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Crystal Structure of Cycloundecanone at —165°C

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The crystals are monoclinic with space group $P2_1/c$, cell dimensions $a = 7.305(2)$ Å, $b = 5.600(2)$ Å, $c = 24.566(7)$ Å, $\beta = 91.29(2)°$, and four molecules in the unit cell. The structure was solved by direct methods and refined by full-matrix least squares technique to an R-value of 4.8 % (R_w = 5.7 %) for 1416 reflections recorded on an automatic four circle diffractometer. The conformation may be described as "triangular" with the carbonyl group in an asymmetric position.

Very little structural information for the eleven- and higher odd-membered saturated rings is available. To the best of the authors' knowledge, an X-ray analysis of cyclodecylamine hydrobromide [1] has not led to a clear picture of the conformation. Strain-minimization calculations of medium and large cycloalkanes have been carried out for a period of more than ten years,[2-5] and it seems to be need for detailed structure information.

Cycloundecanone was synthesized (by T. Ledaal at this university) from *cis* 2,12-dibromocyclododecanone according to the procedure of Garbisch and Wohllebe,[6] and redistilled under reduced pressure (b.p. 111 – 113° (9 mm), m.p. 15°C). Identification was performed by the aid of high resolution NMR and IR-spectra.

The crystals are monoclinic with space group $P2_1/c$, cell dimensions $a = 7.305(2)$ Å, $b = 5.600(2)$ Å, $c = 24.566(7)$ Å, $\beta = 91.29(2)°$, and four molecules in the unit cell. The intensities were measured (at −165°C) on a Syntex $P\bar{1}$ diffractometer with Enraf-Nonius liquid nitrogen cooling device (modified by H. Hope). With an

Table 1. Final fractional coordinates and anisotropic thermal vibration parameters with estimated standard deviations (multiplied by 10^5 for oxygen and carbon atoms, and 10^4 for hydrogens). Hn1 are Hn2 bonded to Cn.

ATOM	X	Y	Z	B	B11	B22	B33	B12	B13	B23
O	79863(25)	18887(38)	28445(7)		1989(44)	1343(59)	107(3)	-298(82)	215(18)	88(22)
C1	83867(32)	31136(41)	19583(8)		1212(50)	1382(62)	47(4)	-172(104)	-53(21)	-8(30)
C2	102386(33)	36682(43)	17114(10)		1073(50)	1463(62)	74(4)	-177(106)	-198(23)	-62(32)
C3	110263(34)	15812(46)	13971(10)		1861(54)	1658(67)	88(4)	488(111)	-78(24)	-48(33)
C4	98142(34)	6645(43)	9192(9)		969(50)	1458(61)	79(4)	95(111)	51(23)	-58(32)
C5	94533(33)	25325(45)	4714(9)		1816(48)	1652(66)	63(4)	-103(106)	63(22)	-16(29)
C6	74731(33)	25232(46)	2425(10)		1284(51)	1756(69)	63(4)	-122(114)	-33(22)	11(32)
C7	68256(34)	31995(43)	6598(10)		988(50)	1637(65)	73(4)	-169(107)	-65(22)	39(33)
C8	62853(34)	57488(43)	8753(10)		982(50)	1584(61)	66(4)	-57(104)	-37(22)	130(32)
C9	49258(34)	63929(45)	13348(10)		995(51)	1485(66)	106(4)	121(106)	18(23)	-88(31)
C10	52165(34)	58364(48)	18665(10)		1894(51)	1847(91)	87(4)	26(113)	121(24)	-171(33)
C11	71782(34)	51912(47)	21185(10)		1357(54)	1388(86)	78(4)	-631(110)	9(24)	-126(31)
H21	18996(32)	3955(42)	2816(10)	1,9(,5)						
H22	10169(30)	4958(46)	1478(10)	2,8(,6)						
H31	11231(31)	212(45)	1652(10)	2,8(,6)						
H32	12256(33)	2698(42)	1251(9)	2,8(,6)						
H41	8628(32)	223(46)	1869(9)	1,6(,6)						
H42	10428(28)	-797(40)	757(8)	1,1(,4)						
H51	10293(29)	2266(48)	181(9)	1,2(,6)						
H52	9718(29)	4191(43)	618(9)	1,8(,5)						
H61	7139(32)	845(46)	186(10)	2,3(,6)						
H62	7379(30)	3596(43)	-92(10)	2,1(,5)						
H71	6151(29)	2889(42)	966(9)	1,6(,5)						
H72	4778(34)	3218(43)	485(10)	2,3(,6)						
H81	5957(31)	6989(44)	558(10)	2,1(,6)						
H82	7468(31)	6802(38)	1803(9)	1,3(,5)						
H91	3616(34)	6188(42)	1285(9)	2,2(,5)						
H92	5141(31)	8179(46)	1415(10)	2,2(,5)						
H101	4937(29)	3285(42)	1814(9)	1,3(,5)						
H102	4341(35)	5635(47)	2137(11)	2,9(,6)						
H111	7115(30)	5031(44)	2498(10)	2,2(,5)						
H112	7785(31)	6655(47)	2928(9)	1,8(,5)						

Table 2. Observed and calculated structure factors on 10 times absolute scale.

Hk	0,Kk	0	16	51	36	19	76	77	16	24	26	-26	113	111	3	181	146	-13	27	14	8	65	70
2	173	173	18	26	29	20	155	154	17	106	113	-24	50	50	4	47	46	-12	38	37	6	126	131
4	281	296	19	69	63	21	151	150	18	69	66	-22	257	257	5	157	153	- 8	117	123	8	314	323
6	154	155	21	49	37	22	120	122	20	90	90	-20	53	52	6	79	69	- 7	35	34	9	59	54
8	628	630	Hk	0,Kk	5	23	30	36	21	34	22	-18	167	157	7	134	134	- 6	62	60	10	67	70
10	379	375	1	71	74	24	164	161	22	34	31	-16	215	223	8	33	35	- 5	82	88	11	143	139
12	280	286	2	51	43	25	51	64	23	86	81	-14	41	44	9	199	201	- 4	38	43	12	74	71
14	171	172	3	108	109	26	96	91	25	43	41	-12	79	80	10	168	165	- 3	87	95	13	57	57
16	277	278	4	171	165	28	40	40	Hk	1,Kk	4	-10	579	566	12	39	43	- 2	104	100	14	83	84
20	95	95	5	207	202	Hk	1,Kk	2	-22	50	50	- 8	97	93	14	89	83	- 1	80	87	16	222	227
22	46	42	6	27	27	-27	98	87	-20	47	42	- 6	231	232	17	29	28	0	148	152	17	83	86
24	48	46	7	203	205	-26	97	79	-19	56	58	- 4	478	462	18	83	81	1	51	54	18	69	67
26	177	170	8	67	72	-25	25	14	-17	114	121	- 2	692	525	19	156	161	2	21	1	19	61	65
28	60	54	10	121	125	-24	95	91	-15	74	76	0	353	361	21	38	37	3	45	40	20	43	43
Hk	0,Kk	1	12	48	48	-23	101	95	-13	43	46	2	102	98	23	130	131	4	97	100	21	132	135
1	281	289	13	97	94	-21	40	38	-12	76	87	4	912	893	24	134	125	5	133	135	22	95	91
2	655	655	15	147	136	-20	54	59	-11	117	125	6	201	207	26	43	44	6	25	24	23	64	66
3	324	335	16	47	41	-19	38	39	-10	187	191	8	182	173	Hk	2,Kk	3	7	41	44	24	76	80
5	270	277	17	44	51	-18	120	120	- 7	136	139	10	211	213	-25	31	15	8	86	88	25	29	20
6	699	705	19	41	36	-17	119	123	- 6	118	125	12	456	454	-24	39	25	9	129	125	26	63	66
7	195	199	Hk	0,Kk	6	-16	91	86	- 4	64	60	14	330	328	-23	58	53	10	88	84	Hk	3,Kk	2
9	203	214	1	42	39	-15	31	34	- 3	152	154	16	42	49	-22	41	43	11	119	121	-24	59	60
9	153	161	2	142	140	-14	97	98	- 2	133	137	18	44	49	-21	76	80	12	58	60	-23	104	103
10	54	51	4	48	50	-13	99	102	- 1	164	169	20	49	45	-19	201	204	13	56	47	-21	24	26
11	71	72	6	98	94	-12	163	172	0	53	45	22	356	355	-17	76	74	14	124	128	-20	128	131
12	126	127	7	32	18	-11	95	99	1	126	131	24	78	79	-16	69	66	15	30	37	-19	124	129
13	55	55	8	30	21	- 9	151	151	2	208	211	26	79	72	-14	239	243	16	51	45	-18	146	150
14	110	111	9	38	43	- 8	396	396	3	39	49	28	48	52	-13	63	69	17	72	67	-16	140	141
15	62	59	10	31	33	- 7	331	329	4	34	45	Hk	2,Kk	1	-12	201	201	Hk	2,Kk	6	-15	162	164
16	52	52	11	45	47	- 6	62	62	5	104	108	-28	38	37	-11	205	208	-11	27	24	-14	65	60
17	110	112	12	36	31	- 5	271	275	6	93	95	-26	91	87	-10	103	113	- 9	106	104	-13	168	173
18	37	36	Hk	1,Kk	0	- 4	16	18	7	125	124	-25	59	56	- 8	224	218	- 8	50	53	-11	197	203
19	76	76	-28	31	26	- 3	43	39	8	215	213	-24	173	170	- 6	117	110	- 7	92	93	-10	77	78
20	33	37	-26	97	86	- 2	104	100	9	69	70	-23	78	68	- 4	40	36	- 6	136	131	- 9	160	164
21	47	49	-24	171	164	- 1	104	103	11	46	45	-20	132	135	- 3	40	48	- 5	62	56	- 7	61	65
22	73	73	-20	61	59	0	158	167	12	77	84	-19	90	98	- 2	34	39	- 1	33	26	- 6	25	25
23	44	42	-18	224	222	1	158	151	13	93	86	-18	76	78	- 1	52	38	0	83	46	- 5	34	42
24	43	44	-16	426	427	2	166	159	14	64	72	-17	86	91	0	108	68	1	42	45	- 4	79	82
28	72	69	-12	244	244	3	146	146	15	142	137	-16	285	287	1	224	221	2	66	63	- 3	177	176
Hk	0,Kk	2	-10	80	85	4	349	338	17	55	48	-15	164	169	2	45	21	3	65	66	- 2	23	19
0	45	47	- 8	379	375	6	318	317	18	32	29	-14	47	48	3	133	129	5	68	72	- 1	191	188
1	358	340	- 6	757	742	7	155	166	19	58	51	-13	71	73	4	110	106	6	69	71	0	75	74
2	485	461	- 4	84	80	8	101	99	20	32	33	-12	182	188	5	146	144	8	48	36	1	57	55
3	178	171	- 2	585	572	9	130	131	21	38	25	- 9	485	484	6	73	79	9	57	60	2	69	75
4	128	127	0	178	178	10	28	26	23	33	33	- 8	172	162	7	83	85	10	33	34	3	74	71
5	118	123	2	564	563	11	123	124	Hk	1,Kk	5	- 7	62	60	8	39	44	Hk	3,Kk	0	4	61	63
6	172	171	4	18	12	12	41	44	-19	34	30	- 5	491	482	9	52	57	-26	121	122	5	247	250
7	371	359	6	967	1071	13	39	39	-18	102	87	- 4	384	366	10	168	169	-24	75	63	6	239	230
8	37	37	8	513	510	14	101	103	-17	138	138	- 3	219	207	11	48	45	-22	38	40	7	133	128
10	279	351	10	22	13	15	114	111	-16	57	59	- 2	60	50	12	29	26	-20	33	33	8	157	151
11	276	291	12	286	274	16	22	28	-15	25	19	- 1	261	262	13	34	26	-18	119	119	9	112	111
12	163	163	14	209	205	17	114	111	-13	127	127	0	35	36	14	131	135	-16	24	26	10	110	108
13	142	144	16	55	62	18	45	47	-11	106	109	1	104	101	15	25	19	-14	39	42	11	105	108
14	53	57	18	90	95	19	133	137	-10	138	144	2	103	103	16	73	73	-12	397	399	12	158	166
15	240	242	20	206	204	20	22	22	- 9	180	177	3	347	335	17	36	38	-10	248	244	13	98	101
16	99	103	22	37	41	21	118	111	- 8	32	35	4	21	23	18	72	69	- 8	78	73	14	248	254
17	37	40	28	106	110	22	136	140	- 7	98	97	5	73	72	19	74	71	- 6	278	280	15	219	226
19	213	223	Hk	1,Kk	1	23	28	21	- 6	170	176	6	65	61	20	23	19	- 4	543	537	16	27	23
20	34	31	-28	66	64	24	62	57	- 5	96	95	7	136	136	21	136	135	- 2	474	474	17	57	60
21	56	52	-27	34	36	25	118	108	- 4	178	183	8	221	221	23	161	148	0	17	15	19	76	82
22	56	56	-26	116	115	27	30	27	- 3	93	97	9	81	83	24	35	28	2	371	377	20	63	60
23	33	45	-25	45	45	Hk	1,Kk	3	- 2	63	63	10	450	454	Hk	2,Kk	4	4	556	554	23	45	41
24	74	77	-24	74	74	-26	31	21	- 1	153	155	11	22	12	-21	26	20	6	421	414	24	37	33
25	139	135	-23	67	70	-25	129	129	1	170	175	13	168	173	-20	183	176	8	117	117	25	69	64
27	25	16	-22	153	147	-24	97	95	2	99	94	14	160	158	-19	25	24	10	428	422	Hk	3,Kk	3
Hk	0,Kk	3	-21	68	71	-23	59	49	3	109	112	15	32	26	-17	90	93	12	249	249	-24	45	41
1	139	135	-20	111	112	-22	64	65	4	171	177	16	180	182	-16	99	100	14	87	92	-22	47	53
2	88	78	-19	118	118	-19	38	38	5	29	32	17	150	157	-15	31	32	16	92	102	-21	66	70
3	88	86	-18	155	163	-18	52	48	6	43	46	19	74	76	-14	39	50	18	226	233	-20	126	134
4	303	290	-17	112	112	-17	77	74	7	98	95	20	94	99	-11	111	121	20	132	134	-18	37	35
5	132	132	-16	58	52	-16	151	158	8	178	175	22	34	37	-10	80	95	22	82	90	-17	49	44
6	73	73	-15	147	149	-15	79	84	10	34	19	23	21	20	- 9	26	25	24	128	118	-16	76	73
8	76	130	-14	84	84	-14	186	189	11	104	102	25	53	53	- 8	213	218	26	43	51	-15	144	154
9	171	166	-12	141	133	-13	89	94	12	43	35	26	30	29	- 5	111	117	Hk	3,Kk	1	-13	185	190
10	104	101	-10	278	277	-12	21	19	13	110	105	Hk	2,Kk	2	- 4	28	24	-27	62	58	-12	63	59
11	72	77	- 9	95	89	-11	353	359	14	97	91	-26	109	109	- 2	75	77	-26	44	39	-11	78	79
15	36	31	- 8	64	66	- 8	145	164	16	23	16	-25	25	16	- 1	63	65	-25	73	71	- 8	155	162
16	25	3	- 7	281	275	- 7	147	139	17	62	59	-24	119	119	0	64	63	-24	116	118	- 6	89	90
17	145	146	- 6	63	64	- 6	151	105	18	62	51	-23	65	73	3	190	191	-21	134	134	- 5	64	66
18	38	49	- 5	158	157	- 5	260	256	Hk	1,Kk	6	-21	209	217	4	164	164	-20	58	63	- 4	32	21
19	27	32	- 4	177	177	- 4	67	42	-12	78	75	-20	136	133	5	95	90	-19	167	170	- 3	44	38
20	53	52	- 3	186	179	- 3	286	283	-11	63	55	-19	26	29	6	93	97	-18	187	195	- 2	72	68
21	31	29	- 2	754	743	- 2	69	78	-10	51	41	-18	219	228	7	95	99	-17	150	151	- 1	89	84
23	27	37	- 1	699	706	- 1	23	12	- 9	34	30	-17	148	154	8	47	53	-14	73	73	0	120	120
Hk	0,Kk	4	0	463	467	0	126	122	- 8	103	108	-15	35	34	9	74	74	-13	107	112	1	125	127
0	86	71	1	363	311	1	113	103	- 7	27	29	-14	169	162	10	54	50	-12	106	111	2	34	40
1	227	228	2	489	497	2	427	430	- 5	81	87	-12	221	226	11	57	56	-11	40	36	3	22	15
2	131	128	3	389	377	3	42	40	- 4	34	30	-10	127	128	12	85	93	-10	113	113	4	62	75
3	90	60	4	583	578	4	85	51	- 3	78	75	- 9	145	150	13	210	213	- 9	63	68	5	82	88
4	219	217	5	291	296	5	190	175	0	80	82	- 8	24	23	15	44	41	- 7	137	139	6	146	150
5	125	124	6	92	117	6	152	146	1	35	30	- 7	361	364	16	29	33	- 6	392	389	8	75	69
7	52	53	7	147	150	7	125	120	2	78	73	- 6	248	248	19	25	17	- 5	57	55	9	109	105
8	72	62	8	371	376	8	163	161	3	35	30	- 5	16	4	20	85	83	- 4	148	143	10	53	57
9	295	300	9	180	184	9	23	19	4	76	68	- 4	163	166	21	122	119	- 3	172	184	11	92	92
10	24	9	10	39	40	10	140	134	5	31	26	- 3	257	251	22	53	48	- 2	157	156	13	130	132
11	41	38	11	191	187	11	202	195	6	95	98	- 2	112	112	Hk	2,Kk	5	- 1	47	55	14	77	79
12	44	43	12	163	166	12	90	91	7	52	51	- 1	203	197	-18	109	107	0	105	107	17	98	104
13	57	59	14	53	57	13	110	110	10	25	26	0	23	34	-17	65	65	1	45	46	18	79	76
14	130	134	15	43	45	14	47	56	Hk	2,Kk	0	1	115	112	-15	78	82	3	108	104	19	25	24
15	51	47	17	144	142	15	225	216	-28	52	52	2	17	14	-14	45	44	4	241	244	20	60	45

Table 2. Continued.

21	68	44	= 8	29	33	3	24	27	= 4	84	76	=14	29	44	14	52	54	= 2	135	139	= 7	97	103
22	67	71	= 6	303	302	4	102	101	= 3	56	66	=13	49	44	15	64	67	= 1	47	47	= 6	84	85
Hs	3,Ks	4	= 4	168	165	5	98	103	= 2	146	147	=11	157	157	Hs	5,Ks	5	0	92	98	= 5	107	108
=21	40	33	= 2	55	57	6	52	52	= 1	81	82	=10	68	54	=10	30	21	1	116	121	= 4	77	79
=20	107	110	0	344	339	8	235	235	0	114	119	= 9	178	183	= 8	67	67	2	68	69	= 3	73	80
=19	46	43	2	283	289	10	78	77	1	39	37	= 8	205	210	= 5	26	24	3	196	196	= 1	30	19
=17	30	45	4	382	385	11	37	41	2	77	80	= 7	106	98	= 2	32	33	4	31	31	1	94	93
=15	89	94	6	189	183	12	29	31	4	151	150	= 6	82	38	0	108	108	5	48	50	2	93	95
=14	236	239	8	38	32	13	132	136	5	24	28	= 5	208	205	1	56	51	6	96	97	3	116	122
=13	109	111	10	216	220	14	106	103	6	51	51	= 4	111	101	2	88	87	7	70	72	4	41	39
=12	132	129	12	143	141	16	168	170	7	98	104	= 3	140	148	3	26	14	8	55	48	5	110	111
=11	44	43	14	49	54	17	25	19	8	46	45	= 2	87	86	7	30	27	10	69	65	8	27	28
= 9	33	34	16	176	174	20	125	127	9	114	116	0	40	44	8	26	27	11	70	67	10	30	42
= 8	59	52	18	84	85	21	88	92	10	104	102	1	77	76	Hs	6,Ks	0	12	75	68	12	91	87
= 7	138	140	20	70	64	24	38	43	13	28	26	2	108	104	=20	81	78	13	40	44	15	43	38
= 5	106	112	22	49	44	Hs	4,Ks	3	Hs	5,Ks	0	3	42	45	=18	45	46	14	60	62	16	33	31
= 4	102	104	24	134	126	=21	65	68	=22	106	90	4	254	262	=14	39	48	17	81	82	Hs	7,Ks	2
= 3	149	153	Hs	4,Ks	1	=20	73	75	=18	89	95	5	37	41	=12	121	125	Hs	6,Ks	3	=14	44	16
= 2	101	101	=25	116	109	=16	90	93	=16	53	51	6	49	51	=10	102	106	=17	84	68	=13	108	90
= 1	109	111	=24	29	37	=17	28	27	=12	139	125	8	23	17	= 8	28	29	=16	35	33	=12	96	97
0	66	61	=23	76	77	=15	67	76	=10	35	35	9	124	127	= 6	57	61	=15	37	38	=11	34	36
1	70	74	=22	26	24	=13	57	58	= 8	250	262	10	102	102	= 4	281	300	=14	121	112	=10	37	17
2	88	94	=21	68	69	=12	159	167	= 6	356	340	11	46	43	= 2	265	274	=11	49	43	= 9	47	42
3	132	130	=20	46	38	=11	51	63	= 4	74	69	12	60	71	4	93	88	=10	78	70	= 8	67	69
4	59	55	=19	27	23	=10	127	128	= 2	64	68	13	129	134	6	55	53	= 9	80	82	= 6	31	45
5	127	136	=17	122	124	= 8	166	133	0	83	81	14	56	60	8	105	98	= 8	22	13	= 3	34	31
6	80	83	=16	117	123	= 7	227	236	2	106	110	15	45	43	10	83	89	= 7	86	86	= 1	45	44
7	72	70	=15	46	44	= 6	58	44	4	105	104	16	36	41	12	44	41	= 6	82	77	1	142	134
8	108	106	=14	61	65	= 5	75	80	6	69	67	17	79	82	14	90	82	= 5	49	45	2	36	39
9	52	48	=12	201	190	= 4	56	44	8	170	166	18	40	35	16	123	118	= 4	116	116	3	28	28
10	68	74	=11	200	206	= 3	48	44	10	242	243	19	93	97	18	91	90	= 3	104	106	5	57	53
11	79	81	=10	131	133	= 2	134	136	12	191	191	20	34	21	20	56	52	= 2	50	45	7	50	51
12	205	208	= 9	108	112	= 1	46	51	14	40	48	Hs	5,Ks	3	Hs	6,Ks	1	1	53	49	8	48	47
15	49	44	= 8	71	76	0	112	114	16	62	65	=20	57	45	=21	67	55	2	121	115	9	63	60
16	68	61	= 7	233	234	1	24	19	22	62	63	=18	102	96	=20	67	66	3	114	109	10	52	45
17	24	21	= 6	55	55	2	34	29	Hs	5,Ks	1	=17	64	63	=19	92	91	4	69	65	14	73	57
18	58	63	= 5	150	146	4	61	59	=22	27	19	=12	65	68	=15	52	49	5	78	76	Hs	7,Ks	3
19	82	87	= 4	107	100	5	164	163	=21	33	30	=11	77	85	=14	79	66	6	35	27	= 8	49	47
Hs	3,Ks	5	= 3	86	84	6	43	48	=20	56	54	=10	128	135	=13	82	82	7	61	57	= 6	93	84
=15	33	34	= 2	173	174	7	162	166	=19	64	67	= 9	135	138	=11	63	57	10	65	81	= 4	68	60
=13	62	61	= 1	86	83	8	230	229	=17	61	59	= 8	29	32	=10	143	156	12	89	90	= 3	24	21
=12	31	28	0	130	127	9	65	71	=16	30	41	= 5	25	10	= 9	103	107	13	55	56	= 2	96	102
= 8	32	33	1	39	38	10	205	213	=13	150	154	= 4	150	150	= 8	160	160	14	43	42	= 1	85	78
= 7	30	38	2	243	254	11	37	40	=12	45	42	= 3	138	141	= 7	174	173	Hs	6,Ks	4	0	82	76
= 6	58	66	3	209	296	12	90	91	=11	43	41	= 1	195	203	= 6	28	25	=12	44	48	1	44	43
= 5	110	111	4	29	33	13	79	71	=10	105	112	1	39	49	= 5	33	17	=11	104	91	3	63	62
= 4	21	15	5	215	221	14	55	59	= 9	146	147	2	114	114	= 4	95	92	=10	72	63	4	113	111
= 3	33	29	6	236	236	15	67	67	= 8	27	24	5	100	97	= 3	219	226	= 9	38	40	5	70	66
= 2	91	94	7	163	163	16	24	24	= 7	77	83	6	115	125	= 1	145	144	= 8	112	101	7	72	68
= 1	25	28	8	20	21	19	106	102	= 6	28	19	7	162	162	0	80	87	= 7	70	64	9	75	69
0	44	46	10	94	95	Hs	4,Ks	4	= 5	141	147	10	75	77	1	124	132	= 5	109	106	10	79	76
1	51	56	11	42	37	=18	37	33	= 4	43	21	12	29	33	2	96	88	= 4	103	95	Hs	8,Ks	0
2	44	41	12	172	172	=17	82	83	= 3	164	168	13	44	38	4	123	125	= 1	96	97	=12	126	127
3	140	138	13	207	214	=16	33	36	= 2	278	289	14	86	89	5	134	135	0	56	57	= 8	74	80
4	81	84	14	260	265	=15	42	39	= 1	35	36	15	70	67	6	86	86	2	99	86	= 4	178	178
5	121	121	15	109	109	=14	31	32	0	79	79	16	24	15	7	27	19	3	61	60	= 2	54	53
7	80	80	16	39	42	=13	61	69	1	27	34	17	24	19	8	55	55	4	58	55	0	138	138
9	46	39	17	64	66	=12	23	19	2	65	67	Hs	5,Ks	4	10	61	62	5	28	38	2	123	116
10	36	42	18	102	101	=10	32	25	3	31	29	=16	56	52	11	94	98	6	37	40	10	76	67
11	164	159	19	25	35	= 9	75	73	4	106	105	=14	32	36	12	174	175	7	85	87	Hs	8,Ks	1
12	82	87	22	146	153	= 7	40	37	5	110	106	=13	22	12	13	27	28	8	55	57	= 6	52	54
13	95	99	23	34	24	= 6	105	106	6	112	118	=12	37	40	14	48	40	9	38	39	= 2	29	22
14	30	26	25	26	26	= 5	101	102	7	107	114	=11	53	48	15	45	51	Hs	7,Ks	0	= 1	35	38
15	94	89	Hs	4,Ks	2	= 4	84	94	8	97	94	=10	120	120	16	79	72	=16	39	47	0	60	60
Hs	3,Ks	6	=23	26	16	= 2	30	41	9	83	90	= 9	47	49	17	50	53	=14	104	93	1	42	38
= 7	37	31	=22	30	32	0	42	46	11	21	27	= 8	73	77	18	51	44	=12	47	54	2	65	51
= 6	39	29	=21	59	52	1	28	12	12	50	49	= 6	83	85	19	42	42	=10	28	33	3	66	61
= 5	63	60	=19	68	55	2	135	137	13	44	47	= 4	97	95	20	58	56	= 4	142	142	5	68	58
= 4	69	55	=17	124	123	3	71	77	14	39	38	= 3	142	143	Hs	6,Ks	2	= 2	28	23	6	37	41
= 3	23	26	=15	36	34	4	36	44	15	85	87	= 1	85	84	=17	72	79	0	41	38	7	27	20
= 1	130	132	=13	93	92	6	96	93	16	82	88	0	94	86	=16	44	42	4	47	52	8	46	47
0	99	94	=11	188	192	7	46	41	17	58	61	1	114	105	=14	142	142	6	31	23	Hs	8,Ks	2
1	114	104	=10	76	82	10	46	44	18	74	70	2	149	151	=13	32	14	8	39	43	= 8	28	6
2	67	66	= 9	120	124	11	56	56	19	26	6	3	59	59	=12	34	31	10	92	89	= 5	66	76
4	56	55	= 8	26	31	12	67	70	20	69	71	4	75	75	=11	34	33	12	56	55	= 4	36	34
6	66	58	= 6	24	27	14	29	36	22	53	47	5	42	43	=10	28	8	14	110	113	= 2	37	28
Hs	4,Ks	0	= 5	37	40	15	68	59	Hs	9,Ks	2	6	82	88	= 9	44	48	16	39	31	= 1	64	60
=26	144	134	= 4	44	33	16	31	27	=22	31	33	7	54	58	= 8	34	21	Hs	7,Ks	1	0	34	24
=24	63	58	= 3	36	30	17	34	33	=21	33	31	8	223	221	= 7	92	86	=16	71	35	2	36	30
=22	88	86	= 2	183	185	18	134	127	=19	62	65	9	85	85	= 6	86	82	=15	117	116	3	113	108
=20	44	52	= 1	91	90	Hs	4,Ks	5	=18	57	62	10	89	89	= 5	59	48	=14	51	53	4	30	14
=18	68	62	0	86	90	=13	56	57	=17	82	81	12	26	10	= 4	29	13	=12	45	35	5	25	23
=16	74	81	1	20	19	=11	84	86	=16	111	115	13	26	21	= 3	45	50	=11	90	93	6	75	71

observed-unobserved cutoff at 2.5 $\sigma(I)$, 1416 reflections were recorded as observed. The radiation was Mo$K\alpha$ and $2\theta_{max} = 50°$. No corrections for absorption or secondary extinction effects have been carried out.

The structure was solved by direct methods [7]* and refined by full-matrix least squares technique. Anisotropic temperature factors were introduced for oxygen and carbon atoms. The weights in least squares were obtained from the standard deviations in intensities, $\sigma(I)$, taken as

$$\sigma(I) = [C_T + (0.02C_N)^2]^{\frac{1}{2}}$$

where C_T is the total number of counts, and C_N the net count (peak minus background). The atomic form factors were those of Hanson *et al.*[8] except for hydrogen.[9] The R-value arrived at was 4.8 % (weighted value $R_w = 5.7$ %) for 1416 observed reflections.

Final fractional coordinates and thermal

* All programs used are included in this reference.

Table 3. Interatomic distances, bond angles and dihedral angles with estimated standard deviations.

DISTANCE	(Å)	DISTANCE	(Å)	DISTANCE	(Å)
O - C1	1,213(3)	C1 - C2	1,515(3)	C2 - C3	1,532(3)
C3 - C4	1,528(3)	C4 - C5	1,536(3)	C5 - C6	1,548(3)
C6 - C7	1,537(3)	C7 - C8	1,523(3)	C8 - C9	1,527(3)
C9 - C10	1,522(3)	C10 - C11	1,536(3)	C11 - C1	1,516(3)

ANGLE	(°)	ANGLE	(°)
O - C1 - C2	120,5(2)	O - C1 - C11	120,1(2)
C11 - C1 - C2	119,3(2)	C1 - C2 - C3	114,3(2)
C2 - C3 - C4	113,8(2)	C3 - C4 - C5	115,4(2)
C4 - C5 - C6	113,8(2)	C5 - C6 - C7	114,2(2)
C6 - C7 - C8	114,0(2)	C7 - C8 - C9	115,6(2)
C8 - C9 - C10	116,0(2)	C9 - C10 - C11	114,6(2)
C10 - C11 - C1	113,9(2)		

DIHEDRAL ANGLE	(°)	DIHEDRAL ANGLE	(°)
C1 - C2 - C3 - C4	-59,8(3)	C2 - C3 - C4 - C5	-61,3(3)
C3 - C4 - C5 - C6	139,8(2)	C4 - C5 - C6 - C7	-64,8(3)
C5 - C6 - C7 - C8	-63,6(3)	C6 - C7 - C8 - C9	173,7(2)
C7 - C8 - C9 - C10	-64,6(3)	C8 - C9 - C10 - C11	-55,7(3)
C9 - C10 - C11 - C1	95,1(3)	C10 - C11 - C1 - C2	-127,2(2)
C11 - C1 - C2 - C3	159,7(2)		

parameters with estimated standard deviations are given in Table 1. The expression for anisotropic vibration is:

$$\exp[-(B11h^2 + B22k^2 + B33l^2 + B12hk + B13hl + B23kl)]$$

A comparison between observed and calculated structure factors is presented in Table 2.

The principal axes of the thermal vibration ellipsoids for oxygen and carbon atoms were calculated from the temperature parameters of Table 1. Maximum root mean squares amplitudes range from 0.17 Å to 0.20 Å for carbon atoms, while that of oxygen is 0.24 Å. Due to the size of the molecule, no rigid-body analysis of translational, librational, and screw motion has been carried out.

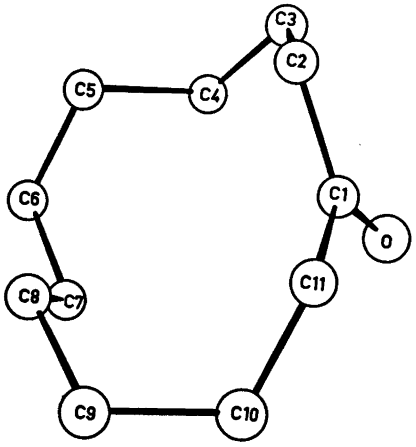
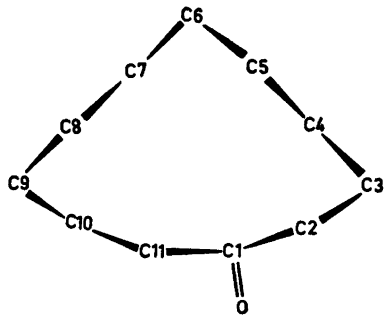


Fig. 1. Schematical drawing of the molecule.

Interatomic distances, bond angles, and dihedral angles are given in Table 3. The standard deviations, in parentheses, are estimated from the correlation matrix of the last least squares refinement cycle. Fig. 1 is a schematical drawing of the molecule.

Temperature dependency of the NMR-spectra of cycloundecanone in the range $-80°$ to $-170°$ indicates a single asymmetric conformation.[10] Strain minimization calculations for cycloundecane [4] give four conformations with about equally low enthalpies, one of which corresponds roughly to the "triangular" ring skeleton of cycloundecanone.

Except for *gauche* bonds, the agreement of calculated dihedral angles of cycloundecane [4] and those of Table 3 is rather poor. The asymmetric position of the carbonyl group confirms the NMR-indication mentioned above. The C=O



bond direction is approximately normal to the plane defined by C1,. . .,C11. H41 – H71 [2.10(4) Å] is the only H – H distance being significantly shorter than the van der Waals contact of 2.4 Å.

As may be seen from Table 3, bond distances and angles are normal. The mean value of C – C bonds (excluding C1 – C2 and C1 – C11 of 1.515 Å and 1.516 Å, respectively) is 1.532 Å. A corresponding average for C – C – C bond angles is 114.6°. C – H bond lengths range from 0.93 Å to 1.03 Å with estimated standard deviations of about 0.04 Å. C – C – H and C – H – C angles are found between 104° and 114° (e.s.d. ~2°).

The (C11 – C1 – C2)-group is planar (to within 0.008 Å) and the C – O bond distance of 1.213(3) Å agrees within error limits with corresponding distances in cyclohexane-1,4-dione [11] [1.220(6) Å; 1.223(8) Å].

No short *inter* molecular contacts are observed.

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