

Table 3. Average bond distances and angles for the cases $n = 3, 4, 5, 7, 8$.

$n =$	3	4	5	7	8
Distance (Å)					
CC—C	1.524	1.525	1.527	1.534	1.530
CC—N	1.351	1.346	1.344	1.343	1.345
CC—O	1.234	1.224	1.228	1.221	1.232
C—N	1.462	1.454	1.456	1.449	1.453
CM—N	1.468	1.466	1.483	1.446	1.487
Angle (°)					
(CM—N—CC) <i>cis</i>	118.0	117.9	118.7	118.6	118.6
(CM—N—CC) <i>trans</i>	—	123.8	123.8	122.8	123.5
(C—N—CC) <i>cis</i>	125.3	123.5	123.8	123.7	122.8
(C—N—CC) <i>trans</i>	—	116.1	117.2	118.4	117.5

^a Due to the more accurate data, the values are taken from the cycloalanyl tetrasarcosyl structure.¹³

sarcosyl is somewhat greater than those of the larger rings. It should also be pointed out that the significantly longer CM—N bonds of cyclopenta- and cyclooctasarcosyl are possibly connected with the fact that for these compounds methyl hydrogens were not included in the calculations.

There are no short inter-molecular contacts. A list of observed and calculated structure factors is available from the author.

Acknowledgement. The author thanks cand. real. K. Titlestad for preparing the crystals.

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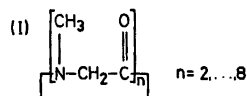
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Crystal Conformation of Cyclodeca-sarcosyl. $4\text{CH}_3\text{OH}$ at -160°C

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With exception for the case $n = 6$, the crystal structures of cyclic oligopeptides of sarcosine with the general formula I are known.^{1a-f} For



$n = 2, 3, 4$ and 8 the conformations could be predicted on the basis of NMR data.² For $n = 5$,

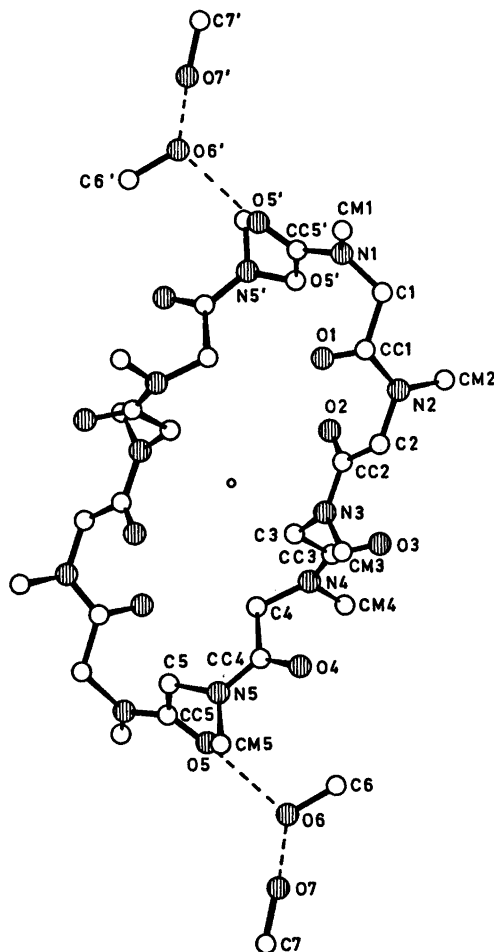


Fig. 1. Schematic drawing of the molecule.

Table 1. Final fractional coordinates and thermal parameters with estimated standard deviations. The expression for anisotropic vibration is $\exp[-2\pi^2(h^2a^{*2}U_{11} + \dots + 2klb^*c^*U_{23})]$. Hmn is bonded to Cm, HMmn to Cmm and HOM to Om.

ATOM	X	Y	Z	U11	U22	U33	U12	U13	U23
O1	-.4071(30)	-.05843(26)	.16415(24)	.0321(16)	.0367(17)	.0406(19)	.0150(14)	.0153(15)	.0087(15)
O2	-.2654(28)	.05991(26)	.4422(23)	.0257(16)	.0355(17)	.0509(19)	.0074(13)	.0045(14)	.0233(15)
O3	-.07281(27)	.34810(26)	.64195(22)	.0186(14)	.0400(17)	.0439(18)	.0163(13)	.0106(13)	.0121(14)
O4	.33845(28)	.47721(25)	.03618(22)	.0246(15)	.0315(15)	.0424(18)	.0132(13)	.0073(13)	.0175(13)
O5	.78970(31)	.44474(26)	.08903(25)	.0585(19)	.0283(16)	.0350(18)	.0205(14)	.0028(15)	.0056(14)
N1	-.76179(36)	-.24623(31)	.14853(28)	.0260(20)	.0269(20)	.0386(22)	.0123(17)	.0009(17)	.0009(17)
N2	-.51474(33)	.10868(30)	.30406(27)	.0107(17)	.0200(19)	.0352(21)	.0086(15)	.0043(16)	.0087(16)
N3	-.09230(34)	.22957(30)	.41900(26)	.0218(18)	.0280(19)	.0362(21)	.0086(15)	.0060(16)	.0111(16)
N4	-.14071(34)	.29895(31)	.71404(27)	.0229(18)	.0356(20)	.0332(20)	.0141(16)	.0064(16)	.0129(16)
N5	.53795(32)	.39386(29)	.66925(26)	.0181(17)	.0266(17)	.0366(21)	.0108(14)	.0080(15)	.0147(15)
C1	.72677(41)	.10860(36)	.02462(33)	.0229(22)	.0247(21)	.0429(26)	.0149(18)	.0054(19)	.0183(19)
C2	-.35785(43)	.19183(36)	.31000(33)	.0273(23)	.0254(22)	.0384(26)	.0074(19)	.0042(20)	.0128(20)
C3	.02067(42)	.20981(37)	.50492(35)	.0175(21)	.0345(24)	.0462(28)	.0090(19)	.0075(20)	.0102(21)
C4	.28131(41)	.27020(38)	.69369(32)	.0206(21)	.0331(23)	.0321(24)	.0093(18)	.0031(18)	.0154(19)
C5	.09896(40)	.20898(36)	.60666(32)	.0183(20)	.0255(22)	.0366(25)	.0100(17)	.0082(19)	.0075(19)
CC1	-.56629(42)	.011731(38)	.22799(34)	.0224(21)	.0299(23)	.0348(25)	.0149(19)	.0056(19)	.0146(20)
CC2	-.2421(42)	.15359(39)	.39929(33)	.0235(23)	.0256(22)	.0336(25)	.0076(19)	.0069(19)	.0062(19)
CC3	.02784(40)	.20481(36)	.6257(34)	.0121(19)	.0247(21)	.0387(26)	.0076(18)	.0094(19)	.0132(19)
CC4	.38907(41)	.38947(36)	.65486(30)	.0217(21)	.0279(22)	.0216(23)	.0119(18)	.0053(17)	.0041(18)
CC5	.69272(41)	.33386(39)	.02178(34)	.0224(22)	.0281(23)	.0370(26)	.0089(19)	.0068(19)	.0124(21)
CM1	-.03739(48)	-.27274(46)	.02780(36)	.0436(27)	.0473(31)	.0423(27)	.0204(24)	.0044(22)	.0181(24)
CM2	-.03640(45)	.14931(38)	.39231(34)	.0309(24)	.0287(23)	.0374(26)	.0097(19)	.0045(20)	.0049(20)
CM3	.03039(47)	.34684(40)	.37535(36)	.0353(25)	.0312(24)	.0479(29)	.0039(20)	.0132(22)	.0146(21)
CM4	.13739(45)	.30994(45)	.02921(35)	.0300(25)	.0660(31)	.0353(27)	.0218(23)	.0133(21)	.0157(24)
CM5	.02784(40)	.20481(36)	.6257(34)	.0121(19)	.0247(21)	.0387(26)	.0076(18)	.0094(19)	.0132(19)
O6	.74667(45)	.20961(28)	.07797(25)	.0589(22)	.0365(18)	.0547(21)	.0217(16)	.0091(17)	.0184(15)
O7	.01895(34)	.80016(31)	.20122(26)	.0390(19)	.0596(22)	.0647(23)	.0212(17)	.0067(17)	.0220(18)
C6	-.60146(50)	.73600(50)	.06490(44)	.0637(36)	.0627(34)	.0619(38)	.0338(29)	.0120(28)	.0162(29)
C7	-.00735(61)	.96746(50)	.14748(43)	.0607(39)	.0579(34)	.0719(37)	.0273(31)	.0123(31)	.0142(28)

ATOM	X	Y	Z	B	ATOM	X	Y	Z	B
HM1	-.7344(35)	-.1116(31)	.3043(26)	1.4(6)	HM12	-.0019(35)	-.0075(30)	.1994(26)	1.3(6)
HM2	-.3460(36)	.2024(31)	.3410(26)	1.6(6)	HM22	-.3296(34)	.1700(30)	.2459(26)	1.2(6)
HM3	.1280(32)	.2422(27)	.4062(23)	4(6)	HM32	-.0138(30)	.1122(31)	.5025(26)	1.9(7)
HM4	.2578(36)	.1922(31)	.6328(26)	1.7(7)	H42	.3285(34)	.2617(30)	.7630(25)	1.1(6)
HM11	.5159(31)	.2037(25)	.6796(22)	3(6)	H52	.6720(39)	.2780(34)	.6417(28)	2.6(8)
HM11	.7641(40)	-.2329(35)	.0117(28)	2.9(8)	HM12	-.0396(45)	-.2404(37)	.0263(31)	3.8(9)
HM13	-.0019(53)	.3845(47)	.0137(39)	7.7(12)	HM21	-.5904(43)	.0083(37)	.4535(31)	3.4(9)
HM22	-.5476(36)	.2441(31)	.4322(26)	1.6(7)	HM23	-.6976(42)	.1327(37)	.3617(31)	3.6(9)
HM31	-.1102(47)	.3928(40)	.3643(34)	4.5(10)	HM32	-.0250(42)	.3250(36)	.2953(31)	3.3(8)
HM33	.0540(47)	.4156(40)	.4265(35)	4.9(10)	HM41	.2312(42)	.4022(36)	.0356(31)	3.5(9)
HM42	.0502(43)	.3944(37)	.0420(31)	3.6(9)	HM43	.1959(45)	.3440(39)	.0039(32)	4.0(9)
HM51	.6334(36)	.4946(32)	.6680(27)	2.1(7)	HM52	.6385(43)	.5076(37)	.0019(31)	3.6(9)
HM53	.7330(50)	.5131(35)	.6780(30)	5.1(11)	H61	-.6458(50)	-.6884(45)	.0595(40)	6.1(11)
H62	-.6070(55)	-.0264(50)	.0332(40)	7.2(13)	H63	-.5198(45)	-.6083(39)	.1289(36)	3.8(9)
HM7	-.1,0931(60)	-.1,0464(53)	.0096(45)	7.6(15)	HM72	-.1,1192(53)	-.1,0403(47)	.0727(40)	7.8(13)
HM73	-.1,1509(60)	-.0262(53)	.1414(43)	7.6(15)	H06	-.7333(50)	-.6325(47)	.0927(39)	6.2(12)
HM7	-.0607(63)	-.0851(53)	.1467(43)	8.7(15)					

6, 7 and $n > 8$ no conformational evidence is obtainable from NMR spectroscopy. The crystal structure of cyclodecasarcosyl, $C_{30}H_{50}N_{10}O_{10}$, (crystallizing with four molecules of methanol per formula unit) is now reported.

The crystals belong to the triclinic system with space group $P\bar{1}$, cell dimensions (for Dirichlet's reduced cell) $a = 9.210(4)$ Å, $b = 10.540(5)$ Å, $c = 12.211(6)$ Å, $\alpha = 103.15(4)^\circ$, $\beta = 99.11(4)^\circ$, $\gamma = 105.82(3)^\circ$, and $Z = 1$ ($D_m = 1.28$ g cm $^{-3}$, $D_x = 1.29$ g cm $^{-3}$). The crystals are unstable at room temperature and data were therefore collected at -160°C (automatic four circle diffractometer, MoK α -radiation, 2570 observed reflections). No corrections for absorption or secondary extinction were applied (crystal size $0.4 \times 0.2 \times 0.3$ mm 3).

The structure was solved by direct methods³ and refined by full-matrix least-squares technique.^{4*} Except for the methylene hydrogens (the positions of which were calculated) all hydrogen atoms were localized in a difference Fourier map. Anisotropic temperature factors were introduced for O, N and C atoms and

* All programs used (except those for phase determination) are included in this reference.

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. The final weighted R -value was 5.3% (conventional $R = 6.4\%$) for 2570 observed reflections. The form factors used were those of Hanson *et al.*⁵ except for hydrogen.⁶

Final fractional coordinates and thermal parameters are given in Table 1. The principal axes of thermal vibration ellipsoids were calculated from the temperature parameters of this table, and the maximum r.m.s. amplitudes range from 0.172 to 0.273 Å (corresponding B -values 2.35 and 5.88 Å 2). Due to the size of the molecule no rigid-body analysis has been carried out.

Bond distances and angles and dihedral angles are listed in Table 2. The standard deviations (in parentheses) are estimated from the correlation matrix of the final least-squares refinement cycle.

Fig. 1 is a schematic drawing of the (centrosymmetric) molecule where the positions of the

Table 2. Bond distances, bond angles and dihedral angles with estimated standard deviations.

DISTANCE	(Å)	DISTANCE	(Å)
O1 - CC1	1.234(4)	O2 - CC2	1.225(4)
O3 - CC3	1.222(4)	O4 - CC4	1.235(4)
O5 - CC5	1.229(4)	N1 - CM1	1.463(5)
N2 - CM2	1.460(5)	N3 - CM3	1.452(5)
N4 - CM4	1.456(5)	N5 - CM5	1.453(4)
N5 - C1	1.451(4)	N2 - C2	1.434(5)
N3 - C3	1.440(5)	N4 - C4	1.453(4)
N5 - C5	1.452(4)	N1 - CC5'	1.337(4)
N2 - CC1	1.344(4)	N3 - CC2	1.345(4)
N4 - CC3	1.362(4)	N5 - CC4	1.345(4)
C1 - CC1	1.517(5)	C2 - CC2	1.536(5)
C3 - CC3	1.524(5)	C4 - CC4	1.523(5)
C5 - CC5	1.528(5)	C6 - O6	1.422(5)
C7 - O7		O6 - O5	2.753(4)
O6 - O7	2.693(4)		

ANGLE	(°)	ANGLE	(°)
O1 - C1 - C1	121.0(3)	O2 - C2 - C2	121.9(3)
O3 - C3 - C3	120.3(3)	O4 - C4 - C4	119.0(3)
O5 - C5 - C5	121.6(3)	O1 - C1 - N2	122.2(3)
O2 - C2 - N3	123.4(3)	O3 - C3 - N4	122.3(4)
O4 - C4 - N5	121.9(3)	O5' - CC5' - N1	122.0(4)
CM1 - N1 - C1	117.5(3)	CM2 - N2 - C2	117.0(3)
CM3 - N3 - C3	116.4(3)	CM4 - N4 - C4	114.0(3)
CM5 - N5 - C5	115.3(3)	CM1 - N1 - CC5'	119.0(3)
CM2 - N2 - CC1	111.1(3)	CM3 - N3 - CC2	124.3(3)
CM4 - N4 - CC3	118.0(3)	CM5 - N5 - CC4	120.1(3)
C1 - N1 - CC5'	121.1(3)	C2 - N2 - CC1	118.1(3)
C3 - N3 - CC2	118.3(3)	C4 - N4 - CC3	121.7(3)
C5 - N5 - CC4	123.6(3)	N1 - C1 - CC1	111.5(3)
N2 - C2 - CC2	111.9(3)	N3 - C3 - CC3	119.6(3)
N4 - C4 - CC4	113.9(3)	N5 - C5 - CC5	112.0(3)
C1 - CC1 - N2	116.9(3)	C2 - CC2 - N3	115.4(3)
C3 - CC3 - N4	117.2(3)	C4 - CC4 - N5	118.2(3)
CC5' - CC5' - N1	115.5(3)	CC5 - C5 - O6	147.4(3)
C6 - O6 - O5	105.6(3)	C6 - O6 - O7	116.6(3)
O5 - O6 - O7	111.5(1)		

DIHEDRAL ANGLE	(°)
N1 - C1 - CC1 - N2	-173.0(3)
C1 - CC1 - N2 - C2	173.5(3)
CC1 - N2 - C2 - CC2	-81.5(4)
N2 - C2 - CC2 - N3	-178.4(3)
CC2 - N3 - C3 - CC3	175.0(3)
CC2 - N3 - C3 - CC3	-83.0(4)
N3 - C3 - CC3 - N4	-172.5(3)
C3 - CC3 - N4 - C4	22.0(5)
CC3 - N4 - C4 - CC4	73.9(4)
N4 - C4 - CC4 - N5	165.3(3)
C4 - CC4 - N5 - C5	1.9(5)
CC4 - N5 - C5 - CC5	-181.3(4)
N5' - C5' - CC5' - N1	-175.9(3)
CC5' - CC5' - N1 - C1	-10.2(5)
CC5' - N1 - C1 - CC1	-72.2(4)

Table 3. Sequences of *cis* and *trans* configurations of the amide groups for cyclic oligopeptides of sarcosine (formula (I)).

<i>n</i>	sequence (c = <i>cis</i> , t = <i>trans</i>)
2	c, c
3	c, c, c
4	c, t, c, t
5	c, c, c, t, t
5 ^a	c, c, c, t, t
7	c, c, c, c, t, t, t
8	c, c, t, t, c, c, t, t

^a Cycloalanyl-tetrasarcosyl hemihydrate.⁷

four methanol molecules and the numbering of atoms are indicated. It may also be seen that the configuration of the ten *N*-methyl amide groups has the sequence *trans, trans,*

cis, cis, cis, trans, trans, cis, cis, cis. Corresponding sequences for other cyclosarcosyl molecules with known structure^{1a-f} are listed in Table 3. Fig. 1 shows that this 30-membered ring has a remarkably open conformation, and thus (in this respect) resembles that of the corresponding 24-membered ring of cyclooctasarcosyl.^{1b} It should, however, be pointed out that in the latter case the inner volume is filled by a cluster of four water molecules, which participate in a network of inter- as well as intramolecular hydrogen bond bridges. In the present compound the solvent molecules are all outside the ring and do not form any intermolecular bridges. Since no direct transannular interactions are present to be held responsible for the rigidity of this rather large and open ring, it must be due to an intrinsic rigidity of the peptide chain itself.⁸

Bond distances and angles are not significantly different from those of earlier findings.^{1f}

A list of observed and calculated structure factors is available from the author.

Acknowledgement. The author would like to thank cand. real. K. Titlestad for preparing the crystals.

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