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The Crystal Structure of the 1,3-Diglyceride of 3-Thiadodecanoic Acid

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The symmetric diglyceride of 3-thiadodecanoic acid crystallizes in the orthorhombic space group $Pca2_1$ with unit-cell dimensions $a = 9{\cdot}15 \pm 0{\cdot}02$, $b = 4{\cdot}99 \pm 0{\cdot}01$, $c = 63{\cdot}1 \pm 0{\cdot}15$ Å and with $Z = 4$. The two hydrocarbon-chain tails in the molecule point in opposite directions and are as usual packed in layers with the chain axes parallel. The direction of the chain-tilt, however, alternates in successive layers.

Introduction

A crystal-structure investigation of glycerides was started by the author a few years ago, but only some general results on monoglycerides have hitherto been presented (Abrahamsson, Aleby, Larsson, Larsson, Ryderstedt-Nahringbauer & von Sydow, 1960). After some work it was obvious that complete determinations of the crystal structures for most forms were extremely complicated. Different glyceride derivatives containing heavy atoms have therefore been synthesized and this diglyceride is one of these. Although it is not isomorphous with any crystal form of the corresponding unsubstituted diglycerides, which are under investigation, there are many structural similarities. Conclusions about the crystal structures of naturally occurring symmetric diglycerides can therefore be based on the present crystal structure of this sulphur-containing diglyceride.

Preparation of crystals

3-Thiadodecanoic acid was prepared by Smith & Hernestam (1954). To obtain the triglyceride of this acid a method intended for normal fatty acids and described by Stacey, Bourne, Tatlow & Tedder (1949) was used, and the final product (dissolved in petroleum spirit, b.p. 40–60 °C) was purified by chromatography over aluminum oxide. The infrared absorption of the sample as well as elementary analyses indicated that a diglyceride instead of the triglyceride had been formed (probably because of the steric differences between a sulphur atom and a CH_2-group in the 3-position). The X-ray analysis confirmed this and showed furthermore that the diglyceride obtained from the synthesis was the symmetric one. Crystals for X-ray work were grown from ethyl ether in the form of needles parallel to [100] with faces (100), (010), and (001). The crystals are biaxially positive with the acute bisectrix perpendicular to the (001)-plane.

Polymorphism

Most glycerides have a complicated phase behaviour. Many polymorphous forms exist, and the literature in this field is rather confusing as different workers have used different nomenclature for the crystal forms.

The phase behaviour of this diglyceride has been studied in a camera built by Stenhagen (1951) for continuous recording of the X-ray diffraction pattern as a function of temperature. Heating of the crystal form obtained from ethyl ether solution shows no transition below the melting point at 51·1–51·8 °C. The persistence of long-spacing lines above the melting point indicates that a mesomorphous state exists with a smectic type of structure. On cooling the melt, another form crystallizes at 45·5–44·5 °C. It is unstable and transforms into the crystal form first described within a day at room temperature. Within the experimental errors these crystal forms have the same long spacing, which indicates that the chains pack in layers with the same angle of tilt in both forms. It was not possible to obtain good single crystals of the unstable form and therefore the crystal-structure determination refers to the stable form only.

X-ray data

Rotation and Weissenberg photographs of the $0kl$ and $h0l$ zones were taken with Cu $K\alpha$ radiation, a calibrated camera being used. The following data were obtained:

Molecular formula: $C_{25}H_{48}O_5S_2$.
Molecular weight: 492·76.
Unit cell: orthorhombic.

$$a = 9{\cdot}15 \pm 0{\cdot}02,\quad b = 4{\cdot}99 \pm 0{\cdot}01,\quad c = 63{\cdot}1 \pm 0{\cdot}15 \text{ Å}\,.$$

Four molecules per unit cell.
Number of electrons: 1080.
Density calculated: $1{\cdot}136 \pm 0{\cdot}006$ g.cm^{-3}.
Density measured: 1·14 g.cm^{-3}.
Absent reflexions: $0kl$ when l odd and $h0l$ when h odd.
Space group: $Pca2_1$.

Intensities were estimated visually by the multiple-film technique and corrected for the polarization and Lorentz factors but not for absorption. Absolute values were later obtained by comparison with calculated structure factors.

Structure determination

No pronounced sublattice, giving information about chain packing, is found in the reciprocal lattice. Patterson (100) and (010) projections, with coefficients sharpened to correspond to atoms at rest, gave the sulphur positions and the chain packing. The origin may be chosen arbitrarily along the *c*-axis and following convention it was placed half way between the sulphur atoms of the molecule. From the methyl contacts a probable arrangement of the chain tails bonded to the sulphur atoms could be derived. Electron-density projections based on phases from the sulphur atoms and chain tails were calculated. The positions of these two chain parts in the projections were pseudosymmetrically related by a two-fold axis through $(0, \frac{1}{4}, 0)$ parallel to *a*. The electron-density projections based on these positions, which have the same symmetry, showed the rest of the structure except for the carbon atom in the middle of the glycerol group and the oxygen atom attached to it. These 'one-fold' atoms appear ambiguously if they do not lie on the axis of pseudosymmetry. The peaks in the electron-density projections correspond to two position sets for this group. One of the two equivalent sets was chosen, as disorder is improbable on the following grounds. The peak positions indicate that there is a hydrogen bond between the hydroxyl group and one of the carbonyl oxygens. If the hydroxyl groups are disordered, the two carbonyl groups will be equivalent with regard to the hydrogen bond. The carbonyl band in the infrared absorption, however, is split (peaks at 1710 and 1750 cm^{-1}), and this indicates that the two carbonyl oxygens in the molecule are not equivalent (*cf.* Chapman, 1956).

The (010) projection was refined to $R=0{\cdot}19$ by successive cycles of structure-factor calculations, electron-density summations and difference syntheses. The refinement could not be performed very far in the (100) projection because of the serious overlap (*cf.* Fig. 2); even the general shape of the molecule was uncertain at this stage. There were four possible ways to join the glycerol group with the carboxyl groups owing to the poor resolution (Fig. 4). A Patterson section through $z=\frac{1}{6}$, corresponding to the intermolecular S–S peak, gave the relative positions of the two chains in the molecule (a displacement of the chains $x=\frac{1}{2}$ does not make any difference in the projections). A three-dimensional electron-density calculation based on all atoms except for the glycerol group showed which one was the true structure of the two remaining possibilities. Another cycle of structure-factor and electron-density calculations was performed and new *y*-coordinates were derived. Anisotropic least-squares refinement was then started. At the fourth round of least-squares refinement a difference synthesis was calculated, which indicated shifts in positional and thermal parameters in accordance with the least-squares results. The hydrogen atoms were not included in the refinement as their large number (48 in the asymmetric unit) would increase the calculation time seriously. After nine rounds of least-squares the *R*-value was 0·16 and all indicated shifts were less than about half the estimated standard deviation. In order to see if the neglect of hydrogen atoms might have influenced the refinement, one round of least-squares was performed with the hydrogen atoms included, their positions being calculated from the data given by Vainshtein & Pinsker (1950). The *R*-value was slightly improved and all positional shifts had the same directions as in the previous round without the hydrogen atoms.

The weighting scheme applied in the refinement was

$$w=\frac{1}{1+[(|F_o|-8|F_{\min}|)/5|F_{\min}|]^2}\;.$$

The scattering factors for carbon and oxygen were taken from Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955), for sulphur from Viervoll & Øgrim (1949) and for hydrogen from McWeeny (1952). The calculations were performed on a Ferranti Mercury computer with programs described by Mills & Rollett (1961).

Table 1. *Atomic coordinates*

	x/a	*y/b*	*z/c*
S(1)	0·3070	0·160	0·08442
S(2)	0·3013	0·343	−0·08448
O(1)	0·3303	0·021	0·03054
O(2)	0·5229	0·288	0·04444
O(3)	0·3500	0·477	−0·03160
O(4)	0·5100	0·212	−0·03790
O(5)	0·2400	0·566	0·01205
C(1)	0·3995	0·227	0·04382
C(2)	0·2776	0·325	0·05901
C(3)	0·4708	0·302	0·09448
C(4)	0·4940	0·210	0·11680
C(5)	0·6331	0·294	0·12720
C(6)	0·6460	0·223	0·15053
C(7)	0·7840	0·285	0·16021
C(8)	0·8065	0·221	0·18398
C(9)	0·9528	0·281	0·19376
C(10)	0·9616	0·216	0·21654
C(11)	0·1076	0·290	0·22637
C(12)	0·3770	0·223	−0·04004
C(13)	0·2754	0·194	−0·05956
C(14)	0·4714	0·197	−0·09440
C(15)	0·4900	0·298	−0·11700
C(16)	0·6367	0·214	−0·12754
C(17)	0·6463	0·286	−0·15025
C(18)	0·7669	0·222	−0·16110
C(19)	0·8082	0·297	−0·18359
C(20)	0·9368	0·189	−0·19445
C(21)	0·9604	0·283	−0·21716
C(22)	0·0917	0·211	−0·22929
C(23)	0·4390	0·886	0·01612
C(24)	0·3290	0·770	0·00135
C(25)	0·4340	0·637	−0·01562

Results and discussion

The atomic coordinates are given in Table 1 and the vibrational parameters in Table 2. The *A*- and *B*-components of the final structure factors are listed in Table 3, and the final electron-density projections along the short axes are given in Figs. 1 and 2.

The bond distances and angles in the molecule are given in Table 4. The standard deviations estimated from the least-squares treatment are 0·01 Å for sulphur atoms, 0·03 Å for oxygen atoms, and 0·04 Å for carbon atoms in the *x*- and *z*-directions and almost twice these values in the *y*-direction. The relatively large number of parameters and also the fact that the intensity data were of rather bad quality limits the accuracy in the atomic positions seriously.

Figs. 3 and 4 show the molecular arrangement. The most striking feature is the chain arrangement

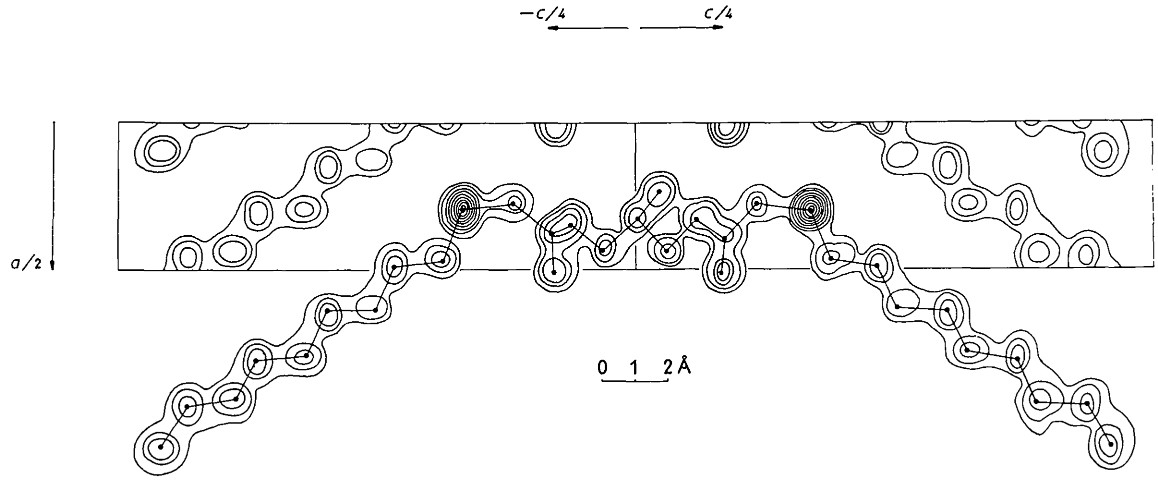

Fig. 1. Electron-density projection along the *b*-axis. Contours are given at intervals of 2 e.Å^{-2}, starting with 2 e.Å^{-2}.

Table 2. *Final anisotropic vibration parameters used in the temperature factor*

$$2^{-(h^2b_{11}+k^2b_{22}+l^2b_{33}+klb_{23}+hlb_{13}+hkb_{12})}$$

	b_{11}	b_{22}	b_{33}	b_{23}	b_{13}	b_{12}
S(1)	0·0150	0·197	0·00031	−0·00044	−0·00044	−0·0120
S(2)	0·0149	0·200	0·00030	0·00093	0·00059	0·0220
O(1)	0·0177	0·102	0·00057	−0·00393	−0·00334	−0·0061
O(2)	0·0149	0·140	0·00019	−0·00529	0·00055	−0·0004
O(3)	0·0220	0·102	0·00063	−0·00622	−0·00295	0·0049
O(4)	0·0155	0·125	0·00035	−0·00823	0·00317	0·0123
O(5)	0·0115	0·097	0·00040	−0·00240	−0·00107	0·0025
C(1)	0·0196	0·113	0·00025	0·00037	−0·00445	−0·0991
C(2)	0·0213	0·231	0·00029	0·00668	0·00035	0·0061
C(3)	0·0207	0·095	0·00036	−0·00843	0·00077	−0·0789
C(4)	0·0116	0·210	0·00020	−0·00378	−0·00030	0·0051
C(5)	0·0267	0·112	0·00038	0·00353	−0·00397	0·0044
C(6)	0·0289	0·079	0·00019	0·00135	−0·00123	0·0372
C(7)	0·0143	0·093	0·00031	0·00138	−0·00140	0·0334
C(8)	0·0174	0·137	0·00044	−0·00474	−0·00165	−0·0197
C(9)	0·0139	0·145	0·00027	0·00591	−0·00507	0·0060
C(10)	0·0228	0·180	0·00038	−0·00055	−0·00147	−0·0198
C(11)	0·0349	0·195	0·00029	0·00556	−0·00414	−0·0340
C(12)	0·0332	0·117	0·00063	−0·01962	−0·00546	0·1164
C(13)	0·0197	0·188	0·00037	0·00809	0·00052	−0·0641
C(14)	0·0192	0·102	0·00029	−0·00409	0·00117	0·1070
C(15)	0·0128	0·187	0·00024	−0·00885	0·00343	−0·0107
C(16)	0·0212	0·097	0·00047	0·00034	0·00240	0·0744
C(17)	0·0211	0·101	0·00036	−0·00180	0·00434	−0·0071
C(18)	0·0211	0·093	0·00038	−0·00233	0·00411	0·0066
C(19)	0·0142	0·128	0·00029	−0·00224	0·00227	0·0087
C(20)	0·0136	0·182	0·00039	0·00330	0·00228	−0·0026
C(21)	0·0199	0·189	0·00030	0·00229	0·00273	0·0438
C(22)	0·0211	0·182	0·00040	−0·00348	0·00052	−0·0058
C(23)	0·0278	0·086	0·00054	−0·01962	0·00592	0·0012
C(24)	0·0230	0·082	0·00025	−0·00504	−0·00121	−0·1964
C(25)	0·0271	0·176	0·00036	−0·00383	0·00198	−0·0273

Table 3. *The A- and B-components of the final structure factors*

An asterisk marks changes in *h* and *l*. Each non-asterisk line gives *k*, $100A_o$, $100B_o$, $100A_c$, and $100B_c$

•	0	0			
	2	−19781	0	−20265	0
•	0	2			
	1	−1135	3277	−1456	4205
	2	4120	−101	3239	−79
•	0	4			
	1	−775	4197	−977	5293
	2	6156	810	5977	783
	4	−56	−1456	−10	−268
•	0	6			
	1	−1280	2188	−1414	2577
	2	2338	950	1327	539
	3	−338	1554	−443	2038
	4	2307	−80	1253	−44
•	0	8			
	0	−4597	1109	−5070	1223
	1	−533	421	−1211	956
	2	3721	1313	3450	1217
	3	−453	2868	−447	2835
•	0	10			
	0	4557	1107	4015	975
	1	−579	663	−872	998
	2	1177	1839	1075	1081
	3	−191	2358	−226	2792
	4	−1453	−217	−1455	−217
•	0	12			
	0	−2626	773	−2379	700
	1	−1422	4286	−1538	4635
	2	1730	2062	1234	1470
	4	−2856	48	−1441	24
•	0	14			
	1	−1359	5024	−1440	5324
	3	165	−1968	126	−1502
•	0	16			
	0	−11517	1663	−12345	1782
	1	−1365	3446	−1530	3860
	3	376	−2423	435	−2603
•	0	18			
	0	−4994	1615	−5606	1813
	1	−663	−349	−1317	−1296
	2	−3468	1342	−3820	1478
	4	1494	−85	824	−47
•	0	20			
	1	−429	−4216	−500	−4913
	2	−3792	343	−4095	371
•	0	22			
	0	11492	4733	10401	4284
	1	−434	−6083	−470	−6583
	2	−6380	−139	−6488	−141
•	0	24			
	0	7128	4607	7490	4641
	1	−56	−2684	−60	−2851
	2	−3650	−375	−3585	−368
•	0	26			
	0	12080	4460	10389	3835
	1	1225	−1812	878	−1299
	2	−4441	−302	−4896	−333
	5	650	−995	429	−657
•	0	28			
	0	−10102	4755	−8464	3984
	1	1739	2561	1447	2131
	2	2553	−1015	2834	−1127
	3	−848	−3234	−1023	−3900
	4	1517	133	948	83
•	0	30			
	0	−10536	1132	−9172	986
	1	1845	−607	1757	−578
	2	3344	365	3797	415
	3	−755	−1200	−951	−1513
	4	1423	−934	629	−413
•	0	32			
	0	8034	28	6572	23
	1	1712	−6768	1589	−6282
	2	−1916	471	−2364	581
	3	−440	3190	−590	4278
	4	1243	−430	1002	−347
•	0	34			
	0	1498	−1114	1467	−1092
	1	845	−1315	1413	−2200
	2	1591	644	1725	699
•	0	36			
	0	1399	−2369	1266	−2144
	2	1304	1213	1461	1360
•	0	38			
	0	−3558	−1742	−3364	−1647
	1	777	2176	893	2502
•	0	40			
	0	−6683	−1361	−6238	−1214
	1	843	1753	864	1827
•	0	42			
	0	−6262	558	−5269	471
	2	−1125	−1423	−639	−1062
	4	771	960	695	883
•	0	44			
	0	−2774	869	−1460	458
	2	−2224	−911	−1923	−787
•	0	46			
	0	2124	1327	2536	1785
	2	−2964	−1396	−2861	−1375
•	0	48			
	0	3459	1605	3661	1910
	2	−2645	−1427	−2769	−1389
•	0	50			
	0	1560	1602	1783	1832
	2	−2335	−1268	−2303	−1250
•	0	52			
	0	−462	775	−558	863
	2	−1907	−815	−1708	−736
•	0	54			
	0	−2853	1237	−2351	1019
•	0	56			
	0	−721	−760	−844	−890
	1	343	−1678	466	−2282
•	0	58			
	0	−693	−1315	−469	−889
	1	439	−1864	578	−2455
	2	1763	445	1622	469
	3	−495	1412	−825	2355
•	0	60			
	0	2867	−1288	3103	−1395
•	0	62			
	0	1385	−1541	1540	−1713
•	0	64			
	0	−1693	−679	−1665	−697
•	0	68			
	0	818	233	1532	437
•	0	70			
	0	2835	392	2970	411
•	0	72			
	0	1646	958	1851	1078
•	1	0			
	1	5652	0	2596	0
	3	−2071	0	−2600	0
	5	818	0	645	0
•	1	1			
	1	393	−12667	379	−12204
	3	−41	2154	−44	2290
•	1	2			
	1	−9097	276	−8856	269
	2	−852	−2533	−1005	−2990
	3	1017	62	1432	87
	5	−467	582	−25	31
•	1	3			
	1	436	−4040	451	−4155
	3	−274	1411	−213	1097
•	1	4			
	1	−2629	1184	−1950	678
	2	−652	−2895	−701	−3113
	3	2398	298	2391	297
•	1	5			
	1	465	22233	398	19053
	2	−1020	−53	−1823	−95
	3	−70	−1290	−116	−2161
•	1	6			
	1	23064	557	21942	530
	2	−647	839	−1077	1396
	5	809	348	214	92
•	1	7			
	1	−588	−3859	−497	−3262
	2	−1052	−59	−1839	−104
	3	96	1587	71	1176
•	1	8			
	1	7773	1228	7219	1140
	2	−425	1894	−452	2012
	3	544	826	202	307
•	1	9			
	1	−297	6632	−277	6179
	2	−1924	94	−2109	103
	3	−243	−709	−324	−945
	5	255	771	74	223
•	1	10			
	1	9	912	8	759
	2	−23	1704	−19	1420
	3	402	672	458	766
•	1	11			
	1	−1746	5492	−1733	5451
	2	−1975	52	−2527	66
	3	339	−2046	295	−1786
	5	−87	1040	−39	469
•	1	12			
	1	−611	799	−612	800
	2	−92	−1625	−89	−1755
	3	−1526	1181	−973	753
•	1	13			
	1	−1091	8854	−1001	8124
	3	−137	−2239	−119	−1948
	5	118	796	65	439
•	1	14			
	1	−90	784	−109	943
	2	352	−3492	405	−4008
•	1	15			
	1	−981	5277	−1087	5847
	3	−150	−1161	−190	−1425
•	1	16			
	1	5480	765	5362	748
	2	320	−3504	356	−3891
	3	841	960	710	810
•	1	17			
	1	−926	5477	−717	4241
•	1	18			
	1	4094	653	3871	617
	3	1940	1623	1001	837
•	1	19			
	1	476	−1014	441	−939
	3	−439	1605	−490	1121
•	1	20			
	1	2372	795	2982	703
	2	533	2724	446	2290
	3	430	582	434	567
•	1	21			
	1	1161	−3616	1188	−3637
	3	−265	671	−506	1190
	4	−1010	−133	−1249	−165
•	1	22			
	1	−4993	1270	−4531	1153
	2	149	4383	138	4043
	3	669	48	706	51
	4	−167	−1393	−122	−1024
•	1	23			
	1	2073	−7615	1664	−6113
	2	−1875	−107	−1678	−95
•	1	24			
	1	−3549	58	−3034	49
	3	−899	1467	−415	677
•	1	25			
	1	2295	−8973	1967	−7689
	2	−2436	−279	−2171	−246
	4	1463	541	456	169
•	1	26			
	1	2310	2727	1444	1079
	2	249	−5076	260	−5305
	3	−1863	615	−1124	371
	4	73	1358	80	1483
•	1	27			
	1	926	111	1912	230
	2	2823	−711	2751	−692
	3	−227	−796	−116	−407
•	1	28			
	1	3429	964	2646	744
	2	92	−2102	90	−2053
•	1	30			
	1	2433	1179	1768	857
•	1	31			
	1	18	3055	17	2946
	2	325	−1118	426	−1468
•	1	32			
	1	4528	911	3958	622
	3	−1532	212	−2650	306
•	1	33			
	1	−1455	−2625	−1577	−2845
	2	−2083	−490	−2788	−656
	3	549	−747	722	−982
•	1	34			
	1	−5538	1379	−5052	1258
	2	621	1752	871	2455
	3	−1841	−268	−1317	−191
•	1	35			
	1	−947	340	−1291	463
	3	420	−2253	360	−2041
•	1	36			
	1	−6694	996	−6025	891
	3	−2196	−370	−736	−124
•	1	37			
	1	−2355	4866	−2330	4813
	2	1636	−75	1188	−69
	3	820	−2719	863	−2862
•	1	38			
	1	1043	154	1602	230
	2	693	−2115	792	−2416
	3	−1896	173	−1852	169
•	1	39			
	1	−916	558	−1398	852
•	1	40			
	1	3120	687	2624	577
	2	731	−1449	937	−1850
•	1	42			
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Table 3 (*cont.*)

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	2	327	-1438	150	-658
•	6	8			
	0	-217	-1521	-297	-2083
	2	-1577	494	-2092	655
•	6	9			
	0	588	-1901	533	-1723
	1	-1310	-513	-787	-300
•	6	10			
	2	-1815	148	-2078	169
•	6	11			
	0	623	-4707	721	-5449
	2	-236	1226	-391	2032
•	6	12			
	0	-1235	-956	-671	-519
	2	-1413	-146	-1805	-187
•	6	13			
	0	425	-4774	457	-5133
	1	1469	342	1523	354
	2	-620	1561	-703	1771
•	6	14			
	0	-2674	-556	-3023	-628
	1	44	1694	50	1894
	2	-1642	-847	-952	-491
•	6	15			
	1	2065	179	2240	194
•	6	16			
	0	-2749	-277	-2933	-295
	2	-1305	-782	-1401	-840
•	6	17			
	0	-183	2614	-183	2619
•	6	18			
	0	-2789	196	-3098	218
	2	-1355	-1303	-1448	-1392
•	6	19			
	0	-859	2839	-784	2592
•	6	21			
	1	-1049	-379	-1302	-471
•	6	24			
	0	3054	-292	4028	-385
•	6	26			
	0	3068	-510	3278	-545
•	6	27			
	0	-1799	2146	-1381	1648
•	6	28			
	0	3938	-616	3025	-473
•	6	29			
	0	-706	4758	-1032	6950
	1	1606	-408	2459	-625
	2	54	-1521	99	-2760
•	6	30			
	0	-1752	-1003	-2624	-1503
	2	2269	-159	1801	-126
•	6	31			
	2	-82	1346	-89	1465
•	6	32			
	0	2110	-673	2985	-1235
•	6	33			
	0	770	-2394	906	-2818
	1	-1409	-114	-1399	-113
•	6	34			
	0	4293	-1691	4399	-1732
•	6	35			
	0	1159	-1718	1030	-1527
	2	-1488	-374	-574	-144
•	6	37			
	0	1278	-2660	1513	-3384
•	6	38			
	0	2092	101	1863	90
•	6	41			
	0	804	1942	936	2263
•	6	43			
	0	227	2335	196	2011
•	7	2			
	1	-1347	-118	-1499	-131
•	7	3			
	1	430	2195	170	2867
•	7	4			
	1	3373	120	3522	126
•	7	5			
	1	-484	-1924	-582	-2311
•	7	6			
	1	3234	574	3104	551
•	7	7			
	1	-791	-3110	-714	-2806
•	7	10			
	1	-2219	142	-2072	133
•	7	11			
	1	-696	2053	-856	2524
•	7	12			
	1	-3560	45	-3402	43
•	7	13			
	1	-96	2729	-114	3247
•	7	14			
	1	-659	-964	-951	-1390
•	7	19			
	1	907	-2022	390	-870
•	7	21			
	1	1354	392	926	268
	2	2171	-339	2787	-436
•	7	24			
	1	-1866	356	-1789	341
•	7	25			
	1	696	-982	811	-1145
•	7	27			
	1	420	-1864	214	-948
•	7	29			
	1	-216	2413	-61	676
•	7	34			
	1	-1318	-555	-1811	-762
•	7	35			
	1	-364	2227	-439	2666
•	7	37			
	1	-367	1858	-424	2148
•	8	0			
	0	-2083	0	-2157	0
•	8	1			
	0	69	1471	57	1208
	2	24	-1978	17	-1363
•	8	5			
	0	-253	-1788	-256	-1806
•	8	8			
	0	2542	-285	3089	-346
•	8	9			
	0	-716	-3549	-702	-3479
	2	406	2243	483	2670
•	8	10			
	0	-3300	-219	-3031	-201
•	8	11			
	2	-164	1792	-159	1739
•	8	12			
	0	-4189	-30	-4364	-31
	2	1575	324	2064	425
•	8	14			
	1	-94	-1208	-120	-1533
•	8	16			
	1	-61	-1211	-74	-1478
•	8	17			
	0	3053	-782	1840	-471
•	9	0			
	1	2058	0	1892	0
•	9	1			
	1	-276	-1853	-320	-2157
•	9	7			
	1	-142	1608	-58	683
•	9	14			
	1	1315	-426	1225	-397
•	9	17			
	1	568	-1007	360	-638
•	9	24			
	1	1576	-43	2073	-50
•	10	0			
	0	1028	0	1719	0
•	10	5			
	0	-1398	1245	-1015	904

Table 4. *Bond distances and angles*

Bond	Distance	Bond	Angle
C(1)–C(2)	1·55 Å	C(1)–C(2)–S(1)	107°
C(2)–S(1)	1·82	C(2)–S(1)–C(3)	105
S(1)–C(3)	1·78	S(1)–C(3)–C(4)	110
C(3)–C(4)	1·50	C(3)–C(4)–C(5)	117
C(4)–C(5)	1·49	C(4)–C(5)–C(6)	115
C(5)–C(6)	1·52	C(5)–C(6)–C(7)	115
C(6)–C(7)	1·44	C(6)–C(7)–C(8)	119
C(7)–C(8)	1·55	C(7)–C(8)–C(9)	118
C(8)–C(9)	1·50	C(8)–C(9)–C(10)	114
C(9)–C(10)	1·48	C(9)–C(10)–C(11)	113
C(10)–C(11)	1·52	C(12)–C(13)–S(2)	126
C(12)–C(13)	1·55	C(13)–S(2)–C(14)	105
C(13)–S(2)	1·76	S(2)–C(14)–C(15)	106
S(2)–C(14)	1·83	C(14)–C(15)–C(16)	114
C(14)–C(15)	1·52	C(15)–C(16)–C(17)	114
C(15)–C(16)	1·56	C(16)–C(17)–C(18)	115
C(16)–C(17)	1·48	C(17)–C(18)–C(19)	115
C(17)–C(18)	1·57	C(18)–C(19)–C(20)	115
C(18)–C(19)	1·47	C(19)–C(20)–C(21)	118
C(19)–C(20)	1·45	C(20)–C(21)–C(22)	123
C(20)–C(21)	1·51	C(2)–C(1)–O(1)	105
C(21)–C(22)	1·47	C(2)–C(1)–O(2)	126
C(23)–C(24)	1·49	O(1)–C(1)–O(2)	128
C(24)–C(25)	1·58	C(13)–C(12)–O(3)	106
O(1)–C(1)	1·47	C(13)–C(12)–O(4)	133
O(1)–C(23)	1·50	O(3)–C(12)–O(4)	100
O(2)–C(1)	1·17	C(23)–C(24)–C(25)	100
O(3)–C(12)	1·40	C(23)–C(24)–O(5)	111
O(3)–C(25)	1·49	C(25)–C(24)–O(5)	111
O(4)–C(12)	1·23		
O(5)–C(25)	1·47		

obvious in the (010) projection. The hydrocarbon chain tails are parallel in double layers and tilted at the same angle towards the end-group planes, but the direction of the chain-tilt alternates in successive double layers of chains. There is also a bend in the chains at the sulphur atom very similar to that found in 3-thiadodecanoic acid (Abrahamsson & Westerdahl, 1963). The two carbon atoms near the carboxyl group as well as the sulphur atom are twisted out of the plane of the hydrocarbon-chain tails. It was pointed out by Abrahamsson & Westerdahl (1963) that bent long-chain molecules are rather common in structures where the hydrocarbon chain packing is disturbed by the presence of groups other than methylene groups in the chains.

The carboxyl groups together with the glycerol group form a roughly straight zigzag chain (which is not planar, however). These chains in adjacent molecules in the *a*-direction are oppositely inclined, forming a crossed-chain structure, which is seen in Fig. 4.

The hydrogen bonds (illustrated in Figs. 3 and 4) link the molecules together in the *a*-direction, and the morphology of the crystals agrees with such an arrangement; the crystals form needles parallel to the *a*-axis. The distance between the hydrogen-bonded oxygens is 2·94 Å and the angle C–OH · · · O is 121°. There are three more short contacts (less than 3·2 Å) between the hydroxyl-group oxygen and carboxyl group oxygens. The corresponding C–OH · · · O angles do not indicate that the hydrogen atom is hydrogen-bonded also to any of these three oxygens, as in the bifurcation of the hydrogen bond supposed to exist in glycine (Albrecht & Corey, 1939) and tartaric acid dihydrate (Parry, 1951), but polar attraction (hydrogen–oxygen) may exist.

The carbon-chain tails are tilted 55° towards the end-group planes. The chain packing is of the common orthorhombic type (O ⊥), and the dimensions of the subcell are (average values):

$$a_s = 4{\cdot}99,\ b_s = 7{\cdot}46,\ c_s = 2{\cdot}56\ \text{Å}\,.$$

The volume per CH$_2$-group is 23·8 Å^3. No significant irregularities have been found in the chain packing. The best least-squares planes through the hydrocarbon chains have been calculated; all atoms lie within 0·1 Å from these planes. The average value of the carbon–carbon distance is 1·50 Å and the average bond angle in the hydrocarbon chain is 115°.

The stacking of the layers is very similar to that found in the *C*-form of lauric acid (Vand, Morley & Lomer, 1951; von Sydow, 1956). This might be expected, as the chain packing is the same in the two compounds as well as the orientation of the orthorhombic subcell in relation to the methyl-group planes. The atoms on different sides of the gap are related by symmetry in the *C*-form but not in this structure. The distances from one methyl group in

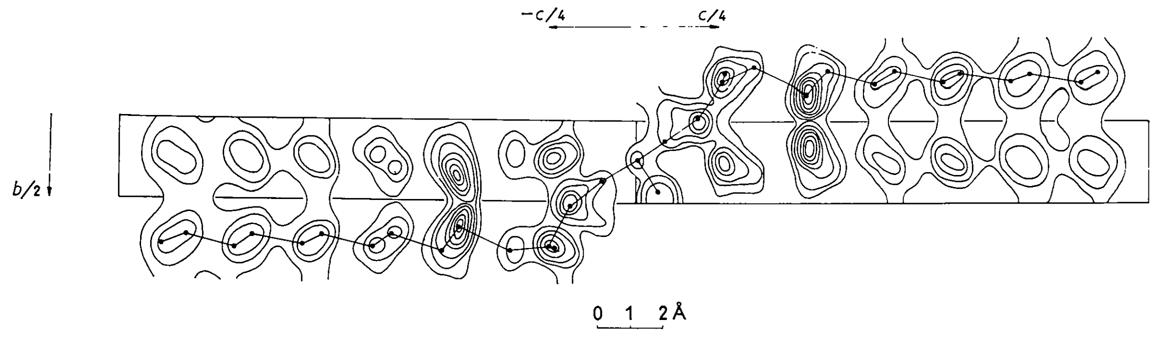

Fig. 2. Electron-density projection along the *a*-axis. Contours are given at intervals of 2 e.Å^{-2}, starting with 2 e.Å^{-2}.

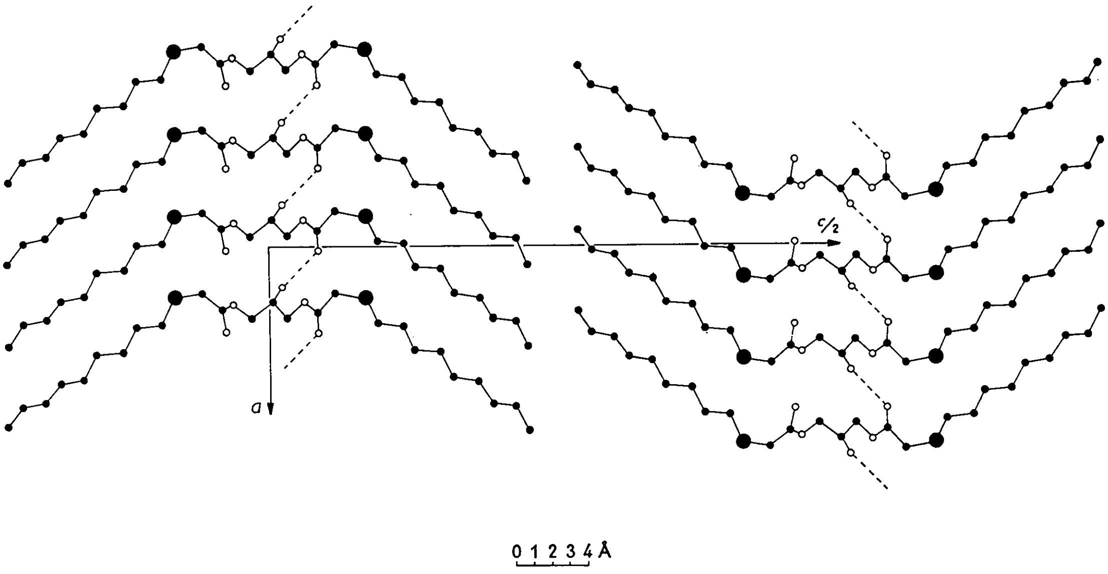

Fig. 3. Projection along the *b*-axis showing the molecular arrangement. The small filled and open circles are carbon and oxygen atoms respectively and the large filled circles are sulphur atoms.

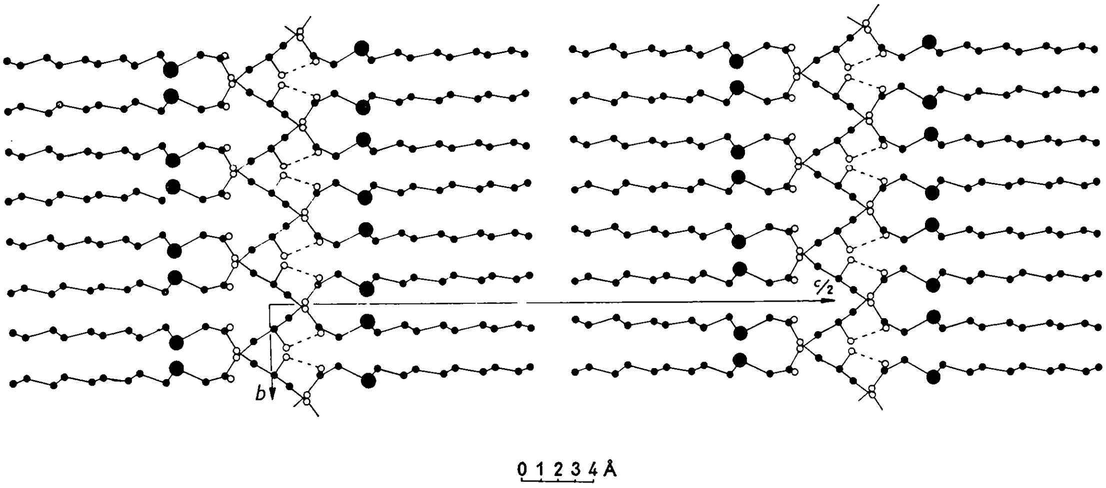

Fig. 4. Projection along the *a*-axis showing the molecular arrangement. The small filled and open circles are carbon and oxygen atoms respectively and the large filled circles are sulphur atoms.

Table 5. *Carbon–carbon distances less than* 5·5 Å *from the carbon atom in one methyl group to its neighbours on the other side of the methyl gap*

	Diglyceride	Lauric acid (*C*-form)
CH_3–CH_3	3·93 Å	3·85 Å
CH_3–CH_3	4·17	4·28
CH_3–CH_3	4·17	4·28
CH_3–CH_2	4·19	4·01
CH_3–CH_2	4·61	4·72
CH_3–CH_2	5·32	5·18

the fatty acid *C*-form to its neighbouring carbon atoms on the other side of the gap have been given by von Sydow (1956). These distances are collected in Table 5 together with the corresponding distances in this diglyceride. There are no significant differences regarding the standard deviations. This is rather surprising in view of the differences in the physical properties of the crystals. The crystals of lauric acid show cleavage along the (001) planes, which has been attributed to the weak van der Waals interaction over the methyl gap, and twinning on these planes is also very common. The crystals of this diglyceride, however, form needles with square cross-sections, and neither (001) cleavage nor twin formation have been observed.

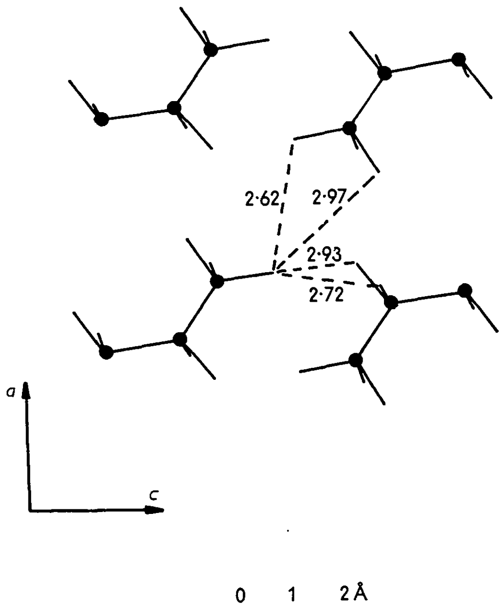

Fig. 5. Projection along the *b*-axis showing the methyl contact region. Non-equivalent hydrogen–hydrogen distances (in Å) less than 3·0 Å over the methyl gap are given.

The hydrogen–hydrogen contact distances over the methyl gap (Fig. 5) give a more informative picture of the packing of the methyl-group planes than the carbon–carbon distances discussed. The shortest distance is 2·62 Å, somewhat longer than the shortest lateral hydrogen–hydrogen contact between the chains, which is about 2·40 Å.

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The Crystal Structure of Monoaquobisacetylacetonatozinc

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The crystal structure of monoaquobisacetylacetonatozinc [$Zn(C_5H_7O_2)_2H_2O$] has been determined by three-dimensional methods. The cell dimensions are $a=10\cdot48$, $b=5\cdot37$, $c=10\cdot94$ Å, $\beta=93°48'$, space group $P2_1$, with $Z=2$. The structure is made up of discrete molecules, containing 5-coordinate zinc with a coordination configuration intermediate between tetragonal pyramidal and trigonal bipyramidal.

Introduction

Lippert & Truter (1960) have recently published a determination of the crystal structure of monoaquobisacetylacetonatozinc, $Zn(C_5H_7O_2)_2.H_2O$. Since we had an independent structure determination in the final refinement stages at the time we learned of their work, and since there appeared to be some significant differences in the results, we have completed our determination.