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The Crystal Structure of Ethyl Stearate. A Correction

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Ethyl stearate has the monoclinic unit cell $a = 5.59$, $b = 7.40$, $c = 55.0$ Å, $\beta = 113.5°$. The cell contains 4 molecules and the space group is Aa. A 3-dimensional least-squares refinement has been performed to an R-value of 0.125. The chains are arranged in the orthorhombic packing O ⊥ (101). The molecules form sheets with the chains tilting 63° towards the end group planes. The alcohol chain forms a continuation of the main chain but a twist occurs at the carboxyl group.

A structure determination of ethyl stearate in two projections has been described in Ref. 1. It was pointed out by Mathieson and Welsh [2] that the space group should probably be the same as they had found in ethyl behenate. This should give a possibility for an arrangement of the atoms in the alcohol group which is more consistent with other data. It was therefore decided to make a new study using 3-dimensional data.

The unit cell given in Ref. 1 has the dimensions $a = 5.59$, $b = 7.40$, $c = 57.1$ Å, $\beta = 118°$.

Due to the bad quality of the reflection data on the original films especially for the higher zones (diffuse reflections and streaking) and the small distances between the spots caused by the long c-axis the space group was erroneously determined to be Aa instead of Ia. The latter is in accordance with Mathieson and Welsh [2] and corresponds to a new unit cell with space group Aa and the following dimensions $a = 5.59 \pm 0.02$, $b = 7.40 \pm 0.02$, $c = 55.0 \pm 0.3$ Å, $\beta = 113.5° \pm 0.5°$. The coordinates in the new cell are related to the coordinates given in Table 1 in Ref. 1 by the transformation matrix

$$\begin{pmatrix} 1 & 0 & \bar{1} \\ 0 & 1 & 0 \\ 0 & 0 & 1 \end{pmatrix}$$

A new set of observed reflections was formed, including those in Tables 2 and 3 in Ref. 1. For $00l$-reflections the average from the $0kl$- and the $h0l$-zones was used. The new reflections belonging to the zones $h1l$ and $h2l$ were estimated visually as the original data. The total number of reflections used

was 316. This corresponds to only 13 % of the reflections within 1/4 of the copper sphere. Within the zones used 25 % of the reflections were observed. It may be remarked that in a regular structure of this type the few subcell reflections are much stronger than the majority of reflections which therefore often fall below the observable limit. It was not possible to obtain better data as the reflections outside the measured zones were of such a bad quality that they could not be estimated with any reasonable accuracy.

REFINEMENT

The coordinates in Ref. 1 were transformed to the new cell. The atoms C_{01} and C_{02} were then considered to form a straight continuation of the main chain by increasing their x/a-coordinates by 0.5. Isotropic temperature factors for each atom were taken as the average of the values B_{yz} and B_{xz} given in Table 1 in Ref. 1. The hydrogen atoms were placed 1.09 Å from the corresponding carbon atoms using tetrahedral angles.

Rounds of least-squares calculations were then performed with a full-matrix programme described by Abrahamsson [3] and Aleby.[4] The weighting function used was $w = [1 + (F_{obs}-a)^2/b^2]^{-1}$ with $a = b = 3.5\ F_{min.}$ 14 very strong reflections showing poor agreement were excluded from the refinement. To avoid singular matrices the x- and z-coordinates of C_{02} were kept fixed during the refinement (*cf.* Templeton [5]). The final R-value for the refined reflections was 0.10 compared with the values 0.13 and 0.16 for $0kl$ and $h0l$, respectively, in Ref. 1. At the end of the refinement the average position shift was about 0.05 of the average standard deviation while the maximum shift was 0.08 of the corresponding standard deviation. The refinement was checked by difference Fourier summations which showed no contradicting features.

The standard deviations (calculated according to Cruickshank [6]), however, were very large, the average being 0.088 Å in the position coordinates. Even considering the limited quality and amount of data this seemed too high. According to Geller [7] and Scheringer [8] a high correlation between the refined parameters could lead to a bad condition of the normal equation matrix which then shows up as high standard deviations. To investigate this the correlation coefficients were calculated from the elements of the inverse of the normal equation matrix. This showed that the average correlation between the x-coordinates was 0.69 and between the z-coordinates 0.72 while the correlation between y-coordinates was on the average 0.09 and between isotropic temperature factors 0.02. The correlations between the parameters within the regular part of the molecule (C_2—C_{18}) did not differ significantly from the corresponding correlations within the group C_1, O_1, O_2, C_{01}, C_{02}. If the two groups of atoms were refined separately the correlations between x- and z-parameters dropped to 0.16 and 0.21, respectively, in both cases with the y- and B-correlations at a low level as before. These figures indicate that for molecules with a high degree of regularity in a polar space group, refinement of many atoms at the same time is likely to cause the normal equation matrix to be badly conditioned.

Table 1. Coordinates and temperature factors.

	x/a	*y/b*	*z/c*	*B*
C_{18}	−0.241	0.201	0.4588	8.75 Å²
C_{17}	−0.126	0.300	0.4437	6.48
C_{16}	−0.218	0.192	0.4153	6.65
C_{15}	−0.100	0.297	0.3976	5.89
C_{14}	−0.193	0.191	0.3710	5.40
C_{13}	−0.062	0.290	0.3542	5.40
C_{12}	−0.158	0.202	0.3278	4.96
C_{11}	−0.038	0.284	0.3072	5.14
C_{10}	−0.129	0.208	0.2816	3.12
C_9	−0.012	0.285	0.2640	5.64
C_8	−0.104	0.212	0.2370	5.34
C_7	0.027	0.285	0.2185	6.61
C_6	−0.085	0.214	0.1897	3.15
C_5	0.056	0.289	0.1744	7.80
C_4	−0.049	0.213	0.1453	4.59
C_3	0.087	0.277	0.1296	4.15
C_2	−0.039	0.216	0.0988	5.21
C_1	0.115	0.246	0.0841	3.48
O_1	−0.042	0.239	0.0573	6.10
O_2	0.301	0.329	0.0902	7.31
C_{01}	0.131	0.254	0.0410	6.80
C_{02}	−0.049	0.248	0.0152	13.22

It was therefore decided to start the refinement from the beginning and refine one of the above groups at a time for some cycles and then the other group. Each group was refined for about 4 cycles. This procedure was repeated 3 times. The weighting scheme used was the same as before but for the last two sets of cycles $b = 4.0\ F_{min}$. This time the final R-value was 0.11 for the refined reflections and for all observed reflections 0.125. In the final cycles the average position shift was about 0.02 of the standard deviations and the maximum position shifts about 0.05 of the standard deviations. The final coordinates did not differ significantly from those of the first refinement. In both cases the differences from the starting coordinates were small except for x- and z-coordinates of the atoms C_1, O_1, O_2, C_{01}, and C_{02} which were shifted up to 0.28 Å from their original positions.

The average standard deviations were 0.065 Å in the position coordinates and 1.56 Å² in the isotropic temperature factors. These high values can be correlated not only with the limited data but also with the temperature factors being high (the average $B = 6$ Å²). This causes the atomic contributions to the structure factors to be small as will the derivatives and the elements of the normal equation matrix. The elements of the inverse matrix will then be large giving high standard deviations.

The final parameters (from the partial refinements) are given in Table 1 and the corresponding observed and calculated structure factors in Table 2 (note that h and l are interchanged because the calculations were performed in space group Cc instead of Aa). Table 3 gives the bond lengths and the angles between the bonds. The average of the C—C bonds between C_2 and C_{18} is 1.53 Å and of the angles at the same atoms is 112°. The average standard

Table 2. Observed and calculated structure factors (corresponding to a unit cell $a = 55.0$, $b = 7.40$, $c = 5.59$ Å, $\beta = 113.5°$, *i.e.* space group Cc). The phase angle FI is given in fractions of a revolution.

H	K	L	FO	FC	FI	H	K	L	FO	FC	FI	H	K	L	FO	FC	FI	H	K	L	FO	FC	FI
4	0	0	58	61	0.417	6	4	0	7	7	0.382	10	0	-4	8	6	0.769	27	-1	-3	11	13	0.459
6	0	0	58	60	0.475	8	4	0	7	12	0.428	8	0	-4	7	7	0.634	23	-1	-3	19	21	0.207
8	0	0	42	39	0.532	10	4	0	5	10	0.523	6	0	-4	6	7	0.410	21	-1	-3	71	79	0.152
10	0	0	19	18	0.604	12	4	0	5	9	0.591	4	0	-4	5	5	0.359	19	-1	-3	42	40	0.679
12	0	0	27	24	0.264	14	4	0	5	6	0.806	2	0	4	13	9	0.151	17	-1	-3	9	7	0.811
14	0	0	37	34	0.295	18	4	0	6	5	0.279	4	0	4	11	10	0.837	13	-1	-3	9	5	0.799
16	0	0	36	34	0.365	20	4	0	6	5	0.752	24	0	4	20	25	0.267	11	-1	-3	9	8	0.825
18	0	0	23	19	0.421	22	4	0	22	20	0.002	26	0	4	16	16	0.752	9	-1	-3	8	12	0.567
20	0	0	23	23	0.328	24	4	0	9	12	0.400	52	0	-6	5	3	0.290	7	-1	-3	9	13	0.390
22	0	0	118	118	0.019	44	4	0	11	12	0.531	20	0	-6	15	14	0.333	5	-1	-3	12	16	0.285
24	0	0	43	33	0.340	21	5	0	30	33	0.959	18	0	-6	25	25	0.840	3	-1	-3	11	10	0.071
26	0	0	35	32	0.436	23	5	0	37	41	0.505	4	0	6	11	12	0.154	1	-1	-3	13	8	0.954
28	0	0	27	24	0.493	43	5	0	7	7	0.065	49	-1	-1	9	7	0.746	1	1	3	50	52	0.265
30	0	0	14	15	0.640	45	5	0	11	13	0.527	47	-1	-1	18	15	0.545	3	1	3	27	29	0.875
32	0	0	10	12	0.825	0	6	0	17	14	0.500	45	-1	-1	42	40	0.530	21	1	3	11	12	0.244
34	0	0	10	12	0.198	2	6	0	9	14	0.755	43	-1	-1	41	43	0.072	23	1	3	32	38	0.340
36	0	0	8	7	0.088	21	7	0	16	20	0.479	41	-1	-1	14	9	0.217	25	1	3	32	34	0.804
38	0	0	7	5	0.364	23	7	0	12	20	0.984	39	-1	-1	18	18	0.021	41	-1	-4	9	8	0.147
40	0	0	6	8	0.468	66	0	-2	7	6	0.597	33	-1	-1	8	6	0.752	21	-1	-4	13	12	0.313
42	0	0	7	11	0.802	64	0	-2	6	7	0.231	29	-1	-1	6	6	0.191	19	-1	-4	10	9	0.757
44	0	0	35	32	0.490	56	0	-2	4	3	0.182	25	-1	-1	8	6	0.391	1	1	4	10	11	0.750
46	0	0	16	18	0.995	54	0	-2	8	9	0.282	23	-1	-1	13	13	0.424	3	1	4	14	13	0.272
48	0	0	7	7	0.946	52	0	-2	11	8	0.009	21	-1	-1	19	16	0.250	41	-1	-5	14	15	0.786
5	1	0	7	6	0.944	50	0	-2	11	7	0.026	19	-1	-1	25	24	0.221	23	-1	-5	13	14	0.273
7	1	0	11	9	0.173	48	0	-2	10	9	0.871	17	-1	-1	30	33	0.956	21	-1	-5	23	17	0.221
9	1	0	12	12	0.305	46	0	-2	19	16	0.614	15	-1	-1	27	33	0.856	19	-1	-5	46	46	0.793
11	1	0	6	7	0.480	44	0	-2	70	64	0.599	13	-1	-1	12	14	0.727	3	1	5	11	13	0.214
15	1	0	9	7	0.998	42	0	-2	42	48	0.119	11	-1	-1	11	10	0.037	30	-2	-1	7	7	0.627
17	1	0	12	11	0.046	40	0	-2	13	10	0.056	9	-1	-1	34	41	0.122	28	-2	-1	10	11	0.573
19	1	0	12	10	0.017	38	0	-2	5	6	0.979	7	-1	-1	55	63	0.039	26	-2	-1	9	10	0.577
21	1	0	35	34	0.948	36	0	-2	6	5	0.424	5	-1	-1	84	73	0.999	22	-2	-1	132	97	0.057
23	1	0	57	52	0.526	34	0	-2	13	11	0.351	3	-1	-1	65	71	0.968	20	-2	-1	59	46	0.508
25	1	0	9	5	0.485	32	0	-2	14	13	0.119	1	-1	-1	99	97	0.017	18	-2	-1	33	30	0.490
27	1	0	5	7	0.052	30	0	-2	13	12	0.964	1	1	1	314	327	0.439	16	-2	-1	16	19	0.428
29	1	0	5	6	0.157	28	0	-2	16	19	0.817	3	1	1	17	20	0.351	14	-2	-1	6	7	0.233
31	1	0	5	3	0.338	26	0	-2	27	28	0.703	5	1	1	18	18	0.135	12	-2	-1	12	12	0.924
45	1	0	10	8	0.535	24	0	-2	45	48	0.570	7	1	1	19	22	0.174	10	-2	-1	20	20	0.767
0	2	0	358	340	0.500	22	0	-2	97	92	0.553	9	1	1	6	6	0.312	8	-2	-1	24	21	0.651
2	2	0	35	36	0.787	20	0	-2	76	84	0.157	11	1	1	10	8	0.935	6	-2	-1	17	15	0.501
4	2	0	41	38	0.839	18	0	-2	24	24	0.184	13	1	1	23	19	0.920	4	-2	-1	7	10	0.239
6	2	0	37	34	0.954	16	0	-2	13	14	0.957	15	1	1	26	22	0.968	2	-2	-1	17	23	0.023
8	2	0	28	26	0.018	14	0	-2	13	12	0.740	17	1	1	19	20	0.049	0	2	1	85	63	0.984
10	2	0	15	15	0.096	12	0	-2	19	20	0.464	19	1	1	11	12	0.112	2	2	1	56	33	0.448
12	2	0	6	7	0.862	10	0	-2	21	18	0.225	21	1	1	17	17	0.195	4	2	1	28	30	0.482
14	2	0	11	10	0.779	8	0	-2	22	22	0.145	23	1	1	92	95	0.453	6	2	1	13	14	0.628
16	2	0	13	16	0.837	6	0	-2	19	20	0.014	25	1	1	10	16	0.704	8	2	1	14	15	0.816
18	2	0	11	12	0.890	4	0	-2	9	14	0.128	27	1	1	13	15	0.855	10	2	1	15	15	0.987
20	2	0	8	12	0.331	2	0	-2	28	28	0.198	29	1	1	8	10	0.910	12	2	1	11	16	0.187
22	2	0	69	69	0.510	0	0	2	86	109	0.835	45	1	1	15	14	0.900	14	2	1	8	9	0.407
24	2	0	26	21	0.874	2	0	2	78	107	0.407	31	-1	-2	8	6	0.693	16	2	1	8	13	0.588
26	2	0	18	20	0.926	4	0	2	19	22	0.121	29	-1	-2	10	6	0.492	18	2	1	8	11	0.720
28	2	0	13	15	0.991	6	0	2	23	23	0.161	27	-1	-2	9	3	0.501	22	2	1	37	33	0.360
30	2	0	7	10	0.140	8	0	2	21	21	0.261	25	-1	-2	6	9	0.748	24	2	1	27	26	0.009
32	2	0	5	7	0.303	10	0	2	13	16	0.523	23	-1	-2	14	17	0.798	44	-2	-2	40	45	0.092
34	2	0	5	7	0.699	12	0	2	13	15	0.798	21	-1	-2	63	54	0.105	42	-2	-2	26	32	0.632
44	2	0	22	23	0.000	14	0	2	18	19	0.904	19	-1	-2	24	23	0.542	26	-2	-2	10	16	0.088
46	2	0	12	13	0.458	16	0	2	15	17	0.957	17	-1	-2	16	17	0.436	24	-2	-2	25	29	0.084
48	2	0	5	6	0.442	18	0	2	10	11	0.957	15	-1	-2	11	11	0.311	22	-2	-2	61	56	0.065
1	3	0	10	12	0.952	20	0	2	13	13	0.874	13	-1	-2	7	6	0.037	20	-2	-2	59	53	0.658
3	3	0	7	9	0.326	22	0	2	34	34	0.936	11	-1	-2	9	9	0.798	18	-2	-2	16	18	0.735
5	3	0	7	9	0.444	24	0	2	55	64	0.375	9	-1	-2	12	13	0.683	16	-2	-2	7	13	0.543
7	3	0	10	12	0.684	68	0	-4	4	3	0.152	7	-1	-2	11	10	0.567	2	-2	-2	16	19	0.627
9	3	0	12	14	0.830	66	0	-4	4	4	0.099	5	-1	-2	7	7	0.377	0	2	2	69	56	0.384
11	3	0	11	11	0.019	64	0	-4	13	7	0.690	1	-1	-2	8	4	0.867	2	2	2	71	56	0.875
13	3	0	7	9	0.163	48	0	-4	7	4	0.861	1	1	2	35	37	0.940	4	2	2	8	11	0.705
15	3	0	9	10	0.444	46	0	-4	11	7	0.780	3	1	2	21	21	0.580	6	2	2	8	11	0.702
17	3	0	12	12	0.553	44	0	-4	11	9	0.773	5	1	2	14	14	0.521	22	2	2	25	25	0.458
19	3	0	12	12	0.561	42	0	-4	30	32	0.237	9	1	2	7	6	0.902	24	2	2	49	49	0.864
21	3	0	40	38	0.451	40	0	-4	13	9	0.729	23	1	2	14	14	0.288	24	-2	-3	10	13	0.341
23	3	0	61	61	0.018	38	0	-4	6	6	0.525	51	-1	-3	9	5	0.609	22	-2	-3	25	26	0.267
25	3	0	7	5	0.023	34	0	-4	6	6	0.963	49	-1	-3	9	6	0.671	20	-2	-3	35	31	0.681
27	3	0	6	8	0.524	32	0	-4	7	7	0.765	47	-1	-3	9	7	0.590	18	-2	-3	12	12	1.000
29	3	0	7	9	0.661	26	0	-4	10	11	0.704	45	-1	-3	55	49	0.167	16	-2	-3	8	11	0.854
31	3	0	6	5	0.854	24	0	-4	17	16	0.680	41	-1	-3	30	24	0.663	0	2	3	14	18	0.777
43	3	0	9	7	0.556	22	0	-4	14	15	0.579	39	-1	-3	10	10	0.502	2	2	3	32	34	0.343
45	3	0	16	16	0.033	20	0	-4	62	68	0.238	35	-1	-3	11	9	0.913	42	-2	-4	15	23	0.727
0	4	0	96	96	1.000	18	0	-4	19	19	0.672	33	-1	-3	16	13	0.788	20	-2	-4	57	52	0.739
2	4	0	21	22	0.321	14	0	-4	8	9	0.000	31	-1	-3	19	15	0.693	18	-2	-4	11	12	0.186
4	4	0	13	15	0.250	12	0	-4	10	10	0.813	29	-1	-3	16	17	0.548	18	-2	-6	18	19	0.342

deviations over the whole molecule are for the bonds 0.09 Å and for angles between the bonds 7°. In Fig. 1 is shown a composite drawing of the final electron density in sections close to the C- and O-atoms and parallel to the *ac*-plane.

The program system used is described by Abrahamsson, Aleby, Larsson, Nilsson, Selin and Westerdahl.[9]

Table 3. Bond lengths and angles.

$C_{18}-C_{17}$	1.44 Å	$C_{18}-C_{17}-C_{16}$	105°
$C_{17}-C_{16}$	1.65	$C_{17}-C_{16}-C_{15}$	107
$C_{16}-C_{15}$	1.58	$C_{16}-C_{15}-C_{14}$	105
$C_{15}-C_{14}$	1.55	$C_{15}-C_{14}-C_{13}$	105
$C_{14}-C_{13}$	1.57	$C_{14}-C_{13}-C_{12}$	107
$C_{13}-C_{12}$	1.48	$C_{13}-C_{12}-C_{11}$	115
$C_{12}-C_{11}$	1.65	$C_{12}-C_{11}-C_{10}$	117
$C_{11}-C_{10}$	1.41	$C_{11}-C_{10}-C_{9}$	115
$C_{10}-C_{9}$	1.48	$C_{10}-C_{9}-C_{8}$	117
$C_{9}-C_{8}$	1.46	$C_{9}-C_{8}-C_{7}$	118
$C_{8}-C_{7}$	1.57	$C_{8}-C_{7}-C_{6}$	116
$C_{7}-C_{6}$	1.55	$C_{7}-C_{6}-C_{5}$	111
$C_{6}-C_{5}$	1.48	$C_{6}-C_{5}-C_{4}$	112
$C_{5}-C_{4}$	1.57	$C_{5}-C_{4}-C_{3}$	115
$C_{4}-C_{3}$	1.44	$C_{4}-C_{3}-C_{2}$	115
$C_{3}-C_{2}$	1.62	$C_{3}-C_{2}-C_{1}$	116
$C_{2}-C_{1}$	1.41	$C_{2}-C_{1}-O_{1}$	109
$C_{1}-O_{1}$	1.39	$C_{2}-C_{1}-O_{2}$	128
$C_{1}-O_{2}$	1.14	$O_{1}-C_{1}-O_{2}$	117
$O_{1}-C_{01}$	1.56	$C_{1}-O_{1}-C_{01}$	109
$C_{01}-C_{02}$	1.38	$O_{1}-C_{01}-C_{02}$	103

DISCUSSION

Despite the low accuracy in the parameters some general remarks can be made. The main features of the structure are as described in Ref. 1. The long chains are packed according to the subcell O ⊥ (101) and have an angle of tilt towards the end group planes of 63°. The subcell dimensions as calculated from the final coordinates are $a_s = 4.99$, $b_s = 7.40$, $c = 2.54$ Å. The shortest distance between carbon atoms across the contact planes is 3.89 Å.

At the carboxyl-group there is no longer any deviation from the direction of the main chain as was stated in Ref. 1. A twisting of the molecular plane occurs so that the OC_2H_5 group lies in a plane approximately parallel to the *ac*-plane. This is shown in Fig. 2, where the unit cell is seen along the *c*-axis (which is pointing down from the plane of the paper). The lines of intersection with the *a*b*-plane and their angles to the *b*-axis are shown for the best least-squares planes through the main chain (atoms $C_2 \cdots C_{18}$), the group $C_2-C_1\begin{matrix}=O_2,\\ -O_1\end{matrix}$ and the group $C_1-O_1-C_{01}-C_{02}$. The projections of the centroids of the different groups are also shown. The planes are nearly parallel to the *c*-axis. The angles between the planes and the *bc*-plane are 44° for the chain plane and 68° for the carboxyl group plane (compared with 44° and 63°, respectively, for methyl stearate in Aleby and von Sydow.[10] For the alcohol chain the angle is 85° (compared with 72° for the plane C_1, O_1, C_{me} in methyl stearate).

The atoms in the main chain lie within 0.09 Å from the plane except C_3 and C_2 (which tend to the more "vertical" arrangement of the carboxyl group). There is an indication of a twist in the chain plane so that the part nearer to the methyl end is more parallel to the *bc*-plane. The carboxyl group

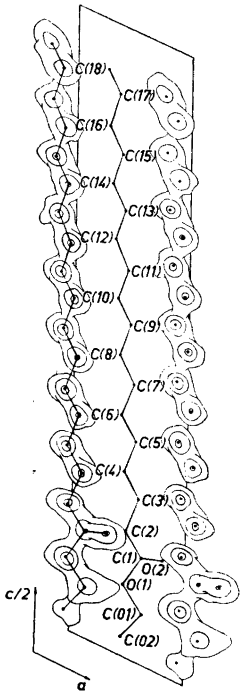

Fig. 1. Electron density in sections close to the atoms. Contours at intervals of 1 e·Å^{-3} starting with 1 e·Å^{-3}.

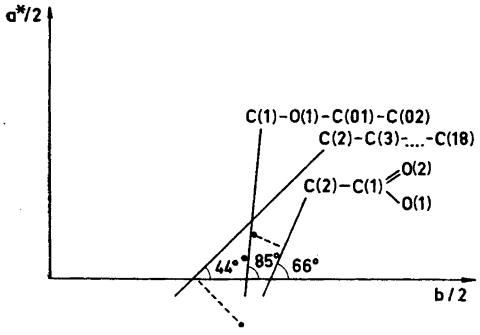

Fig. 2. Least-squares planes through different parts of the molecule (the intersections between the planes and the a^*b-plane are shown as well as the projection of the centroid of the group of atoms).

is planar within the limits of error: the sum of the bond angles is 354° and the maximum deviation from the plane is 0.14 Å. The group $C_1-O_1-C_{01}-C_{02}$ is planar within 0.01 Å.

Malkin [11] has discussed the arrangement of the polar region in various types of esters. To explain the alternation in the increments of the long spacing in series of methyl, ethyl, propyl, butyl, and amyl esters of the same acid he suggested that in ethyl and butyl esters the terminating C—C bond in the alcohol is perpendicular to the end group contact plane while in

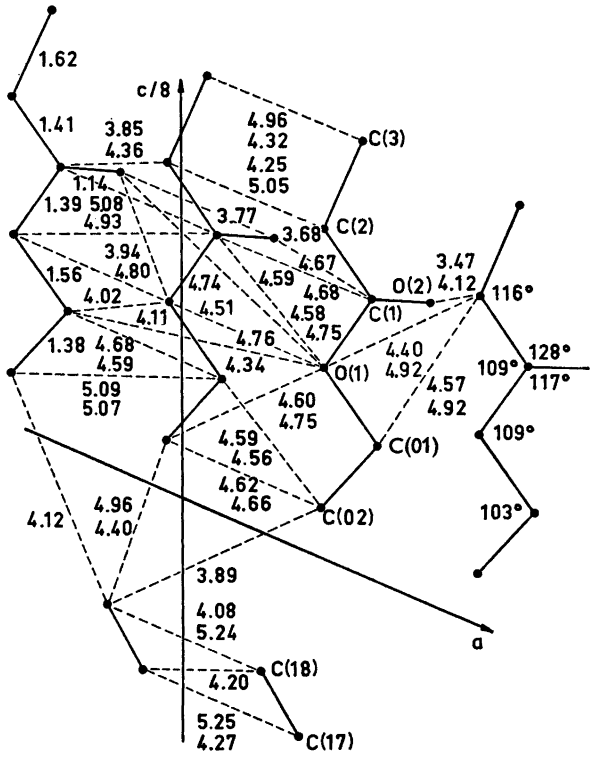

Fig. 3. Detail of the ester group region.

methyl, propyl, and amyl esters it tilts. From Fig. 3 it is seen that this is essentially correct in the case of ethyl stearate.

The same type of alternation can, however, be observed in other series (for example methyl esters and ethyl esters of different acids) and is a consequence of the inclination of the chains to the end group planes (but not a necessary consequence, *cf.* Larsson [12]).

In Fig. 3 are also shown some contact distances (where two distances are given together they refer to atoms displaced one *b*-axis from each other).

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