1962). For molybdenum radiation with the large take-off angle used and small  $2\theta$ 's ( $< 50^\circ$ ), this correction is small.

No absorption correction was applied because the linear absorption coefficient is small ( $2.8 \text{ cm}^{-1}$ ). The weights applied to the structure factors were calculated from the estimated errors in intensities after the method of Williams & Rundle (1964). Reflections for which  $F_o$  was less than  $1.5\sigma(F_o)$  were called unobserved. About 700 reflections remained as observed reflections.

### Structure determination and refinement

The structure was solved by Patterson superposition techniques used in conjunction with the minimum function. The Patterson synthesis was sharpened after the method of Jacobson, Wunderlich & Lipscomb (1961). The coordinates of 19 out of the 20 nonhydrogen atoms were read directly from the weighted

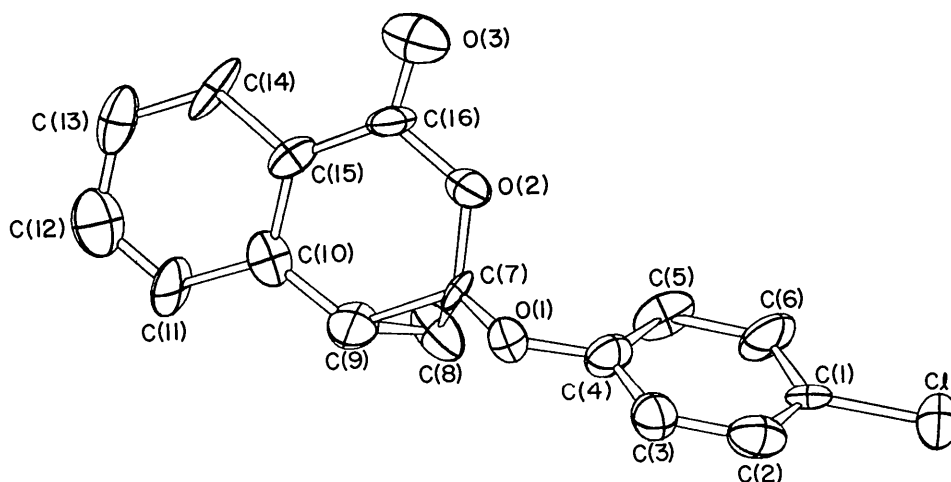


Fig. 1. The molecular configuration of  $C_{16}H_{11}O_3Cl$ .

$M_7(u)$  function. The  $M_7(u)$  function was calculated by use of 3Cl-Cl vectors and 4Cl-O vectors where the 4Cl-O superpositions were weighted according to  $Z_{Cl}/Z_O$ . The procedure used here in the interpretation of the Patterson function has been described previously (Jacobson & Guggenberger, 1966; Jacobson, 1966).

After one cycle of least-squares the conventional reliability index was 0.30. Further study of the  $M_7(u)$  map indicated that four of the phenoxy carbon atoms had been misplaced. After correcting these positions and locating the last nonhydrogen atom on an electron density map the refinement proceeded normally. An  $R(\Sigma ||F_o| - |F_c|| / \Sigma |F_o|)$  of 0.135 was obtained on varying the isotropic thermal parameters. Since it was important to prove that an oxygen atom had been introduced by the photochemical reaction, the lactone oxygen, O(2), was replaced by a carbon atom and a cycle of least-squares run. The isotropic temperature factor of this atom went to  $-1.5$ , indicating that the oxygen assignment was correct. A cycle of least-squares with anisotropic thermal parameters was run after

excluding 9 reflections because of suspected errors resulting from incorrectly positioned angles in the data collection. Hydrogen atoms were then placed in their calculated positions, 1 Å from the associated carbon, which corresponded to peak positions in the electron density difference synthesis. The hydrogen atoms were assigned an isotropic temperature factor of 4.0 and their positions were not refined. A small correction was made in the weighting scheme since the average values of  $w(|F_o| - |F_c|)^2$  in low  $\sin \theta/\lambda$  regions were systematically too high (Cruickshank & Pilling, 1961). The form factors for all atoms were those of Hansen, Herman, Lea & Skillman (1964). Local least-squares and Fourier programs written by D.R. Fitzwater were used.

The refinement converged with a final  $R$  of 0.069 for the observed reflections and 0.171 for all reflections. The maximum peak in the final electron density difference map was  $0.5 \text{ e.}\text{\AA}^{-3}$ . The large  $R$  value for all the data results from the large block of weak high order, unobserved reflections. Although the data were meas-

Table 1. Atomic coordinates and thermal parameters

Standard errors of the coordinates and the  $B_{ij}$  and their standard errors are  $\times 10^5$ .

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

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{11}$	$B_{22}$	$B_{33}$	$B_{12}$	$B_{13}$	$B_{23}$
Cl	0.00847	0.67725	0.49523	376	639	2613	22	-32	-62
	22	27	69	16	18	98	21	44	61
O(1)	0.43914	0.58170	0.58511	324	430	1921	-54	25	56
	54	60	117	45	52	237	43	88	94
O(2)	0.47377	0.43038	0.47420	430	318	2212	-87	176	-65
	46	48	143	44	46	253	37	94	102
O(3)	0.52170	0.27950	0.47428	969	515	2782	-152	-33	121
	77	62	194	75	58	342	58	158	134
C(1)	0.13762	0.64865	0.52129	453	127	2527	-9	28	-103
	77	71	242	67	68	418	53	182	162
C(2)	0.19111	0.68642	0.66432	730	374	1559	-54	192	-99
	98	108	209	95	85	394	90	155	167
C(3)	0.29410	0.66315	0.67417	448	467	1906	-34	-120	97
	87	107	199	77	96	385	74	137	168
C(4)	0.33823	0.60085	0.55312	480	375	1546	87	101	-183
	93	89	187	80	80	374	65	152	150
C(5)	0.28111	0.56265	0.41343	796	481	1235	216	362	-467
	99	102	197	110	95	393	95	167	172
C(6)	0.17786	0.58735	0.39475	691	448	2778	236	-193	-609
	105	107	242	96	91	461	87	195	208
C(7)	0.48857	0.53081	0.44964	223	304	2266	176	156	-170
	80	77	193	64	73	437	63	150	143
C(8)	0.50975	0.57105	0.26659	593	733	1775	-300	-551	563
	103	113	200	76	87	295	89	146	132
C(9)	0.59544	0.56214	0.40852	564	465	2388	24	255	587
	89	98	201	87	88	431	79	154	165
C(10)	0.67781	0.49300	0.39195	421	621	541	-87	87	30
	87	99	182	78	91	286	76	129	143
C(11)	0.77852	0.51720	0.36485	367	714	1899	169	435	-296
	91	111	225	85	112	384	85	155	195
C(12)	0.85106	0.45133	0.35404	587	946	1702	-75	107	-43
	106	132	220	96	132	385	112	162	213
C(13)	0.82705	0.35008	0.35831	389	973	1921	237	178	-213
	94	129	232	85	154	441	97	166	220
C(14)	0.72729	0.32281	0.38752	608	759	1239	542	235	-308
	91	128	205	92	106	350	115	158	217
C(15)	0.65116	0.39357	0.40810	504	357	1272	160	80	-63
	93	86	197	80	88	319	74	134	139
C(16)	0.54949	0.36066	0.45286	548	178	1713	44	265	95
	88	85	192	84	67	413	65	142	133

ured out to a maximum  $2\theta$  of  $50^\circ$  it is clear that most of the high order data from the crystal used were too weak to be considered observed. The relatively poor agreement for the unobserved data probably results from errors in estimating the backgrounds for many of these reflections. Some small background fluctuations were observed for different  $\chi$  and  $\phi$  instrument settings. Noteworthy here is the fact that the weighted  $R$  index,  $\{\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2\}^{1/2}$ , for all the data is much better at 0.090. The lack of good high order data is also reflected to some extent in the errors in the atom positional parameters. It was not possible to obtain better statistics by choosing a different crystal, since the total available supply of this compound was five crystals and the best of these was chosen for this study. In retrospect it is clear that Cu radiation would have given us more usable data without increasing the absorption appreciably and hence would have been a better radiation choice.

The molecular configuration with the anisotropic thermal ellipsoids included (Johnson, 1965) is depicted in Fig. 1. The refined parameters and their standard deviations are given in Table 1. The calculated hydrogen atom positions are given in Table 2. The bond lengths and bond angles are given in Table 3. Estimated standard errors were calculated by use of the variance-covariance matrix and the program of Busing & Levy (1959). The observed and calculated structure factors are listed in Table 4 where asterisks are used to denote unobserved data.

Table 2. *Coordinates of hydrogen atoms*

	<i>x</i>	<i>y</i>	<i>z</i>
H(C2)	0.15736	0.72645	0.75887
H(C3)	0.33707	0.69224	0.77181
H(C5)	0.31275	0.51854	0.32143
H(C6)	0.13543	0.56021	0.29392
H(C8)	0.48244	0.64176	0.22801
H(C8)	0.50867	0.52269	0.14724
H(C9)	0.61613	0.62889	0.46989
H(C11)	0.79720	0.58703	0.35463
H(C12)	0.92498	0.47252	0.34191
H(C13)	0.88056	0.29914	0.34006
H(C14)	0.70891	0.25118	0.39377

Least-squares planes were calculated for several regions of the molecule. The equations of the least-squares planes and the displacements of the atoms from these planes are given in Table 5.

### Discussion

The bond distances and angles are normal in view of the known molecular geometries of other organic compounds. The average benzene carbon-carbon distance is 1.39 Å. The cyclopropane distances found here are similar to those found in a recent structure determination (MacDonald & Trotter, 1965). The C(15)-C(16) distance is the same as that observed in salicylic acid (Sundaralingam & Jensen, 1965).

The C(4)-O(1)-C(7) angle of  $115.6^\circ$  suggests that O(1) is  $sp^2$  hybridized; the driving force for this might be some electron delocalization with the benzene ring.

Table 3. *Intramolecular distances and angles*

Distances		Angles	
Cl(1)-C(1)	1.76 (1) Å	Cl(1)-C(1)-C(2)	119.9 (9)°
C(1)-C(2)	1.37 (2)	Cl(1)-C(1)-C(6)	116.3 (10)
C(1)-C(6)	1.36 (2)	C(2)-C(1)-C(6)	123.8 (11)
C(2)-C(3)	1.40 (2)	C(1)-C(2)-C(3)	117.0 (11)
C(3)-C(4)	1.37 (2)	C(2)-C(3)-C(4)	121.7 (12)
C(4)-C(5)	1.38 (2)	C(3)-C(4)-C(5)	119.3 (10)
C(4)-O(1)	1.38 (1)	C(3)-C(4)-O(1)	115.0 (10)
C(5)-C(6)	1.41 (2)	C(5)-C(4)-O(1)	125.7 (10)
O(1)-C(7)	1.38 (1)	C(4)-C(5)-C(6)	120.6 (10)
C(7)-C(8)	1.48 (2)	C(5)-C(6)-C(1)	117.4 (12)
C(7)-C(9)	1.51 (1)	C(4)-O(1)-C(7)	115.6 (8)
C(7)-O(2)	1.41 (1)	O(1)-C(7)-C(8)	123.4 (9)
C(8)-C(9)	1.54 (2)	O(1)-C(7)-C(9)	116.2 (9)
O(2)-C(16)	1.40 (1)	O(1)-C(7)-O(2)	110.0 (9)
C(16)-O(3)	1.19 (1)	C(8)-C(7)-C(9)	62.2 (7)
C(16)-C(15)	1.46 (1)	C(8)-C(7)-O(2)	120.7 (10)
C(15)-C(10)	1.42 (1)	C(9)-C(7)-O(2)	115.9 (8)
C(10)-C(9)	1.45 (1)	C(7)-C(8)-C(9)	59.8 (7)
C(10)-C(11)	1.39 (1)	C(7)-C(9)-C(8)	58.0 (7)
C(11)-C(12)	1.32 (2)	C(7)-C(9)-C(10)	122.0 (9)
C(12)-C(13)	1.43 (2)	C(7)-O(2)-C(16)	124.3 (7)
C(13)-C(14)	1.39 (2)	O(2)-C(16)-O(3)	114.4 (9)
C(14)-C(15)	1.41 (1)	C(15)-C(16)-O(3)	127.5 (9)
		C(16)-C(15)-C(10)	123.3 (9)
		C(16)-C(15)-C(14)	117.8 (10)
		C(15)-C(10)-C(9)	116.2 (9)
		C(15)-C(10)-C(11)	118.8 (10)
		C(10)-C(11)-C(12)	122.6 (12)
		C(11)-C(12)-C(13)	120.5 (11)
		C(12)-C(13)-C(14)	118.6 (12)
		C(13)-C(14)-C(15)	120.4 (13)



Table 4 (cont.)

5, K, 4	4 125 144	1, K, 5	2* 81 -108	0 101 83	11* 0 36	8 82 -78	1* 83 -106	1* 78 -75
0 0 26	5* 0 46	0 290 277	3 120 -117	1 127 -91		9* 0 40	2* 11 29	2* 6 -32
1 168 -176	6* 43 -59	1 155 162	4* 53 -62	2 127 -124	3, K, 6		3* 42 -54	3* 40 -48
2 102 118	7* 0 -11	2 128 -136	5 187 187	3 91 56		8, K, 6	4* 0 11	4* 27 55
3* 0 10	8* 55 -42	3 183 -173	6* 65 49	4* 37 -52	0 86 -57		5 105 -92	5* 65 53
4 257 -256	9* 77 71	4 125 -135	7* 73 88	5 97 55	1* 63 -61	0 116 99	6* 47 14	6* 47 14
5* 82 -90	10* 0 58	5* 63 79	8* 36 -16	6* 44 53	2 199 205	1 89 95	7* 77 59	7* 77 59
6* 0 -8	11* 0 -43	6* 33 -17	9 126 -122	7* 81 76	3* 54 60	2* 13 27	8* 43 -10	8* 43 -10
7* 74 43		7* 87 93	10* 58 90	8* 0 -15	4* 0 13	3 93 74	9* 0 -16	9* 0 -16
8* 52 32		8 86 96	11* 96 77		5* 43 45	4* 30 50		
9 122 124	10, K, 4	9 95 -100	12* 66 65	11, K, 5	6 105 -93	5* 47 -45		
10* 80 83	0 123 140	10 136 122		0 60 42	7* 40 13	6* 0 23	0 139 134	0, K, 8
11* 78 66	1 183 191	11 116 80	6, K, 5	1 84 51	8* 0 -58	7* 40 38	1* 82 -99	1* 82 -99
12* 0 8	2* 70 -84	12* 41 -54	0 70 60	2 100 95	9* 0 -34	8* 62 -55	2* 49 -71	2* 49 -71
13* 0 43	3* 146 88	13* 0 -26	1 149 -157	3* 62 -5	10* 57 60		3* 0 -49	3* 0 -49
	4* 0 -44		2* 60 -54	4* 64 -52	11* 0 42		4* 0 23	4* 0 23
6, K, 4	5* 53 -63	2, K, 5	3 101 -114	5 112 112			5* 47 52	5* 47 52
0 262 267	6* 50 16	0 72 -43	4* 35 -17	6 21 40	4, K, 6	0 50 5	6* 0 42	6* 0 42
1* 44 85	7 119 99	1 175 151	5 93 61		0 126 120	1 86 56	7* 0 47	7* 0 47
2 89 -109	8* 90 -64	2 151 150	6 110 -109	12, K, 5	1 131 -136	2 102 81	8* 53 57	8* 53 57
3 112 132	10* 0 30	3* 36 -65	7 114 106	0 33 23	2* 82 -83	3* 40 75	9* 57 -44	9* 57 -44
4* 48 -80		4 97 -116	8 113 -108	1 98 -73	3* 0 0	4* 41 -15		
5 152 151	11, K, 4	5 96 -86	9* 48 47	2 121 -113	4* 0 -37	5* 0 -60		
6* 71 28	0 155 163	6* 44 -17	10* 71 39	3 90 -72	5* 50 40	6* 0 0	4, K, 7	4, K, 7
7 120 120	1* 27 -19	7 88 -88	11* 0 -28	4* 0 -21	6* 72 47	7* 0 18	0 0 39	0 0 39
8 214 -214	2* 67 52	8* 57 37			7* 39 -40		1* 24 50	1* 24 50
9* 65 -87	3* 48 46	9* 0 37	7, K, 5	0, K, 6	8* 54 -33	0 97 99	2* 0 26	2* 0 26
10* 70 -67	4 164 -164	10* 80 -64	0 70 73	0 0 15	9* 0 -7	1* 52 44	3* 0 -32	3* 0 -32
11* 48 -69	5* 60 36	11* 8 29	1* 0 -24	1 88 -105	10 84 86	2 86 -95	4* 38 48	4* 38 48
12* 66 -62	6* 36 -27	12* 81 -89	2* 76 -49	2 250 -258	11* 66 -51	3* 0 -2	5* 0 10	5* 0 10
13* 0 27	7* 35 -28	13* 58 -38	3 177 -183	3* 0 5		4* 39 -71	6* 56 89	6* 56 89
	8 82 -53		4 189 -178	4* 0 17	5, K, 6	5* 0 -23	8* 0 -44	8* 0 -44
7, K, 4		3, K, 5	5 127 117	5* 11 19	0 44 -3			
0 0 26	12, K, 4	0 106 -86	6 97 60	6 267 265	1* 0 -47	11, K, 6		
1* 0 -79	0 93 61	1 52 -90	7 167 158	7* 85 -39	2* 36 73	0 0 27	0 0 -46	0 0 -46
2 114 -118	1* 0 -33	2* 0 32	8* 71 71	8* 82 -97	3* 0 58	1* 66 -77	1 92 101	1 92 101
3* 0 -56	2 75 -68	3 173 -196	9* 19 -24	9* 65 -25	4* 0 -62	2* 0 56	2* 0 -37	2* 0 -37
4 259 -264	3* 39 -52	4* 43 -73	10* 32 31	10* 0 -39	5* 81 68	3* 55 63	3* 53 -71	3* 53 -71
5* 26 -52	4* 0 -22	5 105 114	11* 14 22	11* 60 71	6 103 -92		4* 0 -44	4* 0 -44
6 88 102	5* 3 25	6 98 105			7* 36 37		5* 60 59	5* 60 59
7* 28 77	6* 0 41	7* 72 56	8, K, 5	1, K, 6	8* 54 -47		6* 0 -58	6* 0 -58
8 189 -166	7* 0 42	8* 42 56	0 31 -33	0 123 113	9* 54 67	1 24 22	7* 45 52	7* 45 52
9* 69 56		9 115 -122	1 139 141	1 95 -92	10* 49 45	2 119 100	8* 80 -52	8* 80 -52
10* 59 -66	13, K, 4	10* 73 49	2 139 -136	2 215 201		3 136 -142		
11* 0 38	0 54 44	11* 80 65	3 136 -134	3 107 108	6, K, 6	4* 25 37	6, K, 7	6, K, 7
12* 20 44	1* 17 -67	12* 0 -41	4 120 -109	4 105 116	0 139 132	5 167 165	0 62 59	0 62 59
	2* 0 40	13* 62 -32	5* 79 -63	5* 82 87	1* 43 36	6* 62 7	1* 60 -51	1* 60 -51
8, K, 4	3* 0 -47	4, K, 5	6 89 -80	6 145 163	2 86 -113	7* 73 46	2* 0 33	2* 0 33
0 369 372	4* 53 -68	0 190 -177	7* 40 -32	7* 70 -81	3* 30 30	8* 29 -10	3* 49 -59	3* 49 -59
1* 0 21	5* 0 27	1 75 -110	8* 30 61	8* 59 -12	4* 27 -43	9* 0 36	4* 38 -55	4* 38 -55
2* 0 -37		2* 0 -47	9* 64 -62	9* 41 14	5 92 63		5* 23 -66	5* 23 -66
3* 24 68	0, K, 5	3* 81 -94	10* 0 46	10 120 -118	6 101 85		6* 0 -38	6* 0 -38
4* 32 -49	1 196 -194	4* 39 36		11* 48 58	7* 70 87	0 0 24	7* 62 56	7* 62 56
5* 0 -55	2* 0 -10	5 130 -120	0 93 -75	2, K, 6	8 134 -125	1 196 193		
6 91 87	3* 66 36	6* 66 36	1* 52 -31	0 72 90	9* 81 -85	2* 0 30	7, K, 7	7, K, 7
7 82 75	4* 39 66	7 154 154	2 92 -99	1 282 283	0* 56 -61	3 99 -97	0 0 5	0 0 5
8 121 -121	5 380 -382	8* 71 -61	3 153 -143	3 952 992		4 85 -87	1* 0 15	1* 0 15
9 93 -99	6 155 -156	9 102 -84	4 105 71	4 102 84	7, K, 6	5* 51 46	2* 0 38	2* 0 38
10* 57 88	7 188 185	5 105 71	5 102 84	5 102 84	0 8 33	6* 65 15	3 83 -82	3 83 -82
11* 41 -54	8 146 -151	6 82 59	6 82 59	6 82 59	1* 69 -42	7* 66 -24	4* 70 -72	4* 70 -72
	9* 58 49	7* 3 -37	7* 3 -37	7* 3 -37	2* 46 75	8* 76 45	5* 46 30	5* 46 30
0 0 -32	10* 57 88	8* 35 28	8* 35 28	8* 35 28	3* 61 30	9 87 -67	6* 30 37	6* 30 37
1 150 145	9, K, 4	9 102 -98	9 102 -98	9 102 -98	4 181 164			
2 126 134	0 89 92				5 94 -69	2, K, 7		
3 229 227	1 16 -47	0 127 -138			6 56 40	0 0 12	8, K, 7	8, K, 7
	2* 54 -31	1 112 -105	10, K, 5	10* 32 55	7* 49 -37		0 0 1	0 0 1
	13 135 -128							

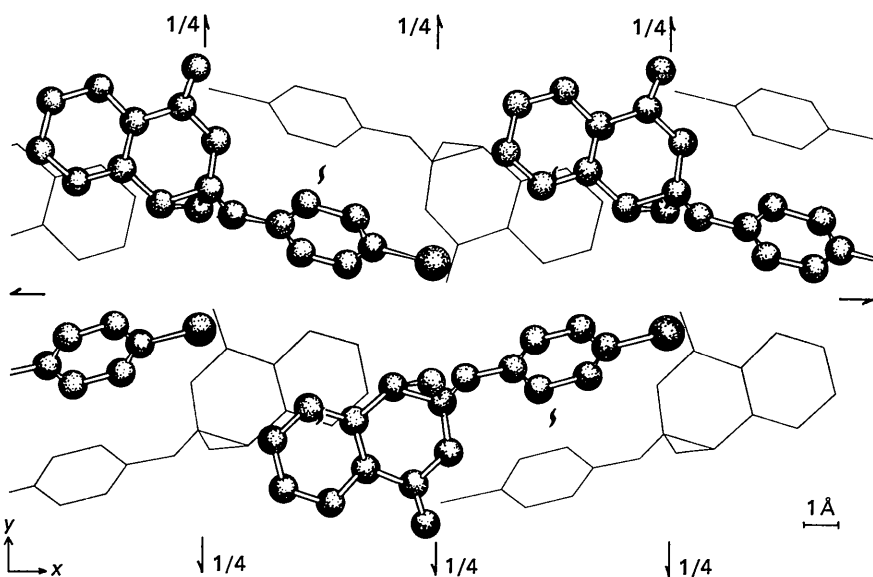
Fig. 2. Projection of the structure along the  $c$  axis. The shaded layer is  $c/2$  above the unshaded layer.

Table 5. *Molecular least-squares planes*

Description of planes					
	I	C(1), C(2), C(3), C(4), C(5), C(6)			
	II	C(10), C(11), C(12), C(13), C(14), C(15)			
	III	C(9), C(7), O(2), C(16), O(3), C(15), C(10)			
	IV	C(9), C(7), O(2), C(16), O(3), C(15), C(10), C(11), C(12), C(13), C(14)			
	V	C(7), C(8), C(9)			
Equations, relative to <i>a, b, c</i>					
	I	$0.2413X + 0.7815Y - 0.5753Z - 5.231 = 0$			
	II	$0.1439X + 0.0227Y + 0.9893Z - 4.299 = 0$			
	III	$0.1976X + 0.0701Y + 0.9778Z - 5.030 = 0$			
	IV	$0.1768X + 0.0561Y + 0.9826Z - 4.809 = 0$			
	V	$-0.2094X + 0.9181Y + 0.3364Z - 6.484 = 0$			
Deviations	I	II	IV	IV	V
C(1)	0.01				
C(2)	-0.02				
C(3)	0.02				
C(4)	0.00				
C(5)	-0.01				
C(6)	0.01				
C(7)			-0.02	-0.02	0.00
C(8)					0.00
C(9)			0.00	-0.04	0.00
C(10)		-0.01	0.02	-0.02	
C(11)		-0.01		0.04	
C(12)		0.03		0.08	
C(13)		-0.02		-0.03	
C(14)		-0.01		-0.07	
C(15)		0.02	-0.02	-0.04	
C(16)			0.00	0.02	
O(2)			0.02	0.05	
O(3)			0.00	0.04	

The  $p_z$  orbital on O(1) makes an angle of  $20.4^\circ$  with the normal of the phenoxy ring, [plane I. The C-O distances for O(1) also compare favorably with those involving O(2) in the lactone ring.

It is evident from the deviations in Table 5 that the combined benzene and lactone rings (plane IV) are nearly planar. This plane is tilted  $10.7^\circ$  out of the  $x$ - $y$  plane. The angle between the phenoxy plane (I) and the molecular plane (IV) is  $64.4^\circ$ . The cyclopropane ring makes an angle of  $69.8^\circ$  with the molecular plane.

The thermal parameters,  $U_{ij}$ , could not be interpreted satisfactorily in terms of rigid-body translational and librational motion. The eight atom phenoxy group was treated as one rigid body and the rest of the molecule as a second rigid body and the center of motion was varied for both groups. Although no good fit could be obtained between the observed and calculated  $U_{ij}$  values, several general conclusions could be drawn. The translational oscillations for both groups were not markedly anisotropic; neither were the librational oscillations of the second group. The root mean square librational amplitude of the phenoxy group about the C(1)-C(4) direction was about  $7^\circ$ .

The intermolecular contacts are of the normal van der Waals type. The molecular packing is illustrated in Fig. 2. The shortest non-hydrogen contact is  $3.35 \text{ \AA}$  and involves C(12) and C(9) on a neighboring molecule. The C(12) atom showed the largest deviations from the least-squares planes and the largest r.m.s. vibrations in the bonding directions. The C(12) atom has probably

been slightly misplaced in this refinement as evidenced by the short C(11)-C(12) distance. This distance remained essentially the same throughout the refinement and probably results from some systematic error in the data. A correction for librational motion increases this bond length by only  $0.01 \text{ \AA}$ .

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