

The Crystal Structure of 1,4-Ethylene-2,8-dichloro-2,4,6,8-tetramethyloctahydronaphthal-5-ene-3,7-dione

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The crystal structure of the title compound, $C_{16}H_{16}Cl_2O_2$, has been determined in three dimensions by X-ray diffraction, from 2093 independent intensities. The crystals belong to the triclinic space group $P\bar{1}$ with two molecules per unit cell of dimensions $a = 8.848 \text{ \AA}$, $b = 9.215 \text{ \AA}$, $c = 10.115 \text{ \AA}$, $\alpha = 98.03^\circ$, $\beta = 92.86^\circ$, and $\gamma = 113.80^\circ$. The structure was solved by a combination of heavy-atom and direct methods and refined by full-matrix least-squares to a final R value of 0.052.

The Diels-Alder product 1,4-ethylene-2,8-dichloro-2,4,6,8-tetramethyloctahydronaphthal-5-ene-3,7-dione is a dimer of 6-chloro-2,6-dimethylcyclohexa-2,4-dien-1-one, formed upon oxidation of 2,6-dimethylphenol by chlorine dioxide. Two molecules with the same absolute configuration about the tertiary carbon atom have dimerised. The structure is rather strained with $C(sp^3) - C(sp^3)$ bond lengths of 1.589 and 1.560 \AA for the bonds $C(4) - C(4a)$ and $C(4a) - C(8a)$, respectively.

Oxidation of 2,6-xylene in carbon tetrachloride solution by chlorine dioxide gives in a low yield the Diels-Alder product 1,4-ethylene-2,8-dichloro-2,4,6,8-tetramethyloctahydronaphthal-5-

ene-3,7-dione which is a dimer of 6-chloro-2,6-dimethylcyclohexa-2,4-dien-1-one.

The steric orientation of the chloro group in the cyclohexenone moiety was established¹ from the high value (1700 cm^{-1}) of the stretching frequency of the conjugated carbonyl group.

The purpose of this investigation, in a program of crystal structure studies of oxidation products of phenols, was to establish the steric configuration at carbon atom C(2) and to confirm the structure of the dimer.

EXPERIMENTAL

Two crystals of the approximate volumes 0.0044 and 0.0014 mm^3 were mounted arbitrarily on the computer-controlled Philips diffractometer PW 1100 equipped with a graphite monochromator to reflect $\text{CuK}\alpha$ radiation. Preceding the measurement of the reflexion intensities, the least-squares unit cell parameters, and crystal-orientation parameters were determined, through measurement of the angular positions of 25 accurately centered reflexions by the "Peak-Hunting Program". Table 1 contains the refined cell parameters.

The intensities of 2562 independent reflexions out to $\theta = 65^\circ$ were measured by the moving-crystal, moving-counter technique ($\theta - 2\theta$ scan). Backgrounds were estimated by stationary counting at $\pm 0.75^\circ 2\theta$ from the peak maxima. The scan speed was $1.2^\circ \text{ min}^{-1}$. Three monitor reflexions were measured every 90 min, which showed that the crystals were not stable to X-rays. Two crystals were used, each with a decrease in intensity of about 10 to 15 % during the period of data collection. Lorentz and polarization factors were applied but not correction for absorption. Only the 2093 reflexions for which $\sigma(I)/I \leq 0.25$ were used in refinement of the structure. $\sigma(I)$ is the estimated standard

Table 1. Crystal data.

Lattice constants	$a = 8.848(2) \text{ \AA}$ $b = 9.215(3) \text{ \AA}$ $c = 10.115(4) \text{ \AA}$ $\alpha = 98.03(1)^\circ$ $\beta = 92.86(1)^\circ$ $\gamma = 113.80(1)^\circ$ $V = 742.08 \text{ \AA}^3$
Cell volume	$d = 1.40 \text{ g cm}^{-3}$
Density (calculated)	$Z = 2$
Molecules per unit cell	$P\bar{1}$
Space group	

Table 2. Observed and calculated structure amplitudes. The columns use the running index l , $10|F_o|$, and $10|F_c|$, respectively.

49	874	854	1	1	1
50	246	236	1	1	1
51	277	267	1	1	1
52	167	157	1	1	1
53	160	150	1	1	1
54	25	15	1	1	1
55	50	40	1	1	1
56	100	90	1	1	1
57	150	140	1	1	1
58	200	190	1	1	1
59	250	240	1	1	1
60	300	290	1	1	1
61	350	340	1	1	1
62	400	390	1	1	1
63	450	440	1	1	1
64	500	490	1	1	1
65	550	540	1	1	1
66	600	590	1	1	1
67	650	640	1	1	1
68	700	690	1	1	1
69	750	740	1	1	1
70	800	790	1	1	1
71	850	840	1	1	1
72	900	890	1	1	1
73	950	940	1	1	1
74	1000	990	1	1	1
75	1050	1040	1	1	1
76	1100	1090	1	1	1
77	1150	1140	1	1	1
78	1200	1190	1	1	1
79	1250	1240	1	1	1
80	1300	1290	1	1	1
81	1350	1340	1	1	1
82	1400	1390	1	1	1
83	1450	1440	1	1	1
84	1500	1490	1	1	1
85	1550	1540	1	1	1
86	1600	1590	1	1	1
87	1650	1640	1	1	1
88	1700	1690	1	1	1
89	1750	1740	1	1	1
90	1800	1790	1	1	1
91	1850	1840	1	1	1
92	1900	1890	1	1	1
93	1950	1940	1	1	1
94	2000	1990	1	1	1
95	2050	2040	1	1	1
96	2100	2090	1	1	1
97	2150	2140	1	1	1
98	2200	2190	1	1	1
99	2250	2240	1	1	1
100	2300	2290	1	1	1
101	2350	2340	1	1	1
102	2400	2390	1	1	1
103	2450	2440	1	1	1
104	2500	2490	1	1	1
105	2550	2540	1	1	1
106	2600	2590	1	1	1
107	2650	2640	1	1	1
108	2700	2690	1	1	1
109	2750	2740	1	1	1
110	2800	2790	1	1	1
111	2850	2840	1	1	1
112	2900	2890	1	1	1
113	2950	2940	1	1	1
114	3000	2990	1	1	1
115	3050	3040	1	1	1
116	3100	3090	1	1	1
117	3150	3140	1	1	1
118	3200	3190	1	1	1
119	3250	3240	1	1	1
120	3300	3290	1	1	1
121	3350	3340	1	1	1
122	3400	3390	1	1	1
123	3450	3440	1	1	1
124	3500	3490	1	1	1
125	3550	3540	1	1	1
126	3600	3590	1	1	1
127	3650	3640	1	1	1
128	3700	3690	1	1	1
129	3750	3740	1	1	1
130	3800	3790	1	1	1
131	3850	3840	1	1	1
132	3900	3890	1	1	1
133	3950	3940	1	1	1
134	4000	3990	1	1	1
135	4050	4040	1	1	1
136	4100	4090	1	1	1
137	4150	4140	1	1	1
138	4200	4190	1	1	1
139	4250	4240	1	1	1
140	4300	4290	1	1	1
141	4350	4340	1	1	1
142	4400	4390	1	1	1
143	4450	4440	1	1	1
144	4500	4490	1	1	1
145	4550	4540	1	1	1
146	4600	4590	1	1	1
147	4650	4640	1	1	1
148	4700	4690	1	1	1
149	4750	4740	1	1	1
150	4800	4790	1	1	1
151	4850	4840	1	1	1
152	4900	4890	1	1	1
153	4950	4940	1	1	1
154	5000	4990	1	1	1
155	5050	5040	1	1	1
156	5100	5090	1	1	1
157	5150	5140	1	1	1
158	5200	5190	1	1	1
159	5250	5240	1	1	1
160	5300	5290	1	1	1
161	5350	5340	1	1	1
162	5400	5390	1	1	1
163	5450	5440	1	1	1
164	5500	5490	1	1	1
165	5550	5540	1	1	1
166	5600	5590	1	1	1
167	5650	5640	1	1	1
168	5700	5690	1	1	1
169	5750	5740	1	1	1
170	5800	5790	1	1	1
171	5850	5840	1	1	1
172	5900	5890	1	1	1
173	5950	5940	1	1	1
174	6000	5990	1	1	1
175	6050	6040	1	1	1
176	6100	6090	1	1	1
177	6150	6140	1	1	1
178	6200	6190	1	1	1
179	6250	6240	1	1	1
180	6300	6290	1	1	1
181	6350	6340	1	1	1
182	6400	6390	1	1	1
183	6450	6440	1	1	1
184	6500	6490	1	1	1
185	6550	6540	1	1	1
186	6600	6590	1	1	1
187	6650	6640	1	1	1
188	6700	6690	1	1	1
189	6750	6740	1	1	1
190	6800	6790	1	1	1
191	6850	6840	1	1	1
192	6900	6890	1	1	1
193	6950	6940	1	1	1
194	7000	6990	1	1	1
195	7050	7040	1	1	1
196	7100	7090	1	1	1
197	7150	7140	1	1	1
198	7200	7190	1	1	1
199	7250	7240	1	1	1
200	7300	7290	1	1	1
201	7350	7340	1	1	1
202	7400	7390	1	1	1
203	7450	7440	1	1	1
204	7500	7490	1	1	1
205	7550	7540	1	1	1
206	7600	7590	1	1	1
207	7650	7640	1	1	1
208	7700	7690	1	1	1
209	7750	7740	1	1	1
210	7800	7790	1	1	1
211	7850	7840	1	1	1
212	7900	7890	1	1	1
213	7950	7940	1	1	1
214	8000	7990	1	1	1
215	8050	8040	1	1	1
216	8100	8090	1	1	1
217	8150	8140	1	1	1
218	8200	8190	1	1	1
219	8250	8240	1	1	1
220	8300	8290	1	1	1
221	8350	8340	1	1	1
222	8400	8390	1	1	1
223	8450	8440	1	1	1
224	8500	8490	1	1	1
225	8550	8540	1	1	1
226	8600	8590	1	1	1
227	8650	8640	1	1	1
228	8700	8690	1	1	1
229	8750	8740	1	1	1
230	8800	8790	1	1	1
231	8850	8840	1	1	1
232	8900	8890	1	1	1
233	8950	8940	1	1	1
234	9000	8990	1	1	1
235	9050	9040	1	1	1
236	9100	9090	1	1	1
237	9150	9140	1	1	1
238	9200	9190	1	1	1
239	9250	9240	1	1	1
240	9300	9290	1	1	1
241	9350	9340	1	1	1
242	9400	9390	1	1	1
243	9450	9440	1	1	1
244	9500	9490	1	1	1
245	9550	9540	1	1	1
246	9600	9590	1	1	1
247	9650	9640	1	1	1
248	9700	9690	1	1	1
249	9750	9740	1	1	1
250	9800	9790	1	1	1
251	9850	9840	1	1	1
252	9900	9890	1	1	1
253	9950	9940	1	1	1
254	10000	9990	1	1	1
255	10050	10040	1	1	1
256	10100	10090	1	1	1
257	10150	10140	1	1	1
258	10200	10190	1	1	1
259	10250	10240	1	1	1
260	10300	10290	1	1	1
261	10350	10340	1	1	1
262	10400	10390	1	1	1
263	10450	10440	1	1	1
264	10500	10490	1	1	1
265	10550	10540	1	1	1
266	10600	10590	1	1	1
267	10650	10640	1	1	1
268	10700	10690	1	1	1
269	10750	10740	1	1	1
270	10800	10790	1	1	1
271	10850	10840	1	1	1
272	10900	10890	1	1	1
273	10950	10940	1	1	1
274	11000	10990	1	1	1
275	11050	11040	1	1	1
276	11100	11090	1	1	1
277	11150	11140	1	1	1
278	11200	11190	1	1	1
279	11250	11240	1	1	1
280	11300	11290	1	1	1
281	11350	11340	1	1	1
282	11400	11390	1	1	1
283	11450	11440	1	1	1
284	11500	11490	1	1	1
285	11550	11540	1	1	1
286	11600	11590	1	1	1
287	11650	11640	1	1	1
288	11700	11690	1	1	1
289	11750	11740	1	1	1
290	11800	11790	1	1	1
291	11850	11840	1	1	1
292	11900	11890	1	1	1
293	11950	11940	1	1	1
294	12000	11990	1	1	1
295	12050	12040	1	1	1
296	12100	12090	1	1	1
297	12150	12140	1	1	

Table 3. Positional and anisotropic thermal parameters of the non-hydrogen atoms. The β -values refer to the temperature factor expression $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + hl\beta_{13} + kl\beta_{23})]$. Estimated standard deviations are given in parentheses. Values are $\times 10^4$.

	<i>x</i>	<i>y</i>	<i>z</i>	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	3375(3)	2821(3)	3005(3)	93(4)	101(4)	81(3)	92(6)	-2(5)	21(5)
C(2)	3265(4)	1096(4)	2602(3)	132(5)	125(4)	85(3)	149(7)	19(6)	24(6)
C(3)	1431(4)	-41(3)	2254(3)	148(5)	102(4)	79(3)	112(7)	3(6)	30(5)
C(4)	262(3)	769(3)	2583(3)	106(4)	101(4)	88(3)	67(7)	23(5)	47(5)
C(4a)	612(3)	2065(3)	1621(3)	98(4)	100(4)	60(3)	80(6)	3(5)	16(5)
C(5)	-553(3)	2848(4)	1878(3)	85(4)	129(4)	95(3)	90(7)	22(5)	57(6)
C(6)	-132(4)	4318(4)	2563(3)	133(5)	138(4)	78(3)	139(7)	62(6)	64(6)
C(7)	1642(4)	5379(3)	2947(3)	165(5)	101(4)	79(3)	135(7)	6(6)	36(5)
C(8)	2760(3)	5001(3)	1987(3)	86(4)	99(4)	99(3)	45(6)	3(5)	70(5)
C(8a)	2498(3)	3223(3)	1833(3)	90(4)	110(4)	65(3)	94(6)	32(5)	33(5)
C(9)	2425(4)	2781(4)	4198(3)	147(5)	121(4)	65(3)	144(7)	-7(6)	11(5)
C(10)	865(4)	1731(4)	3990(3)	150(5)	147(5)	69(3)	150(8)	57(6)	65(6)
C(11)	4108(5)	596(5)	3699(4)	178(6)	150(5)	134(4)	197(10)	-43(8)	44(8)
Cl(12)	4268(1)	956(1)	1103(1)	206(2)	213(2)	129(1)	271(3)	124(2)	31(2)
O(13)	961(3)	-1438(3)	1759(3)	208(5)	112(4)	157(3)	132(6)	-35(6)	-15(5)
C(14)	-1535(4)	-454(4)	2403(4)	122(5)	136(5)	148(5)	43(8)	21(7)	90(8)
C(15)	-1381(5)	4958(5)	2985(4)	206(7)	196(6)	130(4)	280(11)	127(9)	82(9)
O(16)	2134(4)	6503(3)	3870(3)	266(5)	137(4)	118(3)	162(7)	-34(6)	-37(5)
C(17)	2429(5)	5412(5)	610(4)	165(6)	186(6)	117(4)	173(10)	77(8)	164(8)
Cl(18)	4896(1)	6312(1)	2594(1)	107(2)	132(2)	233(2)	5(2)	-62(3)	122(2)

Table 4. Positional and isotropic thermal parameters of the hydrogen atoms, with estimated standard deviations in parentheses.

	<i>x</i> $\times 10^3$	<i>y</i> $\times 10^3$	<i>z</i> $\times 10^3$	<i>B</i> $\times 10^3$
H(C1)	445(4)	350(4)	319(3)	283
H(C4a)	35(4)	142(4)	77(3)	242
H(C5)	-158(4)	220(4)	160(3)	287
H(C8a)	295(4)	299(3)	110(3)	230
H(C9)	295(4)	352(4)	505(3)	281
H(C10)	13(4)	166(4)	464(3)	311
H1(C11)	526(5)	133(4)	385(4)	378
H2(C11)	354(4)	54(4)	449(4)	378
H3(C11)	405(4)	-41(4)	347(4)	378
H1(C14)	-193(5)	-84(5)	157(4)	441
H2(C14)	-164(4)	-134(5)	287(4)	441
H3(C14)	-226(5)	8(5)	274(4)	441
H1(C15)	-254(5)	420(5)	252(4)	432
H2(C15)	-134(5)	508(5)	383(4)	432
H3(C15)	-115(5)	596(5)	257(4)	432
H1(C17)	244(4)	631(5)	65(4)	362
H2(C17)	328(5)	541(4)	9(4)	362
H3(C17)	129(5)	458(4)	16(3)	362

deviation, based on counter statistics, of the net intensity *I*.

Structure determination and refinement

The structure was solved by a combination of heavy-atom and direct methods. The posi-

tional parameters of the two chlorine atoms were found from a 3-D Patterson synthesis. The structure factors were converted to normalized $|E|$'s using an approximate absolute scale and average temperature factor determined by Wilson's ² method. The atomic positions of the chlorine atoms were used to calculate structure factors. The phases of 10 reflexions with $|F_c| \geq 0.2|F_o|$, where $|F_c|$ and $|F_o|$ are calculated and observed structure factor magnitudes, respectively, were used as starting phases together with two reflexions as variables. These

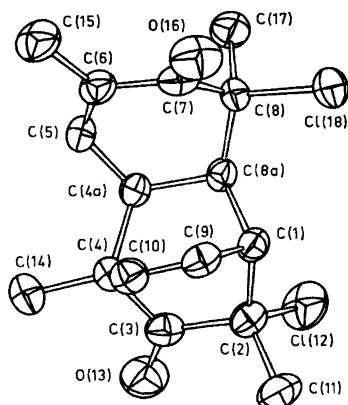


Fig. 1. A perspective view of the molecule.

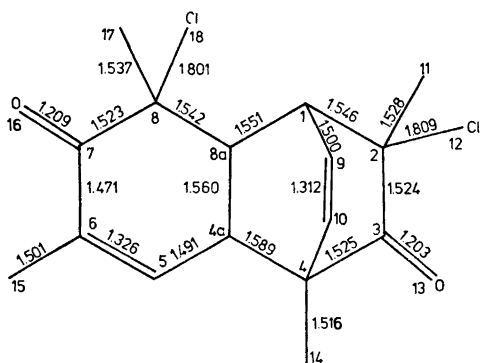


Fig. 2. Bond distances in the molecule.

Table 5. Interatomic angles ($^{\circ}$) with estimated standard deviations in parentheses.

C(2) — C(1) — C(8a)	108.9(2)
C(2) — C(1) — C(9)	105.9(2)
C(8a) — C(1) — C(9)	108.8(2)
C(1) — C(2) — C(3)	107.6(2)
C(1) — C(2) — C(11)	112.6(3)
C(1) — C(2) — Cl(12)	110.2(2)
C(3) — C(2) — C(11)	111.7(3)
C(3) — C(2) — Cl(12)	107.2(2)
C(11) — C(2) — Cl(12)	107.4(2)
C(2) — C(3) — C(4)	113.4(2)
C(2) — C(3) — O(13)	123.0(3)
C(4) — C(3) — O(13)	123.6(3)
C(3) — C(4) — C(4a)	105.4(2)
C(3) — C(4) — C(10)	106.8(2)
C(3) — C(4) — C(14)	111.3(3)
C(4a) — C(4) — C(10)	105.5(2)
C(4a) — C(4) — C(14)	113.3(3)
C(10) — C(4) — C(14)	113.9(3)
C(4) — C(4a) — C(5)	107.1(2)
C(4) — C(4a) — C(8a)	109.6(2)
C(5) — C(4a) — C(8a)	115.7(2)
C(4a) — C(5) — C(6)	125.2(3)
C(5) — C(6) — C(7)	118.9(3)
C(5) — C(6) — C(15)	123.2(3)
C(7) — C(6) — C(15)	117.9(3)
C(6) — C(7) — C(8)	113.0(2)
C(6) — C(7) — O(16)	122.8(3)
C(8) — C(7) — O(16)	124.0(3)
C(7) — C(8) — C(8a)	112.0(2)
C(7) — C(8) — C(17)	109.3(3)
C(7) — C(8) — Cl(18)	109.0(2)
C(8a) — C(8) — C(17)	110.3(3)
C(8a) — C(8) — Cl(18)	110.2(2)
C(17) — C(8) — Cl(18)	105.8(2)
C(1) — C(8a) — C(4a)	109.2(2)
C(1) — C(8a) — C(8)	114.8(2)
C(4a) — C(8a) — C(8)	111.4(2)
C(1) — C(9) — C(10)	114.9(3)
C(4) — C(10) — C(9)	116.0(3)

phases were extended and refined by use of "variance-weighted" Σ_2 -relationships.³ Signs were determined for 178 reflexions ($|E| > 1.8$) without any contradictions in the solution with the highest "reliability index". An E -map based on these phases revealed the complete structure, except for hydrogens. The positional and isotropic thermal parameters of these atoms were refined by full-matrix least-squares computations to an R value of 0.162. Upon assignment of anisotropic thermal parameters to all atoms the value of R fell after three cycles of refinement to 0.075.

The hydrogen atoms were then located from a difference Fourier synthesis. Refinement was continued including all atoms. For the heavy atoms anisotropic temperature factors were used and for the hydrogen atoms isotropic temperature factors equal to those of the final isotropic values of their parent atoms, were applied. The R value converged to 0.052.

The full-matrix least-squares refinements were performed by a modified version of program LALS.⁴ Hughes' weighting scheme⁵ was applied with $|F_{o,\min}| = 1.75$. The atomic scattering factors for oxygen and carbon were taken from Freeman,⁶ that for chlorine from *International Tables for X-Ray Crystallography*⁷ and that for hydrogen from Stewart, Davidson and Simpson.⁸ The observed and calculated structure amplitudes are given in Table 2. The positional and thermal parameters of the non-hydrogen atoms with estimated standard deviations are listed in Table 3 and those for the hydrogen atoms in Table 4.

RESULTS AND DISCUSSION

A perspective view of the molecule together with the numbering of atoms is given in Fig. 1. The stereochemistry of the structure is consonant with considerations given by Adler and Holmberg⁹ and agrees with that found through X-ray investigations of related structures by Karlsson, Pilotti and Wiehager.^{10,11} Intramolecular bond distances and bond angles involving non-hydrogen atoms are given in Fig. 2 and Table 5, respectively.* No corrections for

* The estimated standard deviations, based solely on least-squares parameters, are about 0.004 Å for the bonds.

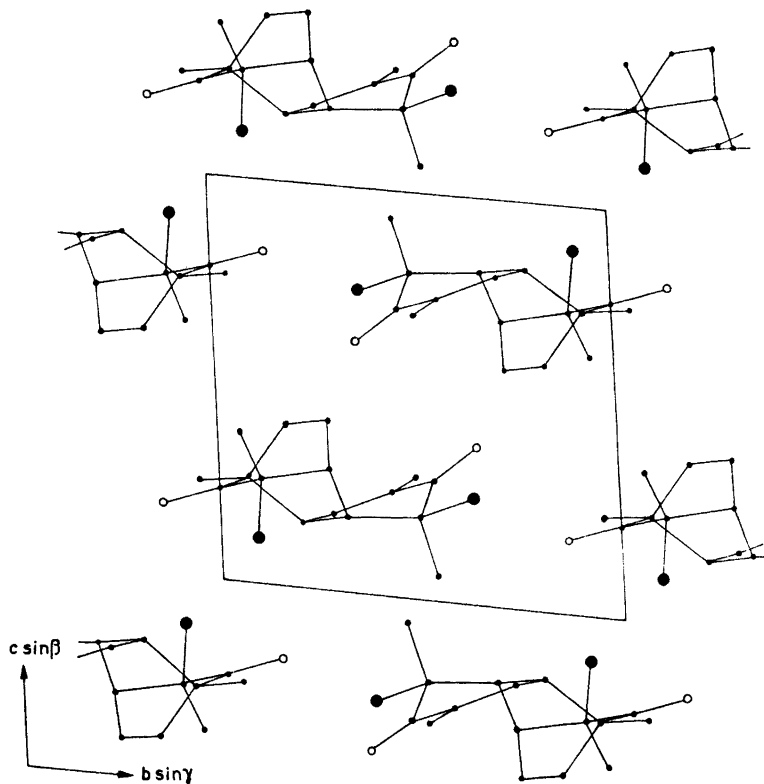


Fig. 3. The unit cell in projection along the a axis. •, carbon; O, oxygen; ●, chlorine.

Table 6. Intermolecular distances (Å) less than 3.8 Å.

Code for symmetry related atoms			
Superscript	Coordinates	Superscript	Coordinates
None	$x; y; z$	iv	$1-x; -y; -z$
i	$x; -1+y; z$	v	$1-x; -y; 1-z$
ii	$-1+x; -1+y; z$	vi	$-x; 1-y; -z$
iii	$-x; -y; -z$	vii	$-x; 1-y; 1-z$
		viii	$1-x; 1-y; 1-z$
C(11)–O(16 ⁱ)	3.492(4)	Cl(12)–Cl(12 ^{iv})	3.285(2)
O(13)–C(7 ⁱ)	3.570(3)	C(11)–C(11 ^v)	3.523(7)
O(13)–C(15 ⁱ)	3.565(4)	C(15)–C(17 ^{vi})	3.641(5)
O(13)–O(16 ⁱ)	3.425(3)	C(9)–C(15 ^{vii})	3.670(5)
O(13)–C(17 ⁱ)	3.703(4)	C(15)–O(16 ^{vii})	3.612(5)
C(14)–Cl(18 ⁱⁱ)	3.393(3)	C(9)–Cl(18 ^{viii})	3.703(3)
C(4a)–O(13 ⁱⁱⁱ)	3.503(3)	C(11)–O(16 ^{viii})	3.797(4)
C(5)–O(13 ⁱⁱⁱ)	3.673(4)		

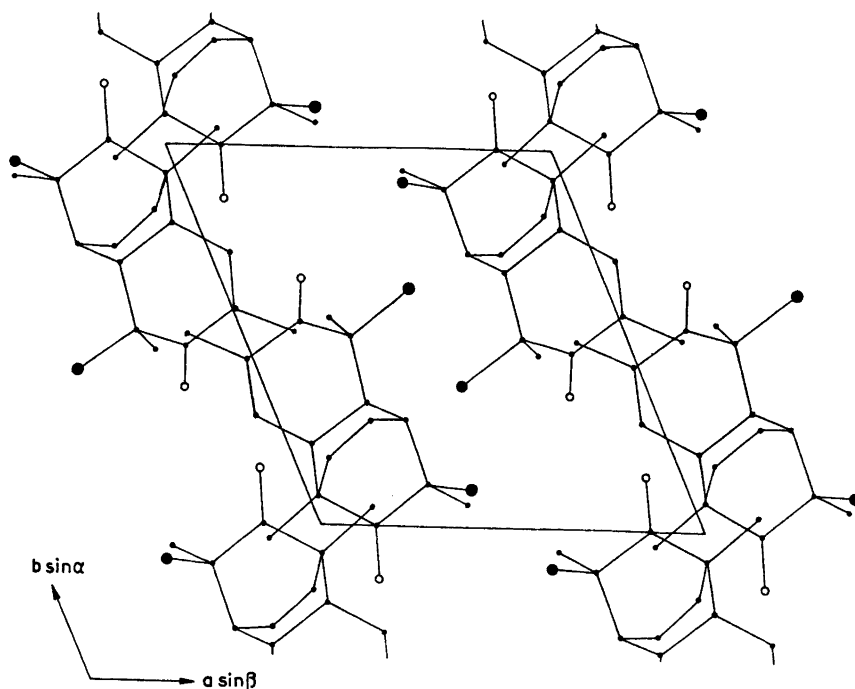


Fig. 4. The unit cell in projection along the c axis. \bullet , carbon; \circ , oxygen; \bullet , chlorine.

Table 7. Least-squares planes and deviations. The planes are described in terms of normalized equations in the orthogonal coordinate system (m, n, p) having $p \parallel c$, n in the bc plane and m in the abc octant.

Plane A	$-0.0307m - 0.3701n + 0.9285p = 0.4227$		
Plane B1	$-0.1205m + 0.3597n + 0.9252p = 1.7490$		
Plane B2	$0.1245m - 0.6029n + 0.7880p = 2.2637$		
Plane B3	$-0.2549m + 0.9538n + 0.1590p = 1.6508$		
Plane A		Plane B1	
Atom	Deviation (\AA)	Atom	Deviation (\AA)
C(4a) ^a	0.198	C(1) ^a	0.742
C(5)	0.001	C(2)	0.034
C(6)	0.001	C(3)	-0.035
C(7)	0.001	C(4) ^a	0.758
C(8) ^a	-0.650	C(4a)	0.034
C(8a)	0.001	C(8a)	-0.033
Plane B2		Plane B3	
Atom	Deviation (\AA)	Atom	Deviation (\AA)
C(1) ^a	-0.740	C(1) ^a	-0.667
C(2)	0.031	C(4) ^a	-0.729
C(3)	-0.031	C(4a)	0.021
C(4) ^a	-0.632	C(8a)	-0.021
C(9)	-0.036	C(9)	0.025
C(10)	0.036	C(10)	-0.025
Angles between the normals to the planes ($^\circ$).		A \wedge B1	60
		A \wedge B2	77
		A \wedge B3	78

^a These atoms were omitted from the calculations of the least-squares planes.

thermal motion have been made. The bond lengths and angles are comparable with values observed in similar structures.^{10,11} Bonds C(4)–C(4a) (1.589 Å) and C(4a)–C(8a) (1.560 Å) are significantly longer than the average value, 1.532 Å, of the other C(sp³)–C(sp³) bonds in the structure, probably in consequence of internal strain in the molecule. Some of the C(sp³)–C(sp³) bond lengths significantly exceed the standard value 1.501 ± 0.004 Å;¹² those of C(2)–C(3), C(3)–C(4), and C(7)–C(8) are 1.524, 1.525, and 1.523 Å, respectively, perhaps because each involves a highly substituted carbon atom. The effect of conjugation is apparent in the C(6)–C(7) bond, 1.471 Å, which is adjacent to double bonds. The two C–Cl bond lengths of 1.801 and 1.809 Å agree with the “normal” value of 1.81 Å given by Gade and Glusker.¹³

The arrangement of the molecules as viewed along the *a* and *c* axis is shown in Figs. 3 and 4, respectively. The molecules are held together in three-dimensional space by van der Waals forces. There are, however, some close intermolecular contacts, a C⋯Cl distance of 3.393 Å and a Cl⋯Cl distance of 3.285 Å (Table 6).

Parameters of least-squares planes through selected atoms of the molecule are given in Table 7. The four atoms C(5), C(6), C(7), and C(8a) in the cyclohexene ring are coplanar within ± 0.001 Å. Atoms C(4a) and C(8) deviate by 0.198 and –0.650 Å from the plane, respectively. Atom C(8) is significantly out of the plane as it is in the dimers of 2,4- and 2,6-dimethyl-*o*-quinol.^{10,11} The three rings containing the ethylene moiety are all boat-shaped. This rather strained three-ring system is connected to the cyclohexene ring in accordance with the endo rule; the angle between the normals to the planes containing atoms C(5), C(6), C(7), and C(8a) and atoms C(4a), C(8a), C(9), and C(10), respectively, is 77.7°.

All hydrogen atoms lie in chemically reasonable positions with apparent bond distances ranging from 0.80 to 1.05 Å, mean value 0.93 Å, in good agreement with values found by Stewart, Davidson and Simpson.⁸

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