

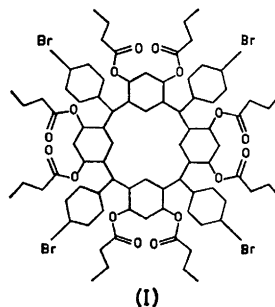
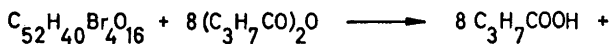
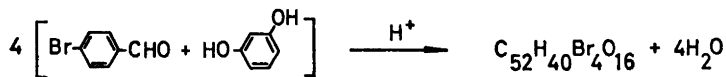
The Crystal and Molecular Structure of the Synthetic Tetramer $C_{84}H_{84}Br_4O_{16}$

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An octabutyrate, $C_{52}H_{40}Br_4O_{16}$, obtained from a condensation product of resorcinol and benzaldehyde, has been subject to an X-ray single crystal analysis. There is one molecule per asymmetric unit (spacegroup $P\bar{1}$). The four carbon atoms of the condensation centra are nearly coplanar and the bromophenyl groups are all directed to the same side of that plane. Most butyrate chain atoms show very high vibrational amplitudes, which are interpreted as due to slight disorder.

In order to establish stereochemical relations of principal interest concerning the condensation of aldehydes with phenols we undertook an X-ray analysis of the tetramer $[C_6H_4Br \cdot CH \cdot C_6H_2(O \cdot CO \cdot C_3H_7)_2]_4$ (I), prepared by Erdtman and Högberg.¹ This octabutyrate is obtained by acylation of a phenolic product obtained by condensation of resorcinol with *p*-bromobenzaldehyde in the presence of hydrogen chloride:



EXPERIMENTAL

Colourless short prismatic crystals of good quality were provided by Prof. Erdtman. They are triclinic, spacegroup $P\bar{1}$, with the following cell dimensions obtained from precession photographs using $CuK\alpha$ radiation (1.5418 Å) at room temperature:

$$\begin{array}{lll} a = 13.99 \pm 5 & b = 15.00 \pm 2 & c = 20.23 \pm 6 \text{ \AA} \\ \alpha = 81.66^\circ \pm 25 & \beta = 75.26^\circ \pm 30 & \gamma = 89.92^\circ \pm 35 \end{array}$$

The errors are obtained by inserting the errors of the reciprocal unit cell into the reciprocal-direct cell transformation equations which were first logarithmically derived. The reciprocal cell errors used were the standard deviations of the reciprocal edges and angles, obtained by about ten measurements of each of them on the precession films. No correction was applied for film shrinkage.

The volume of the unit cell is $4061 \pm 10 \text{ \AA}^3$ and as the molecular weight is 1669.2, the calculated density will be $D_x = 1.365 \pm 4 \text{ g.cm}^{-3}$ for two molecules per unit cell. The measured density, obtained by flotation in water solution of ZnI_2 , is $1.40 \pm 5 \text{ g.cm}^{-3}$ (error estimated).

Using a crystal of the approximate size $0.55 \times 0.55 \times 0.30 \text{ mm}$ data were collected for layers 0–7 about the a axis and for layers 0 and 2 about the b axis using Weissenberg multiple film techniques.

The intensities of 2936 independent reflections were estimated visually and corrected for Lorentz and polarization factors but not for absorption. A sharpened "point atoms at rest" Patterson synthesis² showed several high peaks which could be interpreted as Br–Br vectors. Vector addition and subtraction gave the positions of all four bromine atoms. After four successive cycles of structure factors calculations and Fourier syntheses the whole system of rings and most of the atoms of the butyrate chains were detected. The location of the rest of the chain atoms was not possible from the Fourier maps, since these contained oddly shaped, extended and sometimes overlapping peaks leaving many alternatives for the remaining atoms. Difference syntheses gave clearer pictures, although two of the end methyl carbons were impossible to locate definitely. Except for these two, all heavier atoms could be located and least-squares block diagonal refinement of the structure was initiated. Positional and isotropic temperature parameters were refined for all atoms except the bromines, which were refined with anisotropic vibrational parameters. During the refinement the isotropic B values for many of the butyrate chain atoms increased to high values, especially for the keto oxygens and the two carbons at the end of each chain. In some cases the B values tended to exceed 16 \AA^2 , the upper limit allowed in our programmes. We thought that these high B values might be due to disorder so that the site occupation number should be less than 1 for a number of the chain atoms hitherto found. Several attempts to find alternative chain conformations failed, however. In order to see, then, if the apparently strong thermal motion of these atoms might instead be partly interpreted as an effect of small changes in their positions from one molecule to another, *i.e.* a slight disorder, these atoms were refined anisotropically, and then the magnitudes and directions of the axes of the vibrational ellipsoids were calculated.

The keto oxygens obtained very high vibrational amplitudes ($13\text{--}47 \text{ \AA}^2$) in certain directions, which show no general correlation to the directions of the keto group plane normals. It is unlikely that these atoms, situated near the fixed end of the chains, vibrate with such high amplitudes and most probably the keto group planes have slightly different orientations in different molecules. The carbon atoms of the chains also show extended vibrational ellipsoids with the longest axis below 10 \AA^2 only for four carbons, three of which are carbonyl carbons. The rest of the chain carbons have the longest axis in the range $10\text{--}60 \text{ \AA}^2$ and these atoms most likely also occupy slightly different positions from molecule to molecule. The two methyl carbons that could not be detected from the early difference series are both situated in extended positive regions of low density. These two atoms are evidently more disordered than the other chain atoms. They are denoted C(28) and C(60).

Even the bromine atoms have strong anisotropic vibrations with maximum magnitudes in the range $9\text{--}29 \text{ \AA}^2$, and the carbons of the p -bromophenyl groups have vibrational ellipsoids of about the same orientations as those of the bromines but of smaller magni-

tudes. This is expected as the bromophenyl groups are fixed to the central ring system at one end and free to vibrate at the bromine atom. At least for the bromophenyl group whose bromine was assigned a maximum vibrational amplitude of 29 \AA^2 , it seems reasonable to assume that not only strong vibrations but also slight disorder are encountered.

At this stage it was checked if the apparent disorder might be handled by using a lower symmetry, *i.e.* $P1$ instead of $P\bar{1}$ which was indicated from intensity statistics. All bromines and chain atoms of two molecules in $P1$ were refined independently for two cycles with isotropic B values. For each pair of corresponding atoms in the two molecules the coordinate shifts were centrosymmetrically related — equal in magnitude and opposite in sign — and the high isotropic B value showed no tendency to go down to normal values. This indicates that the choice of the centrosymmetric spacegroup was correct.

The refinement proceeded for some cycles with anisotropic vibrational parameters for all atoms except the hydrogens. These were included at their calculated positions with the C—H distance chosen as 1.09 \AA . They were not refined but recalculated after each cycle. During the refinement 55 strong reflections of low order were found to suffer from extinction and were excluded. The quotient between the number of observed, independent reflections and the total number of refined parameters was slightly larger than 3.

The refinement was discontinued at a stage with the following average quotients between the shifts and standard deviations for positional and vibrational parameters, respectively (the maximum quotients are given within brackets):

for bromines	0.10 (0.28) and 0.21 (0.50)
for carbons of the <i>p</i> -bromophenyl rings	0.15 (0.71) » 0.18 (0.73)
for carbons of the central rings system	0.25 (0.57) » 0.16 (0.62)
for the linking oxygens	0.16 (0.76) » 0.22 (0.59)
for the remainder of the chain atoms	0.28 (1.00) » 0.31 (1.15)

A difference synthesis at this stage showed no electron density outside the range -0.6 to $+0.7 \text{ e\AA}^{-3}$. Two peaks were found at the level $+0.7 \text{ e\AA}^{-3}$. One of them could not corre-

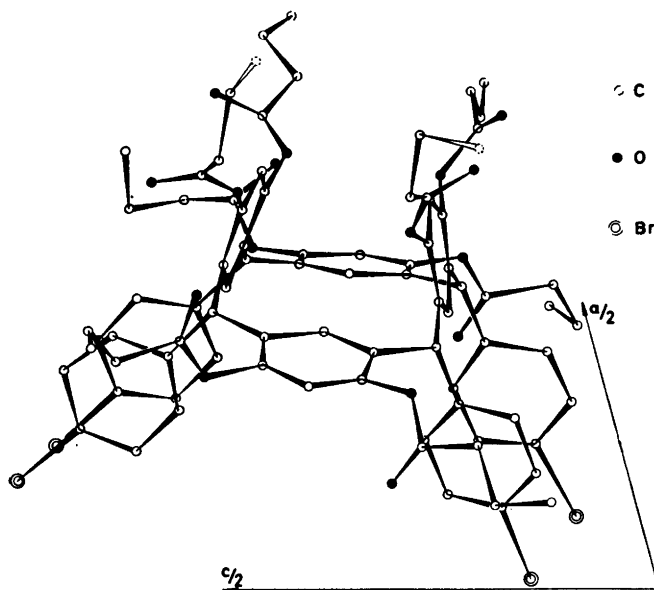


Fig. 1. Stereochemistry of the molecule. The unfilled bonds go to the two not fully localized methyl carbons.

Table 1. Fractional atomic coordinates with standard deviations ($\times 10^4$) for the heavier atoms of the molecule.

	<i>x</i>	<i>y</i>	<i>z</i>
Br(1)	-0.0578 (2)	0.7377 (2)	1.1991 (1)
Br(2)	-0.2328 (3)	0.8270 (4)	0.6456 (2)
Br(3)	-0.1233 (2)	1.1088 (2)	0.5760 (2)
Br(4)	0.0020 (3)	1.0570 (2)	1.1421 (1)
O(1)	0.1207 (10)	0.5159 (9)	0.9568 (8)
O(2)	0.2661 (15)	0.4456 (13)	0.9379 (10)
O(3)	0.0939 (10)	0.5329 (9)	0.7244 (7)
O(4)	-0.0642 (12)	0.5151 (13)	0.7781 (9)
O(5)	0.3729 (14)	0.6717 (12)	0.6756 (10)
O(6)	0.4829 (31)	0.6671 (20)	0.5849 (18)
O(7)	0.4741 (10)	0.9897 (10)	0.6222 (7)
O(8)	0.5790 (12)	0.9350 (11)	0.5353 (8)
O(9)	0.3295 (11)	1.2282 (9)	0.6219 (7)
O(10)	0.1928 (16)	1.2994 (13)	0.6518 (9)
O(11)	0.3454 (10)	1.2070 (8)	0.8606 (7)
O(12)	0.4956 (14)	1.2567 (16)	0.8096 (10)
O(13)	0.5119 (11)	0.9725 (9)	0.7940 (9)
O(14)	0.6178 (24)	0.9522 (23)	0.8540 (18)
O(15)	0.4351 (11)	0.6560 (9)	0.8495 (8)
O(16)	0.4609 (21)	0.6369 (14)	0.9556 (12)
C(1)	0.2373 (12)	0.6809 (11)	0.9259 (8)
C(2)	0.1794 (15)	0.7081 (12)	0.6833 (10)
C(3)	0.2845 (11)	1.0458 (11)	0.6342 (8)
C(4)	0.3332 (14)	1.0213 (11)	0.8825 (9)
C(5)	0.1916 (12)	0.6457 (10)	0.8740 (8)
C(6)	0.1442 (14)	0.5634 (12)	0.8860 (9)
C(7)	0.1074 (13)	0.5260 (10)	0.8402 (8)
C(8)	0.1194 (13)	0.5747 (12)	0.7793 (10)
C(9)	0.1667 (10)	0.6613 (10)	0.7559 (8)
C(10)	0.2013 (13)	0.6946 (11)	0.8064 (9)
C(11)	0.1878 (16)	0.4639 (13)	0.9712 (11)
C(12)	0.1499 (18)	0.4253 (15)	1.0531 (13)
C(13)	0.2071 (24)	0.3454 (21)	1.0729 (18)
C(14)	0.1713 (31)	0.2663 (26)	1.0756 (29)
C(15)	0.0021 (17)	0.5106 (15)	0.7288 (11)
C(16)	0.0015 (24)	0.4628 (18)	0.6674 (14)
C(17)	-0.1006 (28)	0.4404 (25)	0.6656 (18)
C(18)	-0.0978 (29)	0.3922 (28)	0.5990 (23)
C(19)	0.2543 (12)	0.7845 (10)	0.6649 (8)
C(20)	0.3548 (15)	0.7665 (12)	0.6586 (10)
C(21)	0.4316 (12)	0.8318 (11)	0.6411 (9)
C(22)	0.4044 (12)	0.9199 (11)	0.6343 (8)
C(23)	0.3099 (12)	0.9467 (10)	0.6406 (8)
C(24)	0.2359 (12)	0.8731 (11)	0.6582 (8)
C(25)	0.4351 (35)	0.6309 (25)	0.6471 (23)
C(26)	0.4380 (38)	0.5280 (28)	0.6660 (27)
C(27)	0.5453 (60)	0.4774 (60)	0.6372 (40)
C(29)	0.5591 (15)	0.9967 (14)	0.5670 (11)
C(30)	0.6206 (17)	1.0737 (15)	0.5616 (14)
C(31)	0.5706 (24)	1.1552 (22)	0.5652 (18)
C(32)	0.6380 (30)	1.2451 (26)	0.5471 (24)
C(33)	0.3016 (12)	1.0836 (10)	0.6952 (8)
C(34)	0.3159 (13)	1.1761 (12)	0.6894 (9)
C(35)	0.3338 (14)	1.2199 (11)	0.7403 (8)
C(36)	0.3346 (12)	1.1683 (10)	0.8026 (9)

Table 1. Continued.

C(37)	0.3187 (13)	1.0737 (10)	0.8146 (8)
C(38)	0.3016 (13)	1.0360 (11)	0.7596 (8)
C(39)	0.2647 (19)	1.2817 (18)	0.6095 (13)
C(40)	0.2874 (27)	1.3174 (20)	0.5295 (14)
C(41)	0.2108 (35)	1.3866 (26)	0.5130 (20)
C(42)	0.2480 (44)	1.4790 (38)	0.5404 (33)
C(43)	0.4302 (21)	1.2460 (18)	0.8570 (13)
C(44)	0.4306 (20)	1.2785 (15)	0.9242 (14)
C(45)	0.4179 (26)	1.1982 (22)	0.9890 (16)
C(46)	0.5183 (24)	1.1488 (20)	0.9763 (15)
C(47)	0.3541 (12)	0.9188 (10)	0.8720 (7)
C(48)	0.4444 (12)	0.9023 (10)	0.8351 (10)
C(49)	0.4737 (13)	0.8137 (13)	0.8286 (9)
C(50)	0.4046 (12)	0.7473 (10)	0.8580 (8)
C(51)	0.3108 (12)	0.7621 (10)	0.8936 (8)
C(52)	0.2870 (12)	0.8497 (10)	0.9007 (8)
C(53)	0.5787 (28)	0.9911 (18)	0.8095 (18)
C(54)	0.6470 (26)	1.0776 (23)	0.7584 (16)
C(55)	0.7183 (29)	1.1184 (33)	0.7855 (20)
C(56)	0.7520 (24)	1.2080 (21)	0.7445 (20)
C(57)	0.4744 (24)	0.6173 (17)	0.8995 (16)
C(58)	0.4992 (21)	0.5237 (19)	0.8713 (20)
C(59)	0.6180 (27)	0.5284 (27)	0.8386 (28)
C(61)	0.1595 (13)	0.6985 (10)	0.9903 (8)
C(62)	0.0619 (14)	0.7106 (12)	0.9960 (9)
C(63)	-0.0065 (17)	0.7221 (14)	1.0581 (10)
C(64)	0.0315 (18)	0.7231 (14)	1.1155 (10)
C(65)	0.1330 (22)	0.7111 (19)	1.1136 (14)
C(66)	0.1962 (19)	0.6977 (17)	1.0489 (13)
C(67)	0.0770 (16)	0.7410 (14)	0.6750 (11)
C(68)	0.0503 (20)	0.7295 (17)	0.6136 (13)
C(69)	-0.0436 (18)	0.7567 (19)	0.6084 (13)
C(70)	-0.1060 (19)	0.7934 (19)	0.6570 (15)
C(71)	-0.0815 (18)	0.8059 (17)	0.7151 (12)
C(72)	0.0117 (17)	0.7762 (15)	0.7228 (12)
C(73)	0.1820 (15)	1.0603 (13)	0.6229 (10)
C(74)	0.1686 (14)	1.0671 (12)	0.5574 (9)
C(75)	0.0760 (15)	1.0803 (15)	0.5443 (11)
C(76)	0.0041 (12)	1.0883 (12)	0.5986 (9)
C(77)	0.0103 (13)	1.0866 (13)	0.6633 (9)
C(78)	0.1023 (16)	1.0710 (15)	0.6771 (12)
C(79)	0.2444 (14)	1.0269 (13)	0.9425 (10)
C(80)	0.1590 (16)	1.0626 (13)	0.9371 (11)
C(81)	0.0855 (18)	1.0749 (15)	0.9938 (12)
C(82)	0.0992 (16)	1.0451 (14)	1.0593 (10)
C(83)	0.1882 (17)	1.0104 (15)	1.0665 (10)
C(84)	0.2615 (22)	0.9990 (19)	1.0073 (13)
C(28)	0.52	0.45	0.57
C(60)	0.67	0.48	0.80

spond to an atom because of its position, the other might be interpreted as a low-occupancy site for atom C(60), one of the two methyl carbons not located. It refined to a position unacceptably close to its nearest chain neighbour, however.

The final conventional over all *R* index was 0.105, unobserved reflections not taken into account.

The calculations were performed on a Datasaab D21 computer using the programme system by Abrahamsson *et al.*³ extended with programmes by Abrahamsson,⁴ layer

Table 2. Mean-square amplitude tensors. Allowance was made for anisotropic vibrations with

$$\exp -2\pi^2(h^2a^*U_{11} + k^2b^*U_{22} + l^2c^*U_{33} + 2klb^*c^*U_{23} + 2lhc^*a^*U_{31} + 2hka^*b^*U_{12})$$

The tensor elements (Å²), multiplied by 10³, are given with standard deviations.

	U ₁₁	U ₂₂	U ₃₃	U ₂₃	U ₃₁	U ₁₂
Br(1)	139 (3)	115 (2)	67 (2)	-11 (1)	11 (2)	21 (2)
Br(2)	89 (3)	317 (6)	184 (4)	43 (4)	-56 (2)	27 (3)
Br(3)	57 (2)	215 (3)	133 (2)	-56 (2)	-47 (2)	51 (2)
Br(4)	176 (3)	141 (2)	80 (2)	-40 (2)	28 (2)	-20 (2)
O(1)	60 (11)	49 (9)	88 (11)	2 (8)	-17 (8)	15 (8)
O(2)	130 (18)	120 (16)	132 (17)	10 (13)	2 (13)	66 (13)
O(3)	52 (11)	75 (10)	76 (11)	-17 (8)	-20 (8)	-4 (8)
O(4)	66 (12)	153 (17)	115 (15)	-50 (13)	-51 (10)	1 (11)
O(5)	125 (17)	97 (14)	130 (17)	-1 (12)	33 (13)	27 (12)
O(6)	408 (49)	170 (25)	313 (41)	-70 (25)	59 (35)	121 (28)
O(7)	44 (11)	90 (11)	75 (11)	-11 (9)	4 (8)	10 (8)
O(8)	77 (13)	125 (13)	79 (12)	2 (10)	-8 (10)	-11 (10)
O(9)	87 (13)	71 (10)	55 (10)	7 (8)	-16 (8)	29 (9)
O(10)	182 (21)	130 (16)	79 (13)	42 (12)	3 (13)	89 (15)
O(11)	84 (11)	51 (9)	76 (10)	-8 (7)	-20 (8)	0 (8)
O(12)	96 (16)	208 (22)	103 (16)	-41 (15)	-18 (12)	-19 (15)
O(13)	63 (11)	76 (10)	152 (16)	3 (10)	-61 (10)	-2 (9)
O(14)	210 (31)	281 (37)	285 (40)	69 (30)	-152 (29)	-88 (28)
O(15)	76 (12)	60 (10)	84 (12)	-22 (8)	-29 (9)	1 (9)
O(16)	272 (31)	115 (17)	147 (21)	-24 (15)	-106 (20)	80 (18)
C(1)	47 (13)	52 (11)	48 (11)	5 (9)	-25 (9)	10 (10)
C(2)	79 (16)	51 (12)	80 (15)	-12 (11)	-36 (12)	3 (11)
C(3)	27 (11)	62 (12)	39 (10)	-1 (9)	-9 (8)	17 (9)
C(4)	74 (15)	56 (12)	54 (13)	-9 (10)	-46 (10)	16 (11)
C(5)	35 (12)	36 (11)	47 (12)	-19 (9)	-11 (9)	15 (9)
C(6)	57 (15)	59 (13)	55 (13)	-8 (10)	-14 (10)	5 (11)
C(7)	72 (14)	33 (11)	51 (12)	3 (9)	-17 (10)	-8 (10)
C(8)	36 (13)	68 (13)	87 (15)	-39 (11)	-26 (10)	15 (10)
C(9)	-1 (10)	46 (11)	73 (13)	-9 (9)	-2 (8)	24 (8)
C(10)	44 (14)	50 (12)	57 (13)	-15 (10)	-9 (10)	18 (10)
C(11)	77 (17)	52 (14)	97 (18)	-4 (12)	-18 (13)	-8 (12)
C(12)	94 (21)	78 (17)	114 (22)	8 (15)	-34 (16)	0 (15)
C(13)	134 (29)	148 (27)	177 (34)	47 (23)	-28 (24)	36 (22)
C(14)	179 (39)	161 (32)	423 (72)	53 (37)	-98 (41)	39 (27)
C(15)	85 (18)	99 (18)	87 (17)	-44 (14)	3 (13)	-4 (14)
C(16)	177 (31)	114 (22)	107 (23)	-8 (17)	-89 (21)	-19 (20)
C(17)	212 (37)	204 (35)	139 (30)	-51 (26)	-49 (26)	6 (28)
C(18)	168 (36)	243 (40)	212 (47)	-87 (34)	-51 (31)	-62 (30)
C(19)	41 (12)	40 (10)	50 (11)	-17 (9)	-6 (9)	-1 (9)
C(20)	76 (16)	54 (13)	74 (14)	0 (11)	8 (11)	24 (11)
C(21)	32 (12)	48 (12)	67 (13)	8 (10)	16 (9)	-4 (10)
C(22)	37 (12)	49 (11)	50 (11)	-6 (9)	-8 (9)	19 (9)
C(23)	36 (12)	52 (11)	37 (10)	-1 (8)	-13 (8)	22 (9)
C(24)	29 (12)	72 (12)	34 (10)	-9 (9)	6 (8)	23 (10)
C(25)	277 (53)	155 (32)	269 (45)	101 (30)	219 (42)	113 (33)
C(26)	270 (54)	143 (35)	275 (55)	-53 (34)	-79 (42)	39 (33)
C(27)	398 (98)	455(107)	319 (80)	-95 (73)	47 (69)	186 (82)
C(29)	30 (15)	66 (15)	64 (16)	-1 (12)	-27 (12)	11 (13)
C(30)	42 (18)	61 (17)	117 (22)	-1 (15)	-18 (15)	-5 (14)
C(31)	102 (28)	124 (27)	160 (31)	-29 (22)	-60 (23)	-51 (22)
C(32)	155 (37)	143 (31)	207 (47)	-11 (29)	-47 (31)	-38 (26)
C(33)	44 (12)	40 (10)	30 (10)	3 (8)	-1 (8)	2 (9)
C(34)	42 (13)	62 (12)	59 (12)	1 (10)	-14 (9)	-9 (10)

Table 2. Continued.

C(35)	84 (15)	40 (11)	37 (11)	20 (9)	-10 (10)	-21 (10)
C(36)	39 (13)	40 (11)	71 (13)	-11 (9)	-10 (10)	11 (9)
C(37)	49 (14)	31 (11)	47 (12)	1 (9)	-16 (9)	11 (10)
C(38)	48 (13)	42 (11)	36 (11)	-13 (9)	-29 (9)	30 (10)
C(39)	64 (21)	105 (21)	70 (19)	-1 (16)	2 (15)	37 (17)
C(40)	191 (35)	106 (23)	72 (21)	42 (18)	-7 (21)	68 (22)
C(41)	264 (49)	158 (32)	134 (32)	-9 (25)	-39 (31)	-4 (31)
C(42)	244 (59)	236 (55)	332 (74)	-18 (49)	-81 (50)	2 (44)
C(43)	103 (24)	103 (21)	55 (19)	-11 (16)	-20 (16)	21 (18)
C(44)	94 (23)	59 (16)	102 (22)	-23 (15)	-59 (17)	16 (15)
C(45)	159 (34)	129 (27)	96 (25)	25 (20)	-51 (22)	50 (24)
C(46)	135 (30)	131 (26)	96 (24)	1 (19)	-39 (20)	51 (22)
C(47)	43 (12)	40 (10)	27 (10)	2 (8)	-11 (8)	11 (9)
C(48)	42 (13)	27 (10)	94 (15)	14 (9)	-31 (10)	0 (9)
C(49)	37 (14)	68 (13)	58 (14)	-1 (10)	4 (10)	-6 (11)
C(50)	56 (13)	33 (10)	52 (12)	4 (9)	-27 (9)	7 (9)
C(51)	31 (12)	29 (10)	66 (13)	3 (9)	-23 (9)	-8 (9)
C(52)	49 (13)	37 (10)	35 (8)	6 (8)	-14 (8)	-8 (9)
C(53)	218 (40)	89 (22)	184 (33)	10 (21)	-121 (29)	39 (23)
C(54)	133 (32)	156 (28)	102 (25)	24 (21)	-40 (21)	-82 (24)
C(55)	125 (33)	288 (51)	123 (32)	-8 (32)	-71 (25)	-4 (33)
C(56)	110 (28)	104 (25)	177 (37)	2 (24)	-56 (24)	-19 (21)
C(57)	142 (30)	66 (19)	131 (25)	111 (17)	-22 (21)	46 (19)
C(58)	71 (24)	75 (21)	262 (41)	-37 (22)	-8 (23)	21 (18)
C(59)	137 (32)	240 (40)	488 (76)	-191 (45)	-199 (40)	66 (29)
C(61)	63 (14)	31 (10)	47 (11)	-3 (8)	-27 (9)	-2 (9)
C(62)	45 (14)	66 (14)	59 (14)	-5 (11)	-15 (10)	11 (11)
C(63)	73 (19)	63 (16)	32 (14)	0 (12)	-4 (12)	18 (13)
C(64)	95 (21)	47 (15)	37 (14)	-9 (12)	-6 (13)	13 (14)
C(65)	111 (26)	99 (22)	65 (19)	-8 (16)	-3 (16)	4 (18)
C(66)	78 (21)	106 (21)	70 (18)	-25 (16)	-50 (15)	31 (16)
C(67)	47 (17)	66 (16)	52 (15)	-13 (12)	-20 (12)	13 (13)
C(68)	101 (24)	85 (19)	80 (20)	25 (15)	-59 (17)	0 (16)
C(69)	103 (21)	171 (25)	95 (19)	-9 (18)	-65 (16)	16 (18)
C(70)	50 (20)	116 (23)	104 (24)	9 (18)	1 (16)	24 (17)
C(71)	108 (21)	133 (21)	95 (19)	-47 (16)	2 (15)	19 (16)
C(72)	88 (19)	103 (18)	108 (19)	-23 (15)	-31 (14)	3 (14)
C(73)	46 (16)	41 (13)	33 (13)	-1 (10)	-6 (11)	6 (11)
C(74)	71 (15)	77 (14)	40 (11)	4 (10)	-15 (10)	10 (11)
C(75)	67 (16)	134 (20)	78 (15)	-34 (14)	-31 (12)	52 (14)
C(76)	22 (12)	75 (13)	76 (13)	-32 (11)	-24 (9)	44 (10)
C(77)	37 (13)	106 (16)	65 (13)	-24 (11)	-20 (10)	41 (12)
C(78)	38 (17)	79 (18)	69 (17)	-6 (14)	-15 (12)	25 (14)
C(79)	33 (15)	46 (13)	41 (13)	-5 (11)	-14 (10)	9 (11)
C(80)	60 (18)	33 (13)	58 (16)	3 (11)	-4 (12)	8 (12)
C(81)	80 (21)	50 (15)	64 (17)	12 (12)	-12 (14)	-21 (14)
C(82)	68 (18)	56 (15)	41 (14)	-20 (11)	-4 (12)	0 (13)
C(83)	107 (19)	114 (18)	50 (13)	-21 (12)	-28 (12)	36 (15)
C(84)	119 (27)	118 (24)	62 (19)	-10 (17)	-15 (17)	3 (20)

scaling programmes using the method described by Hamilton, Rollett and Sparks,⁵ and a program for determination of the magnitudes and orientations of vibrational ellipsoids. The formfactor curves used are those given in the *International Tables for X-ray Crystallography*, Vol. III, p. 202. The least-squares programmes minimize $\sum w(|F_o| - |F_c|)^2$ where the weight applied to each reflection is

Table 3. Fractional coordinates and isotropic temperature factors for the hydrogens. The number of the parent carbon is obtained by omitting the last digit in the number of the hydrogen atom.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (Å ²)
H(11)	0.278	0.625	0.947	4
H(21)	0.210	0.658	0.651	6
H(31)	0.333	1.083	0.587	4
H(41)	0.394	1.054	0.895	5
H(71)	0.069	0.461	0.852	4
H(101)	0.236	0.762	0.794	4
H(121)	0.158	0.478	1.083	7
H(122)	0.071	0.404	1.066	7
H(131)	0.282	0.352	1.038	11
H(132)	0.214	0.346	1.126	11
H(141)	0.164	0.258	1.019	16
H(142)	0.096	0.252	1.107	16
H(143)	0.218	0.210	1.086	16
H(161)	0.038	0.507	0.619	10
H(162)	0.042	0.401	0.670	10
H(171)	-0.140	0.394	0.713	13
H(172)	-0.146	0.503	0.666	13
H(181)	-0.056	0.334	0.596	16
H(182)	-0.062	0.443	0.550	16
H(183)	-0.173	0.378	0.592	16
H(211)	0.510	0.812	0.634	4
H(241)	0.160	0.893	0.666	4
H(261)	0.418	0.513	0.722	16
H(262)	0.382	0.498	0.646	16
H(301)	0.657	1.063	0.603	5
H(302)	0.676	1.080	0.512	5
H(311)	0.524	1.159	0.529	10
H(312)	0.524	1.153	0.618	10
H(321)	0.684	1.250	0.494	13
H(322)	0.685	1.244	0.583	13
H(323)	0.591	1.303	0.552	13
H(351)	0.347	1.292	0.732	5
H(381)	0.285	0.963	0.768	4
H(401)	0.361	1.350	0.512	9
H(402)	0.285	1.260	0.502	9
H(411)	0.215	1.399	0.458	14
H(412)	0.136	1.364	0.542	14
H(421)	0.324	1.498	0.512	16
H(422)	0.244	1.464	0.595	16
H(423)	0.200	1.534	0.532	16
H(441)	0.501	1.315	0.917	7
H(442)	0.370	1.324	0.936	7
H(451)	0.404	1.226	1.037	10
H(452)	0.357	1.152	0.991	10
H(461)	0.578	1.196	0.974	9
H(462)	0.532	1.122	0.928	9
H(463)	0.515	1.094	1.018	9
H(491)	0.549	0.799	0.800	4
H(521)	0.212	0.864	0.930	4
H(541)	0.687	1.053	0.711	10
H(542)	0.597	1.130	0.746	10
H(551)	0.685	1.126	0.839	13
H(552)	0.781	1.075	0.783	13
H(561)	0.689	1.251	0.747	10

Table 3. Continued.

H(562)	0.786	1.200	0.691	10
H(563)	0.806	1.238	0.766	10
H(581)	0.476	0.467	0.913	9
H(582)	0.463	0.517	0.831	9
H(621)	0.035	0.710	0.949	5
H(631)	-0.085	0.730	1.062	4
H(651)	0.161	0.712	1.159	7
H(661)	0.274	0.686	1.045	7
H(681)	0.102	0.702	0.572	7
H(691)	-0.069	0.743	0.561	9
H(711)	-0.133	0.837	0.754	9
H(721)	0.029	0.783	0.774	8
H(741)	0.232	1.062	0.515	5
H(751)	0.064	1.084	0.492	7
H(771)	-0.056	1.095	0.705	5
H(781)	0.111	1.068	0.729	5
H(801)	0.147	1.083	0.886	4
H(811)	0.018	1.107	0.988	5
H(831)	0.199	0.992	1.118	7
H(841)	0.331	0.969	1.012	8

$$w = \frac{1}{1 + \left[\frac{|F_o| - a}{b} \right]^2}$$

as suggested by Mills and Rollett.⁶

During the last cycles the constants a and b were $3.7 |F_{\min}|$ and $3.1 |F_{\min}|$, respectively. So chosen they gave an approximately equal value of the minimized quantity for reflections in different intensity regions.

RESULTS AND DISCUSSION

The structure factors table is voluminous and is therefore omitted here but may be obtained from this Institute.

The configuration of the molecule is shown in Fig. 1. The two unlocalized methyl carbons C(28) and C(60) are included in this figure and in Table 1 at positions chosen roughly in the middle of the regions of high density. The bonds to them are distinguished from other bonds by just being outlined. The coordinates and anisotropic temperature factors of the atoms, except hydrogens, are given in Tables 1 and 2, respectively.

As seen from Table 2 the following atoms show especially high vibrational amplitudes: the bromines and the carbons next to them in the *p*-bromophenyl groups, the keto oxygens (oxygen with even numbers) and the butyrate chain carbons, in particular the two or three at the free end of each chain. Remarkably high vibrations are assigned to the two atoms C(27) and C(59), which are bonded to the two unlocalized atoms C(28) and C(60), respectively. The reason for these apparently high vibrational amplitudes were discussed above. The hydrogen parameters are given in Table 3. The hydrogens of the unlocalized methyl groups as well as those of the carbons next to them are omitted. The hydrogen isotropic B values were obtained from the vibrational

Table 4(a). Bond distances with standard deviations in Å.

Br(1) —C(64)	1.870	(0.020)	C(21) —C(22)	1.373	(0.023)	
Br(2) —C(70)	1.905	(0.029)	C(22) —C(23)	1.361	(0.023)	
Br(3) —C(76)	1.961	(0.018)	C(23) —C(24)	1.457	(0.022)	
Br(4) —C(82)	1.902	(0.020)	C(25) —C(26)	1.538	(0.055)	
O(1) —C(6)	1.460	(0.022)	C(26) —C(27)	1.689	(0.096)	
	—C(11)	1.282	(0.026)	C(29) —C(30)	1.415	(0.032)
O(2) —C(11)	1.187	(0.028)	C(30) —C(31)	1.410	(0.040)	
O(3) —C(8)	1.469	(0.027)	C(31) —C(32)	1.590	(0.050)	
	—C(15)	1.305	(0.028)	C(33) —C(34)	1.386	(0.023)
O(4) —C(15)	1.187	(0.026)	C(34) —C(35)	1.372	(0.027)	
O(5) —C(20)	1.451	(0.025)	C(35) —C(36)	1.383	(0.023)	
	—C(25)	1.145	(0.047)	C(36) —C(37)	1.412	(0.022)
O(6) —C(25)	1.305	(0.051)	C(37) —C(38)	1.392	(0.026)	
O(7) —C(22)	1.383	(0.022)	C(39) —C(40)	1.580	(0.037)	
	—C(29)	1.401	(0.023)	C(40) —C(41)	1.555	(0.058)
O(8) —C(29)	1.195	(0.029)	C(41) —C(42)	1.695	(0.079)	
O(9) —C(34)	1.438	(0.021)	C(43) —C(44)	1.510	(0.040)	
	—C(39)	1.259	(0.031)	C(44) —C(45)	1.620	(0.038)
O(10) —C(39)	1.200	(0.031)	C(45) —C(46)	1.567	(0.048)	
O(11) —C(36)	1.422	(0.024)	C(47) —C(48)	1.337	(0.022)	
	—C(43)	1.304	(0.032)	—C(52)	1.358	(0.020)
O(12) —C(43)	1.136	(0.029)	C(48) —C(49)	1.405	(0.024)	
O(13) —C(48)	1.429	(0.020)	C(49) —C(50)	1.349	(0.023)	
	—C(53)	1.107	(0.045)	C(50) —C(51)	1.362	(0.022)
O(14) —C(53)	1.245	(0.054)	C(51) —C(52)	1.374	(0.021)	
O(15) —C(50)	1.454	(0.020)	C(53) —C(54)	1.667	(0.042)	
	—C(57)	1.331	(0.037)	C(54) —C(55)	1.431	(0.061)
O(16) —C(57)	1.182	(0.041)	C(55) —C(56)	1.482	(0.053)	
C(1) —C(5)	1.515	(0.027)	C(57) —C(58)	1.596	(0.042)	
	—C(51)	1.548	(0.021)	C(58) —C(59)	1.623	(0.046)
	—C(61)	1.526	(0.021)	C(61) —C(62)	1.354	(0.026)
C(2) —C(9)	1.500	(0.025)	—C(66)	1.405	(0.034)	
	—C(19)	1.494	(0.024)	C(62) —C(63)	1.406	(0.026)
	—C(67)	1.556	(0.031)	C(63) —C(64)	1.397	(0.035)
C(3) —C(23)	1.523	(0.022)	C(64) —C(65)	1.422	(0.040)	
	—C(33)	1.503	(0.024)	C(65) —C(66)	1.422	(0.035)
	—C(73)	1.520	(0.023)	C(67) —C(68)	1.417	(0.039)
C(4) —C(37)	1.542	(0.025)	—C(72)	1.322	(0.030)	
	—C(47)	1.600	(0.023)	C(68) —C(69)	1.400	(0.039)
	—C(79)	1.511	(0.024)	C(69) —C(70)	1.324	(0.037)
C(5) —C(6)	1.363	(0.024)	C(70) —C(71)	1.342	(0.043)	
	—C(10)	1.428	(0.023)	C(71) —C(72)	1.416	(0.036)
C(6) —C(7)	1.352	(0.029)	C(73) —C(74)	1.374	(0.028)	
C(7) —C(8)	1.310	(0.024)	—C(78)	1.377	(0.027)	
C(8) —C(9)	1.419	(0.022)	C(74) —C(75)	1.395	(0.030)	
C(9) —C(10)	1.388	(0.027)	C(75) —C(76)	1.307	(0.025)	
C(11) —C(12)	1.625	(0.032)	C(76) —C(77)	1.331	(0.028)	
C(12) —C(13)	1.504	(0.041)	C(77) —C(78)	1.397	(0.031)	
C(13) —C(14)	1.277	(0.053)	C(79) —C(80)	1.333	(0.030)	
C(15) —C(16)	1.525	(0.040)	—C(84)	1.396	(0.035)	
C(16) —C(17)	1.478	(0.053)	C(80) —C(81)	1.367	(0.029)	
C(17) —C(18)	1.612	(0.063)	C(81) —C(82)	1.396	(0.033)	
C(19) —C(20)	1.410	(0.026)	C(82) —C(83)	1.383	(0.033)	
	—C(24)	1.348	(0.023)	C(83) —C(84)	1.397	(0.032)
C(20) —C(21)	1.395	(0.025)				

Table 4(b). Bond angles with standard deviations in degrees.

C(6) — O(1) — C(11)	114.9	(1.5)	O(5) — C(25) — C(26)	122.0	(3.5)
C(8) — O(3) — C(15)	120.4	(1.5)	O(6) — C(25) — C(26)	117.6	(3.7)
C(20) — O(5) — C(25)	127.8	(2.4)	C(25) — C(26) — C(27)	118.3	(4.2)
C(22) — O(7) — C(29)	121.8	(1.6)	O(7) — C(29) — O(8)	119.2	(1.8)
C(34) — O(9) — C(39)	120.2	(1.6)	O(7) — C(29) — C(30)	114.0	(1.9)
C(36) — O(11) — C(43)	118.4	(1.5)	O(8) — C(29) — C(30)	126.2	(1.9)
C(48) — O(13) — C(53)	120.7	(2.2)	C(29) — C(30) — C(31)	114.6	(2.3)
C(50) — O(15) — C(57)	112.9	(1.8)	C(30) — C(31) — C(32)	116.4	(2.7)
C(5) — C(1) — C(51)	113.0	(1.3)	C(3) — C(33) — C(34)	118.2	(1.4)
C(5) — C(1) — C(61)	112.2	(1.4)	C(3) — C(33) — C(38)	127.1	(1.4)
C(51) — C(1) — C(61)	113.0	(1.4)	C(34) — C(33) — C(38)	114.7	(1.6)
C(9) — C(2) — C(19)	112.2	(1.7)	O(9) — C(34) — C(33)	117.5	(1.6)
C(9) — C(2) — C(67)	108.6	(1.4)	O(9) — C(34) — C(35)	116.9	(1.5)
C(19) — C(2) — C(67)	111.1	(1.5)	C(33) — C(34) — C(35)	124.7	(1.6)
C(23) — C(3) — C(33)	108.7	(1.3)	C(34) — C(35) — C(36)	117.6	(1.5)
C(23) — C(3) — C(73)	112.2	(1.4)	O(11) — C(36) — C(35)	122.5	(1.4)
C(33) — C(3) — C(73)	114.5	(1.4)	O(11) — C(36) — C(37)	115.1	(1.4)
C(37) — C(4) — C(47)	108.5	(1.4)	C(35) — C(36) — C(37)	122.3	(1.7)
C(37) — C(4) — C(79)	111.6	(1.5)	C(4) — C(37) — C(36)	118.2	(1.6)
C(47) — C(4) — C(79)	110.9	(1.3)	C(4) — C(37) — C(38)	126.0	(1.4)
C(1) — C(5) — C(6)	123.2	(1.5)	C(36) — C(37) — C(38)	115.5	(1.5)
C(1) — C(5) — C(10)	121.9	(1.4)	C(33) — C(38) — C(37)	125.1	(1.5)
C(6) — C(5) — C(10)	114.9	(1.7)	O(9) — C(39) — O(10)	125.0	(2.3)
O(1) — C(6) — C(5)	117.0	(1.7)	O(9) — C(39) — C(40)	110.4	(2.1)
O(1) — C(6) — C(7)	116.7	(1.5)	O(10) — C(39) — C(40)	124.5	(2.6)
C(5) — C(6) — C(7)	125.9	(1.6)	C(39) — C(40) — C(41)	111.5	(2.5)
C(6) — C(7) — C(8)	115.7	(1.6)	C(40) — C(41) — C(42)	101.3	(3.8)
O(3) — C(8) — C(7)	118.2	(1.6)	O(11) — C(43) — O(12)	125.5	(2.8)
O(3) — C(8) — C(9)	113.7	(1.5)	O(11) — C(43) — C(44)	111.3	(1.9)
C(7) — C(8) — C(9)	127.6	(1.9)	O(12) — C(43) — C(44)	123.2	(2.8)
C(2) — C(9) — C(8)	122.1	(1.7)	C(43) — C(44) — C(45)	113.9	(2.1)
C(2) — C(9) — C(10)	125.2	(1.4)	C(44) — C(45) — C(46)	105.7	(2.1)
C(8) — C(9) — C(10)	112.7	(1.5)	C(4) — C(47) — C(48)	116.6	(1.3)
C(5) — C(10) — C(9)	123.3	(1.5)	C(4) — C(47) — C(52)	123.3	(1.3)
O(1) — C(11) — O(2)	132.6	(2.1)	C(48) — C(47) — C(52)	120.1	(1.4)
O(1) — C(11) — C(12)	106.2	(1.6)	O(13) — C(48) — C(47)	122.4	(1.4)
O(2) — C(11) — C(12)	121.1	(2.1)	O(13) — C(48) — C(49)	116.1	(1.4)
C(11) — C(12) — C(13)	111.5	(2.0)	C(47) — C(48) — C(49)	121.1	(1.4)
C(12) — C(13) — C(14)	118.8	(3.6)	C(48) — C(49) — C(50)	116.6	(1.5)
O(3) — C(15) — O(4)	123.9	(2.4)	O(15) — C(50) — C(49)	116.1	(1.4)
O(3) — C(15) — C(16)	107.5	(1.9)	O(15) — C(50) — C(51)	120.2	(1.3)
O(4) — C(15) — C(16)	127.6	(2.4)	C(49) — C(50) — C(51)	123.6	(1.5)
C(15) — C(16) — C(17)	111.2	(2.4)	C(1) — C(51) — C(50)	119.5	(1.3)
C(16) — C(17) — C(18)	109.5	(2.7)	C(1) — C(51) — C(52)	123.0	(1.3)
C(2) — C(19) — C(20)	119.7	(1.5)	C(50) — C(51) — C(52)	117.5	(1.3)
C(2) — C(19) — C(24)	126.6	(1.5)	C(47) — C(52) — C(51)	121.0	(1.4)
C(20) — C(19) — C(24)	113.6	(1.5)	O(13) — C(53) — O(14)	130.7	(3.0)
O(5) — C(20) — C(19)	113.6	(1.6)	O(13) — C(53) — C(54)	115.0	(3.0)
O(5) — C(20) — C(21)	121.3	(1.8)	O(14) — C(53) — C(54)	113.7	(3.3)
C(19) — C(20) — C(21)	125.1	(1.6)	C(53) — C(54) — C(55)	116.5	(3.0)
C(20) — C(21) — C(22)	116.1	(1.6)	C(54) — C(55) — C(56)	110.2	(3.5)
O(7) — C(22) — C(21)	120.7	(1.5)	O(15) — C(57) — O(16)	126.3	(2.7)
O(7) — C(22) — C(23)	114.5	(1.4)	O(15) — C(57) — C(58)	97.6	(2.6)
C(21) — C(22) — C(23)	124.8	(1.5)	O(16) — C(57) — C(58)	131.5	(2.7)
C(3) — C(23) — C(22)	122.1	(1.4)	C(57) — C(58) — C(59)	105.2	(2.6)
C(3) — C(23) — C(24)	123.4	(1.4)	C(1) — C(61) — C(62)	126.7	(1.7)
C(22) — C(23) — C(24)	114.4	(1.4)	C(1) — C(61) — C(66)	113.8	(1.7)
C(19) — C(24) — C(23)	125.8	(1.5)	C(62) — C(61) — C(66)	119.4	(1.7)
O(5) — C(25) — O(6)	116.4	(3.6)	C(61) — C(62) — C(63)	123.6	(2.0)

Table 4(b). Continued.

C(62)–C(63)–C(64)	116.3	(2.1)	C(74)–C(73)–C(78)	119.0	(2.0)
Br(1)–C(64)–C(63)	117.4	(1.7)	C(73)–C(74)–C(75)	122.0	(1.7)
Br(1)–C(64)–C(65)	119.3	(1.8)	C(74)–C(75)–C(76)	115.0	(2.0)
C(63)–C(64)–C(65)	123.3	(2.0)	Br(3)–C(76)–C(75)	112.7	(1.6)
C(64)–C(65)–C(66)	116.3	(2.6)	Br(3)–C(76)–C(77)	120.0	(1.2)
C(61)–C(66)–C(65)	121.1	(2.4)	C(75)–C(76)–C(77)	127.3	(1.9)
C(2)–C(67)–C(68)	118.3	(1.9)	C(76)–C(77)–C(78)	118.0	(1.6)
C(2)–C(67)–C(72)	123.9	(2.2)	C(73)–C(78)–C(77)	118.5	(2.1)
C(68)–C(67)–C(72)	117.8	(2.3)	C(4)–C(79)–C(80)	124.9	(1.8)
C(67)–C(68)–C(69)	117.2	(2.3)	C(4)–C(79)–C(84)	114.1	(1.9)
C(68)–C(69)–C(70)	123.0	(2.8)	C(80)–C(79)–C(84)	120.7	(1.9)
Br(2)–C(70)–C(69)	120.0	(2.4)	C(79)–C(80)–C(81)	122.0	(2.2)
Br(2)–C(70)–C(71)	119.4	(2.0)	C(80)–C(81)–C(82)	118.6	(2.2)
C(69)–C(70)–C(71)	120.6	(2.7)	Br(4)–C(82)–C(81)	122.6	(1.7)
C(70)–C(71)–C(72)	117.5	(2.3)	Br(4)–C(82)–C(83)	116.7	(1.6)
C(67)–C(72)–C(71)	123.7	(2.5)	C(81)–C(82)–C(83)	120.5	(1.8)
C(3)–C(73)–C(74)	119.9	(1.5)	C(82)–C(83)–C(84)	119.0	(2.2)
C(3)–C(73)–C(78)	121.0	(1.9)	C(79)–C(84)–C(83)	119.0	(2.5)

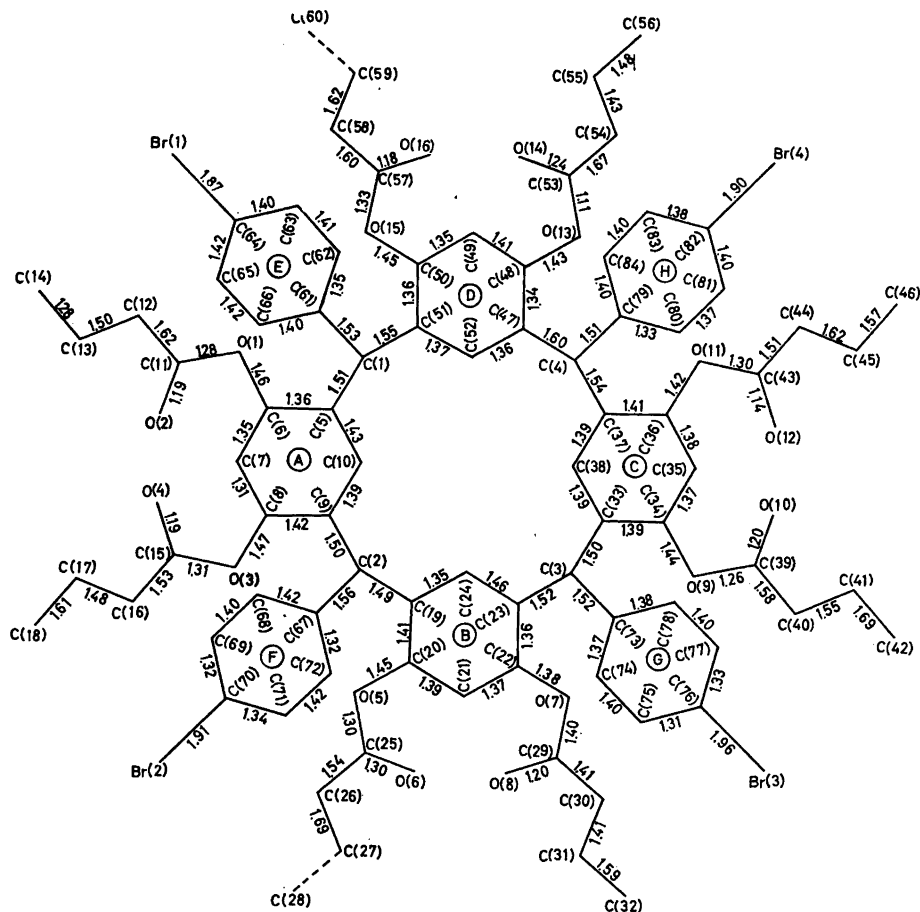


Fig. 2. Atom numbering, plane labelling and bond lengths (Å).

ellipsoids of the carbons to which they are attached by the approximate formula $B = 8\pi^2 \sqrt[3]{U_{11}U_{22}U_{33}}$, but an upper limit of 16 \AA^2 has been fixed.

The bond distances are given in Fig. 2 and (with standard deviations) in Table 4(a). Fig. 2 also shows the numbering of atoms and the labelling of the planes of the aromatic rings. The bond angles are given with standard deviations in Table 4(b). One bond is unacceptably short: C(13)—C(14) is 1.28 \AA and this deviation (5σ) from the expected value is certainly not real but again an effect of chain disorder. The average values within the structural unit are shown in Fig. 3. All distances and most angles given in this figure

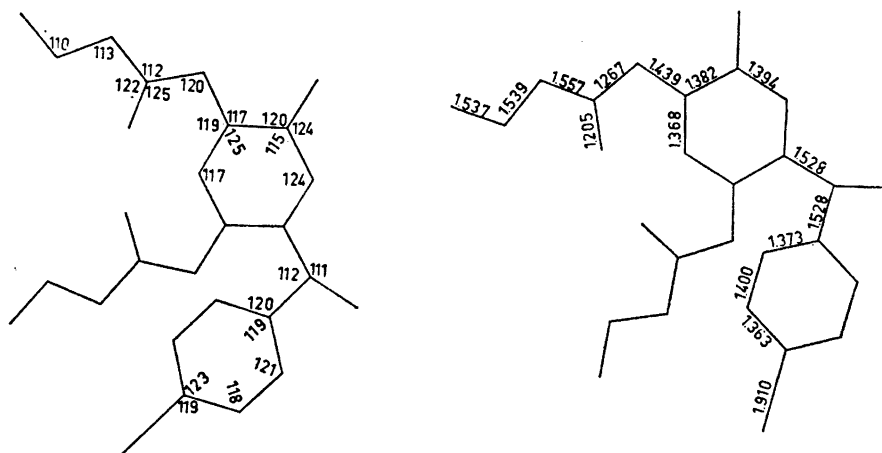


Fig. 3. Average values of angles (degrees) and distances (\AA) within the structural unit.

are of expected magnitudes, but the O—CO—C angle (112°) shows rather large deviation from the ordinary value for an sp^2 hybridized carbon atom. It is hard, however, because of the big range of this angle (98° — 125°), to decide whether this deviation is real. Probably the very low values of three of these angles (98° , 106° , 107°), which keep the average down, are artefacts due to disorder as they cannot be caused by close contacts. The average aromatic C—C distance is 1.379 \AA (the root-mean-square deviation σ being 0.033 \AA), the average chain C—C distance is 1.55 \AA ($\sigma = 0.10 \text{ \AA}$) and the average C_{sp^2} — C_{sp^3} distance at the four condensation centra is 1.528 \AA ($\sigma = 0.028 \text{ \AA}$). The average value for angles within the benzene rings is 120° ($\sigma = 4^\circ$). The average value of the C— C_{sp^2} —C angles at the four carbons of the condensation centra is 112° ($\sigma = 2^\circ$) and for C— C_{sp^3} —C angles of the chains 112° ($\sigma = 5^\circ$).

The least-squares planes fitted to the six atoms of each benzene ring and the atoms immediately attached to them are denoted A-H as shown in Fig. 2. The average (and maximum) deviations from these planes are: for benzene carbons 0.023 (0.070 \AA), for attached bromines 0.020 (0.025 \AA), for attached carbons 0.06 (0.15 \AA) and for attached oxygens 0.10 (0.16 \AA).

The O—CO—C-groups are planar, the atoms diverging on an average 0.03 Å and not more than 0.12 Å from their planes. These planes form angles in the range 49°—88° with the planes of the benzene rings to which they are attached, and they are twisted so that at rings A and C the two keto oxygens are on opposite sides of the ring plane but at rings B and D on the same side.

The four carbon atoms C(1)—C(4) of the condensation centra are planar, the maximum deviation from the plane being 0.04 Å. This plane, denoted P, and the eight planes A-H form angles with each other that are given in Table 5.

Table 5. Intersection angles of the planes.

P—A	13°	A—C	20°	B—F	77°	D—F	82°
P—B	74	A—D	78	B—G	84	D—G	90
P—C	8	A—E	73	B—H	78	D—H	73
P—D	78	A—F	87	C—D	75	E—F	16
P—E	86	A—G	83	C—E	88	E—G	5
P—F	80	A—H	82	C—F	74	E—H	20
P—G	86	B—C	72	C—G	87	F—G	19
P—H	88	B—D	27	C—H	78	F—H	26
A—B	75	B—E	84	D—E	85	G—H	16

The planes of the three benzene rings that surround each condensation centre (that is planes ABF, BCG and so on) are roughly perpendicular to each other with an average intersection angle of 79° (range 72—88°). All four *p*-bromo-

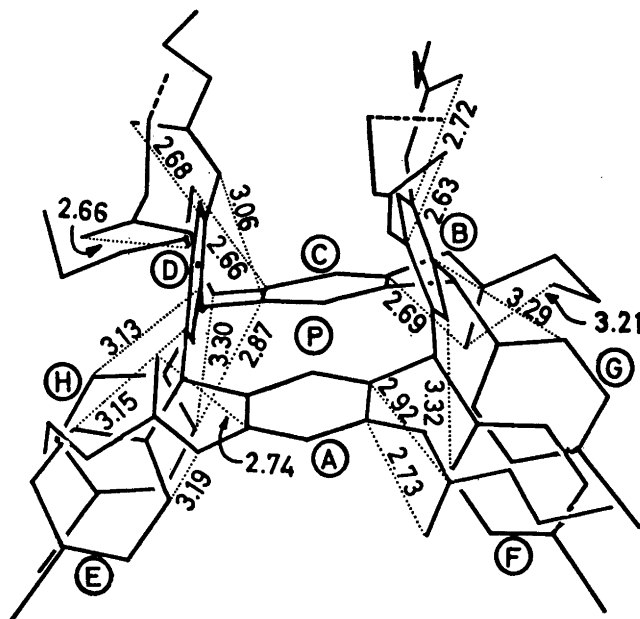


Fig. 4. Projection of the molecule in the positive direction of the *b* axis. Short intramolecular distances are given (Å). The encircled letters are plane labels.

phenyl groups are located on the same side on the plane P, and their planes form rather small angles with each other (range 5–26°).

A projection of the whole molecule along the *b* axis is shown in Fig. 4, where short intramolecular distances of significance for the molecular conformation are also given. The molecular packing with indication of short intermolecular distances is given in Fig. 5. Distances to bromines less than 3.9 Å and other distances less than 3.5 Å are indicated.

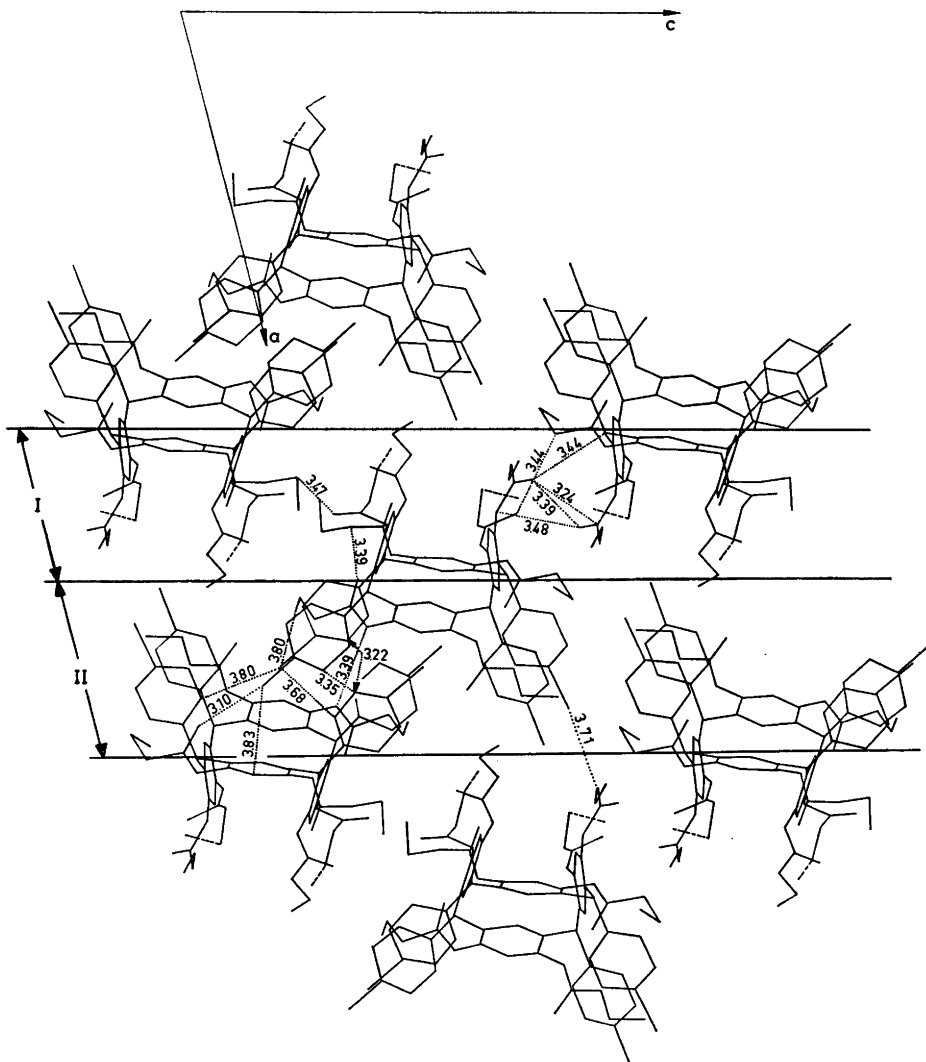


Fig. 5. Molecular packing viewed along the *b* axis with indication of short intermolecular distances. I and II designate the layers dominated by chains and rings, respectively.

The shortest distance between aromatic carbons in rings belonging to different molecules is 3.22 Å ($3\sigma = 0.10$ Å) and the shortest intermolecular Br—C distance is 3.68 Å ($3\sigma = 0.07$ Å) and 3.71 Å ($3\sigma = 0.08$ Å) to sp^2 and sp^3 hybridized carbons, respectively. The shortest intermolecular Br—O distance is 3.10 Å ($3\sigma = 0.05$ Å) between Br(1) and O(10), somewhat shorter than the expected van der Waals' distance which is about 3.35 Å. It is known, however, that the effective radius of an atom in a direction that makes only a small angle with the direction of a covalent bond formed by the atom is smaller than the van der Waals' radius in directions away from the bond.⁷ In our case this angle is 17° at the bromine and 30° at the oxygen.

The molecular packing may be visualized in terms of two alternating layers, parallel to the bc plane, each of thickness about $a/2$. One layer, denoted I in Fig. 5, is dominated by chains, whereas the other layer, denoted II, is dominated by rings. In layer I there are a few short intermolecular distances between chain atoms, mostly including the keto oxygens, but no regular chain packing exists. Except for a methyl-bromine distance of 3.80 Å there are no short distances to the end methyl groups in any of the two layers, and very few to other chain carbons. All chain carbons showing some disorder lack close contacts.

In layer II the two p -bromophenyl groups E and H pack with each other across centres of symmetry, and there are also some short distances from the bromines of these two groups and of group G to a number of atoms at various places in the molecule. Only to the atoms of p -bromophenyl group F there are no close contacts, and the atoms of this group accordingly show specially violent vibrations discussed earlier.

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REFERENCES

1. Erdtman, H. and Högberg, S. *To be published.*
2. Abrahamsson, S. and Maslen, E. N. *Z. Krist.* **118** (1963) 1.
3. Abrahamsson, S., Aleby, S., Larsson, K., Nilsson, B., Selin, K. and Westerdahl, A. *Acta Chem. Scand.* **19** (1965) 758.
4. Abrahamsson, S. *Arkiv Kemi* **25** (1966) 211.
5. Hamilton, W. C., Rollett, J. S. and Sparks, R. A. *Acta Cryst.* **18** (1965) 129.
6. Mills, O. S. and Rollett, J. S. *Computing Methods and the Phase Problem in X-ray Crystal Analysis*, Pergamon, London 1960, p. 107.
7. Pauling, L. *The Nature of the Chemical Bond*, Cornell University Press, Ithaca, New York 1960, p. 263.

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