Acta Cryst. (1968). B**24**, 1399

The Crystal Structure of DL-2-Methyl-7-Oxododecanoic Acid

BY A. M. O'CONNELL

Crystallography Group, Medical Research Council Unit for Molecular Structure Analysis, University of Göteborg, Göteborg, Sweden

(*Received* 12 *February* 1968)

The crystal structure of DL-2-methyl-7-oxododecanoic acid has been determined by direct methods from three-dimensional X-ray diffraction data. The crystals are monoclinic, space group $P2_1/c$, with $a=9{\cdot}122$, $b=5{\cdot}061$, $c=30{\cdot}784$ Å and $\beta=100{\cdot}7°$. Positional and anisotropic thermal parameters of the carbon and oxygen atoms were refined to give a final R index of 0·088. The hydrocarbon chain is bent at C(2) and the methyl group forms a continuation of the zigzag backbone. The chain is twisted by 7° at the keto group. The carboxyl groups are hydrogen bonded across centres of symmetry, the chain packing being the orthorhombic type $O'\perp$.

Introduction

In this laboratory, studies of the effect of a branching methyl group in different positions along a hydrocarbon chain have been undertaken (*e.g.* Abrahamsson, 1959*a*; Abrahamsson & Harding, 1966). The effect of the methyl substituent on the molecular geometry and packing has been reported. The present paper, which describes the X-ray analysis of DL-2-methyl-7-oxododecanoic acid, represents an extension of this work to include other substituent groups.

Experimental

Crystal data

$C_{13}H_{24}O_3$ DL-2-methyl-7-oxododecanoic acid.
Monoclinic, space group $P2_1/c$.
$a=9{\cdot}122\pm20$, $b=5{\cdot}061\pm7$, $c=30{\cdot}784\pm50$ Å, $\beta=100{\cdot}7°\pm0{\cdot}2°$.
($\lambda=1{\cdot}5418$ Å).
$V=1396$ Å^3.
$Z=4$.
m.p. 36·1–36·4 °C. (Ställberg-Stenhagen, 1946.)
Absent spectra $0k0$, k odd; $h0l$, l odd.
$\mu=6{\cdot}54$ cm^{-1} for $\lambda=1{\cdot}5418$ Å .

Crystals suitable for X-ray diffraction measurements were obtained by evaporation of a light petroleum solution. The crystals formed as needles with the longest direction parallel to the b axis. A sample measuring 0·10 × 0·60 × 0·13 mm was selected for data collection about the b axis. Multi-film equi-inclination Weissenberg techniques with Cu $K\alpha$ radiation were used to record intensities on layers $h0l$ to $h4l$. The crystal was then cut with a razor blade to measure 0·10 × 0·25 × 0·13 mm and remounted about the a axis. Layers $0kl$ to $4kl$ were recorded with this orientation.

The data were estimated with the use of the automatic on-line film scanner in this laboratory (Abrahamsson, 1966). Film factor-intensity distributions were used to estimate the linear range of measurement as described previously (O'Connell, 1967). A normal beam film factor of 2·93 (Morimoto & Uyeda, 1963) was assumed, and this value, with the appropriate angular correction for non-zero levels (Grenville-Wells, 1955) was used to reduce the intensities within a film pack to a common scale. Lorentz and polarization corrections were applied to all reflexions; however, absorption corrections were considered unnecessary because of the small size of the crystals and the low value of the linear absorption coefficient ($\mu=6{\cdot}54$ cm^{-1}). Interlayer correlation scales were obtained by the method of Hamilton, Rollett & Sparks (1965). The final set of 827 observed structure factors was obtained from 3835 individual intensity measurements. Unobserved terms were not included in the refinement or R-index calculations (Dunning & Vand, 1966).

Solution and refinement

Initial attempts to solve the structure by using symbolic addition methods failed (see Karle & Karle(1966) for a recent review of these techniques). In retrospect, this appears to have been due to the incorrect indexing of several high order reflexions with large E values. In order to fix the phases of the subcell reflexions, composite three-dimensional structure factor graphs of the two largest normalized structure factors ($E_{31\bar{8}}$ and E_{229}) were computed with use of the two sign combinations $++$ and $-+$. The second of these showed a regular repetition of maxima which was consistent with chain packing considerations. On the basis of this arrangement the phases of the nine largest normalized structure factors were fixed. A further three signs were determined by using the Σ_1 formula (Hauptman & Karle, 1953). With these twelve phases as a starting point the signs of a further 45 reflexions with $E>1{\cdot}7$ were generated through successive application of the Σ_2 formula (Hauptman & Karle, 1953). These phases were in turn used to determine 72 more signs for reflexions with $E>1{\cdot}0$. It was not necessary to introduce symbols at any stage during the application of Σ_2. The

Table 1. *Observed and calculated structure factors* (× 10)

The unobserved terms (indicated by a minus sign on F_{obs}) are included at the threshold values.

H	K	L	Fobs	Fcalc
0	0	4	347	327
0	0	6	232	-211
0	0	8	72	69
0	0	10	340	-339
0	0	12	57	-28
0	0	14	437	-443
0	0	16	110	-92
0	0	18	168	-149
0	0	20	-41	7
0	0	22	-44	-17
0	0	24	-47	57
0	0	26	-49	50
0	0	28	265	286
0	0	30	123	-126
0	0	32	57	-52
0	1	1	293	-305
0	1	2	205	239
0	1	3	250	-264
0	1	4	339	359
0	1	5	285	-280
0	1	6	392	429
0	1	7	68	-46
0	1	8	270	271
0	1	9	100	-104
0	1	10	201	199
0	1	11	185	175
0	1	12	69	-80
0	1	13	64	56
0	1	14	429	-437
0	1	15	-43	-19
0	1	16	105	-83
0	1	17	-48	-39
0	1	18	156	-135
0	1	19	107	94
0	1	20	176	-165
0	1	21	-56	-44
0	1	22	113	-83
0	1	23	-60	42
0	1	24	-61	-71
0	1	25	-63	-44
0	1	26	-63	-5
0	1	27	-64	36
0	1	28	-64	-54
0	1	29	104	-81
0	1	30	153	165
0	2	0	725	-706
0	2	1	252	-250
0	2	2	201	-181
0	2	3	-19	0
0	2	4	131	120
0	2	5	199	-168
0	2	6	163	-142
0	2	7	102	106
0	2	8	157	138
0	2	9	-38	-6
0	2	10	76	-49
0	2	11	310	274
0	2	12	201	199
0	2	13	487	425
0	2	14	210	199
0	2	15	348	-281
0	2	16	100	90
0	2	17	130	112
0	2	18	-61	-40
0	2	19	191	186
0	2	20	-65	50
0	2	21	105	92
0	2	22	-69	14
0	2	23	92	97
0	2	24	-71	36
0	2	25	-72	17
0	2	26	-72	6
0	2	27	-71	-25
0	2	28	138	-118
0	3	1	149	136
0	3	2	-17	15
0	3	3	173	167
0	3	4	97	-77
0	3	5	71	68
0	3	6	77	-47
0	3	7	104	109
0	3	8	126	-102
0	3	9	-43	-25
0	3	10	86	-69
0	3	11	-49	59
0	3	12	105	-86
0	3	13	111	-88
0	3	14	69	-58
0	3	15	187	184
0	3	16	85	-67
0	3	17	134	-107
0	3	18	-66	17
0	3	19	-67	-1
0	3	20	-69	-18
0	3	21	95	-94
0	3	22	-70	18
0	3	23	-70	2
0	3	24	-70	4
0	3	25	-69	-68
0	3	26	-68	3
0	3	27	-66	-28
0	3	28	-63	-13
0	3	29	88	82
0	4	3	21	23
0	4	4	30	44
0	4	5	100	-84
0	4	6	66	-60
0	4	7	29	40
0	4	8	43	42
0	4	9	88	-78
0	4	10	-22	-28
0	4	11	-23	-2
0	4	12	-25	-42
0	4	13	258	-278
0	4	14	182	-197
0	4	15	313	340
0	4	16	-28	6
0	4	17	49	-47
1	0	-32	109	135
1	0	-30	-48	6
1	0	-28	-49	-59
1	0	-26	-48	-11
1	0	-24	93	-93
1	0	-22	-43	31
1	0	-20	-40	-48
1	0	-18	427	410
1	0	-16	149	-118
1	0	-14	265	285
1	0	-12	221	228
1	0	-10	476	523
1	0	-8	286	290
1	0	-6	381	416
1	0	-4	160	110
1	0	-2	-98	-104
1	0	0	-362	-461
1	0	2	-531	-569
1	0	4	-629	-723
1	0	6	583	-665
1	0	8	458	-507
1	0	10	337	-399
1	0	12	269	298
1	0	14	46	39
1	0	16	157	170
1	0	18	59	57
1	0	20	81	86
1	0	22	-45	-63
1	0	24	-48	-38
1	0	26	84	83
1	0	28	99	-104
1	0	30	91	-93
1	1	-32	121	-95
1	1	-31	-61	-29
1	1	-30	157	156
1	1	-29	-63	-28
1	1	-28	77	78
1	1	-27	-64	-2
1	1	-26	-63	33
1	1	-25	-62	27
1	1	-24	-61	0
1	1	-23	-59	52
1	1	-22	-57	-52
1	1	-21	-55	47
1	1	-20	67	-51
1	1	-19	143	127
1	1	-18	-49	-23
1	1	-17	437	-415
1	1	-16	207	-200
1	1	-15	-43	9
1	1	-14	105	-113
1	1	-13	178	-148
1	1	-12	-36	-17
1	1	-11	-34	-16
1	1	-10	56	-44
1	1	-9	258	-228
1	1	-8	-28	-16
1	1	-7	79	78
1	1	-6	313	-316
1	1	-5	254	-244
1	1	-4	1152	-1206
1	1	-3	73	27
1	1	-2	-1338	1641
1	1	-1	-15	9
1	1	0	306	313
1	1	1	546	637
1	1	2	242	-236
1	1	3	236	253
1	1	4	212	-202
1	1	5	571	589
1	1	6	416	-402
1	1	7	120	100
1	1	8	197	-180
1	1	9	258	247
1	1	10	198	-191
1	1	11	793	-836
1	1	12	173	-163
1	1	13	471	445
1	1	14	65	-75
1	1	15	-45	-4
1	1	16	147	141
1	1	17	97	91
1	1	18	126	118
1	1	19	72	-45
1	1	20	145	157
1	1	21	-58	54
1	1	22	-60	74
1	1	23	-61	-20
1	1	24	-62	11
1	1	25	-63	70
1	1	26	328	319
1	2	-31	155	-134
1	2	-30	-67	67
1	2	-29	85	-89
1	2	-28	-70	6
1	2	-27	-71	-43
1	2	-26	-72	76
1	2	-25	-71	-68
1	2	-24	-71	-27
1	2	-23	-69	35
1	2	-22	-68	46
1	2	-21	-66	36
1	2	-20	116	-94
1	2	-19	215	199
1	2	-18	157	-138
1	2	-17	87	-69
1	2	-16	-55	-43
1	2	-15	155	151
1	2	-14	63	61
1	2	-13	138	125
1	2	-12	208	-197
1	2	-11	212	209
1	2	-10	-40	51
1	2	-9	97	83
1	2	-8	162	-150
1	2	-7	155	158
1	2	-6	127	125
1	2	-5	43	29
1	2	-4	58	-39
1	2	-3	202	-208
1	2	-2	129	131
1	2	-1	200	174
1	2	1	228	225
1	2	2	206	190
1	2	3	209	188
1	2	4	144	124
1	2	5	235	214
1	2	6	140	112
1	2	7	194	174
1	2	8	125	102
1	2	9	160	135
1	2	10	-43	-5
1	2	11	228	222
1	2	12	80	-63
1	2	13	231	-202
1	2	14	83	-70
1	2	15	138	-113
1	2	16	-58	0
1	2	17	170	-155
1	2	18	116	-95
1	2	19	114	-91
1	2	20	-67	11
1	2	21	123	-104
1	2	22	87	-73
1	2	23	-71	-28
1	2	24	-72	34
1	2	25	-72	38
1	2	26	85	-89
1	2	27	107	-82
1	3	-27	-67	-4
1	3	-24	88	82
1	3	-23	-70	-6
1	3	-21	-69	-13
1	3	-20	-68	-11
1	3	-19	118	-85
1	3	-18	163	-141
1	3	-17	459	419
1	3	-16	82	69
1	3	-15	-59	21
1	3	-14	85	-70
1	3	-13	-54	26
1	3	-12	-51	-36
1	3	-11	115	115
1	3	-10	222	-184
1	3	-9	-43	19
1	3	-8	58	-33
1	3	-7	120	111
1	3	-6	182	-168
1	3	-5	78	-68
1	3	-4	125	79
1	3	-3	83	70
1	3	-2	258	-227
1	3	-1	225	-193
1	3	0	95	77
1	3	1	84	-63
1	3	2	115	-98
1	3	3	272	-250
1	3	4	99	96
1	3	5	67	-45
1	3	6	141	-119
1	3	7	236	-207
1	3	8	-44	-5
1	3	9	-47	2
1	3	10	167	-138
1	3	11	465	435
1	3	12	266	231
1	3	13	267	-239
1	3	14	-59	19
1	3	15	142	-126
1	4	-19	48	-49
1	4	-18	-29	-45
1	4	-17	74	46
1	4	-16	-28	17
1	4	-15	52	57
1	4	-14	-26	32
1	4	-13	-25	26
1	4	-12	-24	-22
1	4	-11	60	63
1	4	-10	73	55
1	4	-9	-21	-5
1	4	-8	47	-42
1	4	-7	-18	26
1	4	-6	-17	20
1	4	-5	64	-54
1	4	-4	93	-97
1	4	-3	49	64
1	4	-2	-11	23
1	4	-1	104	-99
1	4	0	29	20
1	4	1	54	-51
1	4	2	44	38
1	4	3	53	-56
1	4	4	40	40
1	4	5	-17	-28
1	4	6	40	47
1	4	7	-20	-16
1	4	8	37	52
1	4	9	46	43
1	4	10	64	84
1	4	11	-25	19
1	4	12	-26	8
1	4	13	140	149
1	4	14	-27	26
1	4	15	71	93
1	4	16	77	94
1	4	17	-29	32
1	4	18	-29	54
1	4	19	-29	25
1	4	20	54	70
1	4	21	-28	-8
1	4	22	-27	42
1	4	23	-26	-5
1	4	24	-25	29
1	4	25	79	-76
1	4	26	80	-68
2	0	-34	108	127
2	0	-32	-46	-26
2	0	-30	-49	-18
2	0	-28	-49	27
2	0	-26	-48	51
2	0	-24	93	93
2	0	-22	107	107
2	0	-20	-40	-20
2	0	-18	53	54
2	0	-16	201	-193
2	0	-14	288	-268
2	0	-12	525	-567
2	0	-10	454	-487
2	0	-8	630	-649
2	0	-6	-1525	1824
2	0	-4	-900	-1118
2	0	-2	268	-289
2	0	0	203	-204
2	0	2	316	355
2	0	4	131	153
2	0	6	411	471
2	0	8	54	57
2	0	10	178	199
2	0	12	120	-113
2	0	14	-35	38
2	0	16	177	-167
2	0	18	-41	27
2	0	20	-44	-8
2	0	22	378	389
2	0	24	293	-300
2	0	26	-49	34
2	0	28	-48	60
2	0	30	73	80
2	1	-23	79	-78
2	1	-22	-58	-48
2	1	-21	79	-54
2	1	-20	274	247
2	1	-19	65	-50
2	1	-18	-50	38
2	1	-17	103	108
2	1	-16	68	-58
2	1	-15	64	-75
2	1	-14	-42	27
2	1	-13	224	215
2	1	-12	145	-128
2	1	-11	-36	27
2	1	-10	97	-58
2	1	-9	347	351
2	1	-8	272	-253
2	1	-7	-29	10
2	1	-6	75	6
2	1	-5	642	636
2	1	-4	105	-99
2	1	-3	72	65
2	1	-2	141	144
2	1	-1	204	196
2	1	0	134	125
2	1	1	142	-142
2	1	2	287	295
2	1	3	60	-50
2	1	4	267	263
2	1	5	204	-203
2	1	6	306	329
2	1	7	108	-95
2	1	8	285	297
2	1	9	342	-338
2	1	10	211	- 231
2	1	11	-41	-37
2	1	12	130	138
2	1	13	-45	20
2	1	14	72	71
2	1	15	-49	32
2	1	16	-51	32
2	1	17	84	87
2	1	18	-55	-36
2	1	19	-57	28
2	1	20	-59	-60
2	1	21	-60	53
2	1	22	173	-170
2	1	23	127	-99
2	1	24	94	99
2	2	-26	103	-92
2	2	-25	-71	88
2	2	-24	93	-79
2	2	-23	148	128
2	2	-22	95	-71
2	2	-21	333	302
2	2	-20	167	159
2	2	-19	211	-169
2	2	-18	-61	48
2	2	-17	-58	-31
2	2	-16	126	108
2	2	-15	-54	14
2	2	-14	175	168
2	2	-13	-49	-55
2	2	-12	181	169
2	2	-11	-45	12
2	2	-10	203	187
2	2	-9	62	-56
2	2	-8	60	55
2	2	-7	-37	29
2	2	-6	484	-460
2	2	-5	248	-205
2	2	-4	-33	14
2	2	-3	65	-54
2	2	-2	-31	4
2	2	-1	131	-127
2	2	0	293	-279
2	2	1	70	57
2	2	2	74	-59
2	2	3	114	-97
2	2	4	276	-235
2	2	5	132	118
2	2	6	186	167
2	2	7	186	134
2	2	8	68	61
2	2	9	933	-927
2	2	10	79	74
2	2	11	189	-179
2	2	12	147	-126
2	2	13	-55	8
2	2	14	134	112
2	2	15	89	-81
2	2	16	-62	-19
2	2	17	82	66
2	2	18	86	82
2	2	19	-67	13
2	2	20	-69	-60
2	2	21	-70	66
2	2	22	137	-118
2	2	23	-72	42
2	2	24	105	99
2	3	-24	120	-107
2	3	-23	-70	-14
2	3	-22	-70	-58
2	3	-21	191	187
2	3	-20	155	-135
2	3	-19	92	-75
2	3	-18	-66	-22
2	3	-17	-64	-5
2	3	-16	-62	-31
2	3	-15	133	-132
2	3	-14	-58	60
2	3	-13	-56	-16
2	3	-12	-53	28
2	3	-11	182	-163
2	3	-10	164	148
2	3	-9	-47	22
2	3	-8	134	114
2	3	-7	-43	2
2	3	-6	246	257
2	3	-5	261	-254
2	3	-4	59	59
2	3	-3	129	-118
2	3	-2	-36	20
2	3	-1	88	89
2	3	0	62	-50
2	3	1	-37	-32
2	3	2	-38	-11
2	3	3	116	112
2	3	4	70	-59
2	3	5	57	43
2	3	6	-46	9
2	3	7	90	76
2	3	8	-50	-42
2	3	9	192	174
2	3	10	65	58
2	3	11	-57	-11
2	3	12	104	95
2	3	13	-61	-24
2	3	14	88	91
2	3	15	-65	-37
2	3	16	-66	69
2	3	17	-68	13
2	3	18	-69	44
2	3	19	-70	-6
2	3	20	-70	-1
2	3	21	-70	41
2	3	22	-70	-11
2	3	23	119	120
2	4	-21	159	-157
2	4	-20	87	-70
2	4	-19	185	199
2	4	-18	-29	-9
2	4	-17	-29	-16
2	4	-16	-28	41
2	4	-15	-28	-12
2	4	-14	-27	18
2	4	-13	79	-70
2	4	-12	-25	10
2	4	-11	-24	-16
2	4	-10	-23	34
2	4	-9	61	-43
2	4	-8	56	57
2	4	-7	45	44
2	4	-6	-20	-8
2	4	-5	80	-79
2	4	-4	98	113
2	4	-3	80	83
2	4	-2	137	152
2	4	-1	29	-9
2	4	0	64	74
2	4	1	124	124
2	4	2	119	134
2	4	3	-19	-1
2	4	4	-20	20
2	4	5	76	73
2	4	6	-22	-23
2	4	7	210	-209
2	4	8	215	-226
2	4	9	370	452
2	4	10	70	75
2	4	11	140	-127
2	4	12	-27	0
2	4	13	-28	-14
2	4	14	-28	-5
2	4	15	91	-81
2	4	16	62	-75
2	4	17	-29	-23
2	4	18	-29	-17
2	4	19	59	-59
2	4	20	49	-45
2	4	21	-27	15
2	4	22	50	-49
2	4	23	86	-65
3	0	-24	98	99
3	0	-22	333	-312
3	0	-20	122	-106
3	0	-18	162	-140
3	0	-16	-36	62
3	0	-14	-33	-3
3	0	-12	316	334
3	0	-10	300	286
3	0	-8	494	492
3	0	-6	246	255
3	0	-4	389	456
3	0	-2	42	45
3	0	0	152	186
3	0	2	138	-126
3	0	4	43	55
3	0	6	430	-500
3	0	8	-30	7
3	0	10	-33	-12
3	0	12	83	94
3	0	14	49	63
3	0	16	71	76
3	0	18	-44	63
3	0	20	249	292
3	0	22	73	-59
3	0	24	63	-58
3	0	26	-48	-51
3	0	28	62	-68
3	1	-24	115	113
3	1	-23	128	-130
3	1	-22	-59	-51
3	1	-21	85	84
3	1	-20	-56	30
3	1	-19	135	122
3	1	-18	-52	-7
3	1	-17	94	89
3	1	-16	-48	-22
3	1	-15	98	87
3	1	-14	96	-70
3	1	-13	-43	46
3	1	-12	130	-100
3	1	-11	74	-68
3	1	-10	362	-333
3	1	-9	170	-146
3	1	-8	974	963
3	1	-7	195	-175
3	1	-6	295	272
3	1	-5	-33	-20
3	1	-4	168	163
3	1	-3	201	-202
3	1	-2	200	209
3	1	-1	49	-38
3	1	0	100	97
3	1	1	270	-243
3	1	2	118	115
3	1	3	113	-98
3	1	4	126	-124
3	1	5	801	-834
3	1	6	152	-146
3	1	7	555	561
3	1	8	280	-280
3	1	9	57	54
3	1	10	205	-192
3	1	11	161	147
3	1	12	257	-251
3	1	13	68	-41
3	1	14	189	-173
3	1	15	70	62
3	1	16	193	-180
3	1	17	124	-100
3	1	18	87	-72
3	1	19	-60	25
3	1	20	414	404
3	2	-23	210	-198
3	2	-22	193	162
3	2	-21	84	-65
3	2	-20	116	124
3	2	-19	90	-76
3	2	-18	86	-78
3	2	-17	-61	-19
3	2	-16	-59	22
3	2	-15	111	-84
3	2	-14	211	-206
3	2	-13	-53	6
3	2	-12	-51	-27
3	2	-11	113	-89
3	2	-10	324	-321
3	2	-9	319	-284
3	2	-8	105	-63
3	2	-7	69	58
3	2	-6	205	-207
3	2	-5	98	87
3	2	-4	187	173
3	2	-3	83	-73
3	2	-2	-40	-7
3	2	-1	68	70
3	2	0	289	268
3	2	1	-41	-52
3	2	2	64	42
3	2	3	71	70
3	2	4	185	172
3	2	5	-46	33
3	2	6	198	177
3	2	7	81	-48
3	2	8	90	84
3	2	9	-53	23
3	2	10	111	-83
3	2	11	124	113
3	2	12	-58	-55
3	2	13	116	112
3	2	14	109	-88
3	2	15	151	140
3	2	16	-66	-61
3	2	17	119	115
3	2	18	-69	-5
3	2	19	135	121
3	2	20	92	-70
3	2	21	-72	-30
3	2	22	85	88
3	3	-25	122	-102
3	3	-24	-70	-7
3	3	-23	216	193
3	3	-22	167	168
3	3	-21	171	-149
3	3	-20	132	134
3	3	-19	-68	-40
3	3	-18	122	130
3	3	-17	-66	-33
3	3	-16	78	79
3	3	-15	-63	44
3	3	-14	92	92
3	3	-13	-59	50
3	3	-12	-57	10
3	3	-11	112	109
3	3	-10	83	70
3	3	-9	85	79
3	3	-8	233	-211
3	3	-7	-50	59
3	3	-6	-48	-34
3	3	-5	-47	8
3	3	-4	161	-133
3	3	-3	-46	-1
3	3	-2	-46	-18
3	3	-1	-46	41
3	3	0	137	-105
3	3	1	-47	-6
3	3	2	-48	10
3	3	3	108	107
3	3	4	-51	-1
3	3	5	485	475
3	3	6	334	331
3	3	7	286	-264
3	3	8	-57	52
3	3	9	89	-72
3	3	10	-60	49
3	3	11	-62	80
3	3	12	74	-70
3	3	13	-65	-40
3	3	14	-67	8
3	3	15	-68	57
3	3	16	88	-83
3	3	17	-70	-31
3	3	18	-70	-25
3	3	19	-70	17
3	3	20	130	-132
3	4	-25	43	-52
3	4	-24	-26	-149
3	4	-23	-27	3
3	4	-22	-28	36
3	4	-21	-28	-3
3	4	-20	-29	22
3	4	-19	-29	-46
3	4	-18	-29	28
3	4	-17	-29	0
3	4	-16	69	73
3	4	-15	-28	-33
3	4	-14	-28	20
3	4	-13	43	55
3	4	-12	-27	47

Table 1 (*cont.*)

H	K	L	Fobs	Fcalc
3	4	-11	-26	-1
3	4	-10	59	-51
3	4	-9	226	262
3	4	-8	92	93
3	4	-7	55	-61
3	4	-6	-23	-17
3	4	-5	65	66
3	4	-4	36	-29
3	4	-3	-22	-8
3	4	-2	129	-132
3	4	-1	-22	5
3	4	0	84	-81
3	4	1	104	-100
3	4	2	158	-170
3	4	3	62	-54
3	4	4	74	-72
3	4	5	129	-132
3	4	6	100	-88
3	4	7	38	48
3	4	8	-27	-29
3	4	9	-27	-17
3	4	10	-28	6
3	4	11	-28	-11
3	4	12	47	57
3	4	13	-29	-8
3	4	14	-29	34
3	4	15	-29	24
3	4	16	41	52
3	4	17	-28	31
3	4	18	-28	20
3	4	19	-27	27
3	4	20	-26	-29
3	4	21	-25	51
3	4	22	41	49
4	0	-24	71	81
4	0	-22	-46	61
4	0	-20	-44	44
4	0	-18	64	67
4	0	-16	57	71
4	0	-14	131	133
4	0	-12	714	743
4	0	-10	394	-396
4	0	-8	190	-194
4	0	-6	133	-136
4	0	-4	70	-57
4	0	-2	127	-124
4	0	2	124	-117
4	0	4	106	-80
4	0	6	195	-185
4	0	8	-35	13
4	0	10	94	-96
4	0	12	119	119
4	0	14	64	73
4	0	16	359	391
4	0	18	522	-555
4	0	20	-49	-44
4	0	22	62	-53
4	1	-26	218	220
4	1	-25	-63	30
4	1	-24	-63	-11
4	1	-23	-62	45
4	1	-22	-61	-32
4	1	-21	-59	-53
4	1	-20	-58	50
4	1	-19	-57	-5
4	1	-18	-55	-11
4	1	-17	144	-121
4	1	-16	-52	42
4	1	-15	-50	-20
4	1	-14	66	-56
4	1	-13	222	-187
4	1	-12	76	91
4	1	-11	94	57
4	1	-10	67	-67
4	1	-9	191	-180
4	1	-8	74	-52
4	1	-7	80	77
4	1	-6	202	-189
4	1	-5	117	-118
4	1	-4	168	-132
4	1	-3	164	166
4	1	-2	223	-217
4	1	-1	62	-50
4	1	0	91	-56
4	1	1	211	197
4	1	2	122	132
4	1	3	68	-50
4	1	4	321	-286
4	1	5	233	219
4	1	6	118	-104
4	1	7	-46	26
4	1	8	58	65
4	1	9	152	135
4	1	10	68	64
4	1	11	-52	2
4	1	12	124	122
4	1	13	-55	56
4	1	14	85	80
4	1	15	-58	-15
4	1	16	-59	21
4	1	17	-61	-2
4	1	18	247	250
4	2	-25	210	-180
4	2	-24	130	-108
4	2	-23	-71	-4
4	2	-22	-71	-35
4	2	-21	-70	25
4	2	-20	-69	-60
4	2	-19	-67	46
4	2	-18	-66	39
4	2	-17	-64	62
4	2	-16	-63	13
4	2	-15	72	73
4	2	-14	126	103
4	2	-13	-58	50
4	2	-12	223	-189
4	2	-11	-55	4
4	2	-10	326	285
4	2	-9	-53	30
4	2	-8	139	108
4	2	-7	141	101
4	2	-6	-50	70
4	2	-5	174	151
4	2	-4	-49	3
4	2	-3	175	143
4	2	-2	68	-45
4	2	-1	226	209
4	2	0	-49	20
4	2	1	238	217
4	2	2	-51	-33
4	2	3	534	-471
4	2	4	103	-83
4	2	5	130	108
4	2	6	147	-124
4	2	7	169	141
4	2	8	-58	28
4	2	9	-60	-7
4	2	10	-61	-11
4	2	11	-63	56
4	2	12	101	80
4	2	13	-66	-46
4	2	14	-67	-28
4	2	15	-69	-17
4	2	16	-70	-35
4	2	17	133	-122
4	2	18	177	180
4	3	-27	101	120
4	3	-26	-66	9
4	3	-25	-68	1
4	3	-24	-69	24
4	3	-23	-70	34
4	3	-22	-70	-33
4	3	-21	-70	36
4	3	-20	-70	-63
4	3	-19	-70	89
4	3	-18	133	-115
4	3	-17	-68	41
4	3	-16	89	-74
4	3	-15	91	103
4	3	-14	-65	-64
4	3	-13	99	97
4	3	-12	-62	46
4	3	-11	229	-211
4	3	-10	84	-59
4	3	-9	-59	-42
4	3	-8	-58	17
4	3	-7	-57	74
4	3	-6	-56	12
4	3	-5	72	-80
4	3	-4	120	105
4	3	-3	88	81
4	3	-2	-55	12
4	3	-1	-55	-45
4	3	0	76	68
4	3	1	97	103
4	3	2	100	-73
4	3	3	-58	1
4	3	4	-59	11
4	3	5	-60	-19
4	3	6	93	-83
4	3	7	164	-141
4	3	8	81	-60
4	3	9	-65	-42
4	3	10	131	-114
4	3	11	109	-101
4	4	-27	69	-76
4	4	-26	-22	-12
4	4	-25	91	99
4	4	-24	-25	15
4	4	-23	-26	7
4	4	-22	-27	47
4	4	-21	-27	20
4	4	-20	-28	-6
4	4	-19	-28	45
4	4	-18	-29	-15
4	4	-17	45	57
4	4	-16	63	-72
4	4	-15	51	72
4	4	-14	-29	-46
4	4	-13	58	70
4	4	-12	144	-148
4	4	-11	63	-52
4	4	-10	-28	4
4	4	-9	-27	-18
4	4	-8	-27	-35
4	4	-7	107	-98
4	4	-6	-26	1
4	4	-5	54	-64
4	4	-4	-26	9
4	4	-3	115	-117
4	4	-2	-26	42
4	4	-1	37	-45
4	4	0	-26	-6
4	4	1	160	-150
4	4	2	37	-37
4	4	3	353	400
4	4	4	172	205
4	4	5	56	-52
4	4	6	118	132
4	4	7	93	92
4	4	8	70	74
4	4	9	-29	61
4	4	10	-29	8
4	4	11	80	86
4	4	12	-29	32
4	4	13	-29	14
4	4	14	-28	-19
4	4	15	-28	22
4	4	16	45	-34
4	4	17	78	-61
5	0	-30	86	90
5	0	-28	184	-194
5	0	-26	-49	-49
5	0	-24	-49	-10
5	0	-22	-48	-17
5	0	-20	-46	-16
5	0	-18	-44	32
5	0	-16	-42	30
5	0	-14	-40	-1
5	0	-12	102	-85
5	0	-10	-37	12
5	0	-8	154	-147
5	0	-6	-35	-19
5	0	-4	170	-173
5	0	-2	-35	18
5	0	0	455	-473
5	0	2	125	122
5	0	4	-37	-24
5	0	6	126	118
5	0	8	-41	5
5	0	10	113	111
5	0	12	-45	33
5	0	14	240	253
5	0	16	138	-125
5	0	18	-49	-42
5	0	20	-49	-37
5	0	22	-48	13
5	0	24	-45	36
5	0	26	-41	59
5	0	28	47	63
5	1	-30	74	93
5	1	-29	-60	-34
5	1	-28	-62	-65
5	1	-27	-63	34
5	1	-26	-63	2
5	1	-25	-64	28
5	1	-24	-64	-59
5	1	-23	-63	-23
5	1	-22	84	-78
5	1	-21	-62	10
5	1	-20	151	-147
5	1	-19	-60	-31
5	1	-18	174	-165
5	1	-17	-57	18
5	1	-16	273	-255
5	1	-15	-55	-20
5	1	-14	369	338
5	1	-13	115	119
5	1	-12	89	-60
5	1	-11	145	128
5	1	-10	-50	-12
5	1	-9	173	147
5	1	-8	-48	58
5	1	-7	106	98
5	1	-6	148	132
5	1	-5	82	78
5	1	-4	197	177
5	1	-3	-47	-3
5	1	-2	180	164
5	1	-1	245	-211
5	1	0	152	140
5	1	1	303	274
5	1	2	76	72
5	1	3	-49	17
5	1	4	-50	13
5	1	5	-51	-19
5	1	6	-52	-31
5	1	7	93	-69
5	1	8	71	-61
5	1	9	-55	-18
5	1	10	-57	-67
5	1	11	97	-71
5	1	12	-59	-2
5	1	13	-60	16
5	1	14	393	376
5	1	15	-62	49
5	1	16	175	-159
5	1	17	-63	60
5	1	18	-64	-70
5	1	19	-64	-25
5	1	20	-63	6
5	1	21	-63	13
5	1	22	-61	-21
5	1	23	-60	-46
5	1	24	-58	6
5	1	25	-55	-16
5	1	26	-52	-18
5	1	27	62	-67
5	2	-25	85	89
5	2	-24	-71	22
5	2	-23	97	102
5	2	-22	-72	89
5	2	-21	-71	57
5	2	-20	-71	33
5	2	-19	-70	66
5	2	-18	-69	74
5	2	-17	-68	-4
5	2	-16	82	-85
5	2	-15	160	-134
5	2	-14	-65	-28
5	2	-13	117	106
5	2	-12	131	-121
5	2	-11	-61	63
5	2	-10	-60	-21
5	2	-9	-60	-36
5	2	-8	166	-154
5	2	-7	-58	28
5	2	-6	-58	-9
5	2	-5	78	-61
5	2	-4	132	-114
5	2	-3	-57	-9
5	2	-2	-57	69
5	2	-1	121	-118
5	2	0	239	233
5	2	1	90	-66
5	2	2	135	108
5	2	3	96	-87
5	2	4	-61	-25
5	2	5	-62	-13
5	2	6	114	108
5	2	7	80	-70
5	2	8	-65	-35
5	2	9	-66	-10
5	2	10	-67	32
5	2	11	-68	-71
5	2	12	97	-77
5	2	13	-70	-29
5	2	14	106	-91
5	3	-29	108	116
5	3	-28	-57	-11
5	3	-27	-60	-71
5	3	-26	-63	-42
5	3	-25	-65	0
5	3	-24	-67	-40
5	3	-23	-68	-23
5	3	-22	-69	-15
5	3	-21	-70	13
5	3	-20	-70	22
5	3	-19	-70	-24
5	3	-18	-70	63
5	3	-17	-70	15
5	3	-16	106	100
5	3	-15	-69	-90
5	3	-14	-68	42
5	3	-13	-68	-7
5	3	-12	-67	68
5	3	-11	102	-101
5	3	-10	-65	3
5	3	-9	-65	-5
5	3	-8	-64	-35
5	3	-7	-64	-51
5	3	-6	124	-101
5	3	-5	-63	24
5	3	-4	125	-118
5	3	-3	-63	-3
5	3	-2	100	-87
5	3	-1	294	269
5	3	0	76	84
5	3	1	340	-304
5	3	2	-64	-37
5	3	3	-65	-31
5	3	4	-66	20
5	3	5	-66	20
5	3	6	-67	31
5	3	7	-68	4
5	3	8	137	132
5	3	9	-69	48
5	3	10	90	85
5	3	11	-70	30
5	3	12	124	113
5	4	-17	60	-61
5	4	-16	-29	-3
5	4	-15	108	118
5	4	-14	125	129
5	4	-13	162	-170
5	4	-12	98	106
5	4	-11	-29	-10
5	4	-10	72	83
5	4	-9	-29	-5
5	4	-8	59	66
5	4	-7	-28	51
5	4	-6	77	80
5	4	-5	-28	18
5	4	-4	-28	4
5	4	-3	86	86
5	4	-2	-28	27
5	4	-1	-28	50
5	4	0	-28	-4
5	4	1	142	153
5	4	2	-29	-15
5	4	3	-29	23
5	4	4	-29	-40
5	4	5	-29	3
5	4	6	-29	3
5	4	7	66	-63
5	4	8	64	-58
5	4	9	-29	-38
5	4	10	-28	0
5	4	11	56	-57
5	4	12	-28	-30
5	4	13	-27	-3
5	4	14	-26	9
5	4	15	69	-74
6	0	-32	55	-86
6	0	-30	-43	17
6	0	-28	-46	-1
6	0	-26	-48	3
6	0	-24	-49	7
6	0	-22	-49	0
6	0	-20	-48	42
6	0	-18	211	225
6	0	-16	236	-243
6	0	-14	132	-129
6	0	-12	-43	-10
6	0	-10	-42	-32
6	0	-8	-41	47
6	0	-6	-41	24
6	0	-4	91	81
6	0	-2	-41	35
6	0	0	59	64
6	0	2	110	118
6	0	4	101	107
6	0	6	150	148
6	0	8	123	132
6	0	10	217	226
6	0	12	413	-420
6	1	-32	62	81
6	1	-31	-51	28
6	1	-30	91	-92
6	1	-29	-57	20
6	1	-28	-59	-65
6	1	-27	-60	-1
6	1	-26	-62	12
6	1	-25	-63	-2
6	1	-24	-63	-2
6	1	-23	-64	4
6	1	-22	-64	62
6	1	-21	-64	23
6	1	-20	-63	47
6	1	-19	-63	13
6	1	-18	162	179
6	1	-17	161	155
6	1	-16	139	122
6	1	-15	-60	9
6	1	-14	127	127
6	1	-13	-58	36
6	1	-12	68	74
6	1	-11	-57	-53
6	1	-10	-56	87
6	1	-9	-55	-6
6	1	-8	-55	14
6	1	-7	158	-131
6	1	-6	-54	40
6	1	-5	64	-35
6	1	-4	72	77
6	1	-3	138	-139
6	1	-2	285	-282
6	1	-1	-54	7
6	1	0	137	-128
6	1	1	105	-83
6	1	2	-56	-29
6	1	3	-56	51
6	1	4	-57	-51
6	1	5	-58	-32
6	1	6	-58	10
6	1	7	79	68
6	1	8	-60	-13
6	1	9	-61	-11
6	1	10	-62	18
6	1	11	99	91
6	1	12	151	147
6	2	-18	204	-179
6	2	-17	-71	-66
6	2	-16	-70	93
6	2	-15	-70	-75
6	2	-14	-69	-21
6	2	-13	-68	18
6	2	-12	-68	52
6	2	-11	-67	13
6	2	-10	-67	21
6	2	-9	-66	49
6	2	-8	-66	108
6	2	-7	-65	26
6	2	-6	99	88
6	2	-5	-65	35
6	2	-4	156	140
6	2	-3	351	-325
6	2	-2	-65	-13
6	2	-1	92	73
6	2	0	-65	6
6	2	1	-66	43
6	2	2	80	-59
6	2	3	-67	-2
6	2	4	-68	-48
6	2	5	-68	19
6	2	6	129	-99
6	2	7	-69	12
6	2	8	107	-93
6	2	9	-71	31
6	2	10	124	-113
6	2	11	-71	-25
6	2	12	160	148
6	2	13	127	125
6	2	14	-71	20
6	2	15	95	82
6	3	-17	231	-206
6	3	-16	-70	-68
6	3	-15	-70	30
6	3	-14	-70	-67
6	3	-13	-70	44
6	3	-12	-70	-54
6	3	-11	-70	-9
6	3	-10	-69	-4
6	3	-9	-69	57
6	3	-8	-69	-18
6	3	-7	-69	31
6	3	-6	-69	60
6	3	-5	98	108
6	3	-4	-68	11
6	3	-3	-68	24
6	3	-2	193	181
6	3	-1	-69	41
6	3	0	66	91
6	3	1	-69	-6
6	3	2	110	97
6	3	3	-70	70
6	3	4	-70	20
6	3	5	-70	1
6	3	6	-70	34
6	3	7	-70	66
6	3	8	-70	-41
6	3	9	-70	-26
6	3	10	-70	-27
6	3	11	-69	-60
6	3	12	134	-121
6	4	-16	75	84
6	4	-15	-27	43
6	4	-14	-28	9
6	4	-13	-28	29
6	4	-12	-28	-7
6	4	-11	-28	25
6	4	-10	50	-57
6	4	-9	-29	0
6	4	-8	45	-50
6	4	-7	-29	-17
6	4	-6	110	-123
6	4	-5	75	-74
6	4	-4	-29	-94
6	4	-3	143	176
6	4	-2	-29	36
6	4	-1	130	-125
6	4	0	-29	26
6	4	1	45	-48
6	4	2	-29	-24
6	4	3	-29	-24
6	4	4	-28	4
6	4	5	-28	-6
6	4	6	-28	16
6	4	7	-28	1
6	4	8	-27	24
6	4	9	-27	13
6	4	10	40	-12
6	4	11	-25	12
6	4	12	82	66
7	0	-16	74	88
7	0	-14	97	114
7	0	-12	125	130
7	0	-10	89	93
7	0	-8	72	80
7	0	-6	217	-202
7	0	-4	96	105
7	0	-2	-46	-31
7	0	0	-46	-14
7	0	2	97	-97
7	0	4	-47	-9
7	0	6	66	-45
7	0	8	93	82
7	0	10	130	-123
7	1	-20	127	127
7	1	-19	-64	0
7	1	-18	111	-105
7	1	-17	-64	-32
7	1	-16	104	-86
7	1	-15	-63	-39
7	1	-14	82	-74
7	1	-13	101	-78
7	1	-12	-62	-37
7	1	-11	-62	-52
7	1	-10	-61	-14
7	1	-9	88	-87
7	1	-8	-61	12
7	1	-7	82	-76
7	1	-6	-60	-7
7	1	-5	170	157
7	1	-4	-60	6
7	1	-3	108	91
7	1	-2	-60	-40
7	1	-1	-61	27
7	1	0	-61	-11
7	1	1	-61	63
7	1	2	-61	-39
7	1	3	-62	22
7	1	4	-62	-2
7	1	5	-63	22
7	1	6	-63	21
7	1	7	-63	-1
7	1	8	241	238
7	1	9	-64	50
7	1	10	153	-144
7	2	-21	88	-93
7	2	-20	-70	7
7	2	-19	147	165
7	2	-18	-71	31
7	2	-17	-71	90
7	2	-16	-72	39
7	2	-15	-72	39
7	2	-14	-72	-5
7	2	-13	-71	65
7	2	-12	-71	-2
7	2	-11	-71	12
7	2	-10	-71	-42
7	2	-9	-71	13
7	2	-8	-71	-12
7	2	-7	-70	-54
7	2	-6	131	132
7	2	-5	-70	11
7	2	-4	93	-71
7	2	-3	-70	12
7	2	-2	90	-78
7	2	-1	-70	54
7	2	0	-71	25
7	2	1	-71	26
7	2	2	-71	-21
7	2	3	-71	74
7	2	4	-71	53
7	2	5	-72	16
7	2	6	-72	-8
7	2	7	-72	30
7	2	8	-71	29
7	2	9	140	101
7	2	10	-71	63
7	2	11	-70	7
7	2	12	90	92
7	3	-7	192	181
7	3	-6	-70	78
7	3	-5	221	-206
7	4	-16	50	-56
8	0	-24	97	106
8	0	-22	-46	-23
8	0	-20	-47	38
8	0	-18	-48	37
8	0	-16	-49	-10
8	0	-14	-49	-42
8	0	-12	112	-114
8	0	-10	128	-139
8	0	-8	170	-160
8	0	-6	101	-107
8	0	-4	-49	-29
8	0	-2	-49	37
8	0	0	84	91
8	0	2	133	135
8	0	4	127	140
8	0	6	181	-180
8	1	-12	89	100
8	1	-11	-64	61
8	1	-10	113	119
8	1	-9	-64	73
8	1	-8	192	-186
8	1	-7	-64	68
8	1	-6	-64	-34
8	1	-5	-64	20
8	1	-4	-64	-2
8	1	-3	-64	23
8	1	-2	-64	-35
8	1	-1	-64	-14
8	1	0	-64	-7
8	1	1	-64	-5
8	1	2	-64	-2
8	1	3	-64	-32
8	1	4	-64	48
8	1	5	-63	24
8	1	6	113	107
8	2	-9	191	-163
8	2	-8	-71	0
8	2	-7	-71	96
8	2	-6	-71	61
8	2	-5	-71	52
8	2	-4	-71	0
8	2	-3	-71	19
8	2	-2	-71	33
8	2	-1	-71	3
8	2	0	-71	-44
8	2	1	-71	-13
8	2	2	-71	-21
8	2	3	-70	-40
8	2	4	84	-81
8	2	5	-69	-68
8	2	6	89	93
8	3	7	71	84
8	4	-8	102	59
9	0	-12	127	-140
9	0	-10	154	160
9	0	-8	113	121
9	0	-6	74	21
9	0	-4	71	85
9	0	-2	-48	75
9	0	2	-47	21
9	0	4	93	-87
9	1	2	174	170
9	1	3	-59	64
9	1	4	71	-77
9	2	3	99	89
10	0	-14	59	23
10	0	-12	-41	9
10	0	-10	-42	24
10	0	-8	-42	-4
10	0	-6	-42	4
10	0	-4	-42	-2
10	0	-2	-42	-1
10	0	0	134	-136
10	1	-14	139	-114

E map, computed with the signed normalized structure factors, clearly indicated the twelve carbon atoms of the straight part of the chain. Structure factors based on these positions resulted in an R index of 0·61. Four cycles of block-diagonal least squares ($w=1$ for $|F_{\text{obs}}| < 20$, $w=20/|F_{\text{obs}}|$ for $|F_{\text{obs}}| > 20$) reduced this to 0·47. The positions of the carbon atom C(1) and the keto oxygen atom O(3) were then fixed from chemical and packing considerations. A difference synthesis indicated the positions of the remaining two oxygen atoms of the carboxyl group. The inclusion of these four atoms reduced R to 0·39. After a number of block-diagonal least-squares cycles the residual decreased to 0·17. Full-matrix refinement with anisotropic thermal parameters was then commenced. At $R=0·11$ the 17 hydrogen atoms of the methylene groups were included at their expected tetrahedral positions. Difference syntheses calculated in the plane of the two methyl groups and the carboxyl group indicated the positions of the remaining hydrogen atoms. All hydrogen atoms were included in the structure factor calculations with isotropic temperature factors $B=4·0$ Å^2, but their parameters were not varied in the least-squares procedure. After several more cycles none of the shifts exceeded $0·1\sigma$ and the refinement was terminated. The final R index was 0·088. Observed and calculated structure factors are listed in Table 1, final positional and thermal parameters in Tables 2 and 3. Atomic scattering factors for carbon and oxygen were taken from *International Tables for X-ray Crystallography* (1962). For hydrogen, the values given by Stewart, Davidson & Simpson (1965) were used.

Table 2. *Atomic coordinates and their standard deviations* ($\times 10^4$)

	x/a	y/b	z/c
C(1)	0·3662 (10)	0·5812 (17)	0·0410 (3)
C(2)	0·2601 (9)	0·6479 (15)	0·0705 (3)
C(3)	0·3357 (9)	0·8049 (16)	0·1105 (3)
C(4)	0·4609 (9)	0·6579 (15)	0·1407 (3)
C(5)	0·5339 (9)	0·8194 (15)	0·1803 (3)
C(6)	0·6578 (9)	0·6670 (15)	0·2097 (3)
C(7)	0·7396 (10)	0·8128 (19)	0·2487 (3)
C(8)	0·8634 (9)	0·6699 (15)	0·2782 (3)
C(9)	0·9308 (9)	0·8079 (15)	0·3203 (3)
C(10)	1·0592 (10)	0·6578 (16)	0·3477 (3)
C(11)	1·1265 (10)	0·7921 (18)	0·3902 (3)
C(12)	1·2524 (11)	0·6389 (20)	0·4174 (3)
C(13)	0·1262 (9)	0·7964 (18)	0·0436 (3)
O(1)	0·4706 (7)	0·7278 (11)	0·0359 (2)
O(2)	0·3383 (7)	0·3619 (11)	0·0179 (2)
O(3)	0·7036 (8)	1·0391 (12)	0·2577 (2)
H(1)	0·425	0·354	−0·001
H(21)	0·219	0·479	0·082
H(31)	0·262	0·857	0·129
H(32)	0·379	0·972	0·100
H(41)	0·540	0·615	0·122
H(42)	0·422	0·492	0·150
H(51)	0·460	0·868	0·198
H(52)	0·580	0·986	0·170
H(61)	0·732	0·610	0·190
H(62)	0·613	0·504	0·219
H(81)	0·948	0·632	0·261
H(82)	0·826	0·493	0·286
H(91)	0·853	0·836	0·338
H(92)	0·972	0·984	0·312
H(101)	1·139	0·630	0·329
H(102)	1·020	0·477	0·354
H(111)	1·046	0·813	0·408
H(112)	1·163	0·969	0·383
H(121)	1·348	0·611	0·400
H(122)	1·301	0·741	0·448
H(123)	1·218	0·444	0·428
H(131)	0·038	0·844	0·062
H(132)	0·075	0·683	0·015
H(133)	0·168	0·980	0·031

Discussion

The molecular geometry and atom numbering are illustrated in Fig. 1. The hydrocarbon chain is bent at C(2) and the branching methyl group forms a continuation of the zigzag backbone. Configurations of this type are common in methyl branched fatty acids (Abrahamsson, 1959*a*). The equation of the best plane through C(2) to C(12) and the perpendicular distances of the atoms from it are given in Table 4. There are significant out

Table 3. *Anisotropic thermal parameters in the form*
$\exp\,[-2\pi^2(h^2a^{*2}U_{11}+k^2b^{*2}U_{22}+l^2c^{*2}U_{33}+2hka^*b^*U_{12}+2klb^*c^*U_{23}+2hla^*c^*U_{13})]$

Standard deviations are given in parentheses. All values have been multiplied by 10^4.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{31}	U_{12}
C(1)	506 (54)	340 (48)	243 (46)	57 (40)	−14 (40)	15 (46)
C(2)	495 (56)	295 (48)	468 (56)	−13 (40)	96 (50)	4 (41)
C(3)	494 (55)	352 (46)	471 (54)	−62 (44)	93 (46)	22 (43)
C(4)	522 (56)	336 (49)	409 (54)	32 (41)	51 (48)	68 (43)
C(5)	549 (60)	336 (47)	401 (54)	−10 (44)	80 (48)	25 (44)
C(6)	658 (63)	276 (47)	370 (52)	7 (42)	6 (51)	31 (43)
C(7)	602 (63)	350 (54)	481 (60)	70 (46)	181 (51)	−50 (47)
C(8)	542 (58)	279 (47)	532 (59)	−30 (43)	12 (49)	73 (45)
C(9)	599 (59)	307 (46)	439 (55)	−28 (43)	72 (48)	−26 (45)
C(10)	544 (57)	324 (50)	553 (61)	−99 (46)	−5 (53)	38 (45)
C(11)	617 (61)	486 (55)	572 (62)	−55 (53)	−51 (53)	−59 (53)
C(12)	693 (69)	767 (73)	573 (65)	−121 (57)	−16 (58)	19 (60)
C(13)	379 (50)	566 (57)	655 (62)	19 (52)	−65 (47)	36 (49)
O(1)	641 (39)	427 (37)	554 (41)	−193 (31)	228 (33)	−174 (37)
O(2)	778 (50)	407 (38)	678 (43)	−276 (32)	344 (37)	−248 (33)
O(3)	990 (56)	259 (37)	689 (47)	−163 (33)	−109 (39)	229 (36)

of plane deviations around the keto group and towards the ends of the molecule which arise from a slight twist of the chain at the keto group. Atoms C(2) to C(6) are planar within experimental error as are C(8) to C(12). The angle between these two planar groups is 7·2°. This indicates the degree of distortion of the chain geometry due to the inclusion of the keto group.

The best plane through the carboxyl group is

$$0{\cdot}2058X-0{\cdot}1224Y+0{\cdot}9709Z-0{\cdot}0430=0$$

and none of the atoms deviate significantly from it. The methyl groups are oriented for minimum hindrance with their nearest neighbours. In the case of C(12) the methyl hydrogen atoms are staggered with respect to H(111) and H(112) and for C(13) they are staggered with respect to C(1) and H(21).

The bond lengths and angles are listed in Tables 5 and 6 and shown diagrammatically in Figs. 2 and 3. The mean C–C distance within the chain is 1·511 Å, *i.e.* about 0·03 Å shorter than the normal sp^3–sp^3 carbon bond. The mean C–C–C angle within the chain is 114·2°. Systematically short bonds and large bond angles in the hydrocarbon chains of β-tricaprin (Jensen & Mabis, 1966) have been explained in terms of thermal anisotropy and rotary oscillations of the chains about their long axes. The short C–C bonds and large angles in the present case are probably due to the same effects. There is a tendency for the bonds closer to the carboxyl group to be longer than those towards the end methyl group. The mean C–C distance between C(13) and C(6) is 1·523 Å while between C(6) and C(12) the mean distance is 1·501 Å. These differences are consistent with the general increase in thermal anisotropy away from the carboxyl group. There appears to be some shortening of the bonds adjacent to the keto oxygen, but because of the uncertainty of the thermal corrections it is not possible to say if this is a real effect. The geometry of the carboxyl group is normal and the bond lengths and angles fall within the range of those surveyed by Nardelli, Fava & Giraldi (1962).

Table 4. *Equation of the best plane through atoms* C(2) *to* C(12) *and deviations from it*

The equation is expressed in terms of the crystal axes.

$$0{\cdot}3299X+0{\cdot}0918\,Y-0{\cdot}9395Z-0{\cdot}0813=0.$$

Atom	Deviation
C(2)	−0·048 Å
C(3)	−0·010
C(4)	−0·021
C(5)	0·014
C(6)	−0·001
C(7)	0·078
C(8)	0·077
C(9)	−0·020
C(10)	0·039
C(11)	−0·076
C(12)	−0·033
C(13)	−0·160*
O(3)	0·086*

* Not included in the plane calculation.

Table 5. *Bond lengths and* (*in parentheses*) *their estimated standard deviations*

The standard deviations have been multiplied by 10^3.

C(1)—C(2)	1·485 (13) Å
C(1)—O(1)	1·240 (11)
C(1)—O(2)	1·317 (10)
C(2)—C(3)	1·518 (11)
C(2)—C(13)	1·538 (11)
C(3)—C(4)	1·526 (11)
C(4)—C(5)	1·515 (11)
C(5)—C(6)	1·520 (11)
C(6)—C(7)	1·487 (11)
C(7)—C(8)	1·498 (11)
C(7)—O(3)	1·237 (11)
C(8)—C(9)	1·499 (11)
C(9)—C(10)	1·515 (11)
C(10)-C(11)	1·501 (12)
C(11)-C(12)	1·504 (13)

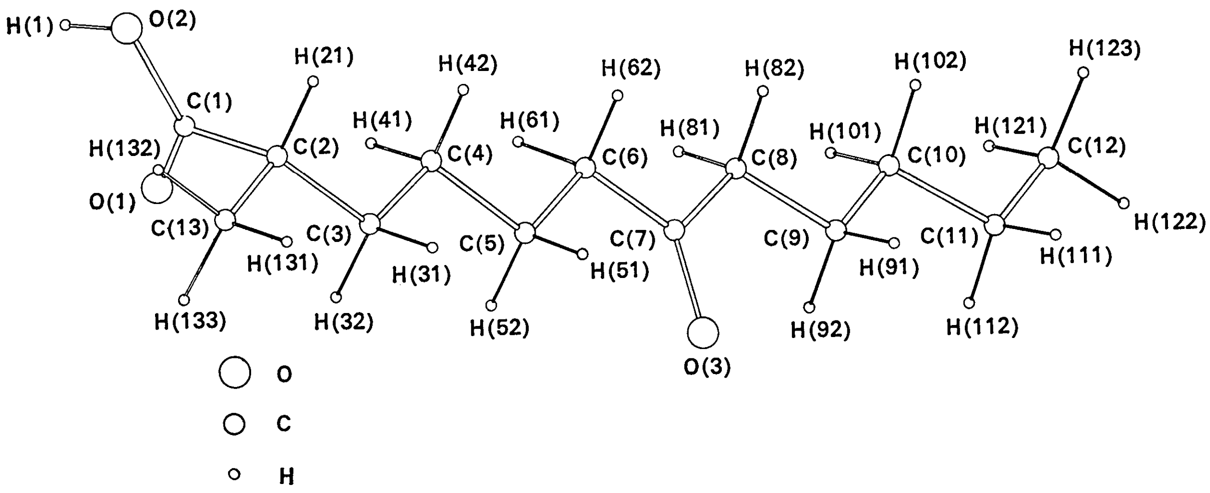

Fig. 1. Molecular geometry and atom numbering.

The packing arrangement is shown in Fig. 4. The molecules are joined by hydrogen bonds between carboxyl groups related across centres of symmetry. The O···O and H···O distances are 2·66 and 1·62 Å respectively. The perpendicular distance between the hydrogen bonded carboxyl groups is 0·10 Å (Jeffrey, 1963). The straight portions of the hydrocarbon chains are arranged with the orthorhombic subcell packing $O'\perp$, which has previously been reported in D-2-methyloctadecanoic acid (Abrahamsson, 1959*b*). The idealized subcell has dimensions $a_s=7{\cdot}57$, $b_s=2{\cdot}53$, $c_s=5{\cdot}06$ Å (Fig. 5). The angle of the chains to the a_sc_s plane is 67·5° (as compared with 59° in D-2-methyloctadecanoic acid) and the angle of the carbon chains to the end group planes $[O'\perp(201)]$ is 58°. The keto group is apparently accommodated in the packing without disturbing the chain arrangement. The oxygen atom packs between the two hydrocarbon chains at $(x,y,1-z)$ and $(1-x,y-\frac{1}{2},\frac{1}{2}-z)$. The shortest intermolecular O(3)–C contacts are 3·45, 3·50 and 3·52 Å to C(5), C(6) and C(8) respectively. The shortest O(3)–H contacts are 2·76, 2·69 and 2·63 Å to H(51), H(62) and H(82) respectively.

The average length of the hydrogen–hydrogen contacts in the subcell region is 2·79 Å. There is a considerable spread in the individual values (~0·2 Å) owing to distortions in the idealized subcell.

There are no layers of weak van der Waals forces in the structure since alternate hydrocarbon chains are arranged head to tail. This explains why the crystals grow as hard needles and not as soft, thin plates as is common with many long chain fatty acids.

Table 6. *Bond angles and (in parentheses) their standard deviations*

C(2)—C(1)—O(1)	123·0 (0·7)°
C(2)—C(1)—O(2)	116·1 (0·7)
O(1)—C(1)—O(2)	120·8 (0·8)
C(1)—C(2)—C(3)	111·5 (0·7)
C(1)—C(2)—C(13)	108·9 (0·7)
C(3)—C(2)—C(13)	112·1 (0·7)
C(2)—C(3)—C(4)	114·5 (0·6)
C(3)—C(4)—C(5)	113·3 (0·6)
C(4)—C(5)—C(6)	111·9 (0·6)
C(5)—C(6)—C(7)	115·8 (0·7)
C(6)—C(7)—C(8)	117·3 (0·7)
C(6)—C(7)—O(3)	121·8 (0·7)
C(8)—C(7)—O(3)	120·8 (0·7)
C(7)—C(8)—C(9)	116·2 (0·7)
C(8)—C(9)—C(10)	113·6 (0·7)
C(9)—C(10)-C(11)	114·1 (0·8)
C(10)-C(11)-C(12)	113·4 (0·8)

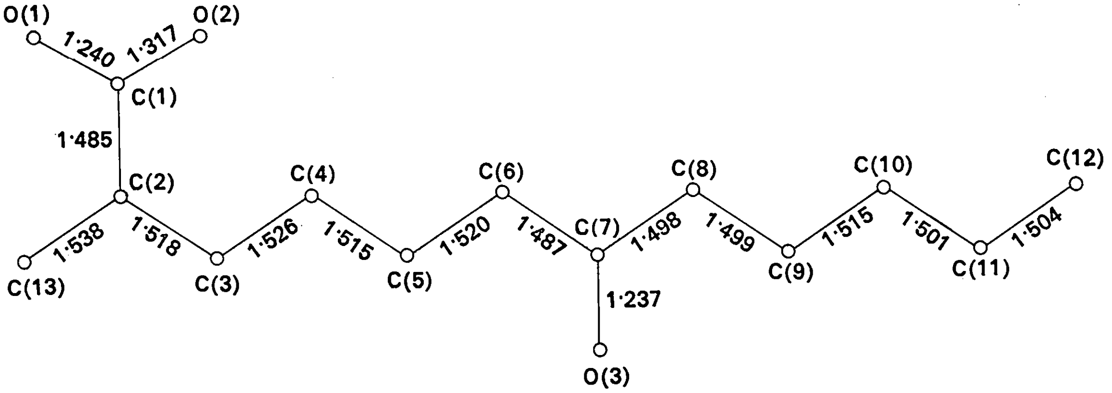

Fig. 2. Intramolecular bond lengths.

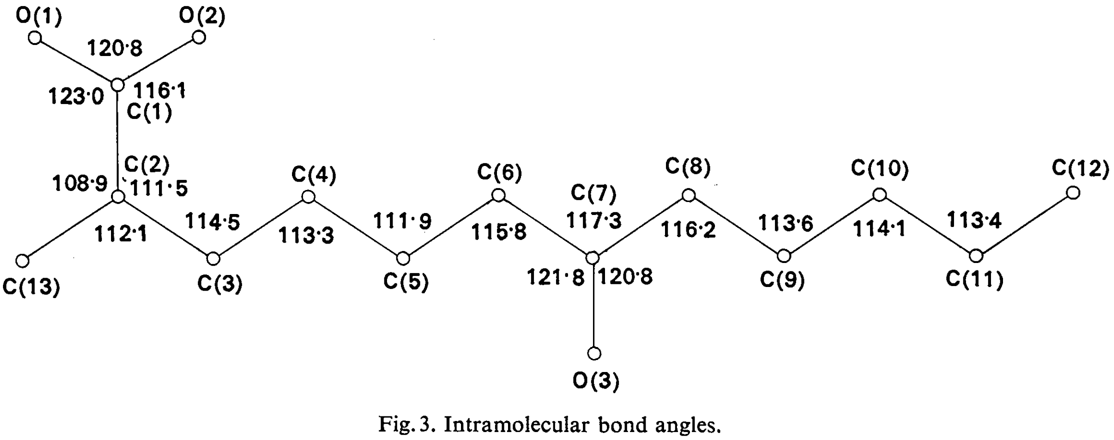

Fig. 3. Intramolecular bond angles.

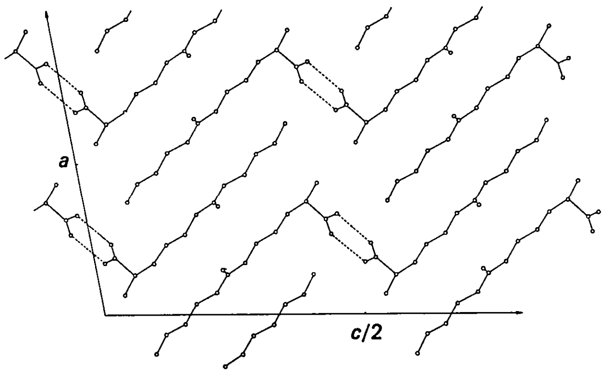

Fig. 4. Molecular packing viewed along the *b* axis. Hydrogen bonds are shown by broken lines.

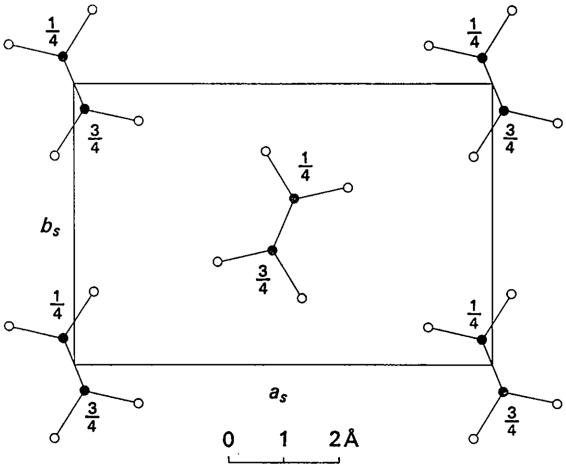

Fig. 5. The idealized subcell $O'\perp$ viewed along $\mathbf{c}_s$.

Crystal samples were kindly provided by Professor Ställberg-Stenhagen. All calculations were performed on a Datasaab D21 computer. The author wishes to thank the National Research Council, Ottawa, Canada, for supplying the symbolic addition program NRC-4 and Dr S. R. Hall for advice on its adaption and operation in our program system. The remaining calculations were made with programs written by Abrahamsson, Aleby, Nilsson, Larsson, Selin & Westerdahl (1965) and additional routines written by the author. Financial support was obtained from the Swedish Medical Research Council, the Swedish National Science Research Council and the U.S. Public Health Service (GM-11653).

References

ABRAHAMSSON, S. (1959*a*). *Ark. Kemi*, **14**, 65.

ABRAHAMSSON, S. (1959*b*). *Acta Cryst.* **12**, 301.

ABRAHAMSSON, S. (1966). *J. Sci. Instrum.* **43**, 931.

ABRAHAMSSON, S., ALEBY, S., LARSSON, K., NILSSON, B., SELIN, K. & WESTERDAHL, A. (1965). *Acta Chem. Scand.* **19**, 758.

ABRAHAMSSON, S. & HARDING, M. (1966). *Acta Cryst.* **20**, 377.

DUNNING, A. J. & VAND, V. (1966). Research Report PPS660105 (CP/P). Unilever Laboratory, Port Sunlight, England.

GRENVILLE-WELLS, H. J. (1955). *Acta Cryst.* **8**, 512.

HAMILTON, W. C., ROLLETT, J. S. & SPARKS, R. A. (1965). *Acta Cryst.* **18**, 129.

HAUPTMAN, H. & KARLE, J. (1953). *Solution of the Phase Problem.* 1. *The Centrosymmetric Crystal.* A.C.A. Monograph No. 3. Pittsburgh: Polycrystal Book Service.

International Tables for X-ray Crystallography (1962). Vol. III, p. 202. Birmingham: Kynoch Press.

JEFFREY, G. A. (1963). *Acta Cryst.* **16**, 430.

JENSEN, L. H. & MABIS, A. J. (1966). *Acta Cryst.* **21**, 770.

KARLE, J. & KARLE, I. L. (1966). *Acta Cryst.* **21**, 849.

MORIMOTO, H. & UYEDA, R. (1963). *Acta Cryst.* **11**, 1107.

NARDELLI, M., FAVA, G. & GIRALDI, G. (1962). *Acta Cryst.* **15**, 737.

O'CONNELL, A. M. (1967). *Acta Cryst.* **23**, 623.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175

STÄLLBERG-STENHAGEN (1946). *Ark. Kemi, Min. Geol.* **23A**, 1.