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The Crystal Structure of 3-Thiadodecanoic Acid

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3-Thiadodecanoic acid is triclinic with

$$a=4\cdot69,\ b=5\cdot10,\ c=29\cdot3\ \text{Å},\ \alpha=95°8',\ \beta=92°36'\ \text{and}\ \gamma=114°48'\ .$$

The space group is $P\bar{1}$. The molecules are bent at the sulphur atom and linked together to dimers, which form a layer structure. The carbon chain tails of the molecules form a new type of chain packing with a nearly monoclinic subcell ($M\|$).

Introduction

The structure determination of long-chain compounds is simplified if a heavy atom is introduced in the molecule. As our main interest lies in the naturally occurring unsubstituted compounds, different synthetic heavy-atom-containing compounds have been studied in order to find out if any of them are isomorphous with or structurally similar to the corresponding unsubstituted ones. The structure analysis of 3-thiadodecanoic acid was performed as part of this investigation, though the acid did not meet these requirements.

Experimental

The sulphur-containing acid was prepared by Smith & Hernestam (1954) by addition of thioglycollic acid to nonene-1. Crystals for the X-ray work were grown from glacial acetic acid. They had a melting point of 51·8 °C.

Rotation and Weissenberg photographs about the a- and b-axes were taken in a calibrated camera using Cu $K\alpha$ radiation.

Molecular formula: $C_{11}H_{22}O_2S$.
Molecular weight: 218·35.
Unit cell: triclinic

$$a=4\cdot69\pm0\cdot05,\ b=5\cdot10\pm0\cdot05,\ c=29\cdot3\pm0\cdot03\ \text{Å};$$
$$\alpha=95°\,8'\pm30',\ \beta=92°\,36',\ \gamma=114°\,48'\pm30'.$$

$d(001)=29\cdot04\pm0\cdot15$ Å.
$Z=2$.
$\varrho_c = 1\cdot134\pm0\cdot011\ \text{g.cm}^{-3}$,
$\varrho_o = 1\cdot128\ \text{g.cm}^{-3}$.

The space group was considered to be $P\bar{1}$ which was later proved to be correct by the refinement.

The crystals decomposed slowly in the X-ray beam and different crystals had to be used for each zone of reflexions. The shape of the reflexions on non-zero layers was very unsatisfactory for intensity estimation. As, moreover, an attempt to scale the three-dimensional data showed that different layers had apparently different temperature factors, it hardly seemed worth while using the full three-dimensional intensity data considering the rather limited accuracy reached in the analysis of phenoxymethylpenicillin (Abrahamsson *et al.*, 1962) where similar experimental difficulties were encountered.

$h0l$ and $0kl$ intensities were estimated visually using the multiple-film technique. The Lorentz and polarization factors were applied but no absorption correction was made.

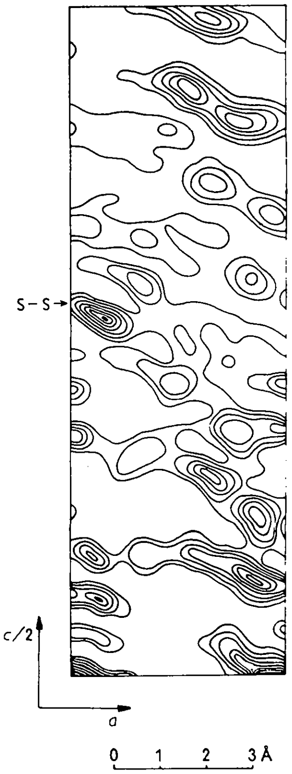

Fig. 1. Sharpened Patterson projection along the b-axis. Contours at arbitrary but equal levels.

Structure determination

From Patterson projections along the two short axes, with coefficients modified to correspond roughly to those from point atoms at rest (Abrahamsson & Maslen, 1962), the S–S vectors were easily recognized (Fig. 1). Electron-density projections from signs given by the sulphur position showed the whole molecule without many spurious features (Fig. 2). A few rounds of difference syntheses reduced R to about 0·25 for both projections. The two sets of data were refined independently by anisotropic least-squares treatment to R about 0·15. An inspection of the vibrational parameters showed that those common for the two projections were similar enough to allow the two sets of data to be refined together. At this stage the hydrogen atoms on the methylene groups and the end methyl group were included in the structure-factor calculations with coordinates calculated with a C–H distance of 1·10 Å and a C–C–H angle of 110°. Isotropic temperature factors only were used for the hydrogen atoms (Table 3). The hydrogen parameters were not refined. Another few rounds of least-squares reduced R to 0·11 after which no further improvement occurred. The carbon shifts were still relatively large being up to half the standard deviations.

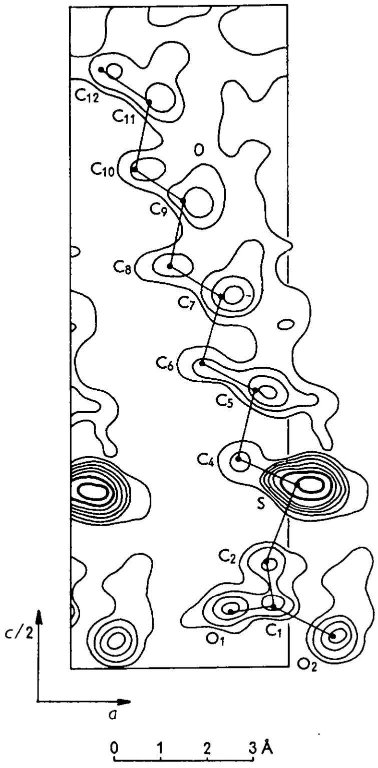

Fig. 2. Sulphur phased electron density projection along the b-axis. Contours at arbitrary but equal levels. The correct structure is outlined.

A Ferranti Mercury computer was used for the calculations with programs described by Mills &

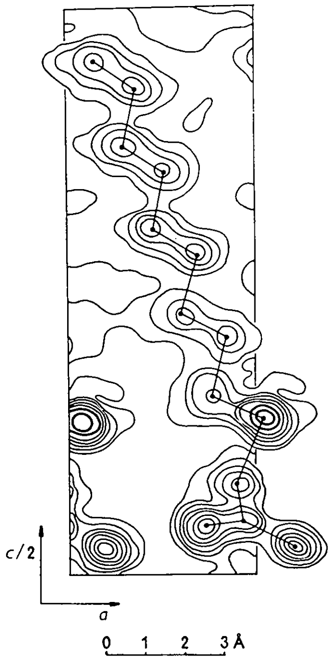

Fig. 3. Final electron density projection along the b-axis. Contours given at intervals of 1 e.Å^{-2} starting at 1 e.Å^{-2} except for the sulphur atom which has heavy lines at intervals of 5 e.Å^{-2}.

Fig. 4. Final electron density projection along the a-axis. Contours given at intervals of 1 e.Å^{-2} starting at 1 e.Å^{-2} except for the sulphur atom which has heavy lines at intervals of 5 e.Å^{-2}.

Rollett (1961). For sulphur the scattering curve of Viervoll & Øgrim (1949) was used, for carbon and oxygen those of Berghuis *et al.* (1955) and for hydrogen that of McWeeny (1952).

Results and discussion

The final coordinates for the carbon and oxygen atoms are listed in Table 1, the vibrational parameters in Table 2. The electron-density projections along the short axes are given in Figs. 3 and 4. Observed and calculated structure factors are given in Table 5.

The bond distances and angles in the molecule are shown in Table 4. The average C–C distance is 1·53 ± 0·06 Å and the average C–C–C angle is 115 ± 3°. This spread of the values is reasonable in comparison with the average standard deviations as estimated from the least-squares treatment (Ahmed & Cruickshank, 1953; Darlow, 1960). They are approximately independent of direction: $\sigma(S) = 0·006$ Å, $\sigma(O) = 0·01$ Å and $\sigma(C) = 0·03$ Å.

The molecular arrangement is shown in Fig. 5. The molecules are as usual for long-chain fatty acids linked together to dimers which form a layer structure.

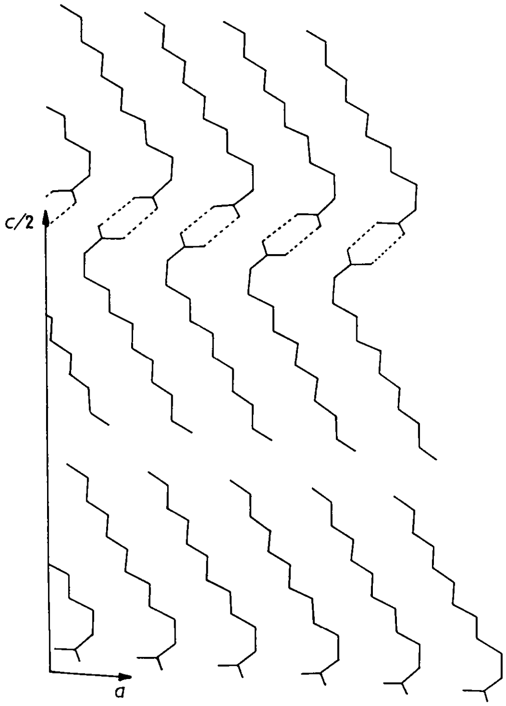

Fig. 5. Molecular arrangement of 3-thiadodecanoic acid as seen along the *b*-axis.

Table 1. *Coordinates of the heavier atoms*

	x/a	y/b	z/c
S	1·0602	1·5290	0·1352
O_1	0·7169	1·0425	0·0422
O_2	1·1962	1·3348	0·0227
C_1	0·9322	1·2762	0·0454
C_2	0·8952	1·5134	0·0802
C_4	0·7652	1·2091	0·1577
C_5	0·8429	1·2230	0·2082
C_6	0·6012	0·9578	0·2299
C_7	0·6859	0·9797	0·2798
C_8	0·4570	0·7180	0·3038
C_9	0·5314	0·7363	0·3538
C_{10}	0·3025	0·4880	0·3780
C_{11}	0·3743	0·5025	0·4279
C_{12}	0·1509	0·2530	0·4539

Table 2. *Vibrational parameters for the heavier atoms*

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

	b_{11}	b_{33}	b_{22}	b_{23}	b_{13}
S	0·12401	0·00175	0·06553	0·00728	−0·00869
C_1	0·11829	0·00187	0·10548	0·02033	−0·00863
C_2	0·18265	0·00231	0·06165	0·00911	−0·01363
C_4	0·12958	0·00343	0·09262	0·01655	−0·01209
C_5	0·13573	0·00271	0·14163	0·00937	−0·00706
C_6	0·12972	0·00286	0·08282	−0·00068	−0·00165
C_7	0·17026	0·00240	0·07390	0·01658	−0·00840
C_8	0·17081	0·00229	0·10728	0·01452	−0·00894
C_9	0·22527	0·00283	0·10427	0·01351	−0·01758
C_{10}	0·18500	0·00269	0·09948	0·01134	−0·01290
C_{11}	0·28786	0·00250	0·10809	0·00686	−0·01846
C_{12}	0·24915	0·00287	0·15894	0·02520	−0·00200
O_1	0·11576	0·00283	0·09604	0·00082	−0·00497
O_2	0·12807	0·00228	0·14525	−0·00092	0·00001

Table 3. *Parameters for the hydrogen atoms*

	x/a	y/b	z/c	B
H_1	0·9567	0·7233	0·0720	5·8 Å^2
H_2	0·6504	0·4724	0·0861	5·8
H_3	0·7618	1·0128	0·1398	5·8
H_4	0·5337	1·2029	0·1514	5·8
H_5	1·0773	1·2364	0·2146	5·8
H_6	0·8551	1·4231	0·2258	5·8
H_7	0·5858	0·7575	0·2119	5·9
H_8	0·3678	0·9486	0·2246	5·9
H_9	0·9219	0·9862	0·2853	5·9
H_{10}	0·7125	1·1753	0·2976	5·9
H_{11}	0·4383	0·5137	0·2869	5·9
H_{12}	0·2194	0·6953	0·2969	5·9
H_{13}	0·7683	0·7569	0·3607	5·9
H_{14}	0·5540	0·9421	0·3706	5·9
H_{15}	0·2774	0·2821	0·3608	6·0
H_{16}	0·0655	0·4667	0·3709	6·0
H_{17}	0·6144	0·5330	0·4351	6·0
H_{18}	0·4029	0·7097	0·4449	6·0
H_{19}	0·1222	0·0458	0·4369	6·0
H_{20}	−0·0893	0·2226	0·4467	6·0
H_{21}	0·2036	0·2637	0·4906	6·0

Table 4. *Bond distances and angles in 3-thiadodecanoic acid*

Bond		Angle	
C_1–O_1	1·20 Å	O_1–C_1–O_2	123°
C_1–O_2	1·37	O_1–C_1–C_2	114
C_1–C_2	1·58	O_2–C_1–C_2	122
C_2–S	1·74	C_1–C_2–S	112
S–C_4	1·85	C_2–S–C_4	103
C_4–C_5	1·50	S–C_4–C_5	110
C_5–C_6	1·58	C_4–C_5–C_6	113
C_6–C_7	1·48	C_5–C_6–C_7	112
C_7–C_8	1·55	C_6–C_7–C_8	115
C_8–C_9	1·47	C_7–C_8–C_9	115
C_9–C_{10}	1·53	C_8–C_9–C_{10}	116
C_{10}–C_{11}	1·47	C_9–C_{10}–C_{11}	116
C_{11}–C_{12}	1·57	C_{10}–C_{11}–C_{12}	118

Table 5. *Final structure factors for 3-thiadodecanoic acid*

An asterisk marks changes of *h* and *k*. Each non-asterisk line contains *l*, $100F_o$ and $100F_c$

*	0	0
3	407	371
4	-478	-478
5	529	429
6	130	80
7	1026	895
8	-107	-88
9	-115	-56
10	-1620	-1554
11	-1161	-1051
12	-2202	-2122
13	204	183
14	1358	1251
15	-456	-431
16	-289	-223
17	-618	-596
18	-645	-673
19	-541	-504
20	-196	-216
21	203	222
22	419	467
23	430	414
24	220	158
27	-319	-269
29	267	200
30	113	99
31	-101	-31
32	-227	-186
33	-275	-199
34	-226	-203
*	0	1
-27	319	327
-26	319	339
-25	727	710
-24	-311	-299
-23	-617	-599
-20	600	582
-19	579	592
-18	558	592
-16	-487	-537
-15	-659	-810
-14	-643	-601
-13	-150	-202
-12	203	196
-11	535	511
-10	261	335
-9	176	204
-8	-168	-65
-7	461	579
-6	915	927
-5	1734	1775
-4	1488	1556
-3	1439	1494
-2	94	138
0	-1563	-1451
1	-255	-178
2	-2384	-2486
3	-254	-235
4	3213	3270
5	-1005	-991
6	-1178	-1070
7	-2109	-2058
8	-1498	-1339
9	-1114	-1077
10	-313	-353
11	-146	-230
12	-265	-275
13	-586	-600
14	-812	-860
15	-465	-524
16	-934	-1005
17	848	873
18	1228	1183
19	402	412
20	207	231
21	-213	-160
*	0	2
-22	380	279
-21	932	847
-20	-1011	-1108
-19	-987	-1003
-18	-448	-405
-16	501	532
-15	318	489
-14	309	500
-13	-637	-490
-12	-652	-575
-11	-1081	-887
-10	-225	-58
-9	-580	-395
-8	1223	1136
-7	885	851
-6	-1775	-1750
-5	-855	-1046
-4	-845	-999
-2	428	393
-1	858	891
0	771	846
1	282	287
2	-286	-391
3	-579	-720
4	-590	-612
6	-438	-381
7	482	462
8	915	601
10	-245	-265
11	-356	-374
13	354	367
14	693	697
15	447	407
16	205	250
17	-298	-226
18	-215	-93
19	-219	-156
20	-445	-331
21	502	521
22	451	468
*	0	3
-23	-388	-357
-20	318	473
-18	442	450
-17	598	557
-16	-826	-808
-15	-668	-588
-14	-467	-420
-12	202	189
-11	199	272
-9	-388	-472
-8	-383	-533
-7	-190	-214
-6	497	420
-5	264	86
-4	1298	1683
-3	989	857
-2	-490	-585
-1	-321	-447
1	374	346
2	608	585
3	783	801
4	534	598
6	-280	-290
7	-449	-426
8	-204	-217
12	-217	-235
13	-439	-373
14	-496	-432
15	-224	-240
*	0	4
-12	-391	-476
-9	317	257
-8	316	257
-7	223	230
0	704	885
2	-387	-371
3	-501	-498
4	-449	-372
9	-225	-236
10	-317	344
14	372	353
*	1	0
-34	-103	-151
-33	-112	-160
-32	-93	-117
-28	-179	-153
-27	-226	-244
-26	-460	-567
-25	-90	-119
-24	402	395
-23	239	232
-22	319	401
-21	110	98
-20	-106	-55
-19	-237	-259
-17	371	336
-16	776	687
-15	869	816
-14	596	600
-13	210	219
-12	-262	-179
-11	-98	-46
-10	128	13
-9	611	568
-8	502	429
-7	315	265
-6	-679	-621
-5	-834	-922
-4	-1262	-1352
-3	-433	-471
-2	-249	-219
-1	1122	1178
0	1006	655
1	2299	2958
2	2153	2768
3	-2319	-2594
4	101	184
5	205	224
6	1017	1082
7	744	773
8	293	357
9	-508	-544
10	-1077	-1171
11	-1063	-1211
12	-950	-954
14	-241	-258
15	1425	1398
16	-312	-360
17	-1649	-1576
18	-780	-818
19	-748	-738
21	204	174
22	354	358
23	197	176
25	-168	-162
26	-127	-116
27	118	70
28	368	301
29	393	373
30	194	213
32	-198	-152
33	-205	-205
*	2	0
-27	-130	-129
-26	-132	-127
-23	392	458
-22	216	263
-21	-126	-108
-20	-106	-142
-19	-151	-178
-18	72	83
17	322	279
-16	542	562
-15	447	463
-14	341	342
-13	103	25
-12	-87	-45
-11	-234	-205
-10	384	394
-9	1394	1333
-8	474	479
-7	89	98
-6	-555	-548
-5	-897	-765
-4	-814	-789
-3	-409	-417
-1	176	137
0	-161	-147
1	-429	-426
2	-684	-840
3	-412	-428
4	-717	-667
5	-528	-479
6	454	466
7	102	137
8	-144	-113
9	-621	-630
10	-662	-703
11	-594	-629
13	318	305
14	558	652
15	364	339
16	182	145
17	198	193
18	-923	-943
19	-319	-265
20	477	380
21	436	373
22	346	352
24	-158	-156
25	-238	-250
27	102	140
28	301	317
29	247	247
30	203	209
32	-276	-290
33	-151	-152
*	3	0
-22	76	113
-21	99	115
-20	-100	-138
-19	-78	-136
-17	110	149
-16	118	163
-14	-76	-145
-13	-317	-325
-12	-267	-272
-11	-235	-198
-8	496	460
-7	189	131
-6	-743	-694
-5	-518	-510
-4	-343	-332
-2	301	294
-1	334	299
1	-217	-182
2	-322	-360
3	-236	-231
5	337	330
6	387	467
7	154	127
8	-252	-264
9	-550	-509
10	-507	-478
11	-261	-209
13	255	245
14	259	270
17	-180	-211
21	447	450
22	265	245
24	-136	-139
25	-134	-128
27	113	147
28	157	222
29	105	182
31	-41	-108
*	4	0
-14	-75	-86
-13	-123	-161
-12	-108	-146
-7	-127	-122
-5	-352	-332
-4	-335	-307
-3	110	63
-2	168	180
-1	182	189
4	201	210
5	365	353
6	333	320
7	272	333
9	-295	-284
10	-224	-196
12	136	156
13	158	180
15	-100	-117
16	-210	-214
17	-171	-195
20	144	140
24	-78	-133
25	-33	-70
*	5	0
1	-106	-73
4	90	79
5	143	121
6	114	108
13	103	115
14	87	36
16	-28	-122

There is a bend at the sulphur atom similar to that found in branched fatty acids (Abrahamsson, 1959), *cis*-cyclopropane fatty acids (Craven & Jeffrey, 1959) *cis*-unsaturated acids (Abrahamsson & Ryderstedt-Nahringbauer, 1962) and in ethyl stearate (Aleby, 1962). Bent long-chain molecules are thus rather common and found in structures where the carbon chain packing is disturbed by the presence of other than methylene groups along the molecule.

The chain part C_4–C_{12} has the usual planar zig-zag conformation. The maximum deviation from the best least-squares plane is 0·04 Å. The sulphur atom also lies in this plane whereas the bond S–C_2 is rotated 12° out of the plane.

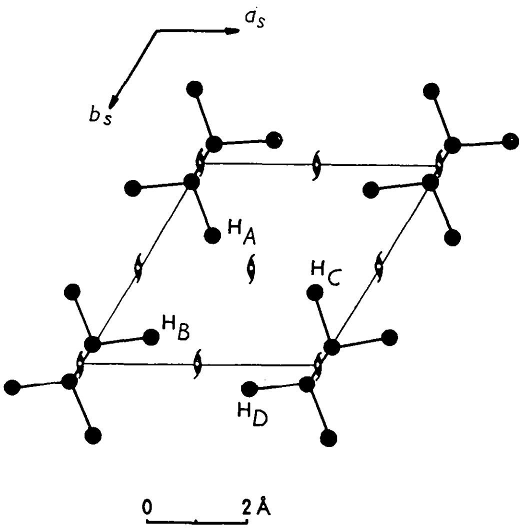

Fig. 6. Idealized subcell *M* || found in 3-thiadodecanoic acid.

The regular chain parts pack side by side with all chain planes parallel. The dimensions of the subcell are calculated to be (average values):

$$a_s = 4{\cdot}69,\ b_s = 4{\cdot}56,\ c_s = 2{\cdot}56\ \text{Å};$$
$$\alpha_s = 90°,\ \beta_s = 84°,\ \gamma_s = 122°.$$

The subcell is not similar to those previously known (Abrahamsson *et al.*, 1962; Abrahamsson & Ryderstedt-Nahringbauer, 1962) but can be regarded as a distortion of the theoretical monoclinic subcell deduced on close-packing considerations by Kitaigorodskii (1957). 3-Thiadodecanoic acid appears to be the first long-chain compound reported to show this packing. The idealized subcell, *M* ||, ($\beta_s = 90$) is shown in Fig. 6. The space group is $P2_1/m$ with 2 CH_2 groups per cell. The volume per CH_2 group (23.2 Å^3) is similar to that in the common orthorhombic (O ⊥) and triclinic (*T* ||) chain packing.

The average values for the hydrogen contacts in the subcell region were calculated from the positions in Table 3. The H_A–H_B distance is 2·68 Å whereas two alternate distances grouped around 2·52 Å and 2·93 Å are found for the H_A–H_C and H_B–H_D distances due to the actual deviations from the idealized subcell. The spread from these mean values is large (~ 0·1 Å) as is to be expected as the hydrogen positions were calculated from carbon positions with appreciable standard deviations.

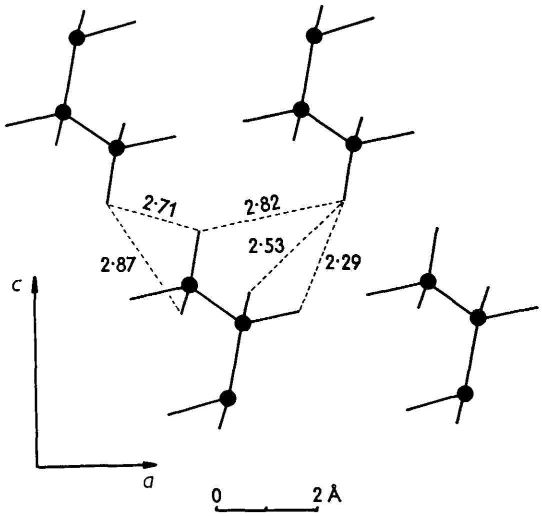

Fig. 7. Hydrogen contacts in the methyl group contact planes. The directions of the *a*- and *c*-axes are indicated.

The angle of tilt of the chain axes to the (001) planes is 57° which is comparable to that in other simple long-chain compounds (Abrahamsson *et al.*, 1962). The methyl end group packing is illustrated in Fig. 7.

The carboxylic groups are as usual around centres of symmetry. The O–H · · · O distance is 2·74 Å. The atoms C_2, C_1, O_1 and O_2 lie in a plane (max. dev. 0·01 Å) which is separated 0·16 Å from that of the hydrogen bonded carboxylic group. A corresponding separation is found in α-pimelic acid (~ 0·3 Å, Kay & Katz, 1958) in oleic acid (0·35 Å, Abrahamsson & Ryderstedt-Nahringbauer, 1962) and in the *D*-form of 11-bromoundecanoic acid (1·17 Å, Larsson, 1962).

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A Direct Determination of the Molecular Structure of Prostaglandin F_{2-1}

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The tri-*p*-bromobenzoate of the methyl ester of prostaglandin $F_{2-1}(C_{42}H_{47}O_8Br_3)$ is orthorhombic with $a=26\cdot14$, $b=33\cdot93$ and $c=4\cdot76$ Å. The space group is $P2_12_12_1$ with $Z=4$. The structure was solved by heavy-atom methods and refined by three-dimensional anisotropic least-squares treatment to give the molecular structure and configuration of prostaglandin F_{2-1}, which belongs to a new class of hormones having smooth-muscle-stimulating and blood-pressure-depressing activity.

Introduction

A smooth-muscle-stimulating and blood-pressure-depressing activity was discovered in human semen by Goldblatt (1933) and by von Euler (1934, 1935) who also found a similar activity in sheep sperm and named the factor 'prostaglandin'.

In 1960 Bergström & Sjövall (1960) isolated two active crystalline compounds $PGE_1(C_{20}H_{34}O_5)$ and $PGF_{1-1}(C_{20}H_{36}O_5)$ from sheep prostate glands. Reduction of PGE_1 with borohydride (Bergström *et al.*, 1962) yielded PGF_{1-1} together with the isomeric compound PGF_{2-1}.

The tri-*p*-bromo- and tri-*p*-iodobenzoates of the methyl ester of PGF_{2-1} were prepared by Prof. S. Bergström who kindly provided them for an X-ray analysis in order to determine *ab initio* the molecular structures.

Experimental

The two benzoates crystallize as beautiful needles from ethanol. All crystals studied gave excellent X-ray photographs, which indicated that the two derivatives are isomorphous. The unit cell is orthorhombic with space group $P2_12_12_1$. The cell dimensions are:

	a	*b*	*c*
Br-derivative	26·14 Å	33·93 Å	4·76 Å
I-derivative	26·49	34·39	4·76

Full three-dimensional data (Cu $K\alpha$ radiation) were collected for the bromo compound. Multiple-film Weissenberg photographs were recorded for the 0–2 layers about *c* (needle direction) and the 0–5 layers about *a*. Only *hk*0 intensities were estimated for the iodo derivative. The intensities were corrected for the Lorentz and polarization factors but not for absorption.

Structure determination

The initial work was done on the (001) projection. From a sharpened Patterson projection of the I-derivative, the relative positions of the heavy atoms of the four molecules per cell were obtained by simple vector addition and subtraction. The origin was then determined from the known symmetry of the projection. As expected the Patterson synthesis was completely dominated by I–I vectors (Fig. 1).

An electron-density projection showed most of the atoms of the molecule (Fig. 2) but also contained some spurious detail. This, however, was still present,