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Refinement of the Structure of *n*-Nonanoic Acid Hydrazide*

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The crystal structure of *n*-nonanoic acid hydrazide has been refined by three-dimensional ΔF syntheses. The structure consists of molecular ribbons laced together by N–H ··· O and N–H ··· N bonds. These ribbons laid face-to-face make up 'molecular double layers' similar to those occurring in many other long-chain structures.

Within the limit of experimental error, the paraffin chain is planar except for a small deviation at the methyl C atom, and the C–C bonds are equal except for that adjacent to the carbonyl group. The average C–C bond length when corrected for angular oscillation is 1·526 Å. Although the maximum corrections in the bond lengths due to angular oscillation of the molecule as a whole are small, it is shown that similar corrections due to oscillation of an atom about another to which it is bonded may be relatively large. Thus the correction to the C–O bond is +0·014 Å or three times its standard deviation.

It has been shown that the melting points of monoacyl derivatives of hydrazine with 4–12 carbon atoms fall on a single smooth curve when plotted against the number of carbon atoms (Kyame, Fisher & Bickford, 1947). This implies that the compounds with even or odd numbers of carbon atoms in the chain have either the same structure or different structures with similar lattice energies and variation of lattice energies with number of carbon atoms.

The length of the C–C single bonds in organic molecules is usually taken to be equal to that in diamond, 1·5445 Å, although until recently this has not been subject to experimental verification with any degree of precision. Furthermore, the C–C bonds in hexamethylene diamine indicate a possible alternation in length (Binnie & Robertson, 1950).

Finally, certain paraffin chain compounds are good models of biological membrane systems and their structures shed light on possible chain configuration and packing in such systems.

For these reasons, the structures of two monoacyl hydrazides have been determined, one with an even number (Jensen, 1956), the other with an odd number of C atoms in the chain. This is to report the structure

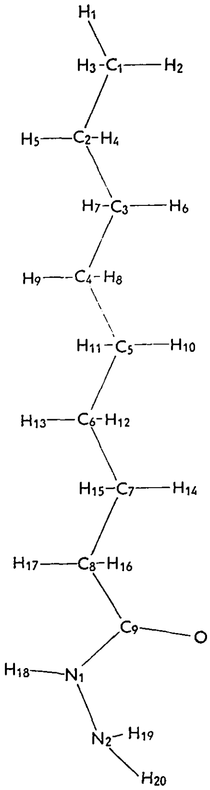

Fig. 1. Structural formula for *n*-nonanoic acid hydrazide.

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

l	F_o	F_c
	00*l*	
6	207	-212
8	800	-852
10	363	403
12	926	-1001
14	270	291
16	413	-452
18	299	-262
20	162	158
22	490	-491
24	95	114
26	198	-172
28	212	-186
30	268	299
32	453	-456
34	473	485
36	342	-347
38	334	335
40	<55	-7
42	143	119
44	781	767
46	218	206
48	761	-777
50	99	-36
52	105	-38
54	187	-178
56	142	157
58	216	-208
60	194	178
62	159	-148
64	94	89
66	<52	-22
68	<48	-32
70	71	57
72	<38	-36
74	89	56
	20*l*	
-74	<30	-13
-72	211	204
-70	84	-70
-68	185	-161
-66	126	117
-64	125	-141
-62	119	122
-60	96	-90
-58	<62	71
-56	<63	-1
-54	<63	-12
-52	<63	53
-50	157	-117
-48	655	-679
-46	241	228
-44	332	320
-42	<58	-83
-40	377	354
-38	157	-139
-36	299	289
-34	<51	49
-32	92	71
-30	240	243
-28	128	95
-26	124	-77
-24	1136	-1177
-22	190	-186
-20	719	752
-18	1036	-1094
-16	1143	1253
-14	1043	-1136
-12	590	644
-10	232	-215
-8	741	-801
-6	773	855
-4	2131	-2784
-2	1552	1853
0	1386	1600
2	471	479
4	221	237
6	81	35
8	124	153
10	<34	9
12	194	-201
14	195	205
16	522	-579
18	193	193
20	1309	-1375
22	231	192
24	622	601
26	201	-200
28	256	256
30	160	-136
32	<52	40
34	67	-38
36	<55	-24
38	93	-62
40	<59	47
42	155	-128
44	921	958
46	231	-219
48	166	-141
50	<63	-18
52	144	-145
54	<62	54
56	180	-152
58	<59	61
60	120	-103
62	<53	0
64	83	-66
66	57	-51
68	236	209
70	<35	-19
	40*l*	
-70	107	-114
-68	39	-35
-66	<42	-4
-64	<47	-34
-62	<50	-2
-60	<54	2
-58	<56	-41
-56	65	83
-54	75	-89
-52	205	214
-50	250	-231
-48	191	-196
-46	116	95
-44	<63	-42
-42	122	87
-40	<62	-9
-38	<61	-2
-36	139	137
-34	97	-88
-32	297	275
-30	<56	-13
-28	611	641
-26	256	-266
-24	832	-898
-22	591	600
-20	646	-680
-18	479	488
-16	281	-280
-14	<48	5
-12	252	257
-10	382	-421
-8	434	469
-6	509	-545
-4	536	-548
-2	381	375
0	253	237
2	257	251
4	<47	-15
6	124	123
8	165	164
10	134	-120
12	349	371
14	268	-261
16	444	448
18	402	-423
20	641	-692
22	<55	79
24	308	286
26	201	-197
28	290	282
30	156	-156
32	93	90
34	<62	42
36	157	-164
38	205	213
40	408	-405
42	330	320
44	129	133
46	<62	31
48	<61	-12
50	<59	-39
52	<57	46
54	72	-60
56	<51	13
58	<47	0
60	85	-73
62	<38	48
64	277	-265
	60*l*	
-62	<28	30
-60	<35	-53
-58	<40	40
-56	<44	-41
-54	<47	13
-52	<50	0
-50	<53	-46
-48	<55	39
-46	<57	-51
-44	<59	22
-42	<60	27
-40	95	-100
-38	125	132
-36	202	-201
-34	192	187
-32	215	-208
-30	306	284
-28	488	484
-26	452	-448
-24	<63	15
-22	130	-122
-20	<62	-7
-18	<62	0
-16	<62	-66
-14	<62	42
-12	<61	-44
-10	<61	-6
-8	95	78
-6	190	-159
-4	<61	89
-2	101	-99
0	101	113
2	<61	-18
4	<61	-20
6	76	76
8	<62	-63
10	85	105
12	<63	3
14	115	115
16	320	315
18	261	-237
20	488	-493
22	298	280
24	200	-186
26	133	135
28	<63	-37
30	<62	-10
32	80	77
34	95	-93
36	119	117
38	91	-88
40	<55	35
42	<52	22
44	<50	-18
46	58	62
48	43	-55
50	<39	35
52	<34	13
54	<26	-41
	80*l*	
-46	<27	-15
-44	64	65
-42	83	-87
-40	99	97
-38	114	-97
-36	65	53
-34	72	-53
-32	106	-98
-30	224	199
-28	84	70
-26	89	-78
-24	<53	-4
-22	<54	-26
-20	<55	10
-18	<55	-14
-16	<56	7
-14	<56	-12
-12	<57	-16
-10	<57	-15
-8	121	-124
-6	133	135
-4	<57	40
-2	<57	-33
0	<57	14
2	<57	8
4	<56	-50
6	<56	60
8	87	-88
10	<54	68
12	94	-71
14	128	93
16	231	197
18	245	-226
20	<49	18
22	<47	-42
24	<45	20
26	<43	-3
28	<40	-12
30	56	18
32	<34	-31
34	<29	14
	01*l*	
1	126	-122
3	196	203
5	299	-292
7	68	55
9	<45	-23
11	271	-279
13	289	306
15	415	-416
17	327	348
19	208	-200
21	460	446
23	1006	1081
25	757	-768
27	<40	74
29	157	-151
31	196	-178
33	199	191
35	326	-319
37	264	257
39	194	-192
41	<53	51
43	98	88
45	198	-186
47	204	219
49	195	-192
51	122	116
53	<59	-48
55	<59	-26
57	102	84
59	84	-90
61	107	103
63	57	-45
65	49	49
67	97	107
69	91	-106
71	86	-78
73	<29	5
	11*l*	
-73	<37	-14
-71	87	67
-69	145	122
-67	131	-143
-65	117	129
-63	95	-104
-61	77	78
-59	<57	17
-57	<58	-61
-55	215	223
-53	175	-192
-51	337	345
-49	<59	12
-47	468	-475
-45	481	-488
-43	260	251
-41	<54	-48
-39	<52	32
-37	134	119
-35	<48	3
-33	124	104
-31	186	188
-29	<42	-30
-27	472	463

Table 1 (*cont.*)

l	F_o	F_c
−25	289	274
−23	441	−434
−21	192	−184
−19	80	−85
−17	143	137
−15	302	−315
−13	389	391
−11	558	−588
−9	541	592
−7	898	−997
−5	664	732
−3	1843	−2281
−1	1251	−1488
1	1313	1539
3	752	816
5	340	361
7	243	246
9	530	573
11	268	−242
13	652	695
15	325	−320
17	423	428
19	< 34	−14
21	367	−340
23	493	−504
25	308	266
27	163	−149
29	178	173
31	232	−208
33	153	145
35	199	−197
37	< 51	2
39	73	−74
41	236	−217
43	198	−207
45	328	311
47	718	725
49	329	−370
51	226	227
53	129	−115
55	< 58	−8
57	< 57	16
59	105	−72
61	< 53	22
63	< 50	−43
65	66	−43
67	73	−57
69	< 39	1
71	62	54
	21*l*	
−73	< 34	42
−71	112	−106
−69	40	−57
−67	83	99
−65	65	−80
−63	91	100
−61	< 54	−52
−59	< 56	34
−57	< 58	53
−55	84	−68
−53	143	141
−51	81	−92
−49	171	168
−47	178	−156
−45	123	−95
−43	163	170
−41	198	−189
−39	184	190
−37	101	−90
−35	< 50	−1
−33	133	141
−31	268	−274
−29	268	282
−27	544	−590
−25	221	−194
−23	517	520
−21	< 38	40
−19	204	201
−17	< 35	14
−15	53	51
−13	104	100
−11	39	−42
−9	55	43
−7	48	34
−5	127	−152
−3	104	−99
−1	600	−671
1	472	488
3	< 29	−7
5	161	−186
7	387	415
9	471	−521
11	375	403
13	312	−337
15	65	−41
17	112	98
19	605	−611
21	906	956
23	115	76
25	61	−49
27	98	132
29	293	−272
31	259	271
33	310	−287
35	153	155
37	103	−73
39	110	−91
41	99	117
43	259	−258
45	237	237
47	< 59	−41
49	< 59	1
51	< 59	38
53	71	−72
55	65	57
57	64	−50
59	< 53	12
61	< 50	15
63	< 47	−39
65	94	87
67	80	63
69	70	−59
	31*l*	
−71	278	268
−69	< 36	−61
−67	< 41	46
−65	< 45	−42
−63	< 48	11
−61	< 51	6
−59	< 54	−35
−57	< 56	42
−55	< 58	−16
−53	< 59	21
−51	147	168
−49	97	111
−47	331	−327
−45	97	−83
−43	< 58	−3
−41	< 57	−40
−39	< 56	−38
−37	58	54
−35	110	−130
−33	142	157
−31	116	−120
−29	131	119
−27	480	446
−25	471	464
−23	589	−549
−21	< 43	31
−19	< 42	−32
−17	226	−210
−15	286	263
−13	383	−393
−11	480	437
−9	425	−422
−7	441	442
−5	173	−172
−3	604	−596
−1	579	−569
1	372	366
3	59	32
5	< 38	−9
7	223	197
9	< 39	−38
11	119	121
13	219	175
15	< 42	−23
17	433	417
19	251	230
21	614	−577
23	224	−200
25	< 49	−35
27	161	155
29	250	−243
31	319	316
33	280	−285
35	235	239
37	160	−165
39	66	63
41	165	−148
43	316	−298
45	395	405
47	< 59	19
49	88	62
51	< 57	25
53	< 56	21
55	< 54	9
57	< 51	29
59	< 48	−15
61	< 45	38
63	< 41	−5
65	100	−72
67	193	−177
	41*l*	
−69	34	42
−67	50	−53
−65	< 40	35
−63	< 44	−17
−61	< 47	−16
−59	< 50	39
−57	58	−54
−55	82	76
−53	< 57	−53
−51	134	148
−49	81	79
−47	191	−199
−45	91	78
−43	75	−72
−41	< 59	8
−39	< 58	47
−37	88	−80
−35	113	130
−33	103	−94
−31	118	106
−29	< 53	9
−27	229	−273
−25	< 51	−32
−23	99	117
−21	< 49	−19
−19	121	123
−17	73	−56
−15	87	107
−13	< 46	31
−11	< 46	−5
−9	170	188
−7	105	−84
−5	332	312
−3	489	−541
−1	145	−155
1	276	254
3	290	−303
5	326	313
7	174	−198
9	77	62
11	127	105
13	244	−269
15	293	308
17	410	−465
19	103	92
21	147	190
23	< 53	−17
25	183	185
27	190	−181
29	192	196
31	115	−116
33	56	47
35	< 59	43
37	114	−102
39	127	122
41	205	−206
43	< 59	−41
45	117	100
47	< 57	−5
49	< 55	30
51	< 53	−6
53	< 51	2
55	< 48	7
57	< 44	−18
59	< 40	11
61	< 36	−19
63	< 30	−21
	51*l*	
−65	< 36	40
−63	< 35	−37
−61	< 40	31
−59	< 43	−31
−57	< 47	18
−55	< 49	−25
−53	< 52	12
−51	70	87
−49	< 56	17
−47	80	−83
−45	< 58	−19
−43	< 59	−7
−41	< 59	−33
−39	< 59	6
−37	< 59	−45
−35	< 59	−26
−33	69	−48
−31	101	−82
−29	207	−163
−27	694	674
−25	< 57	−33
−23	93	−72
−21	< 52	2
−19	73	−61
−17	< 53	44
−15	97	−79
−13	87	84
−11	84	−83
−9	86	90
−7	86	46
−5	92	81
−3	319	−303
−1	73	−87
1	< 52	31
3	< 52	−30
5	68	−40
7	103	75
9	149	−146
11	171	154
13	115	−124
15	93	100
17	319	337
19	201	209
21	353	−363
23	142	−133
25	134	147
27	234	−241
29	204	214
31	175	−178
33	122	116
35	< 59	−52
37	< 58	16
39	< 58	41
41	125	−150
43	56	−24
45	78	68
47	< 51	4
49	< 48	30
51	< 45	−8
53	< 42	32
55	< 38	−6
57	< 40	29
59	< 31	15
	61*l*	
−59	< 39	−44
−57	< 37	27
−55	< 40	−42
−53	< 44	−10
−51	118	151
−49	< 49	−29
−47	< 51	−35
−45	< 53	5
−43	< 55	−34
−41	< 56	13
−39	< 57	−22
−37	< 58	−3
−35	< 58	4
−33	< 59	−17
−31	< 59	35
−29	< 59	−5
−27	< 64	−46
−25	< 64	59
−23	99	−71

Table 1 (*cont.*)

l	F_o	F_c
−21	90	77
−19	< 59	−45
−17	< 59	5
−15	< 58	50
−13	76	−82
−11	117	133
−9	102	−89
−7	205	237
−5	78	65
−3	290	−312
−1	121	118
1	110	−129
3	< 58	49
5	< 58	11
7	84	−83
9	134	134
11	146	−150
13	132	145
15	82	−82
17	< 59	−22
19	< 59	48
21	< 59	−71
23	98	96
25	92	−73
27	< 58	42
29	< 58	5
31	< 57	−29
33	61	63
35	59	−44
37	58	65
39	< 51	15
41	131	−135
43	< 46	−16
45	< 43	38
47	< 40	−4
49	< 36	12
51	< 38	6
	$71l$	
−53	< 36	−15
−51	55	−40
−49	< 37	−14
−47	42	41
−45	66	−55
−43	68	60
−41	48	−50
−39	50	36
−37	< 51	−10
−35	< 52	−41
−33	< 54	58
−31	300	−286
−29	88	100
−27	218	202
−25	< 57	48
−23	58	−61
−21	< 58	56
−19	< 58	−42
−17	< 58	19
−15	< 59	16
−13	< 59	−19
−11	< 59	21
−9	< 59	2
−7	< 59	−103
−5	74	13
−3	< 59	38
−1	< 59	−9
1	< 59	15
3	< 59	−19
5	< 59	31
7	79	−62
9	< 58	43
11	86	−85
13	< 58	−1
15	< 57	−85
17	361	351
19	< 56	17
21	183	−175
23	59	72
25	80	−88
27	< 51	34
29	< 50	−26
31	< 48	−4
33	< 46	−10
35	< 43	−6
37	< 41	−30
39	< 38	−8
41	< 34	25
43	< 36	23
	$81l$	
−43	< 35	−11
−41	< 32	12
−39	< 36	−14
−37	< 38	8
−35	< 40	−4
−33	< 42	−8
−31	< 44	0
−29	< 45	−37
−27	64	68
−25	< 48	−43
−23	< 48	19
−21	< 50	12
−19	< 50	−39
−17	56	53
−15	56	−62
−13	< 52	41
−11	< 52	−33
−9	< 52	−17
−7	150	140
−5	62	−73
−3	< 52	12
−1	< 52	−19
1	< 52	−10
3	< 51	33
5	< 51	−48
7	< 50	56
9	< 50	−50
11	< 49	30
13	< 48	−12
15	< 47	−22
17	50	57
19	< 44	−29
21	< 43	6
23	< 41	0
25	< 39	−15
27	< 37	13
29	< 34	−17
31	< 37	7
33	< 31	0
	$91l$	
−27	76	54
−25	< 35	−18
−23	< 37	6
−21	< 40	−5
−19	< 41	−5
−17	< 43	−1
−15	< 35	19
−13	< 36	−20
−11	71	76
−9	< 37	−40
−7	87	−75
−5	< 37	−5
−3	77	69
−1	< 36	−39
1	< 36	34
3	< 35	4
5	< 37	−20
7	< 33	46
9	40	−51
11	< 37	51
13	111	−93
15	< 30	33
	$02l$	
0	1540	−1770
2	48	5
4	181	154
6	176	203
8	150	−138
10	363	360
12	241	−200
14	252	240
16	< 35	26
18	< 38	−44
20	325	306
22	240	−223
24	303	299
26	124	−135
28	107	111
30	70	−70
32	64	76
34	93	−102
36	119	110
38	176	−166
40	106	101
42	151	−157
44	267	−284
46	158	−159
48	362	404
50	85	66
52	< 55	−58
54	163	167
56	129	−132
58	115	122
60	80	−70
62	< 46	39
64	< 42	−3
66	< 39	−7
68	< 41	−4
70	< 34	12
	$12l$	
−70	67	−62
−68	153	−138
−66	< 39	−6
−64	< 43	32
−62	64	−54
−60	58	61
−58	45	−49
−56	63	56
−54	< 54	−26
−52	< 55	52
−50	< 55	−21
−48	132	122
−46	< 55	7
−44	143	−136
−42	120	121
−40	132	−134
−38	162	165
−36	89	−93
−34	79	87
−32	109	113
−30	180	−157
−28	377	386
−26	382	−367
−24	593	−588
−22	< 40	22
−20	200	166
−18	252	257
−16	236	−256
−14	470	481
−12	307	−321
−10	284	263
−8	94	78
−6	245	−250
−4	547	556
−2	515	−527
0	126	129
2	313	−320
4	181	183
6	116	96
8	205	−213
10	299	308
12	245	−273
14	98	123
16	104	−124
18	131	−123
20	459	−457
22	148	−131
24	797	762
26	< 44	−62
28	60	47
30	114	115
32	122	−115
34	109	116
36	94	−93
38	65	62
40	64	−59
42	< 54	40
44	132	−114
46	< 55	11
48	61	45
50	< 55	5
52	< 54	15
54	< 53	−39
56	< 51	42
58	70	−73
60	< 47	35
62	< 44	−42
64	< 40	−39
66	< 36	−5
68	182	161
70	70	71
	$22l$	
−70	57	67
−68	77	72
−66	62	−52
−64	78	85
−62	95	−94
−60	94	97
−58	90	−84
−56	< 56	42
−54	< 53	−9
−52	< 54	−45
−50	82	88
−48	260	259
−46	74	−72
−44	159	−141
−42	< 54	−22
−40	< 53	−19
−38	90	−74
−36	< 50	10
−34	64	−51
−32	122	−109
−30	117	113
−28	384	−391
−26	381	388
−24	247	268
−22	130	153
−20	183	−192
−18	161	160
−16	144	−147
−14	132	114
−12	79	−96
−10	108	87
−8	< 31	−6
−6	< 30	34
−4	431	415
−2	287	−294
0	478	−436
2	310	−322
4	313	334
6	448	−454
8	359	360
10	271	−287
12	121	131
14	< 37	5
16	54	−28
18	144	149
20	421	457
22	80	−66
24	185	−217
26	< 45	−42
28	< 46	−6
30	< 48	−22
32	< 51	−34
34	112	95
36	127	−127
38	160	172
40	122	−127
42	98	125
44	420	−425
46	< 55	56
48	137	145
50	< 54	−33
52	65	71
54	< 51	12
56	< 49	7
58	51	42
60	< 44	4
62	< 40	16
64	87	83
66	< 31	−33
	$32l$	
−68	37	−45
−66	< 34	3
−64	< 38	−32
−62	< 42	16
−60	45	−46
−58	< 47	34
−56	88	−78
−54	71	68
−52	112	−118
−50	100	87
−48	253	225
−46	140	−138
−44	< 55	41

Table 1 (*cont.*)

l	F_o	F_c
-42	55	-73
-40	<54	19
-38	<53	21
-36	96	-80
-34	157	171
-32	144	-139
-30	184	203
-28	136	136
-26	170	-171
-24	218	-225
-22	163	-174
-20	280	304
-18	331	-341
-16	308	318
-14	163	-171
-12	<41	37
-10	192	180
-8	221	-208
-6	361	359
-4	182	149
-2	<39	35
0	422	-401
2	<40	-1
4	70	86
6	148	-166
8	184	178
10	98	-99
12	92	72
14	<44	9
16	<45	-6
18	<46	-14
20	465	-507
22	188	158
24	141	122
26	<51	14
28	<52	46
30	<52	31
32	<53	12
34	<53	15
36	72	64
38	65	-59
40	169	165
42	140	-116
44	165	-110
46	<54	-35
48	87	79
50	<52	-1
52	<50	-19
54	63	68
56	50	-68
58	39	50
60	<38	-36
62	<33	-11
64	<33	-54
	42*l*	
-66	<31	19
-64	<32	-7
-62	<36	15
-60	<40	-9
-58	<43	10
-56	<46	—21
-54	<48	19
-52	70	-77
-50	111	101
-48	<56	45
-46	<54	33
-44	97	-99
-42	62	71
-40	80	-69
-38	<55	22
-36	<55	12
-34	90	-77
-32	<53	58
-30	143	-142
-28	222	-205
-26	138	125
-24	298	300
-22	124	-115
-20	122	126
-18	97	-92
-16	95	105
-14	101	-92
-12	101	101
-10	80	-79
-8	58	63
-6	51	22
-4	199	177
-2	<49	-24
0	264	-245
2	75	105
4	115	-118
6	<47	34
8	<48	-36
10	<48	-23
12	72	-61
14	<48	38
16	226	-225
18	264	248
20	208	191
22	87	69
24	227	-225
26	117	125
28	96	-106
30	<54	0
32	<57	46
34	87	-103
36	107	113
38	88	-101
40	112	131
42	87	-81
44	105	-104
46	<51	-4
48	69	48
50	66	-49
52	67	60
54	56	-50
56	<33	43
58	<33	-20
60	<33	15
	52*l*	
-62	<34	-47
-60	47	53
-58	72	-74
-56	<39	34
-54	53	-45
-52	147	-156
-50	177	183
-48	77	74
-46	<51	-9
-44	<52	-15
-42	<53	-20
-40	<54	29
-38	82	-79
-36	86	80
-34	83	-90
-32	<55	47
-30	<55	26
-28	<55	43
-26	<57	17
-24	157	-166
-22	116	132
-20	130	-130
-18	83	70
-16	<52	-31
-14	<52	-43
-12	<52	35
-10	<52	-54
-8	<51	-41
-6	130	142
-4	366	346
-2	144	-116
0	183	-185
2	94	104
4	128	-138
6	68	65
8	<52	-38
10	<53	3
12	<53	25
14	<53	-21
16	121	106
18	116	-121
20	<55	-41
22	<55	-38
24	75	70
26	71	-67
28	69	55
30	<55	-24
32	<54	-12
34	77	61
36	66	-63
38	109	107
40	100	84
42	<48	-28
44	170	-175
46	<44	26
48	<41	37
50	48	-69
52	58	81
54	<29	-49
	62*l*	
-56	<34	3
-54	<32	-4
-52	<36	36
-50	<39	-38
-48	<42	44
-46	<44	-33
-44	<46	18
-42	<48	-18
-40	<49	17
-38	<51	-14
-36	<52	42
-34	67	-48
-32	117	100
-30	186	-181
-28	200	-196
-26	172	158
-24	72	76
-22	<55	11
-20	<55	-2
-18	<55	55
-16	48	-64
-14	55	97
-12	56	-71
-10	56	70
-8	<55	-16
-6	<55	11
-4	<55	-6
-2	<55	17
0	60	-42
2	<55	33
4	<55	-28
6	<55	19
8	<55	2
10	<55	-41
12	<55	36
14	139	-119
16	112	-103
18	62	74
20	277	295
22	158	-146
24	<53	53
26	<52	-17
28	<52	-29
30	<50	27
32	<49	-23
34	<47	-1
36	<46	8
38	<44	-25
40	<41	5
42	<39	-9
44	<35	-23
46	<32	17
48	<32	-26
	72*l*	
-50	59	59
-48	38	37
-46	<32	-13
-44	<35	-8
-42	<38	24
-40	<40	-44
-38	<42	48
-36	<44	-40
-34	<45	18
-32	<47	10
-30	<48	-42
-28	<49	-10
-26	<50	-11
-24	<50	49
-22	<51	-39
-20	<52	37
-18	<52	-14
-16	<52	5
-14	<53	-1
-12	<53	-15
-10	<53	-20
-8	146	-116
-6	138	136
-4	141	128
-2	85	-96
0	<53	32
2	<53	-35
4	<53	13
6	<52	-14
8	<52	16
10	<52	-18
12	<51	7
14	<51	-1
16	<50	-52
18	<49	57
20	<48	-10
22	<47	33
24	<45	-34
26	<44	25
28	<42	-14
30	<40	-16
32	<38	17
34	<35	-37
36	<32	0
38	<35	23
40	92	102
	82*l*	
-38	<30	38
-36	<35	-32
-34	38	45
-32	33	21
-30	77	-67
-28	67	-71
-26	62	64
-24	<39	5
-22	<40	-6
-20	<41	21
-18	<42	-24
-16	<43	14
-14	<43	2
-12	<44	-3
-10	<44	39
-8	57	75
-6	79	-104
-4	<44	-20
-2	<44	-3
0	<44	19
2	<44	-29
4	<43	43
6	<43	-30
8	<42	33
10	<41	-6
12	<40	15
14	<39	-28
16	108	-101
18	103	105
20	<34	26
22	<32	-9
24	<36	5
26	<32	6
	92*l*	
-10	74	-49
-8	<29	-30
-6	47	57
-4	<30	-3
-2	<29	1
0	<28	-16
	03*l*	
1	17	-50
3	91	43
5	132	-130
7	213	186
9	166	-158
11	188	173
13	78	-66
15	94	83
17	<26	-4
19	78	75
21	292	-283
23	651	-667
25	413	417
27	180	180
29	<37	-76
31	200	220
33	127	-135
35	129	138
37	67	-61
39	<43	12
41	45	49

Table 1 (*cont.*)

l	F_o	F_c
43	97	−101
45	124	122
47	103	−128
49	89	91
51	61	−46
53	<40	32
55	<38	2
57	<36	−18
59	<34	26
61	57	−48
63	<27	23
65	69	−63
	13*l*	
−65	<39	0
−63	45	−30
−61	50	49
−59	68	−58
−57	46	44
−55	<39	−33
−53	<40	−17
−51	<42	3
−49	105	−111
−47	180	206
−45	158	188
−43	190	−213
−41	185	193
−39	211	−220
−37	146	149
−35	101	−98
−33	<41	−22
−31	64	52
−29	142	−143
−27	<37	11
−25	211	−228
−23	177	211
−21	<30	60
−19	85	−54
−17	116	104
−15	44	−5
−13	77	−82
−11	215	229
−9	310	−307
−7	383	376
−5	334	−299
−3	430	467
−1	389	383
1	480	−484
3	<17	−46
5	106	−129
7	69	−75
9	64	23
11	130	−130
13	48	11
15	<27	−13
17	152	−172
19	150	150
21	169	−135
23	415	437
25	340	−329
27	223	211
29	61	−65
31	71	−69
33	173	163
35	186	−180
37	179	172
39	84	−76
41	<44	58
43	149	153
45	203	−220
47	102	−136
49	<42	62
51	57	−51
53	93	77
55	89	−72
57	90	86
59	69	−64
61	55	58
63	<34	−27
	23*l*	
−65	<25	44
−63	52	−61
−61	<30	28
−59	<33	−27
−57	<35	−16
−55	<38	10
−53	58	−62
−51	<41	18
−49	173	−182
−47	253	263
−45	<44	15
−43	55	−60
−41	63	74
−39	118	−120
−37	88	85
−35	77	−87
−33	<42	−4
−31	89	87
−29	176	−173
−27	471	466
−25	62	76
−23	274	−258
−21	174	−167
−19	<32	18
−17	<31	−8
−15	104	−99
−13	135	147
−11	217	−212
−9	199	198
−7	175	−177
−5	69	64
−3	204	212
−1	244	256
1	169	−177
3	157	−164
5	136	139
7	185	−206
9	145	147
11	77	−78
13	66	58
15	76	79
17	<33	5
19	305	316
21	479	−491
23	277	−254
25	230	216
27	117	−130
29	150	152
31	69	−88
33	106	88
35	<43	−32
37	<44	29
39	<44	29
41	<44	17
43	161	171
45	177	−183
47	<42	−41
49	<40	8
51	<39	13
53	<37	−8
55	<34	32
57	<32	−5
59	<28	18
61	<41	−1
	33*l*	
−63	<36	21
−61	<37	4
−59	45	−38
−57	61	69
−55	79	−86
−53	73	70
−51	53	−69
−49	<41	−58
−47	167	170
−45	79	−80
−43	110	105
−41	94	−98
−39	74	69
−37	<44	−32
−35	<44	15
−33	<43	8
−31	<43	9
−29	<42	−10
−27	123	−127
−25	185	−193
−23	243	244
−21	93	−75
−19	<38	25
−17	111	117
−15	185	−171
−13	221	232
−11	193	−185
−9	113	123
−7	<34	−28
−5	99	−71
−3	258	228
−1	97	85
1	160	−147
3	109	114
5	212	−205
7	180	196
9	178	−183
11	87	79
13	<37	−16
15	128	−135
17	99	88
19	347	−327
21	399	389
23	79	−58
25	<42	4
27	81	65
29	95	−86
31	72	56
33	<44	−8
35	64	−58
37	106	102
39	123	−122
41	143	137
43	<42	−6
45	65	−67
47	<39	−27
49	<37	−40
51	53	57
53	84	−76
55	76	66
57	74	−62
59	<35	37
	43*l*	
−59	<41	10
−57	<28	20
−55	45	−51
−53	61	83
−51	219	−242
−49	<37	−34
−47	201	203
−45	<40	−41
−43	73	61
−41	<42	−31
−39	<43	5
−37	<44	16
−35	81	−76
−33	84	78
−31	135	−143
−29	61	68
−27	122	110
−25	112	112
−23	154	−151
−21	<42	13
−19	<42	−4
−17	58	−63
−15	69	68
−13	98	−102
−11	<40	42
−9	86	−82
−7	81	−79
−5	196	−158
−3	419	416
−1	178	172
1	225	−206
3	105	115
5	114	−99
7	<41	−12
9	<41	46
11	105	−116
13	130	141
15	133	−143
17	220	228
19	<43	37
21	202	−206
23	<44	15
25	70	−69
27	59	68
29	90	−87
31	71	68
33	72	−70
35	48	27
37	<42	−11
39	<41	−49
41	187	187
43	100	96
45	139	−134
47	63	−59
49	<31	44
51	55	−65
53	<35	37
55	<32	−9
	53*l*	
−55	<39	19
−53	<27	1
−51	<30	−40
−49	<32	20
−47	<34	1
−45	<36	41
−43	<38	−24
−41	<39	14
−39	<40	−2
−37	<41	7
−35	<42	1
−33	<43	37
−31	<43	−5
−29	97	99
−27	259	−265
−25	<44	24
−23	<44	45
−21	55	−47
−19	66	70
−17	50	−49
−15	<44	14
−13	<44	33
−11	66	−68
−9	72	77
−7	65	−67
−5	<43	−9
−3	130	147
−1	75	−73
1	122	133
3	171	−165
5	143	152
7	112	−100
9	<44	42
11	<44	35
13	82	−81
15	109	103
17	212	−212
19	<44	−35
21	144	157
23	<43	−40
25	<43	49
27	<42	−4
29	<42	−26
31	57	62
33	70	−77
35	79	83
37	70	−69
39	62	51
41	<33	−8
43	<31	34
45	<28	−30
47	<35	−26
49	<34	16
	63*l*	
−49	37	41
−47	44	48
−45	<40	17
−43	<31	2
−41	<33	14
−37	<36	−6
−35	<37	28
−33	<38	−33
−31	<39	35
−29	<40	−31
−27	<41	17
−25	<41	−47
−23	<42	33
−21	<42	−27
−19	<42	9
−17	<43	19
−15	<43	−17
−13	<43	39
−11	<43	−41
−9	<43	43
−7	246	−231
−5	<43	−63
−3	312	309
−1	<43	−40

Table 1 (*cont.*)

l	F_o	F_c
1	<43	15
3	<43	31
5	<43	−59
7	52	71
9	69	−79
11	57	72
13	71	−72
15	51	40
17	<41	−22
19	<40	−20
21	<40	32
23	<39	−29
25	<38	36
27	<37	−25
29	<35	24
31	<34	−20
33	<32	−4
35	<30	−6
37	49	−57
39	31	−40
41	160	167

73*l*

l	F_o	F_c
−41	<32	15
−39	<39	−4
−37	<36	−16
−35	<28	36
−33	<29	−37
−31	88	108
−29	<32	−25
−27	102	−105
−25	<34	4
−23	<35	11
−21	<36	−26
−19	41	44
−17	37	−60
−15	41	56
−13	<38	−52
−11	<38	28
−9	<38	−9
−7	<38	33
−5	<38	45
−3	77	−88
−1	76	88
1	60	−75
3	<38	38
5	<37	−4
7	<37	−18
9	<37	39
11	<36	−19
13	<35	28
15	<34	31
17	140	−158
19	<32	36
21	<31	22
23	<30	21
25	<28	6
27	<36	−3
29	<40	19
31	<33	−19

83*l*

l	F_o	F_c
−25	<39	31
−23	<44	−12
−21	<48	−16
−19	<51	29
−17	<43	−33
−15	<44	40
−13	<46	−19
−11	<46	38
−9	<47	19
−7	177	−146
−5	<47	56
−3	<47	37
−1	<47	−6
1	<46	14
3	<45	−21
5	<44	19
7	<52	−27
9	<49	33
11	<46	−27
13	<42	49

04*l*

l	F_o	F_c
0	72	101
2	72	91
4	76	−58
6	<31	−48
8	102	105
10	172	−166
12	199	176
14	180	−164
16	125	90
18	<33	−28
20	130	−118
22	142	123
24	168	−177
26	111	93
28	83	−80
30	106	98
32	79	−81
34	86	82
36	64	−49
38	<43	33
40	<42	1
42	<42	−9
44	75	80
46	<39	−21
48	<38	−20
50	<34	−24
52	<32	3
54	<42	0
56	<44	−14

14*l*

l	F_o	F_c
−58	68	70
−56	92	−92
−54	80	75
−52	63	−57
−50	<34	5
−48	57	−67
−46	65	−67
−44	168	190
−42	138	−129
−40	88	94
−38	<43	−25
−36	67	−55
−34	92	79
−32	165	−154
−30	101	84
−28	140	−144
−26	<40	−20
−24	643	659
−22	265	−240
−20	73	−88
−18	<39	−25
−16	115	−111
−14	105	84
−12	132	−121
−10	65	59
−8	48	−36
−6	47	−46
−4	<24	−12
−2	68	−60
0	181	227
2	49	59
4	192	−186
6	225	214
8	254	−243
10	170	153
12	81	−79
14	<32	−38
16	172	157
18	204	−164
20	542	538
22	<39	−77
24	474	−475
26	136	115
28	126	−118
30	64	57
32	<43	5
34	66	−58
36	118	112
38	138	−137
40	139	126
42	97	−94
44	120	125
46	<38	28
48	99	−116
50	86	78
52	78	−74
54	<38	37
56	<38	3

24*l*

l	F_o	F_c
−56	<40	−7
−54	<39	14
−52	45	−43
−50	51	44
−48	63	−74
−46	57	50
−44	<39	−19
−42	<41	38
−40	<42	−28
−38	<42	13
−36	<43	12
−34	49	−39
−32	83	88
−30	129	−109
−28	202	193
−26	180	−175
−24	61	55
−22	<40	−36
−20	56	−39
−18	57	47
−16	82	−69
−14	59	48
−12	36	−26
−10	<33	−12
−8	46	55
−6	86	−86
−4	76	105
−2	83	−76
0	131	101
2	40	60
4	134	−126
6	175	176
8	196	−184
10	172	181
12	118	−122
14	72	81
16	<38	−10
18	<39	−28
20	58	−55
22	<41	−17
24	53	50
26	82	−73
28	89	85
30	72	−69
32	56	56
34	45	−30
36	<42	19
38	<41	−4
40	<40	4
42	<39	5
44	<38	21
46	<34	−3
48	<32	−1
50	61	−48
52	70	57
54	61	−64

34*l*

l	F_o	F_c
−54	<39	−23
−52	75	67
−50	<30	−29
−48	229	−234
−46	113	113
−44	<38	37
−42	55	−50
−40	121	113
−38	111	−107
−36	101	93
−34	58	−54
−32	50	−42
−30	84	64
−28	291	−302
−26	245	232
−24	163	177
−22	<42	−36
−20	<42	−8
−18	<41	−26
−16	<41	−11
−14	<40	27
−12	52	−62
−10	72	71
−8	99	−92
−6	<39	19
−4	268	−290
−2	<39	23
0	379	393
2	247	−229
4	188	188
6	99	−84
8	41	−32
10	98	95
12	173	−171
14	142	138
16	149	−132
18	<42	37
20	376	403
22	267	−222
24	<43	−3
26	91	−82
28	<43	3
30	<42	22
32	90	−85
34	103	95
36	124	−125
38	84	75
40	118	−122
42	<34	18
44	204	210
46	76	−61
48	<38	−38
50	<38	27

44*l*

l	F_o	F_c
−50	<40	−39
−48	<39	30
−46	59	−59
−44	98	77
−42	67	−55
−40	39	32
−38	<38	2
−36	46	−41
−34	76	68
−32	94	−87
−30	105	90
−28	<42	−36
−26	<42	9
−24	<43	14
−22	<43	−30
−20	<43	26
−18	45	−28
−16	<43	−6
−14	<47	19
−12	73	−72
−10	94	88
−8	138	−135
−6	123	111
−4	83	−89
−2	<42	25
0	68	66
2	59	−51
4	68	67
6	67	−69
8	57	73
10	70	−63
12	69	67
14	65	−44
16	76	65
18	52	−40
20	<43	3
22	<42	13
24	<42	−30
26	<41	18
28	<40	−15
30	<39	−4
32	<38	6
34	<36	−16
36	<34	19
38	<33	−19
40	<31	29
42	<40	−6
44	<42	−4

54*l*

l	F_o	F_c
−46	85	99
−44	53	−62
−42	54	61
−40	<30	−18
−38	<32	4
−36	<33	35
−34	<35	−40
−32	47	53
−30	51	−50
−28	66	−61
−26	75	76
−24	<40	7

Table 1 (*cont.*)

l	F_o	F_c	l	F_o	F_c	l	F_o	F_c	l	F_o	F_c	l	F_o	F_c
-22	<41	18	12	<46	2	-34	<44	-19	0	<38	26	-20	51	-63
-20	<41	0	14	49	49	-32	<40	8	2	<38	2	-18	52	59
-18	<46	-36	16	158	-149	-30	<42	24	4	<38	-14	-16	54	-56
-16	72	70	18	159	170	-28	<31	5	6	<36	33	-14	<45	35
-14	86	-80	20	<39	-36	-26	<32	17	8	<36	-37	-12	<38	4
-12	95	111	22	54	52	-24	47	-47	10	49	42	-10	<38	-2
-10	50	-71	24	49	-48	-22	45	31	12	47	-29	-8	155	146
-8	105	114	26	<36	-9	-20	<35	-16	14	<34	31	-6	156	-163
-6	<54	-97	28	48	40	-18	<36	-11	16	<33	9	-4	100	-100
-4	360	-363	30	53	-55	-16	39	36	18	<32	-18	-2	100	98
-2	185	223	32	60	56	-14	49	-66	20	<31	-14	0	58	-49
0	<47	52	34	<41	-40	-12	70	74	22	<42	-3	2	<38	65
2	<47	-29	36	<45	6	-10	70	-84	24	<39	6	4	<45	-39
4	106	111	38	<38	-6	-8	<38	43	26	<44	4	6	<44	25
6	115	-102				-6	<38	-35	28	<38	-15	8	<42	-1
8	108	102		64l		-4	<38	1				10	<39	-20
10	63	-60	-36	<39	26	-2	<38	-12		74l		12	<34	40
									-22	<34	31			

of *n*-nonanoic acid hydrazide, Fig. 1, from three-dimensional photometric X-ray diffraction data.

Experimental

The compound was prepared by the method of Sah as outlined by Kyame, Fisher & Bickford (1947). The product was purified by recrystallization from 95% ethanol. Crystals were grown from an ethanol–water solution by slow evaporation of the solvent. From the mass of ill-defined plates, a crystal was selected and cut to size and shape with a regular histological microtome fitted with a razor blade. A cylindrical crystal with axis coincident with b and diameter about 0·15 mm. was used to collect zero and upper level unidimensionally integrated Weissenberg data. The upper levels were scaled to the zero level from several a axis Weissenberg photographs of another crystal.

The crystals are monoclinic with the following unit cell parameters:

$$a=7{\cdot}44,\ b=4{\cdot}87,\ c=58{\cdot}73\ \text{Å};\ \beta=95°\ 4'.$$

These parameters were determined from Weissenberg and oscillation photographs taken on a camera calibrated with NaCl ($a=5{\cdot}639$ Å). Remeasurement of d_{001} on a photograph with the NaCl pattern superimposed checked within 0·1%. It is reasonable, therefore, to expect errors in unit cell parameters not to exceed 0·3%.

Systematic extinctions (hkl, for $k+l$ odd and $h0l$, for h odd) indicate space group Aa or $A2/a$. Statistical treatment of the $h0l$ date of *n*-heptonoic acid hydrazide (Jensen & Lingafelter, 1953) by the method suggested by Howells, Philips & Rogers (1950) indicates that the space group is $A2/a$. This was assumed for *n*-nonanoic acid hydrazide and confirmed by the subsequent structure determination. With 8 molecules per cell the calculated density is 1·08 g.cm.$^{-3}$. The observed value is 1·07 g.cm.$^{-3}$.

Intensities were measured on unidimensionally integrated photographs with a recording microdensitometer by scanning at right angles to the direction of integration on the camera (Jensen, 1954). The area under the trace was taken as proportional to the integrated intensity. Statistical analysis of about two thousand measurements on other compounds by this method indicates an average mean deviation of about 2% in the F's due to the method of recording and measuring when observing 60–80% of the reflections for crystals of intermediate complexity.

In order to minimize systematic errors, great care was exercised in preparing the crystals and collecting the data. On each film, the range of intensity measurement was limited to assure operation on the linear response curve of the film used. Nevertheless, due to the difficulty of cutting really good cylindrical crystals for a axis photographs, errors in level-to-level scaling probably remain as the greatest single uncertainty.

On the zero through fourth level b axis Weissenberg photographs, 2104 reflections were accessible to Cu $K\alpha$ radiation. Of these, 1233 or 58·7% were observed.

Crystal disorder

Crystals of *n*-dodecanoic acid hydrazide were disordered in a way that could be explained as mistakes in the stacking of the molecular double layers (Jensen, 1956). No crystals of that compound were found which did not exhibit faulting although no extensive search was made. For *n*-nonanoic acid hydrazide, some crystals were obtained which were almost completely free of any disorder. It is to be expected that stacking faults would be less severe for the compounds with shorter chain lengths.

Refinement of the structure

The structure of *n*-heptanoic and *n*-octanoic acid hydrazide projected on (010) and *n*-nonanoic acid

Table 2. *Atomic parameters*

	x/a	y/b	z/c	$U_{11}\times10^2$	$U_{22}\times10^2$	$U_{33}\times10^2$	$U_{12}\times10^2$	$U_{13}\times10^2$	$U_{23}\times10^2$
O	0·6284	0·5727	0·2031	6·9 Å	3·3 Å	4·8 Å	0·00 Å	−0·03 Å	−0·33 Å
N_1	0·7000	0·1435	0·2144	5·6	3·2	2·9	0·00	−0·16	0·00
N_2	0·8053	0·2269	0·2345	6·2	4·2	3·5	0·00	−0·25	0·00
C_1	0·4801	0·2946	0·0266	10·9	10·9	4·8	0·00	0·40	0·00
C_2	0·4132	0·1856	0·0486	8·5	7·3	4·3	−0·31	0·37	0·00
C_3	0·5150	0·3053	0·0701	6·5	5·6	3·9	−0·24	0·34	0·00
C_4	0·4510	0·1913	0·0920	6·1	5·3	3·8	−0·21	0·33	0·00
C_5	0·5499	0·3094	0·1136	5·7	4·8	3·5	−0·24	0·31	0·00
C_6	0·4856	0·1941	0·1356	5·5	4·5	4·4	−0·28	0·28	0·00
C_7	0·5865	0·3153	0·1570	5·3	4·3	3·2	−0·28	0·27	0·00
C_8	0·5180	0·1999	0·1788	5·2	4·2	3·0	−0·28	0·26	0·00
C_9	0·6187	0·3199	0·1996	4·9	3·0	2·9	0·00	0·25	0·00
H_1	0·4069	0·2085	0·0111	—	—	—	—	—	—
H_2	0·4637	0·5191	0·0263	—	—	—	—	—	—
H_3	0·6239	0·2430	0·0265	—	—	—	—	—	—
H_4	0·2500	0·2320	0·0486	—	—	—	—	—	—
H_5	0·4236	−0·0190	0·0486	—	—	—	—	—	—
H_6	0·4956	0·5000	0·0701	—	—	—	—	—	—
H_7	0·6636	0·2750	0·0701	—	—	—	—	—	—
H_8	0·3036	0·2285	0·0920	—	—	—	—	—	—
H_9	0·4648	−0·0250	0·0920	—	—	—	—	—	—
H_{10}	0·5260	0·5250	0·1136	—	—	—	—	—	—
H_{11}	0·7028	0·2725	0·1136	—	—	—	—	—	—
H_{12}	0·3392	0·2420	0·1356	—	—	—	—	—	—
H_{13}	0·5028	−0·0290	0·1356	—	—	—	—	—	—
H_{14}	0·5784	0·5415	0·1570	—	—	—	—	—	—
H_{15}	0·7328	0·2745	0·1570	—	—	—	—	—	—
H_{16}	0·3808	0·2525	0·1788	—	—	—	—	—	—
H_{17}	0·5384	−0·0260	0·1788	—	—	—	—	—	—
H_{18}	0·6850	−0·0750	0·2109	—	—	—	—	—	—
H_{19}	0·9220	0·2750	0·2305	—	—	—	—	—	—
H_{20}	0·7280	0·3500	0·2420	—	—	—	—	—	—

hydrazide projected along [100] have already been reported (Jensen & Lingafelter, 1953). Beginning coordinates used in the present work were derived from the earlier results.

Table 3. *Bond lengths and angles**

C_1–C_2	1·521 Å	C_1–C_2–C_3	113·1°
C_2–C_3	1·529	C_2–C_3–C_4	113·0
C_3–C_4	1·516	C_3–C_4–C_5	113·8
C_4–C_5	1·522	C_4–C_5–C_6	113·7
C_5–C_6	1·524	C_5–C_6–C_7	112·8
C_6–C_7	1·525	C_6–C_7–C_8	111·9
C_7–C_8	1·526	C_7–C_8–C_9	111·2
C_8–C_9	1·494	C_8–C_9–O	122·5
C_9–O	1·249	C_8–C_9–N_1	116·6
C_{12}–N_1	1·329	C_9–N_1–N_2	123·0
N_1–N_2	1·415	O–C_9–N_1	120·9
N_1–$H_{18}\cdots O'$	2·896	N_1–H_{18}–O'	171·7
N_2–$H_{20}\cdots N_2''$	3·188	N_2–H_{20}–N_2''	149·7
C_2–H_4	1·235		
C_2–H_5	0·999		
C_3–H_6	0·959		
C_3–H_7	1·115		
C_4–H_8	1·112		
C_4–H_9	1·058		
C_5–H_{10}	1·065		
C_5–H_{11}	1·152		
C_6–H_{12}	1·114		
C_6–H_{13}	1·094		
C_7–H_{14}	1·103		
C_7–H_{15}	1·106		
C_8–H_{16}	1·052		
C_8–H_{17}	1·111		

* Calculated from coordinates uncorrected for oscillation.

The projections of the structure on (010) and along [100] were first refined by F_o and ΔF syntheses (Cochran, 1951). By including H atoms and individual atom anisotropic thermal parameters, the reliability indices, R, were brought to 6·7% and 6·4% for $0kl$ and $h0l$ reflections respectively.

Coordinates from the refinement in projection were used as the starting point for the refinement by three-dimensional syntheses. In the first such refinement cycle, H atoms were omitted in calculating F_c and individual atom isotropic thermal parameters were used. The thermal parameter for each atom was taken as equal to that for the minimum thermal motion for that atom as determined from the two-dimensional data. For this calculation, R stood at 19·3%.

Three additional refinement cycles including H atoms and individual atom anisotropic thermal parameters brought R to 7·5% for the 1226 observed reflections considered not to suffer appreciably from secondary extinction. The ΔF synthesis from which final changes in the atomic parameters were taken is shown in Fig. 2. In Table 1, F_o and F_c are listed for the unique reflections within that part of the sphere of reflection examined. Final atomic parameters are listed in Table 2 and the bond lengths and angles derived from them in Table 3.

Coordinates for H_{1-17} were calculated on the basis of final coordinates for C_{1-8}. It was assumed that the bonds about each C atom are tetrahedrally arranged with a C–H bond length of 1·1 Å and that the chain is planar with H_1 lying in that plane. Coordinates for H_{18-20} were taken from a ΔF synthesis showing the H atoms, Fig. 3. To minimize the influence of inaccuracies in the thermal parameters, only ΔF for reflections with $\sin\theta/\lambda < 0·48$ were used in calculating this synthesis. For the 1226 observed reflections not appreciably affected by secondary extinction, R = 11·0% when H atoms are omitted.

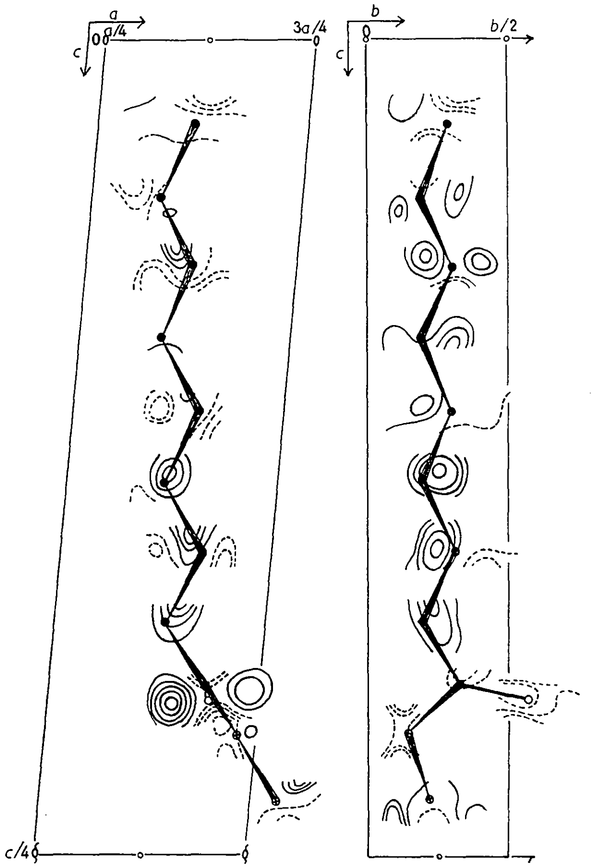


Fig. 2. Composite ΔF synthesis from which final corrections were taken. Contour interval 0·05 e.Å^{-3}. Positive contours solid, negative contours broken and zero contour omitted.

Thermal parameters were estimated by interpolation or extrapolation from the values used in successive pairs of ΔF syntheses. For this method, the sensitivity in root mean square amplitude of vibration is less than 0·003 Å at an amplitude of 0·23 Å. This corresponds to 0·1 Å^2 in B at B = 4·0 Å^2. A ΔF synthesis showing the difference of thermal motion from that corresponding to B = 4·0 Å^2 is shown in Fig. 4. For the 1226 observed reflections not appreciably effected by secondary extinction, R = 16·0% when calculated including H atoms and an overall isotropic temperature factor with B = 4·0 Å^2.

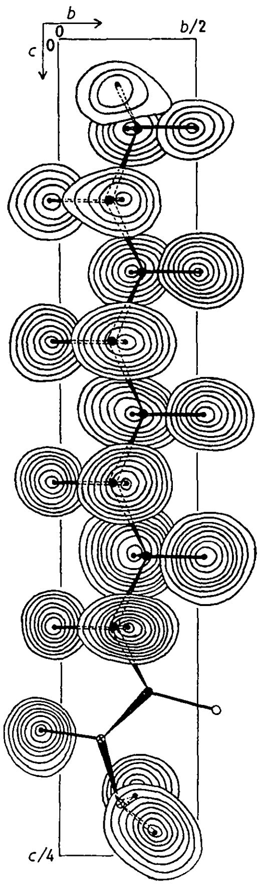


Fig. 3. Composite ΔF synthesis showing H atoms. Contours at intervals of 0·05 e.Å^{-3} beginning at 0·15 e.Å^{-3}. Synthesis calculated using observed F only with $\sin\theta/\lambda < 0·48$.

Precision of the results

For 7 of the 1233 observed reflections, secondary extinction is sufficiently severe that they were coded as extinguished and given zero weight in all ΔF syntheses and included as F_c in F_o syntheses. From the 1226 remaining reflections, 1 scale, 36 positional and 42 thermal parameters were evaluated. Thus, there are 16 observations per parameter.

In order to evaluate standard deviations of the coordinates, it is necessary to know the curvature of the electron density, ϱ, for each atom in the direction of each coordinate (Cruickshank, 1949). In *n*-nonanoic acid hydrazide, not only do curvatures vary appreciably for different atoms of the same type, but also for the same atom in different directions. For such structures, the calculation of exact standard deviations in bond lengths although straight-forward is a matter of some complexity.

Sufficiently good values of the standard deviations in the bond lengths may be obtained by using 'average' curvatures calculated by assuming ϱ to be given by the equation $\varrho = \varrho_0 \exp[-pr^2]$ (Costain, 1941)

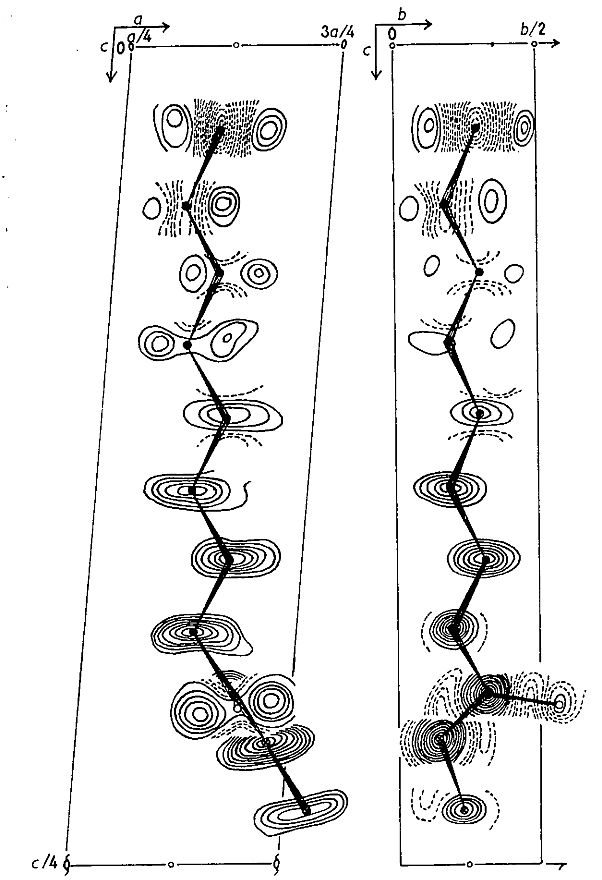


Fig. 4. Composite ΔF synthesis showing difference of electron density from that for spherical atoms with a temperature factor exp $-[4{\cdot}0 \sin^2 \theta/\lambda^2]$. Contour interval 0·1 e.Å^{-3}. Positive contours solid, negative contours broken and zero contour omitted.

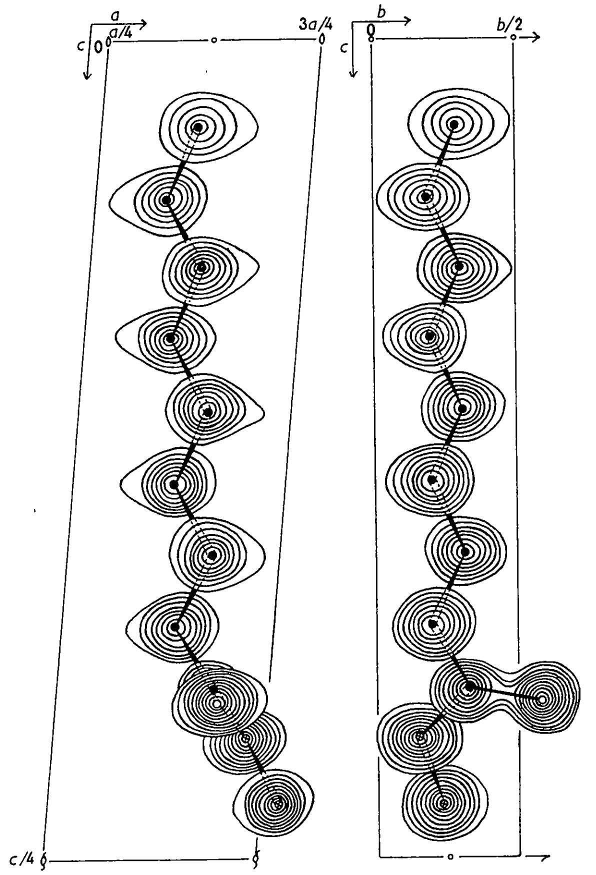


Fig. 5. Composite F_o synthesis. Contour interval 1·0 e.Å^{-3}, zero contour omitted.

where ϱ_0 is the peak electron density, r is the distance from the atomic center and ϱ is assumed to be independent of direction for each atom and a constant for each atom type. Table 4 lists the peak densities taken from the F_o synthesis, Fig. 5, together with calculated values of p and central curvatures for each atom type (Booth, 1946).

For this and many other organic structures, average standard deviations in the coordinates calculated in this way give rise to standard deviations in the bond lengths which are larger than those given by the exact calculations. This follows from the observation that within a molecule, maximum curvature in the electron density tends to be in the bond direction and minimum curvature at right angles to it. Standard deviations for the bonds between the various types of atoms are given in Table 5.

Table 4. *Central densities and curvatures*

Atom type	Central density	p	Curvature	$\sigma_{\text{positional}}$
O	10·3 Å	3·70Å^{-2}	$-59{\cdot}3$ e.Å^{-5}	0·0026 Å
N	9·3	3·80	$-53{\cdot}3$	0·0029
C	7·2	3·52	$-42{\cdot}3$	0·0037
H	0·5	1·98	$-4{\cdot}0$	0·039

It is clear by inspecting the bond lengths in Table 2 that, except for the short C_8–C_9 bond, the C–C bonds in the paraffin chain are essentially of equal length. It is reasonable, therefore, to calculate an average C–C bond length and to take this as the best value. Remembering that the errors in bond lengths are not independent of one another, it can be shown that the standard deviation in the mean of the 7 'equal' C–C bond lengths, $\sigma_{\text{mean C-C}}$, is related to $\sigma_{\text{C-C}}$ by the following equation:

$$\sigma_{\text{mean C-C}} = (\sigma_{\text{C-C}}/7)(6{\cdot}2^2 \cos^2 56{\cdot}5^\circ + 2)^{\frac{1}{2}} = 0{\cdot}0023 \text{ Å}\,.$$

For centrosymmetric structures, H atom positions as determined from ΔF syntheses are essentially independent of the positions assumed in calculating their contribution to the structure factors. In the present case, x and y coordinates were taken from a ΔF synthesis. But due to the coarseness along c of the grid at which $\Delta\varrho$ was evaluated, the z coordinates for H_4–H_{17} atoms were taken equal to that of the C atom to which each is bonded. The lengths of the 14 methylene C–H bonds based on these H atom coordinates range from 0·959–1·235 Å, mean = 1·091 Å, $\sigma_{\text{mean}} = 0{\cdot}018$ Å. This is to be compared with $\sigma_{\text{mean C–H}} = \sigma_{\text{C–H}}/\sqrt{(14)} =$ 0·010 Å calculated from the standard deviations of Table 5 (neglecting the small dependence of C–H bond lengths for H atoms tetrahedrally bonded to the same C atom). The former value is probably a more realistic index of the accuracy since it includes in a direct way the effects on H atom positions of inaccuracies in the heavy atom parameters. Indeed, it is to be expected that when calculated from $\Sigma h_i^2 \Delta F^2$, $\sigma_{\text{positional}}$ for light atoms in the presence of heavy atoms will be too small.

Table 5. *Standard deviations of bonds*

Bond	σ	Bond	σ
C–O	0·0045 Å	N–N	0·0041 Å
C–N	0·0047	C–H	0·039
C–C	0·0052	N–H	0·039

Any precision index is based on the fluctuations of the experimentally observed (or derived) quantities and is, therefore, independent of systematic errors. Thus, precision indices are a lower limit to the error to be expected at a given confidence level. Only in the absence of systematic errors is accuracy equal to precision.

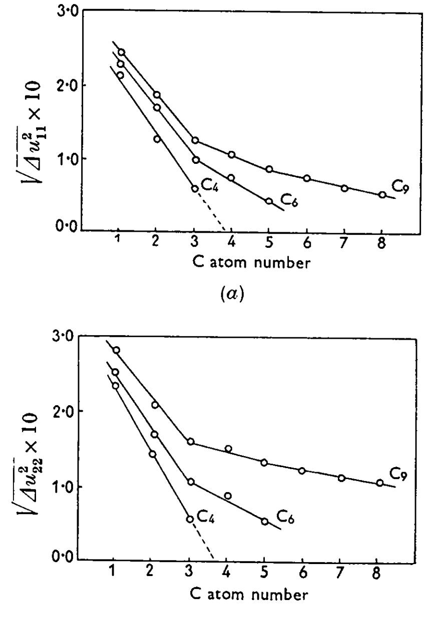
Systematic errors in the observations may enter by way of errors in the apparatus, crystal aberrations or failure of the laws relating the actual quantities measured (optical densities in the present instance) to the structure factors. Not only may systematic errors enter through the observations, but also directly through the method of data processing or indirectly through the failure of the model assumed to fit the physical situation.

The model used in calculating structure factors assumes the electron distribution to be represented by the scattering factors as modified by a general Gaussian function. If the molecule is librating about an axis, this is not correct (Cruickshank, 1956) and bonds with components perpendicular to the axis of rotation will appear shortened.

In the case of long chain molecules, libration about the chain axis would result in shortening the bonds within the chain (Cruickshank, 1959). Although for *n*-nonanoic acid hydrazide it is not possible to distinguish between libration and simple anisotropic motion, we may assume anisotropy of the chain atoms in the *xy* plane to be due to libration and calculate an upper limit for the correction. It turns out to be small, about +0·002 Å in bond length, because the direction of maximum vibration (8° from (010)) is almost 40° from the normal to the plane of the molecule.

Since molecules are not rigid bodies, oscillation of parts of the molecule, particularly terminal atoms or groups of atoms, must not be neglected. In general, however, it is not possible to know with certainty the position of the axes about which such oscillations occur.

If the chain behaved as a rigid body, oscillating about axes parallel to *y* through C_9, a plot of $(u^2_{11C_n} - u^2_{11C_9})^{\frac{1}{2}}$ versus *n*, the number of the C atom in the chain, should be linear; and likewise for an axis parallel to *x* when $(u^2_{22C_n} - u^2_{22C_9})^{\frac{1}{2}}$ is plotted versus *n*. Fig. 6 shows such a plot for axes through C_9, C_6 and C_4. The plot shows that for oscillations perpendicular to the chain axis, the molecule does not vibrate as a rigid body. Approximately, however, C_1, C_2 and C_3 appear to oscillate as a segment about axes close to C_4.



(*a*)

(*b*)

Fig. 6. Square root of Δ m.s. amplitudes of vibration as a function of the number of the C atom in the chain. (*a*) About an axis parallel to *b*, (*b*) about an axis almost parallel to *a*. In both are plots for axes at the level of C_9, C_6 and C_4.

Only for these atoms are corrections appreciable, resulting in an average +0·003 Å for the C_1–C_2, C_2–C_3 and C_3–C_4 bonds.

That the corrections considered here may be relatively large is evident in the case of the C–O bond. By assuming oscillation of the O atom about axes through C_9, the correction to the bond length is +0·014 Å or more than 3σ. Much smaller corrections in the lengths of the C_9–N_1 and N_1–N_2 bonds are to be expected but have not been made because of uncertainty in the position of the axes of oscillation.

It must be emphasized that in *n*-nonanoic acid hydrazide the exact corrections to be made for rotary oscillation of the molecule or its parts are questionable. Nevertheless, their possible magnitudes, as indicated above, is a clear indication that in accurate work *they must be considered.* Collection of diffraction data at sufficiently low temperature would greatly reduce the effects. At liquid nitrogen temperature, for example, errors due to oscillation would be reduced by a factor of about 4, and in addition, precision would be increased by a factor of about 2. Thus for organic structures in the absence of heavy atoms, we could expect standard deviations in the bond lengths involving O, N and C atoms of about 0·002 Å.

Comparison of the structures of the nonanoic and dodecanoic acid hydrazides shows them to be very similar, and the several slight differences between them do not appear to be due to the fact that one has an odd number and the other an even number of carbon atoms. Thus the lack of alternation in the melting points of the homologous series is understandable. Indeed, Malkin pointed out as early as 1931 that alternation is not to be expected when the chains are normal to (001).

The differences between the two structures, although slight, are significantly larger than experimental errors. As indicated in Fig. 1 of a previous article (Jensen, 1956), the terminal hydrogen atoms in 'opposing' layers in the (001) plane in both compounds approximate a closest-packing arrangement, with hydrogen atoms of one layer fitting into nearly equilateral triangles of hydrogen atoms of the other layer. The H–H distances are significantly larger in the dodecanoic than in the nonanoic acid hydrazide. The separation normal to (001) between pairs of equivalent atoms is 0·14 Å greater in the dodecanoic than in the nonanoic acid hydrazide. This is probably a consequence of the larger thermal motion of the terminal atoms in the longer chain of the dodecanoic acid hydrazide.

The plane of the chain also has a slightly different orientation in the two compounds. The angle between the *a*-axis and the line of intersection of the chain plane with (001) is 41° 30′ for the nonanoic and 45° 30′ for the dodecanoic acid hydrazide. These values may be compared with the angle of 41° 23′ reported by Shearer & Vand (1956) for hexatriacontane. Furthermore, although the chain plane is almost exactly normal to (001) in the dodecanoic acid hydrazide (89° 49′), the angle between the chain plane and (001) is decreased to 86° 23′ in the nonanoic acid hydrazide.

Neglecting the C_8–C_9 bond, C–C bond lengths in the chain range from 1·521–1·535 Å (corrected), average $1{\cdot}526 \pm 0{\cdot}0024$ Å. If only non-systematic errors are present, the probability is 0·01 that the true value for the average would be outside the range $2{\cdot}3\sigma = 0{\cdot}0055$ Å. This indicates that the $0{\cdot}0185 = 8\sigma$ difference from the 1·5445 Å value for the C–C bonds in diamond is significant.

The average C–C bond length in *n*-hexatriacontane as determined from X-ray diffraction is $1{\cdot}534 \pm 0{\cdot}006$ Å (Shearer & Vand, 1956). The C–C bond length in gaseous *n*-butane, *n*-pentane, *n*-hexane and *n*-heptane as determined by electron diffraction are respectively

$$1{\cdot}533 \pm 0{\cdot}003,\ 1{\cdot}531 \pm 0{\cdot}002,\ 1{\cdot}533 \pm 0{\cdot}003,\ 1{\cdot}534 \pm 0{\cdot}003\ \text{Å}$$

(Bonham & Bartell, 1959; Bonham, Bartell & Kohl, 1959). If the precision indices quoted are standard deviations, there is no significant difference between these results and the average value from the present work.

The present results for the average C–C bond length differ from the $1{\cdot}550 \pm 0{\cdot}0037$ Å value in *n*-dodecanoic acid hydrazide by more than twice what would be significant at the 1% level. Comparison of the coordinates for the two structures indicates that the crux of the difficulty is in the *y* coordinates. And it is just these that were determined from incomplete data for *n*-dodecanoic acid hydrazide although allowance was made for this in calculating σ_y. The $02l$ and $04l$ reflections (except 020 and 040) were taken from second and fourth level *b* axis Weissenberg photographs. Due to difficulty in cutting satisfactory crystals for *a* axis photographs, the $0kl$ reflections were not scaled experimentally but against F_c in which thermal motion along *b* was assumed to be the same as that along *a*. The possibility that the difficulty lay in the scaling of the $0kl$ reflections was tested for *n*-nonanoic acid hydrazide by calculating three ΔF syntheses: (1) using $0kl$ reflections with k even and no rescaling from final two-dimensional calculations, (2) the same except $02l$ reflections rescaled by 0·9, (3) the same except $04l$ reflections rescaled by 0·9. The results show definitely that incorrect scaling of this type does shift the *y* coordinate and in a direction consistent with the difference found.

The C_8–C_9, C_9–N_1, C_9–O and N_1–N_2 bond lengths are all within experimental error of those for corresponding bonds in the other hydrazides studied in this laboratory (Jensen, 1954, 1956) and compare well with those in *cyclo*propanecarbohydrazide (Chesnut & Marsh, 1958). The significant difference between the lengths of the N–N bonds in the hydrazides and in hydrazine (Collin & Lipscomb, 1951; Giguere & Schomaker, 1943) is confirmed.

The length of the N–H · · · N bond is 3·19 Å, nearly the same as the corresponding bonds in *n*-dodecanoic acid hydrazide and *cyclo*propanecarbohydrazide but considerably longer than the 2·97 Å and 3·04 Å N–H · · · N bonds in isonicotinic acid hydrazide and the 2·92 Å N–H · · · N bond in creatinine (Du Pré & Mendel, 1955). The N–H · · · O bond length is 2·89 Å, appreciably longer than similar bonds in many other compounds (Mendel & Hodgkin, 1954; Pasternak, Katz & Corey, 1954) although shorter than the corresponding bonds in *n*-dodecanoic acid hydrazide and *cyclo*propanecarbohydrazide.

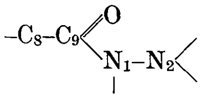
The C–C–C bond angles within the chain average about 3° larger than corresponding angles in *n*-dodecanoic acid hydrazide. The difference is due to the same systematic error in the *y* coordinates that resulted in the long C–C bonds. Angles about C_9 and N_1 appear to differ significantly from 120° and agree well with the corresponding angles in isonicotinic acid hydrazide and *cyclo*propanecarbohydrazide but again are appreciably different from those in *n*-dodecanoic acid hydrazide.

Table 6. *Shortest intermolecular distances*

Atom	Shortest distance	Atom involved	Relationship
O	2·896 Å	N_1	Trans. equivalent
N_1	2·896	O	Trans. equivalent
N_2	3·188	N_2	Trans. equivalent
C_1	3·744	C_1	Across sym. center
C_2	4·092	C_3	Glide equivalent
C_3	4·092	C_2	Glide equivalent
C_4	4·135	C_5	Glide equivalent
C_5	4·135	C_4	Glide equivalent
C_6	4·094	C_7	Glide equivalent
C_7	4·094	C_6	Glide equivalent
C_8	3·440	O	Trans. equivalent
C_9	3·640	O	Trans. equivalent

If C_1 and C_9 are neglected, the least-squares plane through the remaining seven atoms in the chain is $-0{\cdot}5435x+0{\cdot}5948y+0{\cdot}0509z+1=0$ referred to *a*, *b* and *c** as coordinate axes. No chain atom, except C_1, deviates significantly from this plane. But its deviation of $-0{\cdot}022$ Å, while not great, is almost twice what would be significant at the 1% level considering its relatively large thermal motion perpendicular to the plane of the chain. It is remarkable that packing at the ends of the molecule is such that C_9 is within experimental error of and C_1 is so close to the plane determined by the other chain atoms.

The



$$-\mathrm{C_8}-\mathrm{C_9}(=\mathrm{O})-\underset{|}{\mathrm{N_1}}-\mathrm{N_2}<$$

group is very nearly planar. C_8 does not deviate significantly from the plane determined by OC_9N_1 but N_2 does, lying $+0{\cdot}029$ Å $=8\sigma$ from it. This plane makes an angle of 56° 2′ with that of the chain.

In Table 6 are listed the distances of closest intermolecular approach for each atom other than hydrogen in the molecule. There are no abnormally short distances.

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