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The Crystal and Molecular Structure of DL-3-Bromo-octadecanoic acid

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The crystal structure of DL-3-bromo-octadecanoic acid, $C_{18}H_{35}O_2Br$, has been determined from three-dimensional X-ray diffraction data. The crystals are triclinic, with $a=5{\cdot}68$, $b=5{\cdot}63$, $c=32{\cdot}8$ Å; $\alpha=101{\cdot}8°$, $\beta=93{\cdot}1°$, $\gamma=97{\cdot}9°$; space group $P\bar{1}$. Positional and anisotropic thermal parameters have been refined by least-squares methods; the final R index is 8·5 %.

The hydrocarbon chain is bent at C(3) to accommodate the bromine atom; the carbon chain from C(4) to C(18) is planar and regular; the packing of neighbouring chains is of the triclinic type observed in many other long-chain compounds; the carboxyl groups are joined in pairs, by hydrogen bonds, across centres of symmetry.

Introduction

We have earlier studied the effect on the molecular packing of a branching methyl group in different positions along a hydrocarbon chain (Abrahamsson, 1959*a*). A methyl group is either accommodated in the structure between the ends of straight chains or at the apices of V-shaped chains. In the series of racemic methyloctadecanoic acids all acids except DL-3-methyloctadecanoic acid were shown to belong to two main structure types. In order to determine the structure of the β-branched acid we have investigated DL-3-bromo-octadecanoic acid. Compounds with a bromine atom substituted in place of a methyl group are often isotypical with the corresponding unsubstituted ones. This substitution technique is now used systematically in our lipid work to facilitate structure determinations.

The preliminary X-ray work on DL-3-bromo-octadecanoic acid showed, however, that the acid was not isotypical with the corresponding methyl branched acid but isostructural with DL-2-methyloctadecanoic acid (Abrahamsson, 1959*b*). As the latter acid was studied in projection only, we have carried out this analysis to determine the molecular conformation and packing in detail.

Experimental

DL-3-bromo-octadecanoic acid, $CH_3 . [CH_2]_{14} . CHBr . CH_2 . COOH$, was synthesized by Dr I. Pascher from gas-chromatographically pure stearic acid. The final product has a melting point of 53·7–53·8 °C. Crystallization from light petroleum gave lath-shaped crystals suitable for X-ray work.

The crystals are triclinic; rotation and Weissenberg photographs, taken with Cu Kα radiation ($\lambda=1{\cdot}542$ Å) gave

$a=5{\cdot}68\pm0{\cdot}03$ Å	$\alpha=101{\cdot}8°$
$b=5{\cdot}63\pm0{\cdot}03$	$\beta=93{\cdot}1$
$c=32{\cdot}80\pm0{\cdot}2$	$\gamma=97{\cdot}9$

The space group $P\bar{1}$ was assumed, and no indications of non-centrosymmetry have been observed. There are two molecules per unit cell. A subcell was evident from the distribution of intensities; the subcell parameters

$a_s=4{\cdot}27$ Å	$\alpha_s=80{\cdot}4°$
$b_s=5{\cdot}29$	$\beta_s=105{\cdot}9$
$c_s=2{\cdot}55$	$\gamma_s=119{\cdot}5$

have been calculated from the final atomic positions.

*h*0*l* to *h*2*l* and 0*kl* to 3*kl* intensities were estimated visually, and corrected for Lorentz and polarization effects, but not for absorption or spot shape effects. 1486 independent reflexions were recorded; the minimum plane spacing was 1·1 Å, and within this limit another 200 reflexions were too weak to be observed.

Determination and refinement of the structure

The bromine position was determined from sharpened (100) and (010) Patterson projections; most of the carbon and oxygen atom positions were found by superposition of these sharpened Patterson projections, and the remainder from electron-density projections. These parameters were first refined by two three-dimensional difference syntheses; in each of the structure factor calculations the coordinates of the carbon atoms C(4) to C(18) were constrained to be those of a planar, regular chain – deviations suggested by the difference syntheses were ignored. Hydrogen atom peaks appeared at all of the expected positions in the second difference synthesis. Eight cycles of block-diagonal least-squares refinement were carried out, initially treating all atoms as isotropic. In the third and subsequent cycles hydrogen atoms at the stereochemically expected positions on C(2) to C(15), and on O(1) were included in F_c. Anisotropic vibration parameters were introduced for bromine at the fifth cycle, and for carbon and oxygen atoms at the seventh cycle. Reflexions with $F<30$ were given weight 1, and the others weight $(F/30)^2$ in

Table 1. *Observed and calculated structure factors*

A line is either *hk* or *l* F_o F_c; F_o and F_c are 5 times the absolute values for one unit cell.

0 0

3	29	62
4	226	-243
5	196	-184
6	263	-234
7	53	-68
8	19	19
9	161	159
10	159	166
11	152	150
12	59	58
13	31	-33
14	91	-102
15	109	-126
16	81	-86
17	41	-43
18	50	41
19	72	79
20	117	128
21	223	256
22	49	51
25	47	-54
26	29	-37

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-19	60	-57
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-13	235	248
-12	202	209
-11	124	132
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-7	242	-295
-6	249	-319
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-2	24	27
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2	109	82
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4	142	-171
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6	212	189
7	310	312
8	270	266
9	181	188
10	31	31
11	109	-96
12	207	-199
13	229	-217
14	170	-165
15	87	-92
17	55	51
18	78	70
19	32	31
20	16	11
21	55	-46
22	41	-29
23	58	-50
24	199	-187
25	62	-59
26	22	23
27	22	21
28	46	40
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30	18	16
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0	261	-305
1	218	-217
2	146	-131
4	62	69
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6	147	167
7	52	46
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9	121	-127
10	140	-151
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7	109	-108
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13	63	70
14	50	50
17	53	-53
18	62	-46

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10	74	80
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16	58	-69
17	43	-51

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8	214	-206
9	150	-150
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12	78	-79
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15	152	147
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9	190	162
10	248	248
11	279	235
14	71	-72
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16	159	-144
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9	130	126
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11	130	-118
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13	273	-272
14	316	-296
15	207	175
16	118	123
17	83	88
18	142	148
19	83	91
20	71	76
23	34	-45

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28	27	23

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3	106	104
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8	165	-157
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12	128	128
13	118	127
14	88	99
15	28	34
17	49	-58
18	43	-48
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23	10	14
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25	10	-5
26	15	-21
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Table 1 (*cont.*)

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	1	5				16	122	137	4	71	-60	-13	103	102	25	63	63						
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-6	78	77	-25	38	37	21	65	-78	11	102	102	-8	158	-170	-21	55	58						
-5	100	107	-24	58	55	22	50	-61	12	125	128	-7	143	-150	-20	96	112						
-3	112	-125	-23	66	58	23	38	-35	13	99	102	-6	124	-119	-19	28	14						
-2	119	-111	-22	53	52				14	147	-145	-4	204	216	-18	41	44						
1	87	-89	-21	27	17		2	1	15	106	-113	-3	87	85	-17	52	69						
4	105	90	-20	31	-29	-33	34	-30	16	55	-61	-2	60	65	-15	35	-25						
5	122	132	-19	53	-55	-32	28	-26	17	49	-45	-1	62	64	-14	96	-99						
6	130	134	-18	83	-90	-31	22	20	19	28	36	0	46	-39	-13	105	-119						
7	87	87	-17	22	-14	-30	18	18	20	58	55	1	106	-102	-12	87	-105						
10	77	-61	-16	145	130	-29	29	20	21	43	43	2	155	-161	-11	35	-31						
			-14	59	55	-28	32	28				3	149	-156	-10	35	47						
	2	-3	-13	167	166	-27	29	14		2	3	4	103	-99	-9	111	128						
-14	106	-94	-12	134	131	-25	52	-45	-10	56	-53	5	13	-18	-8	158	164						
-13	125	-115	-11	112	114	-24	94	-80	-9	90	-95	6	75	78	-7	159	163						
-12	106	-96	-9	87	-80	-23	96	-97	-8	106	-119	7	118	115	-6	88	97						
-9	136	109	-8	159	-159	-22	88	-86	-7	69	-79	8	128	120	-4	69	-75						
-8	150	150	-7	124	-132	-21	52	-54	-6	69	-66	9	55	53	-3	108	-118						
-7	146	153	-6	75	-79	-19	74	74	-4	97	89	10	12	-24	-2	117	-117						
-6	86	73	-5	74	65	-18	97	100	-3	106	112	11	128	-123	-1	53	-52						
-4	136	-122	-4	150	134	-17	102	101	-2	88	82	12	155	-156	0	58	50						
-3	168	-156	-3	230	231	-16	53	49	1	87	-80	13	170	-181	1	153	155						
-2	215	-209	-2	193	199	-14	94	-87	2	109	-103	14	109	-122	2	46	66						
-1	161	-159	-1	168	175	-13	94	-111	3	97	-96	15	63	-65	3	91	103						
0	209	198	1	96	-82	-12	198	-192	4	59	-50	16	27	35	4	105	99						
1	78	70	2	198	-183	-11	156	-155				17	235	258	6	31	-38						
3	78	66	3	180	-181	-10	274	258		2	4	18	35	45	7	106	-116						
6	150	-146	4	131	-122	-9	158	140	-11	59	-54	20	31	37	8	72	-89						
7	161	-175	6	164	150	-8	106	125	-10	59	-61	21	15	-15	9	63	-74						
8	158	-172	7	215	218	-7	115	115	-6	80	69	22	19	-20	11	60	62						
9	131	-117	8	254	256	-5	75	-70	-5	80	77	23	46	-54	12	103	112						
11	72	61	9	186	193	-4	189	-193	-4	46	65	24	27	-28	13	90	107						
12	133	126	10	131	120	-3	201	-211	-2	81	-82	25	19	-22	14	84	99						
13	133	122	11	15	-20	-2	207	-207	-1	93	-82	26	10	6	15	34	35						
14	99	97	12	56	-61	-1	108	-96	0	81	-79	27	10	14	17	60	-63						
17	91	-97	13	128	-121	0	34	31	1	59	-65	28	10	16	18	53	-66						
			14	65	-69	1	171	167				29	10	11	19	58	-61						
	2	-2	15	37	-36	2	242	247		3	-4	30	10	-10	20	25	-27						
-27	25	-29	16	49	53	3	263	253	-6	80	39	32	25	-22	23	47	57						
-26	41	-36	17	69	70	4	193	190	-3	52	-75	33	21	-21									
-25	46	-41	18	91	97	5	71	67	-2	81	-80	34	21	-21		3	1						
-24	19	-22	19	52	56	6	43	-45	-1	81	-52	35	16	-13	-28	29	-26						
-22	38	36	21	61	-53	7	173	-169	2	88	106				-27	52	-50						

Table 1 (*cont.*)

-26	68	-64	6	46	45	-11	25	-28	-22	55	-55	-19	28	20	-9	31	40
-25	63	-55	7	209	232	-9	74	74	-21	74	-76	-17	13	-19	-8	49	60
-24	38	-28	8	72	73	-8	106	116	-20	19	-20	-16	50	-46	-6	44	-48
-22	38	41				-7	56	52	-19	22	-19	-15	44	-36	-5	18	-19
-21	80	76		3	3	-6	16	23	-17	24	23	-12	40	36	-4	18	-20
-20	84	76	-11	114	-102	-5	46	-39	-16	37	35	-11	71	65	-3	41	-43
-19	80	82	-10	80	-76	-4	97	-102	-15	50	51	-10	66	63	-2	28	-29
-17	145	-141	-6	71	70	-3	137	-136	-14	43	41	-9	46	45	-1	25	-24
-16	40	-34	-5	86	82	-2	128	-130	-12	21	-18	-7	46	-44			
-15	49	-44	-4	71	70	-1	84	-82	-11	50	-46	-6	77	-73		6	-2
-14	50	-54	5	58	51	1	55	54	-10	55	-54	-5	84	-87	-17	19	-21
-12	32	28	11	131	-68	2	105	103	-9	35	-37	-4	68	-68	-16	21	-26
-11	86	82				3	105	100	-8	10	-2	-3	29	-25	-15	21	-26
-10	75	79		4	-2	4	62	65	-7	31	36	-2	27	26	-7	21	-22
-9	31	48	-18	28	30	5	16	-15	-6	60	55	-1	63	60	-6	24	-25
-8	38	-32	-17	56	60	6	81	-80	-5	65	58	0	75	70	-5	22	-29
-7	109	-105	-16	71	74	7	119	-133	-4	37	36	1	50	48	-4	13	8
-6	107	-162	-15	69	69	8	112	-123	-3	15	-18	2	13	14	-3	29	31
-5	170	-173	-14	32	31	9	96	-100	-2	63	-64	3	41	-39	-1	24	21
-4	130	-133	-12	81	-87	10	29	-24	-1	149	-156	4	62	-62	0	52	57
-3	53	-54	-11	122	-129	11	32	30	0	164	-170	5	97	-94	1	29	36
-2	44	39	-10	34	-25	12	99	106				6	65	-59	3	16	-23
-1	106	106	-9	46	-39	13	204	188		5	-2	7	97	94	4	44	-40
0	133	138	-8	32	-30	14	22	-18	-20	16	21	8	47	45	5	49	-55
1	96	99	-7	44	47	15	31	-35	-19	34	43	9	41	38	6	41	-48
2	41	36	-6	75	80	16	32	-30	-18	22	25	10	83	65	7	24	-21
3	118	-118	-5	114	107	17	71	-66	-17	15	-15	11	43	40	8	13	2
4	285	-287	-4	87	78	18	62	-55	-14	40	-43	12	29	23	9	16	19
5	63	-60	-3	32	24	19	46	-50	-13	49	-46	13	22	-21	10	22	29
6	52	-53	-2	37	-39	22	31	30	-12	44	-45	14	52	-40	11	19	30
7	60	-55	-1	87	-93	23	35	34	-11	10	-15	15	66	-58	12	12	18
8	40	37	0	99	-102	24	29	25	-10	15	20	16	55	-51	13	12	-9
9	50	45	1	75	-75	27	16	-18	-9	43	45	17	32	-29	14	15	-23
10	80	86	3	55	51	28	18	-20	-8	50	60	19	21	22	15	18	-26
11	44	51	4	114	104	29	19	-14	-7	44	46	20	35	28	16	19	-24
13	96	-47	5	114	107	33	24	21	-6	15	15	21	40	29	21	19	26
14	97	-69	6	96	88		4	1	-5	27	-23	22	18	18			
15	71	-80	7	18	14				-4	50	-52	23	15	4		6	-1
	3	2	8	38	-40	-29	22	-20	-3	68	-60	24	19	-15	-15	18	21
-28	21	-25	9	130	-127	-28	21	-20	-2	43	-47	25	34	-24	-14	32	33
-27	24	-23	10	176	-177	-25	50	48	-1	19	-11	26	43	-30	-13	38	32
-26	15	-16	11	63	62	-24	55	54	0	37	41	27	22	15	-12	22	25
-24	19	19	14	71	59	-22	29	24	1	78	70	28	40	37	-11	12	2
-23	27	32	15	58	58	-21	32	30	2	97	108				-10	28	-25
-22	19	20	16	74	75	-19	19	-14	3	72	70		5	1	-9	40	-44
-20	40	-36	17	32	22	-18	37	-35	4	40	-45	-17	28	29	-8	41	-48
-19	56	-60	19	32	-36	-17	29	-24	5	41	31	-16	35	31	-7	40	-35
-18	81	-79	20	37	-51	-15	44	44	6	15	14	-15	22	25	-6	16	-14
-17	60	-63	21	38	-50	-14	80	70	7	53	-51	-12	44	-45	-4	41	40
-16	68	-70	22	31	-30	-13	90	76	8	49	-49	-11	65	-60	-3	46	45
-15	18	15	25	27	25	-12	56	58	9	52	-50	-10	46	-35	-2	52	48
-14	150	151	26	27	28	-11	22	16	11	19	13	-6	58	50	0	47	-45
-13	87	82	27	22	21	-10	37	-41	12	46	45	-5	60	53	3	29	-25
-12	59	55	29	16	-13	-9	83	-83	13	43	49	-4	62	59	6	37	33
-11	40	41	30	24	-30	-8	102	-105	14	31	32	-3	37	31	7	41	40
-10	29	-20	31	31	-36	-7	90	-94	16	18	-16	-1	19	-23	8	40	40
-9	12	-48	32	15	15	-6	47	-44	17	29	-26	0	27	-19	9	28	24
-8	83	-79		4	-1	-5	12	17	18	29	-33		5	2	11	22	-22
-7	96	-89	-25	37	-26	-4	93	95	19	16	-25	-24	15	-17	12	34	-31
-6	32	-30	-24	53	-48	-3	108	108	21	13	23	-23	15	-17	13	27	-28
-5	25	23	-23	55	-54	-1	38	40	22	29	34	-22	12	-16	14	22	-18
-4	84	81	-22	47	-44	0	27	30	23	32	40	-20	13	18	16	13	16
-3	114	117	-21	18	-20	1	27	-24	24	18	21	-19	28	30	17	31	27
-2	118	119	-19	34	33	2	25	-23	25	19	-21	-18	34	38	18	25	24
-1	71	65	-18	59	50	3	43	-41	26	10	9	-17	31	31	19	18	16
1	71	-70	-17	52	48	4	22	-8	28	16	-21	-16	18	15	20	12	-11
2	118	-112	-16	25	22		4	2		5	-1	-14	22	-23	21	32	-33

the refinement. The atomic scattering factors given in *International Tables for X-ray Crystallography* (1962), p. 202, were used for carbon, oxygen and hydrogen atoms; the bromine scattering factor on p. 211 (from a Thomas–Fermi–Dirac statistical wave function) was used, corrected for anomalous dispersion(p. 214). The final R index, for observed reflexions only, was 0·085. No shift in the last two cycles was greater than half the appropriate estimated standard deviation.

Observed and calculated structure factors are given in Table 1, positional and thermal parameters in Table 2, and bond lengths and angles in Table 3. The positional standard deviations were estimated from the reciprocals of the diagonal elements of the last least-squares matrix, and will therefore be slightly underestimated. The estimated standard deviations in bond lengths take account of the possible errors in cell dimensions, as well as the above positional errors (the former are only important in the C–Br bond).

Discussion of the structure

Hydrocarbon chain configuration

The mean C–C bond length is 1·526 Å and the mean C–C–C angle 113°. The conformation around the bond C(3)–C(4) is such that bromine is approximately *trans* to C(5) (so occupying the position that C(2) would have occupied in the absence of a branch); the dihedral angle Br–C(3)–C(4)–C(5) is 172°. The mean plane of the atoms C(4) to C(18) in terms of the orthogonal coordinates of Table 2(*a*) is

$$0{\cdot}7013X+0{\cdot}2157Y-0{\cdot}6794Z-0{\cdot}652=0\,.$$

Table 2(*a*). *Fractional coordinates, x, y, z, and orthogonal coordinates, X, Y, Z, of atoms other than hydrogen, and their estimated standard deviations*

The orthogonal coordinates refer to axes with X parallel to **a***, and Z parallel to **c**. σ is $\sigma(X)$ or $\sigma(Y)$ or $\sigma(Z)$, which are equal.

	x	y	z	X (Å)	Y (Å)	Z (Å)	σ (Å)
Br	0·2552	1·2491	0·09687	1·431	6·207	2·989	0·0015
O(1)	0·288	0·675	−0·0047	1·615	3·621	−0·288	0·008
O(2)	0·074	0·638	0·0486	0·414	3·222	1·525	0·008
C(1)	0·259	0·713	0·0359	1·452	3·583	1·029	0·012
C(2)	0·479	0·846	0·0626	2·686	3·988	1·782	0·012
C(3)	0·423	0·972	0·1054	2·370	4·453	3·184	0·011
C(4)	0·631	1·071	0·1368	3·537	4·642	4·093	0·011
C(5)	0·748	0·857	0·1491	4·191	3·261	4·431	0·013
C(6)	0·936	0·947	0·1862	5·248	3·371	5·535	0·012
C(7)	1·049	0·740	0·1974	5·878	2·042	5·839	0·011
C(8)	1·242	0·819	0·2341	6·960	2·094	6·926	0·012
C(9)	1·368	0·615	0·2442	7·668	0·774	7·189	0·012
C(10)	1·552	0·700	0·2812	8·697	0·861	8·292	0·012
C(11)	1·672	0·491	0·2910	9·374	−0·475	8·547	0·013
C(12)	1·861	0·567	0·3280	10·432	−0·440	9·646	0·013
C(13)	1·983	0·358	0·3382	11·116	−1·782	9·915	0·013
C(14)	2·170	0·437	0·3756	12·164	−1·734	11·028	0·014
C(15)	2·293	0·228	0·3860	12·853	−3·081	11·305	0·015
C(16)	2·473	0·311	0·4239	13·864	−3·007	12·434	0·016
C(17)	2·594	0·094	0·4321	14·540	−4·381	12·642	0·017
C(18)	2·775	0·167	0·4694	15·554	−4·357	13·752	0·021

Table 2(*b*). *Anisotropic thermal vibration parameters*

The temperature factor of each atom is
$\exp(-b_{11}h^2-b_{22}k^2-b_{33}l^2-b_{23}kl-b_{31}lh-b_{12}hk)$

	$10_4\times b_{11}$	b_{22}	b_{33}	b_{23}	b_{31}	b_{12}
Br	401	379	17·7	41	−2	246
O(1)	498	546	10·4	11	−4	−30
O(2)	350	425	11·7	15	−1	−80
C(1)	423	342	12·2	22	1	170
C(2)	425	479	10·1	37	−21	118
C(3)	257	346	13·3	6	−16	161
C(4)	309	166	16·5	15	−26	79
C(5)	341	563	15·3	−8	−29	80
C(6)	415	349	14·5	33	−4	490
C(7)	291	401	11·5	23	−39	−184
C(8)	353	403	13·2	30	−12	114
C(9)	435	422	12·6	25	−24	339
C(10)	478	357	17·5	46	−22	659
C(11)	362	637	13·6	−7	−38	−118
C(12)	407	480	14·6	26	−5	234
C(13)	468	458	12·5	22	−32	192
C(14)	435	616	17·3	53	−4	178
C(15)	502	539	16·7	42	−34	437
C(16)	606	795	17·8	53	−64	369
C(17)	817	867	21·6	85	−101	522
C(18)	844	1314	26·5	96	−99	502

C(3) and Br are very significantly displaced from this plane, by 0·19 and 0·34 Å as shown in Fig. 1; C(4) and C(7) are displaced by small amounts which may just be significant.

There is a suggestion that, within its plane, the hydrocarbon chain is slightly curved. The odd carbon atoms deviate from collinearity by amounts just greater than three standard deviations, and in a systematic way; so do the even carbon atoms. This can be seen in their distances from the mean axis of the chain:

Carbon atom no.	Distance	Carbon atom no.	Distance
3	−0.53 Å	4	0.35 Å
5	−0.49	6	0.39
7	−0.43	8	0.39
9	−0.42	10	0.43
11	−0.41	12	0.41
13	−0.43	14	0.40
15	−0.44	16	0.42
17	−0.48	18	0.32

Table 3. *Bond lengths and angles*

Bond		Angle	
C(3)–Br	2·00 Å	Br–C(3)–C(2)	106°
C(1)–O(1)	1·33	Br–C(3)–C(4)	108
C(1)–O(2)	1·21	O(1)–C(1)–O(2)	122
C(1)–C(2)	1·50	O(1)–C(1)–C(2)	113
C(2)–C(3)	1·51	O(2)–C(1)–C(2)	126
C(3)–C(4)	1·49	C(1)–C(2)–C(3)	112
C(4)–C(5)	1·56	C(2)–C(3)–C(4)	116
C(5)–C(6)	1·53	C(3)–C(4)–C(5)	110
C(6)–C(7)	1·50	C(4)–C(5)–C(6)	112
C(7)–C(8)	1·53	C(5)–C(6)–C(7)	112
C(8)–C(9)	1·52	C(6)–C(7)–C(8)	114
C(9)–C(10)	1·51	C(7)–C(8)–C(9)	115
C(10)–C(11)	1·52	C(8)–C(9)–C(10)	113
C(11)–C(12)	1·53	C(9)–C(10)–C(11)	112
C(12)–C(13)	1·53	C(10)–C(11)–C(12)	114
C(13)–C(14)	1·53	C(11)–C(12)–C(13)	115
C(14)–C(15)	1·54	C(12)–C(13)–C(14)	114
C(15)–C(16)	1·52	C(13)–C(14)–C(15)	114
C(16)–C(17)	1·54	C(14)–C(15)–V(16)	113
C(17)–C(18)	1·50	C(15)–C(16)–C(17)	110
O(1)–H · · · O(2)	2·66	C(16)–C(17)–C(18)	112

Estimated standard deviations.
In bond lengths:

C–Br	0·014 Å	
C–O	0·014	
C–C	0·018	between C(1) and C(12), rising to
	0·021	at C(15) and
	0·028	at C(18)

In bond angles 1° to 1·5°.

Carboxyl group

The dimensions of the carboxyl group are in accord with those in other saturated fatty acids, for example with those collected by Higgs & Sass (1963). The mean plane of the atoms C(1), C(2), O(1), and O(2) is (in orthogonal coordinates)

$$-0{\cdot}3186X+0{\cdot}9479Y-0{\cdot}0029Z-2{\cdot}922=0\,,$$

and none of these atoms is significantly displaced from the plane. The carboxyl group related by the symmetry centre at $0\frac{1}{2}0$ is joined to this one by two hydrogen bonds of length 2·66 Å. The two carboxyl groups are not coplanar, but are in planes 0·49 Å apart; Such a displacement of the planes is common in solid fatty acids and has been commented on by Jeffrey & Sax (1963).

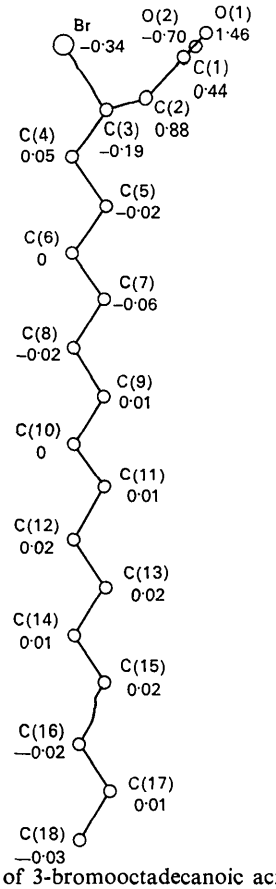

Fig. 1. One molecule of 3-bromooctadecanoic acid, projected onto the mean plane of the atoms C(4) to C(18). The displacements, (Å) of all atoms from this plane are shown.

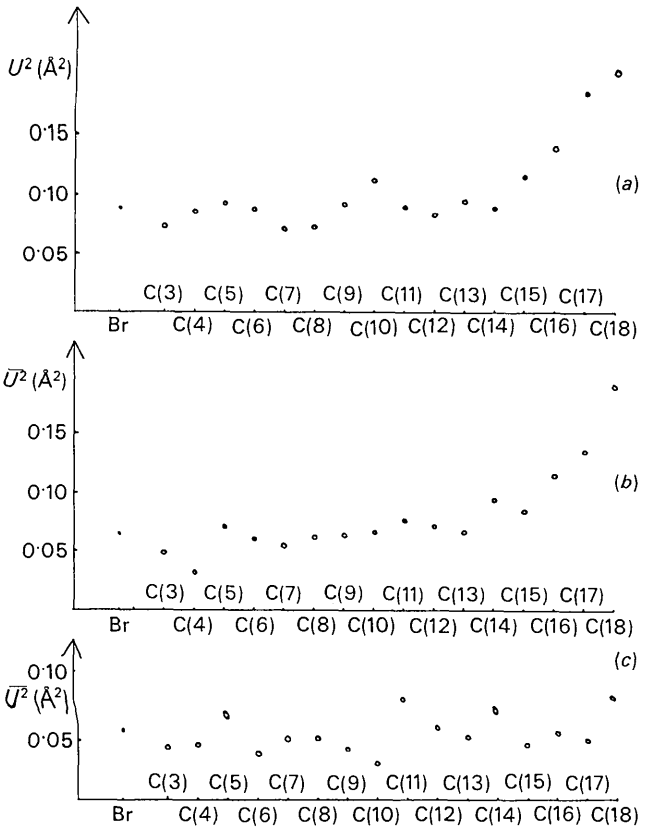

Fig. 3. The mean square amplitudes of vibration of atoms (*a*) perpendicular to the plane of the carbon chain, (*b*) in the plane of the chain and perpendicular to the chain axis, (*c*) parallel to the chain axis.

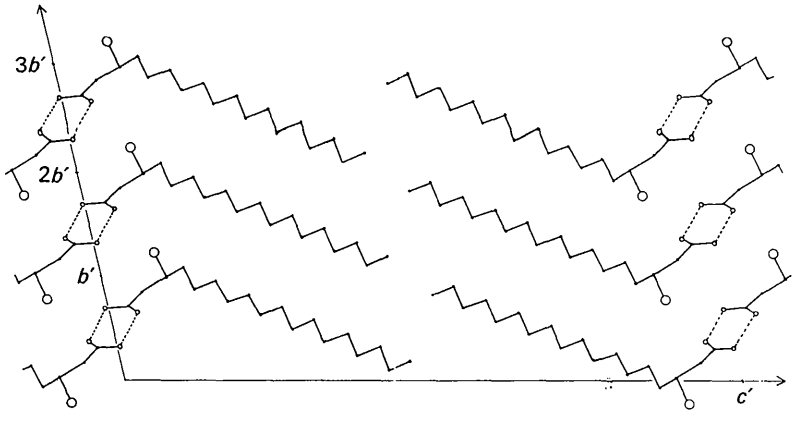

Fig. 2. (100) projection showing the packing of the molecules. (*b*′ and *c*′ are the projections of *b* and *c*.)

Packing arrangement

Fig. 2 shows the packing of the molecules in the lattice. The straight portions of the hydrocarbon chains are arranged in the triclinic form found in many other long chain compounds. The dimensions of the triclinic subcell (T ||) are given in the experimental section; they are close to those observed by Lomer (1963) in lauric acid.

There are two short intermolecular distances:

Br · · · C(1) at $010+xyz$	3·60 Å
Br · · · O(2) at $010\times xyz$	3·21

No others (except those between carboxyl groups) are less than 3·8 Å.

The angle of tilt of the chain axes to the end group planes is 36° as compared to 46° in DL-2-methyloctadecanoic acid.

Thermal vibrations

The anisotropic thermal parameters, b_{ij}, were converted to mean square amplitudes of vibration in the directions of the principal axes of the hydrocarbon chain [C(4)–C(18) portion], and these are shown in Fig. 3. Although the estimated standard deviations of the b_{ij} parameters suggest that variations between neighbouring atoms are not significant, the general pattern is physically sensible. The perpendicular vibrations [Fig. 3(*a*)] increase considerably towards the C(18) end of the chain; the increase is particularly marked from about C(14) onwards, *i.e.* for that part of the chain which is not flanked on all sides by other chains (see Fig. 2). The transverse vibrations [Fig. 3(*b*)] also increase towards the C(18) end, while the longitudinal vibrations [Fig. 3(*c*)] do not.

The initial calculations were done on the Saab D21 computer of the Institute of Medical Biochemistry, University of Göteborg. The remainder were done on the Atlas Computers at Manchester University and at the National Institute for Research in Nuclear Science in England. We are grateful to all these establishments and to the Edinburgh University Computer Unit for their cooperation, to Dr R. Diamand for his least-squares refinement program, and to Dr L. Hodgson for a bond-length and angle program. We also thank Mrs M. Innes, Mr U. Lövås and Mr A. Westerdahl for technical assistance. Financial support has been obtained from the Swedish Natural Science and Swedish Medical Research Councils.

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An Experimental Determination of $\Delta f''$ for Iodine

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The imaginary component of the anomalous contribution to the scattering factor ($\Delta f''$) of iodine for Cu $K\alpha$ radiation has been determined with the intensity data from the structure analysis of methyl melaleucate iodoacetate by Hall & Maslen (1965). The reliability of the calculation is shown to depend critically on the weighting of all terms, but particularly on those where the Bijvoet inequality is immeasurably small. A method for deriving the correct weighting scheme from an assessment of the errors is developed.

The results obtained are in reasonable agreement with those from theoretical calculations. The angular dependence of the $\Delta f''$ curve corresponds closely to that predicted. There is a small discrepancy in scale, but this may have resulted from an error in the experimental value.

Introduction

In recent years several experimental determinations of the anomalous dispersion corrections to the atomic scattering factors have been carried out. These were necessary both to confirm the theoretical values and to investigate the dependence of $\Delta f''$ on the presence of more than one anomalous scatterer in the unit cell. In general the experimental zero Bragg angle values are in good agreement with values calculated by James (1954) from the wave mechanical theory of Hönl (1933) and by Dauben & Templeton (1955) and Cooper (1963) from the work of Parratt & Hempstead (1954) and Eisenlohr & Muller (1954). Recently the corrections at zero Bragg angle have been re-evaluated with the

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