HAMILTON, W. C. (1964). *Statistics in Physical Science*, pp. 157–162. New York: Ronald.

HAUPTMAN, H., KARLE, I. L. & KARLE, J. (1960). *Acta Cryst.* 13, 451–453.

HINGERTY, B., SUBRAMANIAN, E., STELLMAN, S. D., BROYDE, S. B., SATO, T. & LANGRIDGE, R. (1975). *Biopolymers*, 16, 227–236.

LINDAHL, T., ADAMS, A. & FRESCO, J. R. (1966). *Proc. Natl. Acad. Sci. U.S.* 55, 941–948.

NAGASHIMA, N. & IITAKA, Y. (1968). *Acta Cryst.* B24, 1136–1138.

POWELL, M. (1964). *Comput. J.* 7, 155–162.

ROGERS, D. (1965). *Computing Methods in Crystallography*. Edited by J. S. ROLLETT, pp. 117–132. New York: Pergamon Press.

ROSENBERG, J. M., SEEMAN, N. C., DAY, R. O. & RICH, A. (1973). Amer. Cryst. Assoc. Meeting, Vol. 1, Ser. 2, 130.

RUBIN, J., BRENNAN, T. & SUNDARALINGAM, M. (1972). *Biochemistry*, 11, 3112–3128.

SIEKER, L. C. & JENSEN, L. H. (1974). Amer. Cryst. Assoc. Meeting, Vol. 2, Ser. 2, 79.

SHEFTER, E. & TRUEBLOOD, K. N. (1965). *Acta Cryst.* 18, 1067–1077.

STELLMAN, S. D., HINGERTY, B., BROYDE, S. B., SUBRAMANIAN, E., SATO, T. & LANGRIDGE, R. (1973). *Biopolymers*, 12, 2731–2750.

STOUT, G. H. & JENSEN, L. H. (1968). *X-ray Structure Determination: A Practical Guide*, p. 321. New York: Macmillan.

SUNDARALINGAM, M. (1969). *Biopolymers*, 7, 821–860.

SUNDARALINGAM, M. (1973). *Jerusalem Symposia on Quantum Chemistry and Biochemistry*. Edited by E. BERGMANN & B. PULLMAN, Vol. V, pp. 417–455. New York: Academic Press.

SUSSMAN, J. (1971). Ph.D. Dissertation, Massachusetts Institute of Technology.

SUSSMAN, J., SEEMAN, N. C., KIM, S. H. & BERMAN, H. J. (1972). *J. Mol. Biol.* 66, 403–421.

TROTTER, J. (1965). Univ. British Columbia, Vancouver, Canada.

TRUEBLOOD, K. N., HORN, P. & LUZZATI, V. (1961). *Acta Cryst.* 14, 965–982.

WATSON, J. D. & CRICK, F. H. C. (1953). *Nature, Lond.* 171, 737–738.

WUENSCH, B. J. & PREWITT, C. T. (1965). *Z. Kristallogr.* 122, 24–59.

Structural Studies of Polychlorinated Hydrocarbons. II.* Hexachloroxanthene and Hexachloroxanthone

BY M. SÖDERHOLM AND U. SONNERSTAM

University of Stockholm, S-104 05 Stockholm, Sweden

AND R. NORRESTAM† AND T.-B. PALM

Department of Structural Chemistry, Arrhenius Laboratory, University of Stockholm, S-104 05 Stockholm, Sweden

The crystal structures of the two related compounds 1,3,4,6,7,9-hexachloroxanthene ($C_{13}H_4Cl_6O$) and 1,3,4,6,7,9-hexachloroxanthone ($C_{13}H_2Cl_6O_2$) have been determined by X-ray diffraction techniques. Both structures are monoclinic, space group $P2_1/n$ with $a = 16.114$ (4), $b = 11.950$ (2), $c = 7.254$ (1) Å, $\beta = 92.29$ (2)°, $Z = 4$, $D_x = 1.85$ g cm^{-3} and $a = 15.960$ (3), $b = 11.833$ (2), $c = 7.348$ (2) Å, $\beta = 93.11$ (2)°, $Z = 4$, $D_x = 1.93$ g cm^{-3}, for the xanthene and xanthone derivatives respectively. Both structures were refined to R values of 0.06. The molecules of both compounds were found to be almost planar, with the largest deviation of the nonhydrogen atoms from planarity being shown by the pyranoid ring of the hexachloroxanthene molecule. The crystal structures are built up by successive layers (separation 3.4 Å) of nearly coplanar molecules, where the centroids of the molecules are approximately hexagonally distributed within one layer.

Introduction

The structure investigations of the two related hexachloro-substituted xanthene derivatives, 1,3,4,6,7,9-hexachloroxanthene and 1,3,4,6,7,9-hexachloroxanthone (*cf.* Fig. 1), reported here are part of a research project

---

* Part I: Norrestam, Palm, Jensen & Sundström (1976).

† Present address: Chemistry Department B, The Technical University of Denmark, DTH 301, DK-2800 Lyngby, Denmark.

concerning structural studies of polychlorinated hydrocarbons. Suitable specimens (Göthe & Wachtmeister, 1972; Eklund-Westlin, Göthe & Wachtmeister, 1976) were kindly supplied by Dr R. Göthe, University of Stockholm. The following experimental details apply to studies of both compounds. Single crystals suitable for X-ray studies were obtained by slow evaporation of ethanolic solutions. Preliminary examination of the crystals by photographic X-ray techniques revealed monoclinic symmetry with the systematic extinctions characteristic of the centrosymmetric space group

$P2_1/n$. Unit-cell parameters with e.s.d.'s were determined both from Guinier powder photographs taken with potassium chloride as internal standard, and from diffractometer measurements of the selected single crystals. All deviations between these two sets of parameters were within three e.s.d.'s. Full three-dimensional single-crystal X-ray diffraction data were collected by the ω–2θ scan technique on a computer-controlled four-circle diffractometer (PW 1100) with graphite-monochromatized Cu $K\alpha$ radiation.

The intensity data collected were corrected for geometrical as well as for absorption effects. The structures were solved quite straightforwardly by conventional direct methods (Karle & Karle, 1966). The resultant E maps clearly showed the positions of all nonhydrogen atoms. The structural models, including the predictable hydrogen positions, were refined by full-matrix least-squares methods. In the final refinement cycles, with Hughes's weighting scheme, the non-hydrogen atoms were allowed to vibrate anisotropically and the hydrogens isotropically. All parameters shifted less than one e.s.d. in the last cycle. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Some further details of the computations are given in Table 1. The final structural parameters, with e.s.d.'s, are listed in Tables 2, 3, 4 and 5.*

---

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31847 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Discussion

The molecular arrangements in the unit cells are shown in Figs. 2 and 3. The crystal structures may be regarded as comprised of successive layers of approximately coplanar fairly close-packed molecules with the molecular centroids within one layer in hexagonal arrangements. The set of planes containing the molecules has

Table 2. *Hexachloroxanthene: fractional atomic coordinates and hydrogen isotropic temperature factors with e.s.d.'s*

	x	y	z	B (Å^2)
O	0·4285 (3)	0·4821 (5)	0·6838 (8)	
Cl(1)	0·2720 (3)	0·5764 (2)	0·5711 (4)	
Cl(3)	0·4195 (2)	0·9717 (2)	0·7004 (5)	
Cl(4)	0·5856 (2)	0·8469 (2)	0·8197 (4)	
Cl(6)	0·7296 (2)	0·4547 (2)	0·9289 (4)	
Cl(7)	0·7070 (2)	0·1929 (2)	0·9020 (4)	
Cl(9)	0·3898 (2)	0·2513 (2)	0·6714 (4)	
C(1)	0·3587 (5)	0·6520 (7)	0·6405 (12)	
C(2)	0·3545 (6)	0·7682 (7)	0·6427 (12)	
C(3)	0·4260 (6)	0·8265 (7)	0·6977 (12)	
C(4)	0·4982 (5)	0·7730 (7)	0·7504 (12)	
C(5)	0·5825 (5)	0·5954 (7)	0·7996 (14)	
C(6)	0·6357 (5)	0·3990 (7)	0·8551 (11)	
C(7)	0·6258 (5)	0·2821 (7)	0·8466 (12)	
C(8)	0·5509 (5)	0·2378 (7)	0·7907 (12)	
C(9)	0·4853 (5)	0·3051 (7)	0·7401 (12)	
C(1a)	0·4318 (5)	0·5982 (6)	0·6914 (11)	
C(5a)	0·5027 (5)	0·6556 (6)	0·7458 (11)	
C(6a)	0·5703 (5)	0·4692 (6)	0·8005 (11)	
C(9a)	0·4965 (5)	0·4215 (6)	0·7423 (11)	
H(2)	0·299 (8)	0·802 (10)	0·617 (17)	8·5 (33)
H(8)	0·543 (5)	0·153 (6)	0·787 (10)	3·9 (15)
H(5)1	0·608 (6)	0·624 (7)	0·933 (13)	5·0 (21)
H(5)2	0·631 (6)	0·619 (8)	0·704 (13)	5·7 (21)

Table 1. *Some relevant details of the data collection and of the structure determinations*

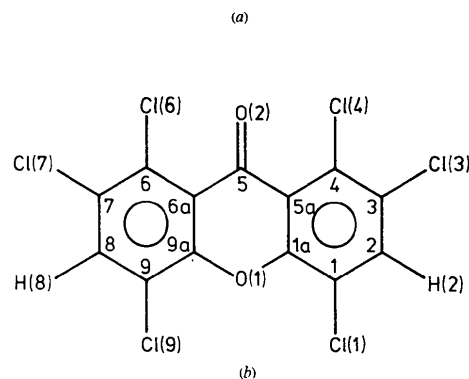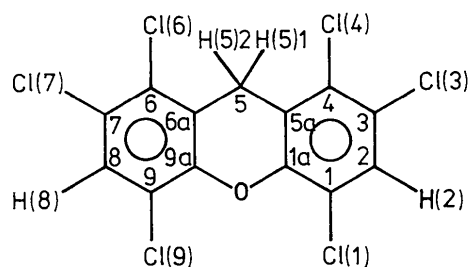|	Hexachloroxanthene	Hexachloroxanthone
Crystal size	0·16 × 0·16 × 0·12 mm	0·08 × 0·08 × 0·25 mm
Linear absorption coefficient (μ)	114·6 cm^{-1}	116·2 cm^{-1}
Total number of measured independent reflexions	1914	1757
Acceptance limit $\sigma(I_{net})/I_{net}$	0·303	0·25
Accepted reflexions	1073	1180
Normalized structure factors used	($|E| \geq 1\cdot7$) 171	($|E| \geq 1\cdot4$) 250
Best triple relations	885	1800
Number of solutions of triple relations	2	4
Parameters to describe the final structural model	197	198
Final linear R value based on accepted reflexions (strongest reflexion, $\overline{2}02$, omitted)	0·064	0·061

Fig. 1. Schematic structural formulae for (a) hexachloroxanthene and (b) hexachloroxanthone.

Table 3. *Hexachloroxanthene: anisotropic thermal parameters* ($\times 10^4$) *with e.s.d.'s*

The temperature factor expression used is
$$\exp\,[-(h^2b_{11}+k^2b_{22}+l^2b_{33}+hkb_{12}+hlb_{13}+klb_{23})].$$

	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
O	27 (3)	52 (5)	263 (16)	−3 (6)	−24 (10)	−1 (13)
Cl(1)	26 (1)	67 (2)	220 (6)	−5 (2)	−16 (4)	−3 (5)
Cl(3)	50 (2)	43 (2)	452 (10)	7 (3)	10 (6)	−26 (6)
Cl(4)	36 (1)	57 (2)	279 (7)	−26 (2)	0 (4)	−33 (6)
Cl(6)	25 (1)	83 (2)	213 (6)	−1 (2)	−17 (4)	−12 (6)
Cl(7)	36 (1)	77 (2)	295 (7)	43 (3)	0 (4)	12 (6)
Cl(9)	32 (1)	51 (2)	253 (6)	−14 (2)	−14 (2)	11 (6)
C(1)	22 (4)	58 (7)	202 (21)	−1 (8)	6 (13)	1 (19)
C(2)	32 (4)	58 (7)	167 (20)	14 (9)	13 (14)	12 (19)
C(3)	39 (5)	45 (7)	250 (25)	6 (9)	32 (16)	−16 (22)
C(4)	34 (4)	56 (7)	189 (22)	−16 (9)	31 (14)	−17 (20)
C(5)	24 (4)	53 (7)	230 (25)	0 (8)	−1 (15)	−23 (20)
C(6)	27 (4)	71 (8)	143 (20)	−9 (8)	45 (13)	19 (18)
C(7)	23 (4)	68 (7)	159 (20)	12 (8)	15 (13)	−3 (18)
C(8)	33 (4)	51 (7)	178 (21)	12 (9)	48 (14)	10 (20)
C(9)	39 (4)	44 (6)	158 (19)	1 (8)	38 (14)	3 (18)
C(1a)	29 (4)	40 (6)	162 (20)	−9 (8)	14 (13)	12 (17)
C(5a)	35 (4)	45 (7)	154 (19)	−10 (8)	29 (14)	3 (17)
C(6a)	19 (4)	53 (7)	157 (20)	1 (8)	37 (13)	−7 (17)
C(9a)	24 (4)	54 (7)	160 (19)	17 (8)	9 (13)	13 (18)

the indices ($\overline{2}02$) with the interplanar spacings of 3·35 and 3·39 Å for the hexachloroxanthene and hexachloroxanthone structures respectively. The intermolecular distances listed in Tables 6 and 7 predominantly indicate van der Waals interactions between molecules in both structures.

Tables 8 and 9 give the deviations of the atoms from least-squares planes through the ring systems of the two molecules. Both molecules are roughly coplanar as regards the nonhydrogen atoms, despite the fact that the C(5) and O atoms of the pyranoid ring in the xanthene derivative are expected to be tetrahedrally sp^3-hybridized (*cf.* Fig. 4). The largest deviations from the

least-squares planes, 0·10 for the xanthene and 0·15 Å for the xanthone molecules, are exhibited by the Cl substituents. However, there are other small but significant deviations from planarity. To analyse these in more detail, all the torsion angles and their e.s.d.'s (Stanford & Waser, 1972) within the ring systems were calculated. The torsion angles in the benzenoid rings of both molecules are all less than three e.s.d.'s, while some in the pyranoid rings deviate significantly from 0°. Thus, in the xanthene derivative the torsion angles within the pyranoid ring about the bonds C(5a)–C(5), C(5)–C(6a), C(9a)–O and O–C(1a) differ significantly

Table 4. *Hexachloroxanthone: fractional atomic coordinates and hydrogen isotropic temperature factors with e.s.d.'s*

	x	y	z	B (Å²)
O(1)	0·3031 (4)	0·4925 (5)	0·5574 (9)	
O(2)	0·5152 (5)	0·6494 (6)	0·7603 (13)	
Cl(1)	0·1462 (2)	0·5821 (3)	0·4319 (4)	
Cl(3)	0·2929 (2)	0·9839 (2)	0·5388 (4)	
Cl(4)	0·4568 (2)	0·8702 (3)	0·6866 (4)	
Cl(6)	0·6030 (2)	0·4483 (2)	0·8508 (4)	
Cl(7)	0·5697 (2)	0·1908 (3)	0·8271 (4)	
Cl(9)	0·2670 (2)	0·2622 (2)	0·4988 (4)	
C(1a)	0·3063 (6)	0·6075 (7)	0·5643 (12)	
C(1)	0·2329 (6)	0·6602 (8)	0·5005 (13)	
C(2)	0·2290 (6)	0·7763 (8)	0·4948 (13)	
C(3)	0·3003 (6)	0·8372 (7)	0·5544 (14)	
C(4)	0·3727 (6)	0·7865 (8)	0·6208 (12)	
C(5a)	0·3771 (6)	0·6669 (8)	0·6293 (12)	
C(5)	0·4518 (6)	0·6032 (8)	0·7007 (14)	
C(6a)	0·4435 (5)	0·4769 (8)	0·6943 (12)	
C(6)	0·5075 (6)	0·4017 (8)	0·7574 (12)	
C(7)	0·4946 (6)	0·2862 (8)	0·7456 (13)	
C(8)	0·4212 (6)	0·2425 (10)	0·6662 (14)	
C(9)	0·3589 (6)	0·3134 (8)	0·6020 (12)	
C(9a)	0·3706 (6)	0·4297 (8)	0·6179 (12)	
H(2)	0·168 (6)	0·824 (7)	0·441 (11)	3·1 (1·9)
H(8)	0·413 (6)	0·173 (7)	0·662 (11)	2·6 (1·9)

Table 5. *Hexachloroxanthone: anisotropic thermal parameters* ($\times 10^4$) *for the nonhydrogen atoms with e.s.d.'s*

The temperature factor expression used is $\exp\,[-(h^2b_{11}+k^2b_{22}+l^2b_{33}+hkb_{12}+hlb_{13}+klb_{23})].$

	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
O(1)	25 (3)	55 (5)	238 (15)	2 (7)	−36 (10)	−11 (15)
O(2)	37 (4)	66 (6)	510 (26)	−21 (8)	−125 (15)	−23 (21)
Cl(1)	27 (1)	65 (2)	331 (8)	−1 (3)	−91 (5)	−14 (7)
Cl(3)	44 (2)	44 (2)	317 (8)	6 (3)	−59 (5)	16 (6)
Cl(4)	35 (2)	57 (2)	280 (7)	−18 (3)	−60 (5)	−25 (6)
Cl(6)	25 (1)	72 (3)	226 (6)	0 (3)	−61 (4)	−8 (6)
Cl(7)	33 (2)	71 (2)	256 (6)	30 (3)	−53 (5)	46 (6)
Cl(9)	29 (1)	53 (2)	272 (7)	−8 (3)	−70 (4)	−15 (6)
C(1a)	26 (4)	45 (8)	166 (20)	0 (9)	−11 (14)	1 (19)
C(1)	27 (4)	52 (8)	210 (23)	18 (9)	−29 (15)	2 (20)
C(2)	22 (4)	54 (8)	220 (22)	0 (9)	−19 (15)	16 (21)
C(3)	39 (5)	38 (7)	216 (23)	8 (9)	−16 (17)	13 (19)
C(4)	34 (5)	63 (9)	152 (20)	−2 (10)	−13 (15)	4 (21)
C(5a)	30 (5)	60 (9)	158 (21)	1 (9)	−11 (15)	−10 (19)
C(5)	33 (5)	53 (9)	194 (22)	−7 (10)	−19 (16)	−7 (21)
C(6a)	27 (4)	50 (7)	141 (18)	2 (9)	−11 (14)	−8 (19)
C(6)	23 (4)	79 (10)	146 (19)	0 (10)	−32 (14)	4 (21)
C(7)	27 (4)	60 (8)	177 (20)	9 (9)	−25 (15)	19 (21)
C(8)	33 (5)	50 (9)	222 (24)	12 (11)	−29 (16)	18 (24)
C(9)	23 (4)	55 (8)	180 (22)	−1 (9)	−35 (15)	−34 (21)
C(9a)	25 (4)	63 (8)	162 (20)	9 (10)	−21 (15)	−18 (21)

from 0°, *viz* 4·8, −5·3, 3·3 and −3·8° (e.s.d.'s 1·1°) respectively. This distribution of ring torsion angles indicates that the hexachloroxanthene pyranoid ring deviates from planarity towards a boat conformation. In the hexachloroxanthone pyranoid ring only the

torsion angle about the C(6a)–C(9a) bond, 4·1° (e.s.d. 1·3°), indicates a significant out-of-plane deviation: a very small twist along the elongation of the xanthone molecule is suggested. Due to the geometry of the pyranoid rings and possibly also to steric factors as-
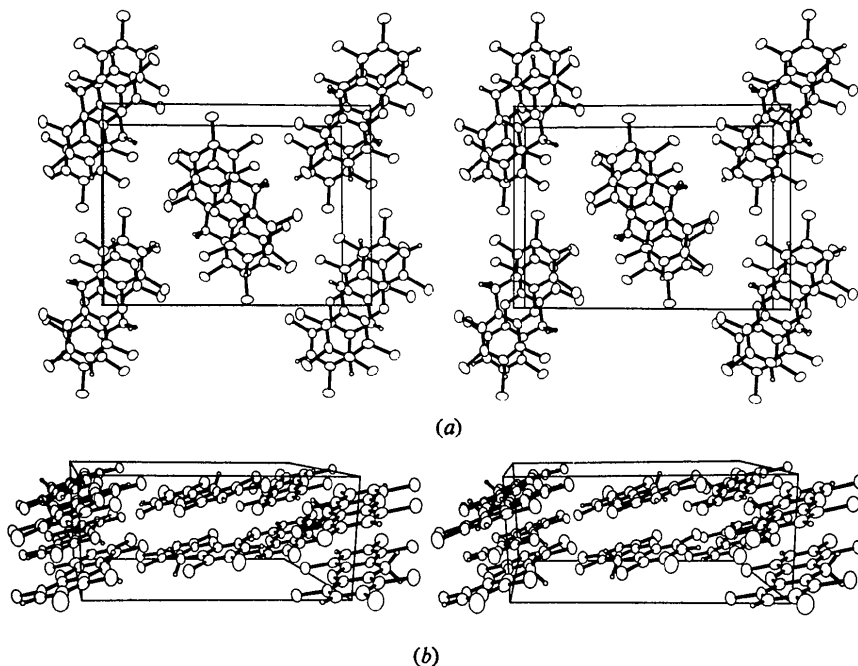

(a)

(b)

Fig. 2. Stereoscopic packing diagrams (origin in upper right corner) of the hexachloroxanthene structure viewed (a) along the c axis and (b) along the b axis.
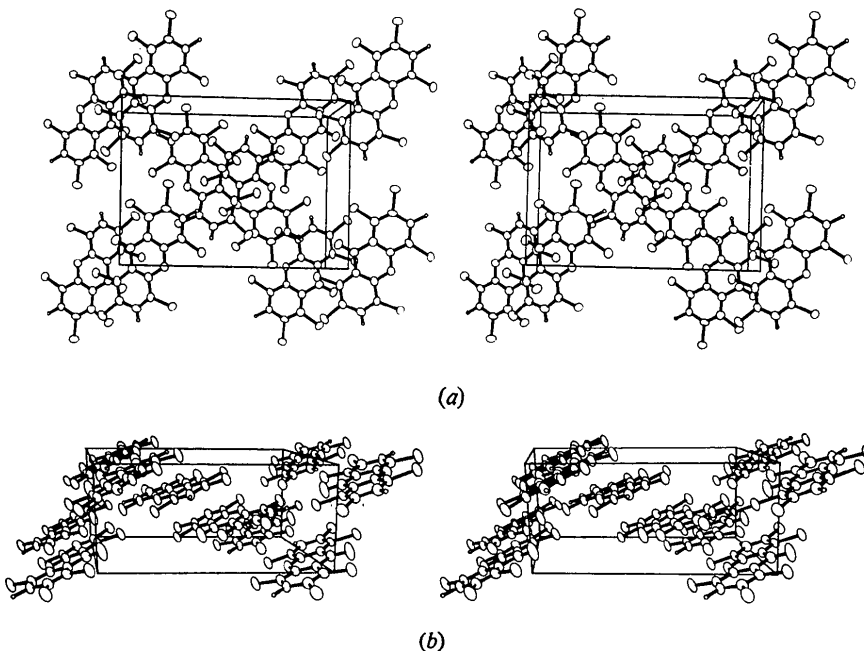

(a)

(b)

Fig. 3. Stereoscopic packing diagrams (origin in upper right corner) of the hexachloroxanthone structure viewed (a) along the c axis and (b) along the b axis.

Table 6. *Hexachloroxanthene: intermolecular distances less than 3·6 Å with e.s.d.'s*

O · · · · · C(6A)[i]	3·562 (10)	Cl(7) · · · Cl(9)[ii]	3·534 (4)
O · · · · · C(9A)[i]	3·555 (10)	Cl(9) · · · H(5)2[i]	3·139 (88)
O · · · · · H(5)2[i]	3·171 (89)	C(2) · · · · C(8)[i]	3·549 (12)
Cl(1) · · · Cl(4)[iv]	3·570 (4)	C(3) · · · · H(8)[i]	3·579 (69)
Cl(1) · · · Cl(7)[v]	3·586 (4)	C(9) · · · · C(5a)[i]	3·579 (12)
Cl(1) · · · C(6)[i]	3·496 (9)	C(1a) · · · C(9a)[i]	3·402 (11)
Cl(1) · · · H(5)2[i]	3·478 (87)	H(2) · · · H(5)1[iv]	3·414 (144)
Cl(6) · · · H(2)[iii]	3·383 (116)		

Symmetry code

None	x, y, z	(iii)	$\frac{1}{2}+x, 1\frac{1}{2}-y, \frac{1}{2}+z$
(i)	$1-x, 1-y, 1-z$	(iv)	$-\frac{1}{2}+x, 1\frac{1}{2}-y, -\frac{1}{2}+z$
(ii)	$\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$	(v)	$-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$

Table 7. *Hexachloroxanthone: intermolecular distances less than 3·6 Å with e.s.d.'s*

O(1) · · · Cl(6)[i]	3·496 (7)	Cl(7) · · · Cl(9)[ii]	3·375 (5)
O(2) · · · C(8)[i]	3·583 (14)	Cl(7) · · · C(3)[i]	3·593 (10)
O(2) · · · C(9)[i]	3·450 (13)	Cl(7) · · · C(4)[i]	3·476 (9)
O(2) · · · C(9a)[i]	3·532 (13)	C(5) · · · C(6)[i]	3·463 (14)
Cl(1) · · · Cl(4)[iv]	3·480 (4)	C(5) · · · · C(6a)[i]	3·555 (13)
Cl(1) · · · Cl(7)[v]	3·522 (5)	C(6) · · · · C(5a)[i]	3·562 (13)
Cl(3) · · · Cl(6)[iv]	3·360 (4)	C(7) · · · · C(5a)[i]	3·564 (13)
Cl(4) · · · C(8)[i]	3·583 (11)	C(6a) · · C(6a)[i]	3·501 (17)
Cl(6) · · · C(1a)[i]	3·510 (9)		

O(2) · · · H(2)[iii]	2·73 (9)
Cl(4) · · · H(8)[i]	3·43 (8)
Cl(6) · · · H(2)[iii]	2·95 (8)
Cl(9) · · · H(2)[vi]	3·53 (8)

Symmetry code

None	x, y, z	(iv)	$-\frac{1}{2}+x, 1\frac{1}{2}-y, -\frac{1}{2}+z$
(i)	$1-x, 1-y, 1-z$	(v)	$-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$
(ii)	$\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$	(vi)	$1\frac{1}{2}-x, \frac{1}{2}+y, 1\frac{1}{2}-z$
(iii)	$\frac{1}{2}+x, 1\frac{1}{2}-y, \frac{1}{2}+z$		

Table 8. *Hexachloroxanthene: deviations of the non-hydrogen atoms from least-squares planes*

The atoms indicated with an asterisk were omitted from the calculations of the planes.

O	−0·066 Å	C(4)	0·029 Å
Cl(1)*	−0·024	C(5)	−0·074
Cl(3)*	0·097	C(6)	0·030
Cl(4)*	0·049	C(7)	0·024
Cl(6)*	0·047	C(8)	0·030
Cl(7)*	−0·020	C(9)	0·025
Cl(9)*	0·051	C(1a)	−0·031
C(1)	0·001	C(5a)	−0·027
C(2)	0·039	C(6a)	−0·004
C(3)	0·043	C(9a)	−0·019

R.m.s. deviaton of the atoms from the plane 0·027 Å.

sociated with the chlorine substitution, both molecules are slightly bent within the molecular planes. This bending gives rise to different Cl(1) · · · Cl(9) and Cl(4) · · · Cl(6) intramolecular distances, 4·37 and 5·28 Å in hexachloroxanthene and 4·26 and 5·61 Å in hexachloroxanthone.

Table 9. *Hexachloroxanthone: deviations of the non-hydrogen atoms from a least-squares plane*

The atoms indicated with an asterisk were omitted from the calculation of the plane.

C(1a)	0·014 Å	C(9)	−0·055 Å
C(1)	0·055	C(9a)	−0·029
C(2)	0·033	O(1)	−0·006
C(3)	−0·028	*Cl(1)	0·155
C(4)	−0·043	*Cl(3)	−0·095
C(5a)	−0·008	*Cl(4)	−0·148
C(5)	−0·003	*O(2)	−0·012
C(6a)	0·014	*Cl(6)	0·046
C(6)	0·033	*Cl(7)	0·116
C(7)	0·043	*Cl(9)	−0·158
C(8)	−0·018		

R.m.s. deviation of the atoms from the plane 0·024 Å.

Table 10. *Hexachloroxanthene: intramolecular bond distances (Å) and angles (°) with e.s.d.'s*

Cl(1)–C(1)	1·722 (9)	C(5)—C(6a)	1·521 (11)
Cl(3)–C(3)	1·738 (9)	C(6a)–C(6)	1·392 (11)
Cl(4)–C(4)	1·720 (9)	C(6)—C(7)	1·406 (12)
Cl(6)–C(6)	1·719 (9)	C(7)—C(8)	1·366 (12)
Cl(7)–C(7)	1·722 (9)	C(8)—C(9)	1·366 (12)
Cl(9)–C(9)	1·722 (9)	C(9)—C(9a)	1·403 (11)
C(1a)–C(1)	1·380 (11)	C(9a)–C(6a)	1·370 (11)
C(1)—C(2)	1·390 (12)	C(9a)–O	1·367 (9)
C(2)—C(3)	1·391 (13)	O——C(1a)	1·390 (9)
C(3)—C(4)	1·369 (12)	H(2)—C(2)	1·00 (12)
C(4)—C(5a)	1·406 (11)	H(8)—C(8)	1·02 (8)
C(5a)–C(1a)	1·376 (11)	H(5)1–C(5)	1·09 (10)
C(5a)–C(5)	1·510 (11)	H(5)2–C(5)	1·11 (9)

O——C(1a)–C(5a)	122·6 (7)	H(5)2–C(5)—C(6a)	110 (5)
O——C(1a)–C(1)	115·1 (7)	H(5)1–C(5)—H(5)2	103 (7)
C(5a)–C(1a)–C(1)	122·4 (7)	C(5)—C(6a)–C(9a)	121·4 (7)
C(1a)–C(1)—Cl(1)	120·5 (7)	C(5)—C(6a)–C(6)	120·2 (8)
C(1a)–C(1)—C(2)	120·2 (8)	C(9a)–C(6a)–C(6)	118·4 (7)
Cl(1) –C(1)—C(2)	119·2 (7)	C(6a)–C(6)—Cl(6)	120·2 (7)
C(1)—C(2)—H(2)	117 (7)	C(6a)–C(6)—C(7)	120·2 (8)
C(1)—C(2)—C(3)	117·6 (9)	Cl(6) –C(6)—C(7)	119·7 (7)
H(2)—C(2)—C(3)	125 (7)	C(6)—C(7)—Cl(7)	121·4 (7)
C(2)—C(3)—Cl(3)	117·0 (7)	C(6)—C(7)—C(8)	119·7 (8)
C(2)—C(3)—C(4)	122·1 (8)	Cl(7) –C(7)—C(8)	118·9 (7)
Cl(3) –C(3)—C(4)	121·0 (7)	C(7)—C(8)—H(8)	120 (4)
C(3)—C(4)—Cl(4)	121·2 (7)	C(7)—C(8)—C(9)	121·1 (8)
C(3)—C(4)—C(5a)	120·3 (8)	H(8)—C(8)—C(9)	119 (4)
Cl(4) –C(4)—C(5a)	118·5 (7)	C(8)—C(9)—Cl(9)	122·0 (7)
C(4)—C(5a)–C(1a)	117·4 (8)	C(8)—C(9)—C(9a)	118·9 (8)
C(4)—C(5a)–C(5)	120·9 (7)	Cl(9) –C(9)—C(9a)	119·1 (7)
C(1a)–C(5a)–C(5)	121·7 (7)	C(9)—C(9a)–C(6a)	121·7 (8)
C(5a)–C(5)—H(5)1	112 (5)	C(9)—C(9a)–O	114·9 (7)
C(5a)–C(5)—H(5)2	110 (5)	C(6a)–C(9a)–O	123·4 (7)
C(5a)–C(5)—C(6a)	111·4 (7)	C(9a)–O——C(1a)	119·2 (6)
H(5)1–C(5)—C(6a)	110 (5)		

The average values of the intramolecular C–C bond distances (cf. Tables 10 and 11) within the benzenoid rings, 1·385 (15) and 1·388 (16) Å for xanthene and xanthone respectively, agree well with the normal aromatic value, 1·395 Å (Sutton, 1965). The C–Cl bond distances average 1·724 (7) and 1·726 (10) Å respectively, in close agreement with the 1·74 (2) Å found in similar compounds (De Lacy & Kennard, 1972). The C(6a)–C(5) and C(5a)–C(5) bond distances,

Table 11. *Hexachloroxanthone: intramolecular bond distances (Å) and angles (°) with e.s.d.'s*

Cl(1)–C(1)	1·717 (10)	C(5)–O(2)	1·211 (13)	
Cl(3)–C(3)	1·743 (9)	C(5)–C(6a)	1·501 (13)	
Cl(4)–C(4)	1·717 (10)	C(6a)–C(9a)	1·383 (13)	
Cl(6)–C(6)	1·727 (10)	C(6a)–C(6)	1·413 (13)	
Cl(7)–C(7)	1·729 (10)	C(6)–C(7)	1·385 (13)	
Cl(9)–C(9)	1·724 (10)	C(7)–C(8)	1·380 (14)	
C(1a)–C(1)	1·386 (13)	C(8)–C(9)	1·365 (14)	
C(1a)–C(5a)	1·394 (13)	C(9)–C(9a)	1·393 (13)	
C(1)–C(2)	1·376 (13)	C(9a)–O(1)	1·363 (11)	
C(2)–C(3)	1·396 (13)	O(1)–C(1a)	1·362 (10)	
C(3)–C(4)	1·368 (13)	C(2)–H(2)	1·17 (9)	
C(4)–C(5a)	1·417 (13)	C(8)–H(8)	0·84 (9)	
C(5a)–C(5)	1·481 (13)			

O(1)–C(1a)–C(1)	114·0 (8)	O(2)–C(5)–C(6a)	122·0 (9)
O(1)–C(1a)–C(5a)	123·0 (9)	C(5)–C(6a)–C(6)	123·8 (8)
C(5a)–C(1a)–C(1)	123·0 (9)	C(5)–C(6a)–C(9a)	119·0 (8)
C(1a)–C(1)–Cl(1)	120·7 (8)	C(9a)–C(6a)–C(6)	117·2 (9)
C(1a)–C(1)–C(2)	119·6 (9)	C(6a)–C(6)–Cl(6)	122·4 (8)
Cl(1)–C(1)–C(2)	119·7 (8)	C(6a)–C(6)–C(7)	119·9 (9)
C(1)–C(2)–H(2)	121 (4)	Cl(6)–C(6)–C(7)	117·7 (8)
C(1)–C(2)–C(3)	118·2 (9)	C(6)–C(7)–Cl(7)	121·6 (8)
H(2)–C(2)–C(3)	120 (4)	C(6)–C(7)–C(8)	121·1 (10)
C(2)–C(3)–Cl(3)	116·2 (8)	Cl(7)–C(7)–C(8)	117·3 (8)
C(2)–C(3)–C(4)	122·9 (9)	C(7)–C(8)–H(8)	121 (6)
Cl(3)–C(3)–C(4)	120·9 (8)	C(7)–C(8)–C(9)	120·1 (11)
C(3)–C(4)–Cl(4)	118·7 (8)	H(8)–C(8)–C(9)	119 (8)
C(3)–C(4)–C(5a)	119·6 (9)	C(8)–C(9)–Cl(9)	121·1 (8)
Cl(4)–C(4)–C(5a)	121·8 (8)	C(8)–C(9)–C(9a)	119·1 (10)
C(4)–C(5a)–C(5)	124·1 (9)	Cl(9)–C(9)–C(9a)	119·4 (8)
C(4)–C(5a)–C(1a)	116·8 (9)	C(9)–C(9a)–O(1)	114·3 (9)
C(1a)–C(5a)–C(5)	119·1 (9)	C(9)–C(9a)–C(6a)	122·8 (9)
C(5a)–C(5)–O(2)	122·6 (9)	C(6a)–C(9a)–O(1)	123·0 (9)
C(5a)–C(5)–C(6a)	115·4 (8)	C(8a)–O(1)–C(1a)	120·4 (8)

which are about 1·52 Å in hexachloroxanthene, are shortened to about 1·49 Å in hexachloroxanthone, probably due to the adjacent C(5)–O(2) double bond. The observed C–O distances in the pyranoid rings, ranging from 1·36 to 1·39 Å, agree with the usual single-bond distance of 1·37 Å (Sutton, 1965).

This investigation was performed during a course in structural chemistry given at the University of Stockholm. The authors are indebted to Professor Peder Kierkegaard for his continous interest in this study. They are also indebted to Dr Rolf Göthe at the Wallenberg Laboratory, Stockholm, for supplying the crystals.
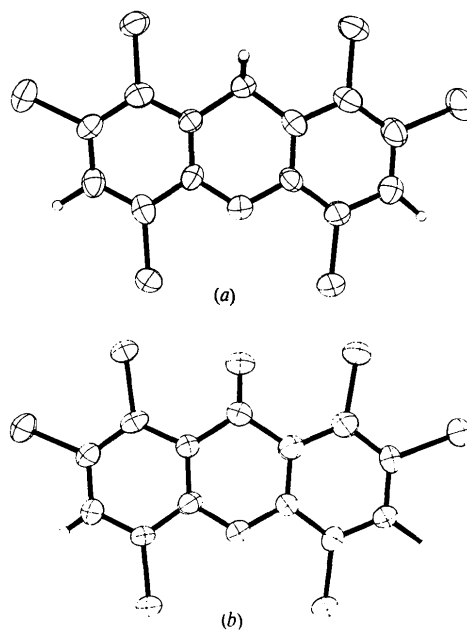

Fig. 4. The molecular geometries of (*a*) hexachloroxanthene and (*b*) hexachloroxanthone. The nonhydrogen atoms are represented by their thermal ellipsoids, the hydrogens are drawn with constant radii.

References

DE LACY, T. P. & KENNARD, C. H. L. (1972). *J. Chem. Soc. Perkin* II, pp. 2141–2153.
EKLUND-WESTLIN, U., GÖTHE, R. & WACHTMEISTER, C. A. (1976). To be published.
GÖTHE, R. & WACHTMEISTER, C. A. (1972). *Acta Chem. Scand.* **26**, 2523.
International Tables for X-ray Crystallography (1974). Vol. IV, pp. 72–102. Birmingham: Kynoch Press.
KARLE, J. & KARLE, I. L. (1966). *Acta Cryst.* **21**, 849–859.
NORRESTAM, R., PALM, T.-B., JENSEN, S. & SUNDSTRÖM, G. (1976). *Acta Chem. Scand.* In the press.
STANFORD, R. H. JR & WASER, J. (1972). *Acta Cryst.* **A28**, 213–215.
SUTTON, L. E. (1965). *Tables of Interatomic Distances and Configuration in Molecules and Ions.* London: The Chemical Society.