

The Crystal Structure of 13-Oxoisostearic Acid

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13-Oxoisostearic acid (16-methyl-13-oxoheptadecanoic acid) is triclinic ($P\bar{1}$) with $a=4.933$, $b=5.623$, $c=34.460$ Å, $\alpha=95.65$, $\beta=94.01$ and $\gamma=103.60^\circ$. The molecules are arranged in hydrogen-bonded dimers in which the chain axes are tilted 44° to the end-group planes to allow the methyl branches to be accommodated between the methyl ends of the chains. The keto oxygen is directly incorporated into the chain packing with a resulting strain at the sp^2 carbon atom. This leads to a bend of the chain axis in the zigzag plane of the molecule. The carboxyl group forms an angle of 13° with the chain plane.

Introduction

As a part of crystal structure investigations of compounds with long hydrocarbon chains at this research unit, branched-chain fatty acids have been studied (Abrahamsson, 1959*b*; Abrahamsson & Harding, 1966). The effect of a keto group along the carbon chain in an acid branched close to the carboxyl group (DL-2-methyl-7-oxododecanoic acid) was studied by O'Connell (1968). In connexion with work on the crystal structure of isostearic acid (Abrahamsson & Lundén, 1972) a single-crystal analysis of 13-oxoisostearic acid was performed to see the influence of the keto group on the molecular geometry and packing in the case of ω -branching.

Crystal data

Molecular formula	$C_{18}H_{34}O_3$, 16-methyl-13-oxoheptadecanoic acid
Unit cell:	
Triclinic	$a=4.933$ (2), $b=5.623$ (4), $c=34.460$ (7) Å; $\alpha=95.65$ (5), $\beta=94.01$ (3), $\gamma=103.60$ (5) $^\circ$
V	920.25 Å ³
Molecular weight	298.47
Z	2
D_c	1.077 g.cm ⁻³
D_m	1.071 g.cm ⁻³
λ	1.54051 Å (Cu $K\alpha_1$ radiation)
Space group	$P\bar{1}$

The space group was assumed to be $P\bar{1}$ and this was not contradicted in the structure determination.

Experimental

13-Oxoisostearic acid was synthesized by Stenhagen & Tägtström-Eketorp (1944). The crystals grow from ether as soft thin plates. They have a melting point of 76.9 – 77.2°C .

A single crystal with the dimensions $0.07 \times 0.59 \times 0.24$ mm was mounted along the b axis and data were

collected on a Picker four-circle automatic diffractometer using graphite monochromated Cu $K\alpha$ radiation. The $\theta/2\theta$ scanning mode was used to measure the reflexions at a scanning rate of $1^\circ/\text{min}$ and with 10 sec background counts at the scan limits. 2758 reflexions having 2θ less than 120° were recorded. 458 of these intensities I , with $I \leq 2\sigma(I)$ were considered to be unobservably weak and assigned an intensity of $2\sigma(I)$. The unobserved reflexions were not used in the least-squares refinement but included in the final SF-calculation.

The Lorentz and polarization corrections were applied to all reflexions, but no absorption correction was made as the linear absorption coefficient was small ($\mu=5.63$ cm⁻¹).

Structure determination and refinement

The carbon-chain direction was clearly shown in the three-dimensional sharpened Patterson function. The acid was considered to exist as hydrogen bonded dimers around centres of symmetry, as is usual for higher fatty acids. Therefore the assumption was made that the carbon atoms C(1) and C(2) and their symmetry-related equivalents were all on one line passing through the inversion centre. There were then two possible positions for the carboxyl carbon. The coordinates for the chain carbon atoms were calculated for the two different alternatives and from a calculation of distances and angles one of them seemed to be more plausible.

A structure-factor calculation including the 17 carbon atoms in the chain gave an R value of 0.8 for the 1237 reflexions whose F value exceeded 30. All these atoms appeared in the Fourier map but in addition there were fairly large peaks at a distance of about 1 Å from every odd-numbered carbon atom. The whole chain was therefore moved half the distance towards the 'ghost peaks' and after two cycles of structure-factor and Fourier calculation all carbon and oxygen atoms could be located. The resulting R value was then 0.36.

Table 1. Observed and calculated structure factors ($\times 100$)The unobserved terms, indicated by a minus sign on F_{obs} , are included at the threshold values.

h	k	l	F_{obs}	F_{calc}	h	k	l	F_{obs}	F_{calc}	h	k	l	F_{obs}	F_{calc}	h	k	l	F_{obs}	F_{calc}	
1	0	0	451	466	1	0	0	450	466	1	0	0	114	246	244	1	0	0	114	246
1	0	1	243	228	1	0	1	243	228	1	0	1	115	590	603	1	0	1	115	590
1	0	2	278	281	1	0	2	278	281	1	0	2	116	1102	1105	1	0	2	116	1102
1	0	3	378	386	1	0	3	378	386	1	0	3	117	1700	1700	1	0	3	117	1700
1	0	4	478	472	1	0	4	478	472	1	0	4	118	2292	2291	1	0	4	118	2292
1	0	5	578	572	1	0	5	578	572	1	0	5	119	2880	2880	1	0	5	119	2880
1	0	6	678	672	1	0	6	678	672	1	0	6	120	3468	3468	1	0	6	120	3468
1	0	7	778	772	1	0	7	778	772	1	0	7	121	4056	4056	1	0	7	121	4056
1	0	8	878	872	1	0	8	878	872	1	0	8	122	4644	4644	1	0	8	122	4644
1	0	9	978	972	1	0	9	978	972	1	0	9	123	5232	5232	1	0	9	123	5232
1	0	10	1078	1072	1	0	10	1078	1072	1	0	10	124	5820	5820	1	0	10	124	5820
1	0	11	1178	1172	1	0	11	1178	1172	1	0	11	125	6408	6408	1	0	11	125	6408
1	0	12	1278	1272	1	0	12	1278	1272	1	0	12	126	6996	6996	1	0	12	126	6996
1	0	13	1378	1372	1	0	13	1378	1372	1	0	13	127	7584	7584	1	0	13	127	7584
1	0	14	1478	1472	1	0	14	1478	1472	1	0	14	128	8172	8172	1	0	14	128	8172
1	0	15	1578	1572	1	0	15	1578	1572	1	0	15	129	8760	8760	1	0	15	129	8760
1	0	16	1678	1672	1	0	16	1678	1672	1	0	16	130	9348	9348	1	0	16	130	9348
1	0	17	1778	1772	1	0	17	1778	1772	1	0	17	131	9936	9936	1	0	17	131	9936
1	0	18	1878	1872	1	0	18	1878	1872	1	0	18	132	10524	10524	1	0	18	132	10524
1	0	19	1978	1972	1	0	19	1978	1972	1	0	19	133	11112	11112	1	0	19	133	11112
1	0	20	2078	2072	1	0	20	2078	2072	1	0	20	134	11700	11700	1	0	20	134	11700
1	0	21	2178	2172	1	0	21	2178	2172	1	0	21	135	12288	12288	1	0	21	135	12288
1	0	22	2278	2272	1	0	22	2278	2272	1	0	22	136	12876	12876	1	0	22	136	12876
1	0	23	2378	2372	1	0	23	2378	2372	1	0	23	137	13464	13464	1	0	23	137	13464
1	0	24	2478	2472	1	0	24	2478	2472	1	0	24	138	14052	14052	1	0	24	138	14052
1	0	25	2578	2572	1	0	25	2578	2572	1	0	25	139	14640	14640	1	0	25	139	14640
1	0	26	2678	2672	1	0	26	2678	2672	1	0	26	140	15228	15228	1	0	26	140	15228
1	0	27	2778	2772	1	0	27	2778	2772	1	0	27	141	15816	15816	1	0	27	141	15816
1	0	28	2878	2872	1	0	28	2878	2872	1	0	28	142	16404	16404	1	0	28	142	16404
1	0	29	2978	2972	1	0	29	2978	2972	1	0	29	143	16992	16992	1	0	29	143	16992
1	0	30	3078	3072	1	0	30	3078	3072	1	0	30	144	17580	17580	1	0	30	144	17580
1	0	31	3178	3172	1	0	31	3178	3172	1	0	31	145	18168	18168	1	0	31	145	18168
1	0	32	3278	3272	1	0	32	3278	3272	1	0	32	146	18756	18756	1	0	32	146	18756
1	0	33	3378	3372	1	0	33	3378	3372	1	0	33	147	19344	19344	1	0	33	147	19344
1	0	34	3478	3472	1	0	34	3478	3472	1	0	34	148	19932	19932	1	0	34	148	19932
1	0	35	3578	3572	1	0	35	3578	3572	1	0	35	149	20520	20520	1	0	35	149	20520
1	0	36	3678	3672	1	0	36	3678	3672	1	0	36	150	21108	21108	1	0	36	150	21108
1	0	37	3778	3772	1	0	37	3778	3772	1	0	37	151	21696	21696	1	0	37	151	21696
1	0	38	3878	3872	1	0	38	3878	3872	1	0	38	152	22284	22284	1	0	38	152	22284
1	0	39	3978	3972	1	0	39	3978	3972	1	0	39	153	22872	22872	1	0	39	153	22872
1	0	40	4078	4072	1	0	40	4078	4072	1	0	40	154	23460	23460	1	0	40	154	23460
1	0	41	4178	4172	1	0	41	4178	4172	1	0	41	155	24048	24048	1	0	41	155	24048
1	0	42	4278	4272	1	0	42	4278	4272	1	0	42	156	24636	24636	1	0	42	156	24636
1	0	43	4378	4372	1	0	43	4378	4372	1	0	43	157	25224	25224	1	0	43	157	25224
1	0	44	4478	4472	1	0	44	4478	4472	1	0	44	158	25812	25812	1	0	44	158	25812
1	0	45	4578	4572	1	0	45	4578	4572	1	0	45	159	26400	26400	1	0	45	159	26400
1	0	46	4678	4672	1	0	46	4678	4672	1	0	46	160	26988	26988	1	0	46	160	26988
1	0	47	4778	4772	1	0	47	4778	4772	1	0	47	161	27576	27576	1	0	47	161	27576
1	0	48	4878	4872	1	0	48	4878	4872	1	0	48	162	28164	28164	1	0	48	162	28164
1	0	49	4978	4972	1	0	49	4978	4972	1	0	49	163	28752	28752	1	0	49	163	28752
1	0	50	5078	5072	1	0	50	5078	5072	1	0	50	164	29340	29340	1	0	50	164	29340
1	0	51	5178	5172	1	0	51	5178	5172	1	0	51	165	29928	29928	1	0	51	165	29928
1	0	52	5278	5272	1	0	52	5278	5272	1	0	52	166	30516	30516	1	0	52	166	30516
1	0	53	5378	5372	1	0	53	5378	5372	1	0	53	167	31104	31104	1	0	53	167	31104
1	0	54	5478	5472	1	0	54	5478	5472	1	0	54	168	31692	31692	1	0	54	168	31692
1	0	55	5578	5572	1	0	55	5578	5572	1	0	55	169	32280	32280	1	0	55	169	32280
1	0	56	5678	5672	1	0	56	5678	5672	1	0	56	170	32868	32868	1	0	56	170	32868
1	0	57	5778	5772	1	0	57	5778	5772	1	0	57	171	33456	33456	1	0	57	171	33456
1	0	58	5878	5872	1	0	58	5878	5872	1	0	58	172	34044	34044	1	0	58	172	34044
1	0	59	5978	5972	1	0	59	5978	5972	1	0	59	173	34632	34632	1	0	59	173	34632
1	0	60	6078	6072	1	0	60	6078	6072	1	0	60	174	35220	35220	1	0	60	174	35220
1	0	61	6178	6172	1	0	61	6178	6172	1	0	61	175	35808	35808	1	0	61	175	35808
1	0	62	6278	6272	1	0	62	6278	6272	1	0	62	176	36396	36396	1	0	62	176	36396
1	0	63	6378	6372	1	0	63	6378	6372	1	0	63	177	36984	36984	1	0	63	177	36984
1	0	64	6478	6472	1	0	64	6478	6472	1	0	64	178	37572	37572	1	0	64	178	37572
1	0	65	6578	6572	1	0	65	6578	6572	1	0	65	179	38160	38160	1	0	65	179	38160
1	0	66	6678	6672	1	0	66	6678	6672	1	0	66	180	38748	38748	1	0	66	180	38748
1	0	67	6778	6772	1	0	67	6778	6772	1	0	67	181	39336	39336	1	0	67	181	39336
1	0	68	6878	6872	1	0	68	6878	6872	1	0	68	182	39924	39924	1	0	68	182	39924
1	0	69	6978	6972	1	0	69	6978	6972	1	0	69	183	40512	40512	1	0	69	183	40512
1	0	70	7078	7072	1	0	70	7078	7072	1	0	70	184	41100	41100	1	0	70	184	41100
1	0	71	7178	7172	1	0	71	7178	7172	1	0	71	185	41688	41688	1	0	71	185	41688
1	0	72	7278	7272	1	0	72	7278	7272	1	0	72	186	42276	42276	1	0	72	186	42276
1	0	73	7378	7372	1	0	73	7378	7372	1	0	73	187	42864	42864	1	0			

Table 1 (cont.)

h	k	l	FOMS	FVALC	h	k	l	FOMS	FVALC	h	k	l	FOMS	FVALC	h	k	l	FOMS	FVALC							
1	-22	1734	-1735	340	353	3	3	0	222	240	4	-3	17	170	-211	4	0	-7	187	198						
1	-21	675	-680	3	1	-10	233	-213	3	3	10	-80	46	4	-3	10	122	115	4	0	-7	187	198			
1	-20	886	-894	3	1	-14	130	-84	3	3	11	-88	6	4	-3	10	123	133	4	0	-5	270	293			
1	-19	255	-270	3	1	-12	871	-894	3	3	12	-109	-149	4	-3	10	122	175	4	0	-5	270	293			
1	-18	-84	-75	3	1	-12	807	-809	3	3	13	110	-95	4	-3	11	240	204	4	0	-3	370	-382			
1	-17	1408	-1408	3	1	-10	3515	-3505	3	3	14	109	-103	4	-3	12	106	-106	4	0	-1	270	-375			
1	-16	-82	-20	3	1	-10	3515	-3505	3	3	15	170	-134	4	-3	13	277	-75	4	0	-1	310	-284			
1	-15	177	-177	3	1	-8	1020	-1020	3	3	16	110	-112	4	-3	14	170	-172	4	0	-1	310	-284			
1	-14	670	-670	3	1	-8	402	-80	3	3	17	200	-200	4	-2	-26	282	320	4	0	-1	310	-284			
1	-13	156	156	3	1	-7	1070	1175	3	4	-10	80	-141	4	-2	-25	804	818	4	0	-1	155	-102			
1	-12	845	-876	3	1	-6	1404	-1404	3	4	-15	110	-71	4	-2	-24	91	76	4	0	-1	155	-102			
1	-11	655	-655	3	1	-5	239	-250	3	4	-14	-87	30	4	-2	-23	156	100	4	0	-1	521	409			
1	-10	330	-330	3	1	-5	500	533	3	4	-12	580	-80	4	-2	-21	183	-148	4	0	-1	136	-109			
1	-9	648	-648	3	1	-4	1000	-1000	3	4	-11	142	-112	4	-2	-19	262	-230	4	0	-1	700	-698			
1	-8	-77	-74	3	1	-4	151	158	3	4	-10	170	-167	4	-2	-18	161	-85	4	0	-1	700	-698			
1	-7	687	-690	3	1	-3	200	-400	3	4	-9	200	-83	4	-2	-16	222	-252	4	0	-1	15	208	273		
1	-6	889	-887	3	1	-2	-81	-80	3	4	-7	400	-411	4	-2	-15	108	-292	4	0	-1	11	168	-100		
1	-5	926	-924	3	1	-2	227	245	3	4	-6	137	-82	4	-2	-14	108	-292	4	0	-1	12	112	170		
1	-4	-2	-208	3	1	-1	448	-610	3	4	-5	239	-292	4	-2	-14	95	32	4	0	-1	13	183	203		
1	-3	1	968	-964	3	1	-1	1000	-1000	3	4	-4	239	-292	4	-2	-13	131	-239	4	0	-1	14	216	-37	
1	-2	935	937	3	1	0	0	80	3	4	-3	111	-97	4	-2	-12	-91	-18	4	0	-1	15	185	5		
1	-1	83	80	3	1	0	0	80	3	4	-2	111	-97	4	-2	-11	-10	-18	4	0	-1	16	185	5		
1	0	1475	1487	3	1	0	81	-956	3	4	-1	135	-114	4	-2	-10	146	-99	4	0	-1	17	154	111		
1	0	339	353	3	1	0	262	244	3	4	0	-82	-43	4	-2	-9	188	92	4	0	-1	18	-84	86		
1	0	333	74	3	1	0	163	419	3	4	1	-88	43	4	-2	-8	104	311	4	0	-1	19	294	32		
1	0	5	1466	1495	3	1	11	444	-692	3	4	2	233	-214	4	-2	-7	155	260	4	0	-1	20	112	-58	
1	0	6	874	784	3	1	12	87	-87	3	4	3	185	-185	4	-2	-6	107	98	4	0	-1	21	202	32	
1	0	7	419	380	3	1	13	895	-955	3	4	4	85	-85	4	-2	-5	162	150	4	0	-1	22	104	69	
1	0	8	1221	1152	3	1	14	295	-310	3	4	5	97	-97	4	-2	-4	107	85	4	0	-1	23	104	69	
1	0	9	708	729	3	1	15	155	-136	3	4	6	507	854	4	-2	-3	294	291	4	0	-1	24	143	-134	
1	0	10	359	353	3	1	16	409	-416	3	4	7	84	-84	4	-2	-2	136	583	4	0	-1	25	104	-905	
1	0	11	1002	976	3	1	17	-95	122	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	26	104	-905	
1	0	12	264	327	3	1	18	77	-95	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	27	177	-110	
1	0	13	158	380	3	1	19	429	-435	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	28	104	-905	
1	0	14	521	498	3	1	20	106	-43	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	29	104	-905	
1	0	15	380	327	3	1	21	259	-265	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	30	104	-905	
1	0	16	1721	-1709	3	1	22	157	-110	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	31	104	-905	
1	0	17	1817	1813	3	1	23	107	-205	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	32	104	-905	
1	0	18	1999	2005	3	1	24	101	192	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	33	104	-905	
1	0	19	1250	-1240	3	1	25	104	-104	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	34	104	-905	
1	0	20	237	-351	3	1	26	300	300	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	35	104	-905	
1	0	21	1265	-1270	3	1	27	91	34	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	36	104	-905	
1	0	22	488	-484	3	1	28	116	-116	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	37	104	-905	
1	0	23	488	-484	3	1	29	116	-116	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	38	104	-905	
1	0	24	287	-288	3	1	30	107	-107	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	39	104	-905	
1	0	25	414	-377	3	1	31	227	221	317	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	40	104	-905
1	0	26	149	1280	3	1	32	104	-104	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	41	104	-905	
1	0	27	-91	-71	3	1	33	225	80	74	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	42	104	-905
1	0	28	271	-258	3	1	34	224	552	744	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	43	104	-905
1	0	29	130	-130	3	1	35	185	-185	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	44	104	-905	
1	0	30	121	-107	3	1	36	227	188	-168	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	45	104	-905
1	0	31	115	-107	3	1	37	227	188	-168	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	46	104	-905
1	0	32	124	-112	3	1	38	225	430	-652	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	47	104	-905
1	0	33	127	-108	3	1	39	225	430	-652	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	48	104	-905
1	0	34	202	214	3	1	40	108	108	-53	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	49	104	-905
1	0	35	127	-108	3	1	41	225	430	-652	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	50	104	-905
1	0	36	239	241	3	1	42	106	145	-335	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	51	104	-905
1	0	37	242	244	3	1	43	163	188	-99	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	52	104	-905
1	0	38	242	244	3	1	44	163	188	-99	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	53	104	-905
1	0	39	242	244	3	1	45	163	188	-99	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	54	104	-905
1	0	40	242	244	3	1	46	163	188	-99	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	55	104	-905
1	0	41	242	244	3	1	47	163	188	-99	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	56	104	-905
1	0	42	242	244	3	1	48	163	188	-99	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	57	104	-905
1	0	43	242	244	3	1	49	163	188	-99	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	58	104	-905
1	0	44	242	244	3	1	50	163	188	-99	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	59	104	-905
1	0	45	242	244	3	1	51	163	188	-99	4	0	-9	-209	327	4	-2	-1	-91	-45	4	0	-1	60	104	-905
1	0	46	242	244	3	1	52	163	188	-99	4	0	-9	-209	327	4	-2	-1	-91	-45	4					

Table 2. Fractional atomic coordinates with standard deviations ($\times 10^4$) for the non-hydrogen atoms

	<i>x</i>	<i>y</i>	<i>z</i>
C(1)	1.1154 (5)	0.8035 (4)	0.0378 (1)
C(2)	1.2182 (5)	0.6553 (5)	0.0667 (1)
C(3)	0.9980 (5)	0.4688 (5)	0.0828 (1)
C(4)	1.1211 (5)	0.3477 (4)	0.1149 (1)
C(5)	0.9104 (5)	0.1586 (5)	0.1326 (1)
C(6)	1.0358 (5)	0.0453 (4)	0.1655 (1)
C(7)	0.8301 (5)	-0.1390 (5)	0.1845 (1)
C(8)	0.9611 (5)	-0.2537 (5)	0.2165 (1)
C(9)	0.7582 (5)	-0.4361 (4)	0.2364 (1)
C(10)	0.8972 (5)	-0.5521 (4)	0.2675 (1)
C(11)	0.6942 (5)	-0.7337 (4)	0.2881 (1)
C(12)	0.8389 (5)	-0.8419 (5)	0.3193 (1)
C(13)	0.6537 (5)	-1.0260 (5)	0.3405 (1)
C(14)	0.7886 (5)	-1.1332 (5)	0.3726 (1)
C(15)	0.5899 (5)	-1.3223 (5)	0.3919 (1)
C(16)	0.7305 (5)	-1.4331 (5)	0.4242 (1)
C(17)	0.5170 (7)	-1.6428 (6)	0.4380 (1)
C(18)	0.8692 (7)	-1.2475 (6)	0.4581 (1)
O(1)	0.8768 (4)	0.7479 (4)	0.0220 (1)
O(2)	1.3036 (4)	0.9933 (4)	0.0308 (1)
O(3)	0.4024 (4)	-1.0873 (4)	0.3322 (1)

Table 3. Anisotropic thermal parameters in the form

$$\exp [-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2lha^*c^*U_{31} + 2hka^*b^*U_{12})]$$

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)	564 (15)	492 (14)	477 (14)	178 (11)	122 (12)	151 (12)
C(2)	568 (15)	607 (16)	530 (14)	284 (12)	72 (11)	151 (12)
C(3)	517 (14)	556 (15)	519 (14)	222 (12)	42 (11)	136 (12)
C(4)	495 (14)	499 (14)	506 (14)	196 (11)	26 (11)	119 (11)
C(5)	536 (14)	503 (14)	498 (14)	199 (11)	20 (11)	84 (11)
C(6)	500 (14)	479 (14)	541 (14)	203 (11)	21 (11)	80 (11)
C(7)	491 (14)	493 (14)	579 (15)	208 (12)	21 (11)	85 (11)
C(8)	522 (14)	498 (14)	526 (14)	210 (11)	34 (11)	84 (11)
C(9)	525 (14)	497 (14)	549 (15)	199 (12)	27 (11)	96 (12)
C(10)	518 (14)	497 (14)	522 (14)	162 (11)	13 (11)	102 (11)
C(11)	474 (13)	514 (14)	523 (14)	202 (12)	39 (11)	77 (11)
C(12)	445 (13)	570 (15)	544 (14)	226 (12)	36 (11)	110 (11)
C(13)	444 (14)	586 (15)	509 (14)	156 (12)	69 (11)	184 (11)
C(14)	489 (14)	565 (15)	573 (15)	275 (12)	55 (11)	86 (12)
C(15)	524 (14)	539 (15)	505 (14)	167 (12)	67 (11)	83 (12)
C(16)	634 (15)	504 (14)	520 (14)	248 (12)	131 (12)	137 (12)
C(17)	926 (23)	678 (19)	802 (20)	340 (16)	114 (17)	156 (17)
C(18)	1031 (24)	735 (20)	564 (17)	221 (15)	-90 (16)	49 (18)
O(1)	592 (12)	740 (13)	814 (14)	476 (11)	-30 (10)	105 (10)
O(2)	649 (12)	680 (12)	780 (13)	450 (10)	54 (10)	44 (10)
O(3)	425 (11)	979 (16)	949 (15)	572 (13)	27 (10)	72 (10)

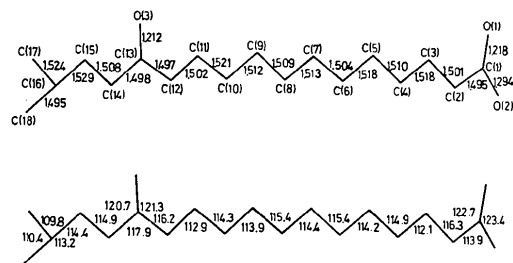


Fig. 2. Bond lengths and angles.

Table 4. Fractional atomic coordinates and isotropic temperature factors with their estimated standard deviations for the hydrogen atoms

The first appended number refers to that of the parent atom.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
H(21)	1.345 (6)	0.574 (6)	0.055 (1)	7.3 (7)
H(22)	1.313 (5)	0.754 (5)	0.087 (1)	4.8 (6)
H(31)	0.856 (5)	0.528 (5)	0.092 (1)	5.3 (6)
H(32)	0.898 (5)	0.343 (5)	0.061 (1)	5.1 (6)
H(41)	1.264 (5)	0.274 (5)	0.104 (1)	5.2 (6)
H(42)	1.219 (5)	0.472 (5)	0.136 (1)	5.1 (6)
H(51)	0.769 (5)	0.228 (5)	0.144 (1)	5.6 (6)
H(52)	0.818 (5)	0.026 (4)	0.110 (1)	4.4 (5)
H(61)	1.181 (6)	-0.028 (5)	0.155 (1)	5.6 (6)
H(62)	1.144 (6)	0.178 (5)	0.185 (1)	5.6 (6)
H(71)	0.685 (5)	-0.062 (5)	0.196 (1)	5.9 (6)
H(72)	0.734 (5)	-0.266 (5)	0.166 (1)	5.2 (6)
H(81)	1.102 (5)	-0.326 (4)	0.205 (1)	3.9 (5)
H(82)	1.062 (5)	-0.129 (5)	0.235 (1)	5.0 (6)
H(91)	0.627 (5)	-0.353 (5)	0.248 (1)	5.1 (6)
H(92)	0.665 (5)	-0.556 (5)	0.216 (1)	5.7 (6)
H(101)	1.034 (5)	-0.629 (5)	0.255 (1)	5.5 (6)
H(102)	1.006 (5)	-0.422 (5)	0.286 (1)	5.1 (6)
H(111)	0.551 (5)	-0.665 (5)	0.299 (1)	5.2 (6)
H(112)	0.586 (6)	-0.869 (5)	0.269 (1)	5.5 (6)
H(121)	0.976 (6)	-0.926 (5)	0.309 (1)	5.7 (6)
H(122)	0.932 (5)	-0.724 (5)	0.338 (1)	5.7 (6)
H(141)	0.926 (6)	-1.202 (5)	0.362 (1)	6.6 (7)
H(142)	0.870 (6)	-1.004 (5)	0.391 (1)	6.7 (7)
H(151)	0.449 (6)	-1.256 (6)	0.404 (1)	7.4 (7)
H(152)	0.479 (5)	-1.460 (5)	0.372 (1)	5.5 (6)
H(161)	0.896 (6)	-1.478 (5)	0.413 (1)	7.2 (7)
H(171)	0.447 (6)	-1.740 (5)	0.418 (1)	7.5 (7)
H(172)	0.355 (8)	-1.587 (7)	0.450 (1)	10.1 (10)
H(173)	0.592 (6)	-1.725 (5)	0.458 (1)	6.5 (7)
H(181)	0.707 (6)	-1.194 (5)	0.468 (1)	5.9 (6)
H(182)	1.018 (6)	-1.103 (5)	0.451 (1)	6.4 (7)
H(183)	0.938 (7)	-1.325 (6)	0.481 (1)	8.4 (8)
H(24)*	1.243 (8)	1.085 (7)	0.009 (1)	11.4 (11)

* Refers to the hydroxyl hydrogen.

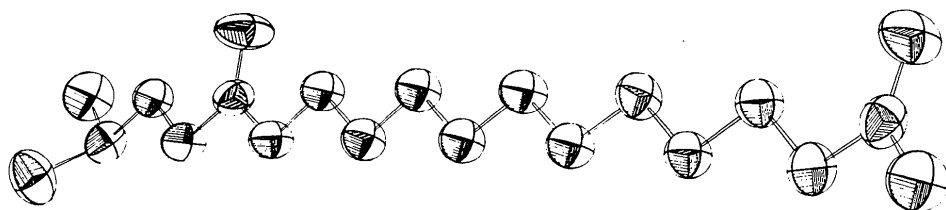
Fig. 1. Drawing of 13-oxoisostearic acid showing the thermal ellipsoids as viewed along the *b* axis.

Table 5. Bond distances and angles and their estimated standard deviations for the non-hydrogen atoms

C(1)—O(1)	1.218 (3) Å	O(1)—C(1)—O(2)	123.4 (3)°
C(1)—O(2)	1.294 (3)	O(1)—C(1)—C(2)	122.7 (2)
C(1)—C(2)	1.495 (4)	O(2)—C(1)—C(2)	113.9 (2)
C(2)—C(3)	1.501 (3)	C(1)—C(2)—C(3)	116.3 (2)
C(3)—C(4)	1.518 (4)	C(2)—C(3)—C(4)	112.1 (2)
C(4)—C(5)	1.510 (3)	C(3)—C(4)—C(5)	114.9 (2)
C(5)—C(6)	1.518 (4)	C(4)—C(5)—C(6)	114.2 (2)
C(6)—C(7)	1.504 (3)	C(5)—C(6)—C(7)	115.4 (2)
C(7)—C(8)	1.513 (4)	C(6)—C(7)—C(8)	114.4 (2)
C(8)—C(9)	1.509 (3)	C(7)—C(8)—C(9)	115.4 (2)
C(9)—C(10)	1.512 (4)	C(8)—C(9)—C(10)	113.9 (2)
C(10)—C(11)	1.521 (3)	C(9)—C(10)—C(11)	114.3 (2)
C(11)—C(12)	1.502 (4)	C(10)—C(11)—C(12)	112.9 (2)
C(12)—C(13)	1.497 (3)	C(11)—C(12)—C(13)	116.2 (2)
C(13)—C(14)	1.498 (4)	C(12)—C(13)—C(14)	117.9 (2)
C(13)—O(3)	1.212 (3)	C(12)—C(13)—O(3)	121.3 (2)
C(14)—C(15)	1.508 (3)	C(14)—C(13)—O(3)	120.7 (2)
C(15)—C(16)	1.529 (4)	C(13)—C(14)—C(15)	114.9 (2)
C(16)—C(17)	1.524 (4)	C(14)—C(15)—C(16)	114.4 (2)
C(16)—C(18)	1.495 (3)	C(15)—C(16)—C(17)	109.8 (2)
		C(15)—C(16)—C(18)	113.2 (2)
		C(17)—C(16)—C(18)	110.4 (2)

The mean $C(sp^3)-C(sp^3)$ bond distance is 1.512 (4) Å and the mean $C-C-C$ angle for the same carbon atoms is 113.9 (2)° (see, however, below). These values are in agreement with those found in other long hydrocarbon chains (Jensen & Mabis, 1966; O'Connell, 1968; O'Connell & Pascher, 1969). The systematically short bond lengths and large bond angles are most probably due to thermal motion and rotatory oscillations of the chain around the long axis. The distances and angles have not been corrected for thermal motion as this would require an analysis of the complete vibrational pattern of all the atoms. However, if a riding motion correction is considered applicable for the two $C-C$ bonds in the iso-group the bond lengths should be increased by about 0.025 Å.

The equation of the best plane through C(2) to C(16) is given in Table 6 with the out-of-plane deviations as well as the in-plane deviations from the chain axis. From the Δ_1 and Δ_2 values it can be seen that the molecule is clearly bent in the zigzag plane. This is also shown by the interatomic angles where the $C-C-C$ angle for the even-numbered carbon atoms are all somewhat larger than those of the odd-numbered atoms. This type of bending is rather unexpected but is a combined effect of the valence angles at the keto group and the molecular packing (Fig. 3). The keto oxygen atom is in the zigzag plane of the chain. The sp^2 carbon atom exerts a pressure on the chain to bend or twist locally to preserve the 120° valence angle. However, as the molecules are heavily tilted with respect to the end-group planes, each keto group is isolated in regions where normal carbon chain packing exists. The strain forces at the keto group are not strong enough to deform the chains locally. The strain is instead taken up by a successive bend of the chain axis. The $C-C-C$ angle of the keto carbon atom has also decreased to 117.9°. In DL-2-methyl-7-oxododecanoic acid (O'Connell, 1968) the corresponding angle

is 117.3°. Here the geometric requirements of the keto group resulted in a twist of 7.2° of the zigzag planes of the two chain parts on each side of the keto group. This is compatible with the less pronounced chain domination in the smaller molecule.

Table 6. The best least-squares plane through C(2) to C(16)

The equation is expressed in terms of the crystal axes.

$$-0.0798X + 0.1873Y + 0.9791Z - 0.0886 = 0.$$

Δ_1 are the out-of-plane deviations, Δ_2 and Δ_3 are the in-plane distances from the chain axes and the 'worst' plane.

	Δ_1	Δ_2	Δ_3
C(2)	0.048	0.560	8.841 Å
C(3)	0.014	-0.358	7.655
C(4)	-0.009	0.405	6.343
C(5)	-0.036	-0.460	5.105
C(6)	-0.015	0.319	3.803
C(7)	-0.006	-0.516	2.552
C(8)	-0.018	0.284	1.267
C(9)	0.015	-0.532	-0.002
C(10)	-0.037	0.298	-1.264
C(11)	0.015	-0.513	-2.550
C(12)	-0.013	0.344	-3.783
C(13)	0.010	-0.382	-5.092
C(14)	0.022	0.435	-6.348
C(15)	0.008	-0.364	-7.627
C(16)	0.000	0.480	-8.901
*C(1)	0.214	-0.091	10.177
*C(17)	-0.187	-0.420	-10.116
*C(18)	1.226	1.322	-9.058
*O(1)	0.066	-1.287	10.349
*O(2)	0.508	0.734	11.131
*O(3)	0.018	-1.592	-5.141

* Atoms not included in the plane calculation.

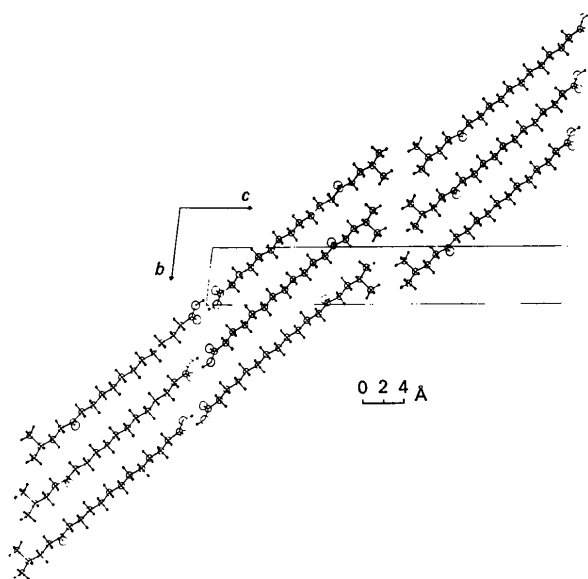


Fig. 3. Molecular arrangement of 13-oxoisostearic acid viewed along the a axis.

The closest intermolecular contacts between the keto oxygen and carbon atoms are 3.609, 3.403 and 3.387 Å to C(10), C(12) and C(14) respectively and the shortest keto oxygen-hydrogen distances are 2.67, 2.59 and 2.60 Å to H(102), H(121) and H(141) respectively. The

corresponding values in DL-2-methyl-7-oxododecanoic acid for the oxygen-carbon contacts are 3.45, 3.50 and 3.52 Å and for oxygen-hydrogen contacts 2.76, 2.69 and 2.63 Å.

Fig. 3 shows the molecular packing which agrees in principle with that reported for 17-methyl-octadecanoic acid (Abrahamsson, 1959a). The molecules are arranged in layers but tilted by as much as 44° to the end-group planes to allow each branching methyl group to be accommodated in the space between two chain ends.

Some short contacts around the carboxyl group and at the end methyl groups are shown in Fig. 4. Table 7 gives all non-hydrogen intermolecular distances less than 3.8 Å. The molecules form hydrogen-bonded dimers and the two carboxyl groups are coplanar within the experimental errors. The perpendicular distance between the two carboxyl groups is thus only 0.016 Å. The hydroxyl hydrogen atom deviates from the plane by 0.083 Å. This hydrogen atom has a fairly large temperature factor and less definite positional parameters than the other hydrogen atoms which is often the case in O-H...O hydrogen bonds.

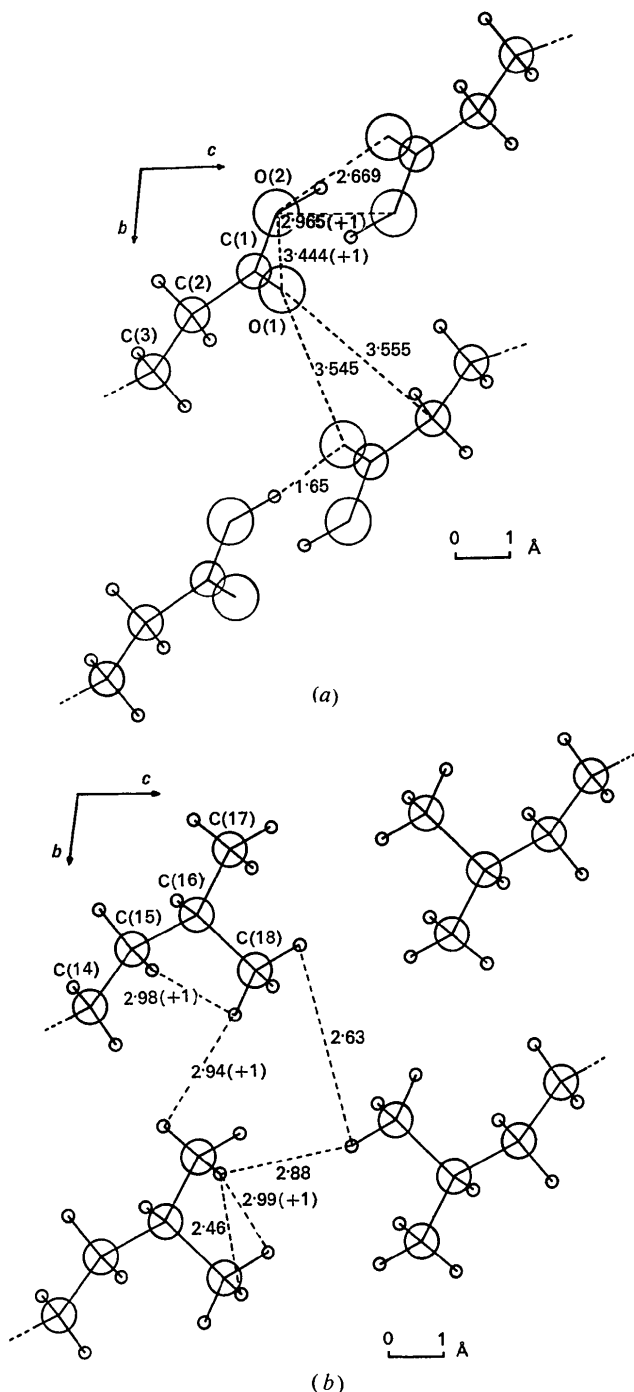


Fig. 4. (a) The carboxyl group contact planes as seen along the *a* axis with the shorter intermolecular distances indicated. (b) The methyl group contact planes as seen along the *a* axis with the shorter hydrogen contacts indicated.

Table 7. Intermolecular distances less than 3.8 Å

C(1)····O(1)	(2, 1, 0) 2*	3.560 (3) Å
C(1)····O(2)	(2, 2, 0) 2	3.406 (3)
C(1)····O(2)	(2, 2, 0) 2	3.481 (3)
C(2)····O(1)	(2, 1, 0) 2	3.555 (3)
C(2)····O(1)	(1, 0, 0) 1	3.641 (3)
C(3)····O(2)	(0, -1, 0) 1	3.737 (3)
C(4)····O(2)	(0, -1, 0) 1	3.641 (3)
C(10)····O(3)	(1, 1, 0) 1	3.609 (3)
C(12)····O(3)	(1, 0, 0) 1	3.403 (3)
C(14)····O(3)	(1, 0, 0) 1	3.387 (3)
C(18)····C(18)	(2, -2, 1) 2	3.737 (4)
O(1)····O(1)	(2, 1, 0) 2	3.545 (3)
O(1)····O(1)	(2, 2, 0) 2	3.363 (3)
O(1)····O(2)	(-1, 0, 0) 1	3.444 (3)
O(1)····O(2)	(2, 2, 0) 2	2.669 (3)
O(2)····O(2)	(2, 2, 0) 2	3.567 (3)
O(2)····O(2)	(3, 2, 0) 2	2.965 (3)

* Unit-cell translation of the second atom.

The carboxyl group has normal bond lengths and angles and is tilted 13° to the plane of the hydrocarbon chain. The O-H bond distance is 1.03 Å and the O...O and O...H distances are 2.669 and 1.65 Å respectively. The O...H-O angle is 171°. These values are in a good agreement with those found in neutron-diffraction studies of O-H...O bonded compounds (Hamilton & Ibers, 1968).

The closest interlayer van der Waals contact is 3.74 Å between branching methyl carbons. As shown in Fig. 4 the shortest hydrogen-hydrogen contacts between the layers are H(183)···H(183) and H(172)···H(183) which are 2.63 and 2.88 Å respectively.

The chains are arranged in the common triclinic subcell packing (*T*||) (Abrahamsson, 1959b), illustrated

in Fig. 5, with the dimensions

$$\begin{aligned} a_s &= 4.27, & b_s &= 5.39, & c_s &= 2.55 \text{ \AA} \\ \alpha_s &= 73.9, & \beta_s &= 108.6, & \gamma_s &= 119.6^\circ. \end{aligned}$$

The volume of the CH₂ group is 23.9 Å³.

The mean C-H bond distance in the subcell regions is 0.96 Å ($\sigma = 0.04$ Å), where $\sigma = [\sum_N (X_N - \bar{X})^2 / (N - 1)]^{1/2}$

and the average value of the angles involving hydrogen atoms in the same part of the molecule is 108° ($\sigma = 3^\circ$). The shortest H...H interatomic contacts are H_A...H_B 2.89 (6) Å, H_A...H_B (0,0,-1)* 2.77 (6) Å, H_A...H_C 2.98 (4) Å, H_A...H_C (0,0,1)* 2.88 (6) Å and H_A...H_D (0,0,1)* 2.73 (9) Å. (The standard deviations are calculated with the formula given above.)

A Fourier difference map was calculated in the plane of the molecule. Even if it showed the expected pattern of positive electron density in the middle of every carbon-carbon bond arising from the valence electrons these peaks were less than three standard deviations above the background level.

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* Subcell edge translation of the second atom.

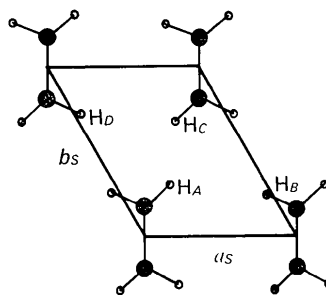


Fig. 5. The idealized subcell viewed down c_s .

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The Crystal Structure of Isostearic Acid

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Crystals of isostearic acid, C₁₈H₃₆O₂, are triclinic (PT) with $a = 4.9356$, $b = 5.6522$, $c = 34.408$ Å, $\alpha = 95.22$, $\beta = 95.21$ and $\gamma = 103.62^\circ$. The molecules are, as is usual for long-chain fatty acids, held together by hydrogen bonds to dimers. The molecular packing is dominated by the space requirements of the methyl branches, which are accommodated between the ends of the carbon chains. The chain axes then become tilted 44° to the end group planes. The chain packing is of the common triclinic type T[†].

The structures of branched-chain fatty acids have earlier been studied by this research group (Abrahamsson, 1959a; Abrahamsson & Harding, 1966). Two isoacids have so far been investigated by single-crystal methods. 17-Methylcetadecanoic acid (Abrahamsson,

1959b) had a superstructure of the carboxyl groups and was treated only in one projection. Isopalmitic acid was also studied only in projection (Stenhagen, Vand & Sim, 1952) and the reported structure is probably wrong as pointed out by Abrahamsson (1959b).