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Crystal Structure of Cycloalanyltetrasarcosyl Hemihydrate

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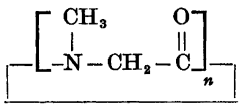
The crystals of $C_{15}O_5N_5H_{25}\cdot\frac{1}{2}H_2O$ belong to the monoclinic system with space group $C2/c$ and cell dimensions $a=19.427(5)$ Å, $b=10.804(3)$ Å, $c=17.731(7)$ Å, $\beta=100.27(3)°$. There are eight molecules in the unit cell. The phase problem was solved by direct methods and the R-value arrived at for 1774 observed reflections was 6.3 % ($R_W=5.2$ %). The conformation is *cis, cis, cis, trans, trans*. Inter-molecular N···O hydrogen bonds (2.962 Å) connect centrosymmetrically related molecules, forming dimers. Water molecules, situated at two-fold axes of rotation, link the dimers to endless chains along [001]. The results are compared with those of cyclotetra-, cyclopenta-, and cycloocta-sarcosyl.

Cyclic oligopeptides of sarcosine, glycine, and alanine are studied by Dale and Titlestad, mainly by spectroscopic methods.[1-3] For the sarcosine compounds



with $n=2$, 4, 5, 8, the crystal structures are known.[4-7] The *cis, cis, cis, trans, trans*-conformation found for cyclopentasarcosyl is also predominant in solution, whereas many other cyclic pentapeptides are conformer mixtures in solution.[3] For example, by dissolving crystals of cycloalanyltetrasarcosyl, (AS4), in $CHFCl_2$ at low temperature (−75°), and slowly heating the solution, drastic changes in the NMR-spectra recorded at different temperatures are observed. A new set of lines due to a second conformer develops at −40°. At the final equilibrium, the crystal conformer spectrum is completely replaced by these lines together with a third set, belonging to the dominant solution conformer. These findings, and a comparison of the (−75°)-spectrum of AS4 with the (−50°)-spectrum of cyclopentasarcosyl, suggest different crystal conformations. In order to settle the conformational problem, and to obtain detailed information of the molecular geometry, an X-ray crystallographic investigation of cycloalanyltetrasarcosyl has been carried out.

The crystals belong to the monoclinic system and the systematic absences lead to the space group $C2/c$.* The cell parameters, measured by means of a four circle diffractometer, and their estimated standard deviations are: $a=19.427(5)$ Å, $b=10.804(3)$ Å, $c=17.731(7)$ Å, $\beta=100.27(3)°$. The unit cell contains eight AS4, and four water molecules. With $2\theta(\max)=50°$ and Mo$K\alpha$-radiation, about 3400 independent reflections were measured on an automatic four circle diffractometer. Using an observed-unobserved cutoff at $2.0\sigma(I)$, 1774 were recorded as observed. No corrections have been made for absorption or secondary extinction effects.

The structure was solved by direct methods [8] and refined by full-matrix least squares technique.[9]** Hydrogen atom positions were calculated. Anisotropic temperature factors were introduced for O, N, and C-atoms, and weights in least squares were calculated 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 conventional R-value arrived at was 6.3 % (weighted value $R_W=5.2$ %) for 1774 observed reflections. The form factors were those of

* Since L-alanine was used in the synthesis, the centrosymmetric space group shows that racemization has occurred.

** All programs used are included in this reference.

Table 1. Final fractional coordinates and anisotropic thermal vibration parameters with estimated standard deviations (multiplied by 10^5 for non-hydrogens and 10^4 for hydrogens). The symbols CC, CM, and OW are used for carbonyl carbons, methyl carbons and water oxygen, respectively. Hmn is bonded to Cm, HMmn to CMm, H4 to N4, and HW to OW.

ATOM	X	Y	Z	B	B11	B22	B33	B12	B13	B23
OW	0(0)	46453(47)	25000(0)		478(24)	937(60)	768(31)	0(0)	440(44)	0(0)
O1	4926(16)	35157(29)	4789(18)		211(11)	718(35)	360(14)	118(33)	96(20)	-171(37)
O2	11057(18)	29460(34)	25890(19)		374(15)	1238(46)	271(14)	25(41)	210(23)	-168(42)
O3	19070(18)	65947(31)	26294(19)		364(14)	763(37)	339(14)	-138(36)	-63(22)	-307(39)
O4	7465(18)	87466(31)	5710(20)		431(15)	597(36)	451(17)	297(38)	223(25)	129(41)
O5	19549(18)	48424(31)	-1265(20)		204(12)	901(40)	488(17)	118(35)	-63(23)	-256(42)
N1	10568(20)	18176(35)	15110(22)		297(15)	644(43)	274(17)	-24(41)	195(25)	-41(45)
N2	20966(21)	45420(38)	22929(22)		276(15)	749(48)	293(17)	98(43)	-107(25)	-94(46)
N3	9986(20)	70501(34)	12895(22)		285(14)	518(40)	298(16)	80(39)	88(24)	-122(44)
N4	18393(18)	61737(35)	-3441(21)		174(12)	639(41)	315(16)	30(37)	31(23)	-30(41)
N5	10457(20)	28841(35)	-4593(22)		271(14)	606(41)	262(17)	121(40)	75(25)	-123(43)
CC1	8708(24)	27624(43)	2379(27)		196(16)	530(48)	261(19)	-98(47)	21(28)	-223(53)
CC2	13198(24)	27344(46)	19906(28)		257(18)	719(53)	229(20)	239(52)	107(31)	108(57)
CC3	17888(24)	56560(50)	21686(28)		217(17)	750(56)	273(21)	-158(51)	18(30)	-11(58)
CC4	10286(25)	77297(47)	6503(29)		257(18)	553(52)	308(21)	-82(50)	-12(31)	-59(59)
CC5	13468(27)	50698(45)	-4050(26)		239(18)	670(54)	234(19)	-36(50)	70(29)	59(51)
C1	12470(24)	17061(42)	7484(27)		246(18)	605(50)	283(20)	-22(49)	144(29)	-109(52)
C2	19245(24)	34921(44)	17872(26)		230(16)	674(53)	263(19)	83(48)	44(28)	-98(53)
C3	12771(25)	58024(41)	14171(26)		290(18)	494(49)	306(20)	6(47)	-16(31)	-81(50)
C4	14387(25)	72077(41)	513(27)		286(18)	470(47)	305(20)	-163(49)	131(30)	-58(54)
C5	8825(25)	40760(45)	-8389(26)		244(17)	712(55)	265(19)	88(48)	-55(30)	-12(53)
CM1	4417(29)	11592(49)	16802(32)		432(21)	911(57)	474(27)	-332(60)	370(39)	-35(65)
CM2	25684(28)	43252(53)	30285(30)		398(21)	1192(69)	349(22)	339(61)	-274(36)	-57(66)
CM3	5745(26)	75193(47)	18304(28)		289(18)	874(62)	359(21)	2(53)	119(32)	-159(59)
CM4	15329(27)	82184(50)	-5178(31)		305(20)	963(67)	439(24)	-209(59)	193(34)	-88(67)
CM5	15169(28)	20379(48)	-7652(27)		443(22)	904(63)	326(20)	246(61)	338(35)	-95(60)
H11	1428(17)	869(32)	535(18)	2,9(,8)						
H12	1729(16)	1703(29)	746(17)	1,7(,7)						
H21	2334(17)	2921(32)	1817(18)	3,1(,8)						
H22	1812(16)	3798(31)	1240(18)	3,0(,8)						
H31	1523(18)	5589(34)	982(19)	4,2(,9)						
H32	890(19)	5215(33)	1381(21)	3,9(,9)						
H51	368(15)	4295(27)	-857(16)	2,1(,7)						
H52	985(15)	3995(29)	-1354(16)	2,0(,7)						
HM11	292(19)	456(36)	1294(23)	10,6(1,0)						
HM12	59(23)	1743(44)	1684(26)	8,3(1,3)						
HM13	569(24)	732(45)	2142(28)	8,8(1,3)						
HM21	2715(27)	3485(54)	3024(31)	10,8(1,6)						
HM22	2327(27)	4584(50)	3422(29)	10,4(1,5)						
HM23	2928(20)	4845(38)	3061(23)	10,7(1,0)						
HM31	436(20)	6815(40)	2136(24)	6,3(1,1)						
HM32	164(21)	7986(38)	1535(23)	6,5(1,1)						
HM33	841(24)	8162(42)	2175(26)	5,6(1,2)						
HM41	1830(22)	8904(41)	-232(24)	6,2(1,2)						
HM42	1074(20)	8616(37)	-758(22)	5,4(1,0)						
HM43	1791(19)	7874(36)	-904(21)	5,8(1,0)						
HM51	1573(20)	2298(37)	-1247(22)	9,6(1,0)						
HM52	1368(25)	1200(49)	-735(26)	9,9(1,5)						
HM53	1965(21)	2025(38)	-414(22)	10,8(1,0)						
H4	580(19)	6248(35)	-473(20)	4,9(1,0)						
HW	382(17)	4212(35)	2642(22)	9,0(1,0)						

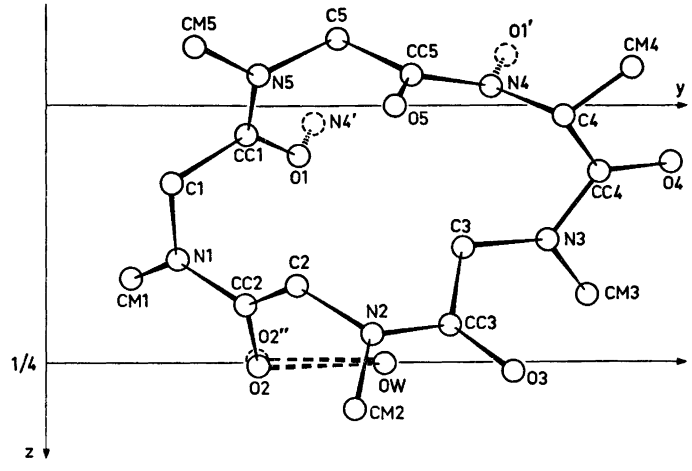


Fig. 1. The molecule viewed along [100].

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

K= 0,L= 0		
2	184	205
4	4091	4070
6	1149	1146
10	1246	1245
12	499	479
14	524	528
16	158	154
20	416	421
22	142	134
K= 0,L= 2		
-20	196	221
-16	646	667
-14	263	295
-12	376	379
-8	428	411
-6	176	232
-4	1160	1209
-2	3189	3178
0	896	834
2	823	807
4	3595	3600
6	1135	1131
8	463	461
10	393	447
14	483	487
16	172	151
18	160	217
20	197	204
K= 0,L= 4		
-20	187	210
-16	176	156
-14	157	173
-12	308	323
-8	767	728
-6	861	822
-4	1376	1398
-2	3236	3308
0	2220	2132
2	1058	1140
4	874	879
6	418	391
8	525	508
10	733	731
14	118	65
16	227	200
18	215	175
K= 0,L= 6		
-16	211	195
-14	143	172
-12	951	957
-10	823	828
-6	1558	1545
-4	1081	1089
-2	682	703
0	532	521
2	331	356
4	112	102
6	1404	1373
8	236	270
12	359	367
18	301	295
K= 0,L= 8		
-18	244	207
-16	130	103
-10	671	699
-8	264	283
-6	836	825
-4	1215	1281
-2	305	319
0	836	834
2	626	619
4	848	809
6	330	358
10	903	909
K= 0,L= 10		
-20	236	262
-18	470	462
-16	320	315
-14	817	838
-10	700	699
-8	182	141
-6	454	446
-4	724	712
-2	1105	1144
0	1237	1213
2	402	427
4	717	695
6	397	422
8	191	194
10	206	203
12	397	421
K= 0,L= 12		
-18	135	47
-14	166	144
-12	289	304
-8	245	260
-6	332	352
-2	219	285
0	135	67
4	294	300
6	215	235
8	438	428
10	238	289
K= 0,L= 13		
6	141	0
K= 0,L= 14		
-14	245	204
-8	283	308
-4	423	432
-2	452	477
4	149	98
6	173	198
K= 0,L= 16		
-14	169	102
-12	144	169
-10	224	214
-6	715	728
-2	397	411
0	207	176
2	357	388
6	575	542
10	327	280
K= 0,L= 18		
-14	145	49
-8	454	419
-4	328	334
2	173	170
6	289	286
K= 0,L= 20		
-10	155	106
-6	185	171
-2	274	285
K= 1,L= 0		
1	1783	1772
3	1868	1864
5	925	874
7	403	374
9	534	497
11	444	449
13	607	616
15	304	301
19	129	19
21	152	194
K= 1,L= 1		
-15	203	232
-13	276	260
-11	624	622
-9	639	665
-7	314	303
-5	587	569
-3	301	313
-1	123	36
1	371	427
5	135	85
7	744	732
9	410	400
13	257	278
21	180	155
K= 1,L= 2		
-13	393	395
-11	408	415
-7	441	465
-5	1756	1765
-3	251	217
-1	219	200
1	308	312
3	1096	1142
7	749	724
9	107	140
11	269	269
21	196	187
K= 1,L= 3		
-17	274	252
-15	178	202
-13	279	276
-9	641	625
-5	298	308
-1	191	231
1	1259	1218
3	583	549
5	2196	2199
7	315	331
9	929	957
11	356	344
K= 1,L= 4		
-21	139	174
-17	189	197
-13	439	446
-11	191	208
-9	972	978
-7	224	202
-5	1747	1774
-3	1486	1447
-1	330	315
1	1768	1759
5	358	352
7	636	602
9	449	458
11	609	600
13	382	370
15	233	235
17	143	135
K= 1,L= 5		
-21	147	48
-19	180	233
-11	836	835
-9	602	604
-7	462	413
-5	757	711
-3	461	397
-1	387	368
1	237	297
3	305	255
5	413	381
7	272	210
9	380	386
K= 1,L= 6		
-17	201	204
-15	224	225
-11	193	198
-9	127	87
-7	1097	1061
-5	198	239
-3	1318	1301
-1	886	888
1	318	314
5	340	356
7	605	594
K= 1,L= 7		
-19	146	115
-13	458	466
-11	173	162
-9	226	220
-5	389	366
-3	406	385
-1	836	857
3	412	411
5	790	809
7	165	165
9	558	538
13	226	206
17	234	220
K= 1,L= 8		
-15	136	144
-9	369	389
-7	171	208
-5	423	444
-3	480	484
-1	206	189
1	1023	1020
3	539	541
11	305	319
K= 1,L= 9		
-19	175	168
-17	220	227
-15	556	552
-13	259	277
-11	434	439
-9	188	200
-7	488	482
-5	638	635
-3	783	756
-1	717	737
1	160	183
5	300	340
7	146	178
9	250	237
13	159	129
15	147	79
K= 1,L= 10		
-17	240	207
-11	131	147
-9	283	300
-7	249	254
-5	356	378
-3	143	155
1	254	245
3	190	200
5	171	177
9	210	207
11	334	337
K= 1,L= 11		
-17	243	255
-7	150	150
-5	305	321
-3	225	268
-1	166	168
1	165	170
3	134	126
5	348	334
7	312	320
9	133	86
11	190	198
13	260	231
K= 1,L= 12		
-13	277	306
-11	180	213
-7	274	297
-5	305	326
-3	357	355
-1	439	456
3	182	171
5	251	276
7	165	167
11	200	207
K= 1,L= 13		
-19	136	30
-15	217	207
-13	202	198
-11	427	455
-9	486	492
-7	292	282
-5	426	446
-1	172	198
1	435	409
3	194	186
5	271	249
11	230	256
15	190	137
K= 1,L= 14		
-7	231	212
-5	188	214
-3	160	154
-1	210	197
1	117	140
3	215	206
9	217	203
11	139	104
K= 1,L= 15		
-11	148	134
-7	128	149
-3	166	146
-1	199	195
1	132	126
3	128	69
5	343	363
7	159	169
9	309	309
K= 1,L= 16		
-9	132	154
-3	287	301
-1	148	143
5	185	236
9	162	125
K= 1,L= 17		
-15	171	168
-9	249	230
-7	242	263
-3	331	368
1	183	181
3	384	383
7	274	262
K= 1,L= 18		
-7	167	140
-5	170	175
5	210	210
K= 1,L= 19		
-1	138	25
1	189	190
3	168	184
5	228	192
K= 1,L= 20		
-5	153	133
-3	155	96
K= 1,L= 21		
-5	148	167
K= 2,L= 0		
0	2315	2271
2	1325	1344
4	2754	2708
6	948	951
8	560	561
14	338	361
16	273	268
18	211	251
20	216	167
22	142	11
K= 2,L= 1		
-20	172	187
-16	145	140
-14	222	237
-12	264	265
-10	521	538
-8	300	315
-6	1580	1590
-4	342	399
-2	447	499
0	390	402
2	345	356
4	1297	1285
6	877	875
8	128	112
10	824	834
12	208	216
14	272	289
16	166	174
K= 2,L= 2		
-16	405	413
-12	711	726
-8	311	330
-6	223	254
-4	1306	1287
-2	888	921
0	712	717
2	835	862
4	812	858
6	134	153
8	704	723
10	228	226
12	171	209
14	165	161
16	274	289
K= 2,L= 3		
-22	151	132
-16	321	311
-10	391	334
-8	1438	1465
-6	197	215
-4	693	650
-2	565	604
0	351	340
2	1048	1015
4	1684	1666
6	243	272
8	882	891
10	364	367
K= 2,L= 4		
-20	214	194
-12	268	270
-10	406	413
-8	130	80
-6	215	214
-4	942	905
-2	607	615
0	368	382
2	374	409
4	633	650
6	586	547
8	669	685
10	469	480
K= 2,L= 5		
-20	164	149
-18	176	161
-16	283	255
-14	153	169
-12	228	225
-10	276	269
-8	352	356
-6	1288	1311
-4	399	410
-2	1488	1459
0	1335	1324
2	1635	1608
4	1185	1192
6	916	923
8	181	177
10	120	118
12	221	227
14	235	232
18	251	259
K= 2,L= 6		
-20	222	193
-16	200	195
-12	188	185
-8	382	397
-6	136	130
-4	746	769
-2	746	729
0	135	126
2	337	350
4	126	105
8	411	436
10	235	243
12	383	419
14	566	541
16	313	336
K= 2,L= 7		
-18	315	296
-16	180	175
-10	190	196
-8	989	976
-6	379	367
-4	639	641
-2	859	823
0	917	941
2	994	983
4	559	561
6	423	436
12	135	106
K= 2,L= 8		
-18	136	138
-14	177	162
-12	314	297
-4	625	589
-2	655	622
0	588	595
2	696	668
4	275	294
6	595	606
8	402	381
16	188	176
18	147	37
K= 2,L= 9		
-18	192	158
-12	193	182
-10	238	237
-6	229	220
-4	312	302
-2	871	861
0	136	96
2	345	321
4	377	360
6	256	246
8	193	198
10	272	289
12	139	132
K= 2,L= 10		
-20	312	269
-18	286	296
-16	356	359
-14	711	728
-12	271	258
-10	619	632
-8	349	368
-6	267	232
-4	122	80
0	381	383
2	185	198
6	453	472
10	285	276
14	251	233
K= 2,L= 11		
-14	268	256
-12	500	537
-8	579	596
-6	229	240
-4	120	103
-2	115	98
0	288	294
4	168	129
6	464	471
12	204	200
K= 2,L= 12		
-16	246	271
-12	185	189
-10	241	284
-8	181	151
-6	353	347
-4	481	478
-2	270	267
0	140	153
2	654	692
4	143	140
6	128	18
8	183	138
10	144	129
14	144	146
K= 2,L= 13		
-14	211	260
-10	480	494
-8	546	555
-4	218	232
0	117	70
2	344	349
4	342	335
6	261	258
12	161	125
K= 2,L= 14		
-14	264	292
-10	294	300
-8	295	302
-6	144	192
-4	212	181
2	159	142
4	205	204
8	340	321
K= 2,L= 15		
-6	186	195
0	439	461
4	311	325
K= 2,L= 16		
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-6	122	109
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0	380	377
2	339	331
4	228	230
8	226	239
K= 2,L= 17		
-4	156	154
-2	308	308
2	154	127
4	141	101
K= 2,L= 18		
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-10	194	190
-4	165	131
K= 2,L= 19		
-4	189	185
K= 3,L= 0		
1	312	321
3	418	489
5	437	459
7	643	597
11	211	184
13	328	317
15	288	293
21	228	178
K= 3,L= 1		
-19	404	400
-13	185	175
-11	159	132
-9	217	227
-7	1101	1109
-5	273	240
-3	1254	1225
-1	426	446
1	119	97
3	779	797
5	703	728
7	232	202
9	146	139
13	144	140
17	145	141
19	217	196
K= 3,L= 2		
-15	255	264
-11	416	429
-9	373	358
-7	516	510
-5	677	701
-3	1144	1120
1	376	398
3	2320	2359
5	109	146
7	789	780
9	177	172
11	190	158
13	394	397
15	391	427
19	372	392
K= 3,L= 3		
-21	219	183
-15	244	266
-13	248	246
-11	214	191
-9	505	498
-7	454	486
-5	522	500
-3	151	149
1	448	447
3	1787	1772
5	1253	1232
7	420	423
9	976	957
13	554	542
15	342	365
17	443	434
K= 3,L= 4		
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-13	480	463
-11	324	320
-9	497	494
-7	1060	994
-5	1126	1097
-3	269	218
-1	929	898
1	317	329
5	667	671
7	556	566
9	253	289
15	239	280
17	218	187
K= 3,L= 5		
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-13	440	430
-11	138	121
-9	420	417
-7	323	292
-1	292	270
1	186	232
3	456	446
5	243	254
7	1088	1117
9	634	645
11	225	240
13	657	648
K= 3,L= 6		
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-11	425	436
-9	584	597
-7	1062	1022
-5	258	276
-3	436	393
-1	174	176
7	139	129
9	491	525
13	603	638
17	195	187
K= 3,L= 7		
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-15	135	144
-13	412	426
-11	249	257
-9	342	346
-7	485	483
-5	210	190
-3	180	205
-1	337	363
1	720	703
3	253	250
5	560	554
7	180	154
9	192	181
11	273	261
13	285	236
15	161	183
17	233	209
K= 3,L= 8		
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-17	166	156
-15	145	149
-9	152	155
-7	880	859
-5	911	907
-3	886	898
-1	1031	1005
1	153	108
3	115	112
5	452	464
7	172	141
9	218	194
13	348	340
17	242	179
K= 3,L= 9		
-15	251	264
-11	144	145
-9	351	346
-7	476	482
-5	287	283
-3	171	117
-1	244	232
1	439	429
3	109	107
5	150	114
7	341	333
11	195	215
K= 3,L= 10		
-17	144	100
-13	337	371
-11	165	163
-9	153	179
-7	188	165
-3	430	402
1	144	174
3	482	491
5	283	268
7	510	505
11	162	110
13	309	299
K= 3,L= 11		
-11	127	119
-9	239	230
-7	150	77
-5	290	294
-3	470	465
-1	179	158
1	127	144
3	508	513
5	291	291
13	128	17
K= 3,L= 12		
-19	177	175
-13	233	221
-11	374	374
-9	573	560
-5	120	143
-1	287	303
1	853	900
3	246	249
5	236	271
7	168	148
9	216	188
11	235	238
15	196	190
K= 3,L= 13		
-15	153	145
-11	443	449
-9	187	192
-7	444	486
-5	262	259
-3	155	184
-1	347	360
1	393	392
5	213	230
7	164	167
9	248	244
K= 3,L= 14		

Table 2. Continued.

-15	280	237	-4	398	386	-2	322	327	-3	394	365	13	151	156	-2	753	758	K=	7,L=	0	K=	7,L=	10
-13	337	328	-2	526	533	2	187	146	-1	792	799	K=	5,L=	13	0	199	160	1	449	440	-13	135	135
-11	344	350	0	695	714	4	352	364	1	719	704	-15	143	156	4	113	102	5	890	913	-9	524	536
-9	309	308	2	310	297	6	273	280	3	1030	991	-13	138	120	10	465	457	7	178	183	-7	133	127
-7	177	193	4	185	188	8	141	112	5	142	129	-11	279	234	K=	6,L=	6	9	355	341	-1	530	528
-5	171	179	6	431	412	10	164	125	7	131	119	-7	161	194	-20	160	116	13	196	202	1	300	328
-3	182	180	8	521	514	12	162	188	9	559	560	-5	387	399	-16	232	261	15	127	113	3	127	138
-1	573	582	10	144	141	K=	4,L=	13	11	240	215	-3	232	237	-14	233	213	K=	7,L=	1	5	142	133
3	350	338	12	576	577	-14	201	119	13	247	256	-1	218	216	-12	149	144	-15	270	272	11	226	223
7	205	182	16	126	115	-10	363	386	K=	5,L=	5	3	343	336	-10	527	540	-11	225	226	K=	7,L=	11
11	140	101	K=	4,L=	5	-6	530	566	-19	142	161	5	265	272	-8	149	118	-9	355	340	-15	160	68
K=	3,L=	15	-20	153	161	-2	571	579	-15	177	182	7	260	235	-4	393	376	-3	329	330	-11	131	61
-13	152	88	-16	156	122	0	185	166	-9	230	209	K=	5,L=	14	-2	523	532	1	710	720	-9	373	354
-11	173	187	-14	371	369	2	453	477	-7	141	153	-15	157	114	0	196	197	3	240	235	-5	429	442
-9	134	124	-10	350	325	4	155	153	-5	611	594	-5	252	242	2	326	336	5	122	118	-3	238	222
-7	296	314	-6	503	512	8	132	143	-3	347	336	-3	231	223	4	149	157	7	213	214	-1	375	342
-5	142	181	-4	570	562	12	156	58	-1	316	305	-1	378	347	6	250	224	11	291	287	1	323	345
-3	139	130	0	118	29	K=	4,L=	14	1	120	108	1	134	149	8	242	243	K=	7,L=	2	7	217	167
-1	132	169	2	1082	1056	-8	264	228	5	143	152	3	163	115	10	235	293	-13	128	127	11	203	188
1	400	393	6	796	803	-6	251	249	7	668	682	5	173	131	12	136	174	-11	176	224	K=	7,L=	12
3	229	228	10	164	195	-4	171	181	11	448	455	K=	5,L=	15	K=	6,L=	7	-9	146	106	-11	166	193
9	176	136	12	394	396	-2	127	50	15	365	354	-7	406	397	-18	226	262	-7	181	220	-3	428	425
11	146	119	14	192	200	0	125	54	17	205	147	-3	188	184	-14	148	146	-5	472	465	7	217	241
K=	3,L=	16	16	213	170	2	241	199	K=	5,L=	6	3	177	75	-12	138	119	-3	294	298	11	250	227
-9	157	55	K=	4,L=	6	10	185	200	-15	159	206	K=	5,L=	16	-8	149	156	1	396	388	K=	7,L=	13
-1	211	229	-18	177	199	K=	4,L=	15	-11	386	389	-11	134	95	-6	364	362	3	113	68	-7	160	153
K=	3,L=	17	-16	166	111	-12	234	231	-9	240	230	-7	209	219	-4	138	150	5	301	307	-5	247	218
-9	208	201	-14	372	354	0	176	134	-5	396	403	-5	184	229	0	686	679	7	188	155	-1	295	290
-7	168	155	-8	536	531	2	134	140	-3	401	390	-1	255	286	4	210	229	9	166	153	5	178	154
-5	252	259	-6	417	391	K=	4,L=	16	-1	277	246	3	269	240	6	378	391	11	267	242	9	169	59
-3	191	171	-4	514	522	-14	200	118	1	154	150	7	174	99	8	273	255	13	317	353	K=	7,L=	14
-1	422	419	0	460	486	-6	151	146	3	330	334	K=	5,L=	17	10	625	610	17	172	202	-7	234	223
1	255	244	6	148	147	-4	138	149	9	400	421	-9	164	119	K=	6,L=	8	K=	7,L=	3	-5	180	149
3	145	118	8	260	259	-2	293	298	11	170	225	-3	216	166	-16	228	204	-11	135	131	-3	257	287
K=	3,L=	18	10	159	146	0	164	112	13	141	159	-1	134	109	-14	138	110	-9	138	109	-1	247	263
-9	180	194	18	183	143	4	350	330	17	223	217	K=	5,L=	18	-12	333	350	-5	359	372	K=	7,L=	15
7	161	110	K=	4,L=	7	K=	4,L=	17	K=	5,L=	7	-11	184	98	-10	188	163	-3	287	273	-9	145	106
K=	3,L=	19	-18	190	197	-4	225	207	-19	219	222	K=	6,L=	0	-8	197	209	-1	118	95	-3	172	158
-1	200	168	-14	211	173	4	161	90	-15	291	267	0	241	239	-2	115	98	1	283	319	5	160	108
K=	3,L=	20	-12	163	155	K=	4,L=	18	-11	128	41	2	345	327	2	110	49	5	262	251	K=	7,L=	16
-3	144	38	-10	191	161	-10	185	109	-9	216	244	4	115	110	4	244	246	7	139	60	-5	292	245
K=	4,L=	0	-8	539	568	2	189	164	-5	597	600	6	583	605	6	424	422	9	190	167	-3	229	189
2	474	458	-6	198	194	K=	4,L=	19	-3	360	353	8	247	246	10	317	290	K=	7,L=	4	-1	197	163
4	600	478	-4	533	533	-2	208	188	-1	611	628	10	160	140	16	175	172	-17	171	157	1	149	94
6	290	321	-2	337	334	2	154	105	1	514	482	12	331	332	K=	6,L=	9	-13	131	166	3	187	179
8	781	775	2	376	363	K=	5,L=	0	5	352	383	K=	6,L=	1	-18	166	151	-11	179	175	K=	7,L=	17
10	209	200	4	504	513	1	487	470	7	505	498	-16	192	132	-14	230	227	-7	142	120	-5	166	145
14	294	278	6	251	245	7	338	327	9	134	116	-12	571	586	-12	244	241	-5	213	212	K=	8,L=	0
18	138	75	8	565	578	9	180	152	13	479	465	-10	135	170	-10	210	192	-3	240	258	2	337	344
K=	4,L=	1	10	312	377	15	215	230	15	289	279	-8	588	603	-8	152	114	3	262	251	4	131	153
-20	162	118	12	389	388	17	148	117	K=	5,L=	8	-6	216	218	-6	160	151	7	154	136	6	183	185
-18	122	37	16	162	216	K=	5,L=	1	-19	156	189	-4	333	330	-4	401	400	9	115	39	8	432	422
-16	229	216	K=	4,L=	8	-17	220	209	-17	260	220	0	443	463	-2	158	141	11	174	200	14	155	143
-10	296	278	-20	218	194	-15	154	100	-13	158	168	2	306	317	0	321	324	13	200	231	K=	8,L=	1
-8	343	317	-16	153	127	-11	174	150	-11	171	130	4	250	243	2	220	229	15	199	165	-16	131	53
-4	1011	994	-14	364	372	-9	779	737	-7	148	155	12	197	205	4	251	271	17	214	204	-14	293	271
-2	301	249	-12	136	121	-7	146	144	-5	131	138	14	234	238	6	492	496	K=	7,L=	5	-12	188	192
0	142	124	-8	373	368	-5	428	443	-3	356	350	16	153	122	8	246	239	-19	169	70	-8	517	537
2	367	365	-6	571	597	-3	177	134	1	519	527	K=	6,L=	2	12	194	188	-13	173	124	-6	360	360
4	321	321	-4	227	273	-1	228	185	3	378	390	-16	155	129	K=	6,L=	10	-11	125	156	-4	540	547
6	366	378	-2	133	161	1	507	506	5	174	177	-14	164	174	-18	143	131	-9	244	230	2	173	166
10	368	360	0	579	570	5	490	470	9	217	186	-10	617	629	-12	203	157	-7	115	94	4	145	108
12	134	122	2	535	536	7	159	182	11	281	308	-8	317	302	-10	262	236	-3	199	218	6	265	274
20	153	112	6	226	219	9	186	196	13	148	168	-6	376	342	-4	488	514	1	320	332	8	253	297
K=	4,L=	2	8	178	172	13	330	360	K=	5,L=	9	-4	324	323	-2	386	378	5	177	156	12	191	205
-20	143	85	10	156	160	15	504	515	-17	135	95	-2	147	152	0	525	528	7	188	128	14	142	150
-18	413	428	14	217	227	17	145	124	-15	221	212	0	363	367	2	281	285	9	227	277	K=	8,L=	2
-14	179	198	16	166	77	K=	5,L=	2	-13	250	250	4	148	161	4	218	213	11	158	112	-12	219	200
-10	257	278	K=	4,L=	9	-19	357	324	-7	311	312	8	224	257	K=	6,L=	11	K=	7,L=	6	-8	211	198
-8	613	630	-18	172	192	-17	198	199	-3	116	133	10	434	440	-18	228	217	-17	168	152	-2	202	181
-6	340	346	-16	210	180	-15	481	481	1	187	193	12	496	528	-14	314	293	-15	162	138	0	590	602
-4	156	113	-12	179	220	-11	247	258	5	659	572	16	271	295	-6	294	310	-11	288	327	4	304	294
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2	965	956	-2	434	433	-7	115	121	11	371	365	-18	133	45	4	220	224	-7	572	579	8	217	224
4	297	291	0	432	409	-5	657	661	13	163	158	-12	535	533	6	125	27	-5	309	309	10	387	414
6	1038	1052	2	365	392	-1	111	85	K=	5,L=	10	-10	145	161	10	221	215	-3	211	253	12	218	215
8	229	222	4	120	106	1	263	276	-17	153	156	-8	601	615	K=	6,L=	12	-1	187	167	K=	8,L=	3
10	291	302	6	205	210	3	465	458	-13	133	166	-6	698	701	-14	135	101	3	132	132	-12	123	121
12	147	144	8	339	325	5	164	168	-9	150	180	-4	209	212	-10	204	233	5	228	206	-10	120	103
14	302	296	10	410	413	7	342	352	-7	127	162	-2	628	638	-8	153	118	13	176	208	-8	410	420
18	256	232	16	175	127	11	467	467	-5	202	218	0	1301	1274	-4	278	315	15	139	165	-6	206	226
K=	4,L=	3	K=	4,L=	10	13	128	109	-1	228	221	2	264	281	-2	249	264	K=	7,L=	7	-2	210	229
-20	138	41	-16	134	110	15	127	10	3	297	292	4	167	141	2	206	201	-9	344	346	0	215	204
-16	176	183	-14	156	172	K=	5,L=	3	7	399	404	8	627	658	4	274	235	-5	253	259	4	172	212
-12	211	189	-10	351	367	-21	160	104	11	259	214	10	504	503	10	225	216	-3	140	35	6	127	122
-10	290	269	-8	497	498	-19	189	156	13	157	122	12	784	835	K=	6,L=	13	-1	141	89	8	209	227
-8	192	196	-6	133	122	-15	145	166	K=	5,L=	11	14	168	147	-8	140	100	5	131	110	12	274	271
-6	485	491	-4	156	108	-9	135	124	-13	190	157	16	234	154	-6	609	628	9	252	239	14	157	161
-4	218	208	-2	253	275	-7	118	119	-11	234	211	18	205	185	-2	599	590	K=	7,L=	8	K=	8,L=	4
-2	296	305	0	541	535	-5	188	214	-5	276	292	K=	6,L=	4	0	330	323	-9	150	133	-14	208	184
2	221	207	8	164	160	-3	300	311	-1	228	222	-14	158	119	6	136	115	-7	256	217	-4	341	342
4	1984	1972	12	264	264	-1	1718	1702	5	429	439	-12	311	325	K=	6,L=	14	-5	155	172	-2	229	256
6	219	183	K=	4,L=	11	1	116	151	7	253	242	-8	115	77	-6	490	480	-1	188	218	0	304	306
8	1034	1037	-20	177	148	3	736	729	11	133	51	-6	519	516	-2	224	234	1	279	279	4	278	291
10	115	71	-10	137	131	9	857	861	15	147	138	-4	561	571	K=	6,L=	15	3	309	336	6	171	193
12	160	173	-8	217	244	11	156	136	K=	5,L=	12	0	275	287	-8	175	173	5	270	272	8	233	221
14	587	572	-2	265	276	13	595	604	-11	200	181	2	274	238	-2	204	184	7	425	433	10	274	305
16	276	266	2	391	398	15	182	153	-9	123	94	4	284	308	0	132	147	15	187	210	14	205	196
18	364	362	6	338	338	19	248	235	-7	440	431	8	295	315	4	144	153	K=	7,L=	9	K=	8,L=	5
20	165	177	8	402	398	K=	5,L=	4	-5	244	224	14	247	225	K=	6,L=	16	-13	219	201	-14	211	203
K=	4,L=	4	10	138	179	-19	150	99	-3	411	407	16	265	287	-12	146	75	-11	205	158	-12	160	141
-20	138	54	K=	4,L=	12	-17	184	149	1	355	342	18	172	186	K=	6,L=	17	-5	207	202	-10	200	167
-18	363	357	-18	178	172	-15	498	496	3	224	244	K=	6,L=	5	-2	168	183	-3	388	387	-6	207	233
-14	553	549	-12	144	117	-13	144	167	5	362	397	-10	433	448	0	166	163	1	160	162	-4	691	607
-12	328	342	-8	127	97	-11	365	331	7	366	347	-8	603	621	4	196	136	3	323	314	0	270	260
-10	127	145	-6	235	239	-7	284	293	9	236	221	-6	172	185	K=	6,L=	18	7	421	435	2	424	434
-6	128	142	-4	664	671	-5	248	252	11	151	118	-4	363	370	-4	208	119	11	256	229	4	171	136

Table 2. Continued.

6	158	183	8	179	126	7	302	316	-9	145	90	-3	171	170	2	142	110	-2	154	209	9	144	167
8	151	106	10	167	126	9	414	428	-7	161	105	K=	9,L=	11	6	190	234	4	236	219	K=	11,L=	4
10	246	260	K=	8,L=	10	K=	9,L=	1	-3	291	315	-9	179	109	10	176	186	K=	10,L=	8	1	237	237
K=	8,L=	6	-10	267	194	-13	141	63	-1	242	244	-1	207	187	K=	10,L=	3	-6	187	177	K=	11,L=	5
-12	238	270	-8	219	195	-7	174	176	1	300	306	3	231	180	-10	212	196	0	160	136	-11	245	194
-10	177	121	-6	125	63	-3	206	220	7	125	92	K=	9,L=	12	-8	207	203	K=	10,L=	9	-9	200	177
-8	138	128	-2	442	406	1	127	142	9	147	140	-9	138	62	-6	522	527	-6	281	277	6	183	199
-6	125	68	2	318	328	3	316	316	K=	9,L=	6	-3	197	193	-4	405	428	-4	259	238	K=	11,L=	6
-4	432	428	4	231	211	7	373	379	-9	235	233	1	227	226	-2	188	237	4	180	164	-9	147	104
0	264	302	6	189	173	K=	9,L=	2	-3	486	490	K=	9,L=	13	0	341	299	K=	10,L=	10	-5	271	267
2	220	218	K=	8,L=	11	-9	157	187	1	473	484	-8	131	85	8	165	104	-8	206	172	-1	209	193
4	405	427	-14	153	131	-7	147	152	9	134	123	5	174	5	10	154	163	-4	204	215	K=	11,L=	7
6	275	299	-10	137	84	-3	559	556	11	215	255	K=	9,L=	14	K=	10,L=	4	4	145	136	-5	233	213
K=	8,L=	7	4	232	241	-1	216	218	K=	9,L=	7	-3	165	147	-6	204	186	K=	10,L=	11	K=	11,L=	9
-12	301	294	8	149	117	1	399	391	-15	150	80	K=	10,L=	0	-4	240	238	-8	147	81	3	183	188
-10	259	268	K=	8,L=	12	3	258	297	-13	181	189	2	148	131	-2	239	257	-4	159	128	K=	12,L=	0
-6	509	524	-14	182	157	5	241	259	-3	338	343	4	500	523	0	176	205	0	195	152	6	168	138
-4	126	72	-8	145	165	11	132	130	3	159	138	6	201	209	2	267	266	K=	10,L=	12	K=	12,L=	1
-2	353	371	-4	141	142	K=	9,L=	3	5	129	34	8	363	364	4	177	108	2	159	95	-8	153	159
2	241	240	2	135	162	-7	199	205	K=	9,L=	8	12	162	148	10	148	42	K=	11,L=	0	-4	275	276
6	234	220	4	198	193	-5	382	380	-9	286	296	K=	10,L=	1	K=	10,L=	5	1	180	165	-2	135	116
8	261	242	K=	8,L=	13	-1	376	402	-7	205	164	-12	226	175	-10	152	145	3	186	186	0	151	135
K=	8,L=	8	-6	175	190	7	147	169	1	298	254	-10	189	177	-6	239	243	K=	11,L=	1	2	225	214
-10	261	253	-4	287	275	11	152	134	3	160	156	-8	400	404	0	360	348	-3	166	147	4	152	163
-8	200	215	0	145	130	13	150	142	5	144	179	-4	476	472	2	151	138	3	198	181	K=	12,L=	2
-6	703	682	4	171	166	K=	9,L=	4	11	184	155	-2	203	211	4	185	161	K=	11,L=	2	2	157	91
-2	410	407	K=	8,L=	14	-11	171	150	K=	9,L=	9	0	397	415	8	186	189	-9	175	139	4	159	167
0	289	288	-4	181	200	-7	478	484	-11	219	185	2	279	276	12	196	177	-5	138	158	6	183	187
2	171	190	0	309	309	-5	219	225	-9	178	182	4	164	151	K=	10,L=	6	-3	152	111	K=	12,L=	3
4	220	227	4	160	102	-3	427	427	-7	159	167	8	200	183	-12	195	147	5	183	129	-4	264	190
K=	8,L=	9	6	137	73	1	330	323	1	315	312	12	175	115	-2	473	479	7	167	141	-2	154	119
-12	353	330	K=	8,L=	15	3	261	248	3	239	211	K=	10,L=	2	2	349	349	K=	11,L=	3	0	177	193
-10	316	303	0	146	129	5	165	195	5	139	156	-14	178	125	4	180	163	-11	196	187	4	160	119
-8	598	601	4	154	71	7	202	185	K=	9,L=	10	-10	259	237	10	145	127	-9	241	204	K=	12,L=	4
-6	247	300	K=	8,L=	0	11	169	92	-11	225	235	-8	150	152	K=	10,L=	7	-3	249	248	0	142	161
-2	200	198	1	286	280	K=	9,L=	5	-9	188	215	-2	481	449	-6	188	203	-1	255	250	K=	12,L=	7
2	278	316	3	356	362	-11	147	127	-5	313	281	0	441	435	-4	220	180	1	330	318	0	176	204
4	272	278	5	687	681																		

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

DISTANCE	(Å)	DISTANCE	(Å)	DISTANCE	(Å)
O1 - CC1	1,224(5)	O2 - CC2	1,228(5)	O3 - CC3	1,222(5)
O4 - CC4	1,225(5)	O5 - CC5	1,222(5)	N1 - CM1	1,467(6)
N2 - CM2	1,473(6)	N3 - CM3	1,462(6)	C4 - CM4	1,520(7)
N5 - CM5	1,463(6)	CC1 - N5	1,346(5)	CC2 - N1	1,345(6)
CC3 - N2	1,344(6)	CC4 - N3	1,348(6)	CC5 - N4	1,347(6)
CC1 - C1	1,529(6)	CC2 - C2	1,527(6)	CC3 - C3	1,522(6)
CC4 - C4	1,555(7)	CC5 - C5	1,520(6)	N1 - C1	1,438(5)
N2 - C2	1,448(5)	N3 - C3	1,455(5)	N4 - C4	1,466(5)
N5 - C5	1,461(6)	O1 - N4′	2,962(5)	OW - O2	2,809(5)

ANGLE	(°)	ANGLE	(°)
N5 - CC1 - O1	121,4(4)	N1 - CC2 - O2	122,3(5)
N2 - CC3 - O3	122,5(4)	N3 - CC4 - O4	120,6(5)
N4 - CC5 - O5	123,8(5)	C1 - N1 - CM1	117,4(4)
C2 - N2 - CM2	117,5(4)	C3 - N3 - CM3	117,2(4)
C5 - N5 - CM5	118,6(4)	N5 - CC1 - C1	117,8(4)
N1 - CC2 - C2	117,8(4)	N2 - CC3 - C3	116,0(4)
N3 - CC4 - C4	118,7(4)	N4 - CC5 - C5	115,8(4)
CC1 - C1 - N1	110,9(4)	CC2 - C2 - N2	112,1(4)
CC3 - C3 - N3	113,5(4)	CC4 - C4 - N4	108,4(4)
CC5 - C5 - N5	109,2(4)	C1 - N1 - CC2	122,5(4)
C2 - N2 - CC3	123,6(4)	C3 - N3 - CC4	124,3(4)
C4 - N4 - CC5	120,6(4)	C5 - N5 - CC1	116,1(4)
O1 - CC1 - C1	120,6(4)	O2 - CC2 - C2	119,8(5)
O3 - CC3 - C3	121,5(5)	O4 - CC4 - C4	120,7(5)
O5 - CC5 - C5	120,4(5)	CM1 - N1 - CC2	117,3(4)
CM2 - N2 - CC3	118,4(4)	CM3 - N3 - CC4	117,9(4)
CM5 - N5 - CC1	123,8(4)	CC4 - C4 - CM4	109,7(4)
N4 - C4 - CM4	110,2(4)	CC1 - O1 - N4′	134,8(3)
C4 - N4 - O1	113,0(3)	CC5 - N4 - O1′	123,2(3)
OW - O2 - CC2	116,5(3)	O2 - OW - O2″	98,4(2)

DIHEDRAL ANGLE	(°)	DIHEDRAL ANGLE	(°)
C1 - N1 - CC2 - C2	14,4(6)	N1 - CC2 - C2 - N2	-172,5(4)
CC2 - C2 - N2 - CC3	87,9(5)	C2 - N2 - CC3 - C3	4,9(7)
N2 - CC3 - C3 - N3	175,9(4)	CC3 - C3 - N3 - CC4	-125,5(5)
C3 - N3 - CC4 - C4	5,3(6)	N3 - CC4 - C4 - N4	-71,3(5)
CC4 - C4 - N4 - CC5	128,3(4)	C4 - N4 - CC5 - C5	179,8(4)
N4 - CC5 - C5 - N5	141,3(4)	CC5 - C5 - N5 - CC1	-68,1(5)
C5 - N5 - CC1 - C1	163,1(4)	N5 - CC1 - C1 - N1	-170,8(4)
CC1 - C1 - N1 - CC2	68,1(5)		

Hanson *et al.*[10] except for hydrogen.[11] The final fractional coordinates and thermal vibration parameters are given in Table 1. The expression for anisotropic vibration is:

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

The principal axes of the thermal vibration ellipsoids for oxygen, nitrogen, and carbon atoms were calculated from the temperature parameters of Table 1. Maximum root mean square amplitudes range from about 0.23 Å for ring atoms to about 0.33 Å for methyl carbon atoms and the water oxygen. Due to the size of the molecule, no rigid-body analysis of translational, librational, and screw motion has been carried out. A comparison between observed and calculated structure factors is presented in Table 2.

Interatomic distances, bond angles and dihedral angles are given in Table 3. The standard deviations, given in parentheses, are estimated from the correlation matrix of the last least squares refinement cycle. Fig. 1 shows the molecule viewed along [100].

By averaging bond distances of Table 3, and comparing with the results of the corresponding tetrameric [5] (I), pentameric [7] (II), and octameric [6] (III) compounds of sarcosine, no significant differences are observed:

Distance	(I)	(II)	(III)	AS4
CC–C	1.531 Å	1.527 Å	1.530 Å	1.525 Å
CC–N	1.358	1.344	1.345	1.346
CC–O	1.225	1.228	1.232	1.224
C–N	1.458	1.456	1.453	1.454
CM–N	1.467	1.483	1.487	1.466

The somewhat longer CM–N distances of (II) and (III) are possibly connected with the fact that for these compounds, methyl hydrogens were not included in the calculations. The geometry of the *cis* and *trans* *N*-methyl amide groups, respectively, is also roughly the same:

Angle	(I)	(II)	(III)	AS4
(CM–N–CC)*cis*	119.8°	118.7°	118.6°	117.9
(CM–N–CC)*trans*	124.3	123.9	123.5	123.8
(C–N–CC)*cis*	123.9	123.8	122.8	123.5
(C–N–CC)*trans*	120.1	117.2	117.5	116.1

Fig. 1 shows that the ring conformation is *cis, cis, cis, trans, trans*; quite unexpectedly the same as that of cyclopentasarcosyl [7] (II). Dihedral angles of AS4 and (II) agree closely except for CC3–C3–N3–CC4 (−125.5°) which has a value of −102.8° for the latter compound. Since the shortest CC···N distance across the ring is longer than 3.5 Å, no direct transannular contact can be held resposible for the rigidity of this 15-membered ring. As in the case of cyclooctasarcosyl and cyclopentasarcosyl, the explanation must be sought in the intrinsic conformation of the peptide chain itself.[2]

As indicated in Fig. 1, hydrogen bonds N4···O1′ and O1···N4′ of length 2.962 Å connect centrosymmetrically related molecules, forming dimers. Water molecules, situated at two-fold axes of rotation, link the dimers to endless chains along [001] with OW···O2 bonds of length 2.809 Å, the angle O2···OW···O2″ being 98.4°.

The OW–HW and N4–H4 bonds are 0.88 Å, while the C–H bond distances range from 0.89 Å to 1.03 Å.

Apart from the hydrogen bonds, there are no short inter-molecular contacts.

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