the central double bond, do indeed enable the relief of the overcrowding. The resulting C(1)—C(1)′ and C(8)—C(8)′ distances, which are 3·38 (1) and 3·39 (1) Å, respectively, are below the usual van der Waals distance between carbon atoms in a linear C—H···H—C arrangement (4·0 Å; Bondi, 1964; Baur, 1972); however, they are significantly longer than the calculated distances in a hypothetically coplanar bistricyclic ethylene. It should be noted that even with these compensating modes of distortion, molecule (1) does not cease to be overcrowded, although the degree of overcrowding has been considerably lowered.

We could not find a simple correlation between the degree of overcrowding, folding, pyramidalization and twisting in (1). Although various such modes should be considered concomitantly, in many cases it may be expected that no more than one of these modes will be predominant (Greenberg & Liebman, 1978). We expect, however, that as more structures of different bistricyclic ethylenes are analyzed, we will be able to understand and correlate distortion modes and overcrowding in strained molecules in general, and in bistricyclic ethylenes in particular.

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Structure of *cis*-4-Hydroxy-*N*-triphenylmethyl-L-proline Methyl Ester

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Abstract. $C_{25}H_{25}NO_3$, $M_r = 387\cdot5$, monoclinic, $P2_1$, $a = 14\cdot7875\,(28)$, $b = 8\cdot7717\,(13)$, $c = 17\cdot5559\,(19)$ Å, $\beta = 114\cdot833\,(8)^\circ$, $V = 2066\cdot6\,(6)$ Å^3, $Z = 4$, $D_x = 1\cdot24$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1\cdot5418$ Å, $\mu = 6\cdot58$ cm^{-1}, $F(000) = 824$, $T = 293$ K. Final $R = 0\cdot049$ for 2763 observed reflections. The asymmetric unit contains two crystallographically independent molecules which show no significant differences in corresponding bond lengths or angles. These molecules are linked by an O—H···O hydrogen bond. The *N*-trityl group is oriented *trans* to the hydroxyl and methoxycarbonyl substituents.

Introduction. In a recent communication we reported the synthesis of *N*-tritylated derivatives of *cis*-4-

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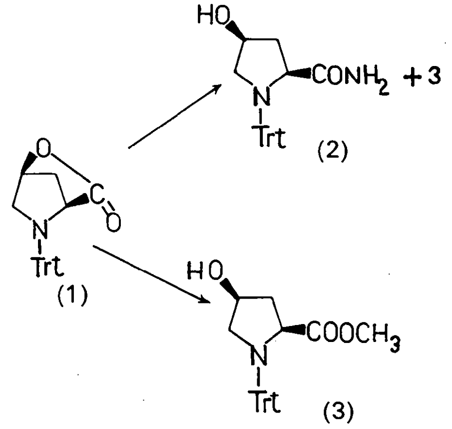
hydroxy-L-proline (Papaioannou, Stavropoulos & Karagiannis, 1988) by inversion of configuration at C(4) of *trans-N*-trityl-4-hydroxy-L-proline *via* an intramolecular dehydration reaction (Bowers-Nemia & Joullie, 1983; Portoghese & Turcotte, 1971). Amongst the derivatives prepared was *N*-trityl-2-oxa-5-azabicyclo[2.2.1]heptan-3-one (1) (Papaioannou, Stavropoulos, Nastopoulos, Voliotis & Leban, 1989) which proved to be a key intermediate for the synthesis of many other derivatives. The bridged bicyclic heterocycle (1) is a conformationally rigid molecule and exhibits some unexpected chemical behaviour, *e.g.* treatment of this compound with saturated ammoniacal methanol affords the expected *cis-N*-trityl-4-hydroxy-L-prolinamide (2) and the corresponding *cis-N*-trityl-4-hydroxy-L-proline methyl ester (3) in the ratio 3:1. On changing the organic solvent from methanol to 2-propanol only (2) was obtained. On the other hand, catalytic treatment of (1) with the system triphenylphosphine/diethyl azodicarboxylate (Bittner, Barneis & Felix, 1975) gave only the methyl ester (3) in 87% yield after purification by flash chromatography. The IR spectrum of (3) in Nujol showed a broad hydroxyl band at *ca* 3420 cm^{-1} due to intermolecular H bonding and an absorption band at 1715 cm^{-1} attributed to the carbonyl group. Compound (3), an important intermediate in the synthesis of biologically interesting peptides and of *trans*-4-substituted L-proline derivatives, is unexpectedly resistant to ammonolysis. We have determined its crystal structure in order to establish its conformation.



Experimental. The synthesis of the title compound (3) was effected by treatment of (1) (5 g, 14 mmol) in dry THF (80 ml) and anhydrous MeOH (20 ml), at 273 K, with triphenylphosphine (1·84 g, 7 mmol) and diethyl azodicarboxylate (1.1 ml, 7 mmol). The reaction mixture was left to attain room temperature and kept there for 48 h. Evaporation of the solvent under reduced pressure afforded an oil which was flash chromatographed on silica gel 60 (230–400 mesh) Merck, using as eluent toluene/ethyl acetate 9/1. The fractions containing the product were pooled, the solvent was evaporated *in vacuo* and the residue was recrystallized from diisopropyl ether/petroleum ether 60–80°: yield 4·7 g (87%), m.p. 402–403 K, $[\alpha]_D^{30°C}$ − 35·3° (c 1, $CHCl_3$). Colourless crystal 0·15 × 0·30 × 0·45 mm; Siemens Kristaloflex 805 diffractometer; graphite-monochromated Cu $K\alpha$ radiation; 31 reflections with $18 < \theta < 39°$ used for determining lattice parameters; data collected using ω–2θ scans up to $2\theta = 118°$; one standard reflection monitored after every 50 measurements showed no significant deviation from its mean intensity; 3189 unique reflections measured of which 2763 with $I > 2·5\,\sigma(I)$ used in refinement; range of hkl: $-16 \leq h \leq 14$, $-9 \leq k \leq 0$, $0 \leq l \leq 19$. Structure solved by direct methods (*MULTAN*87; Main, Germain & Woolfson, 1987) and refined by the full-matrix least-squares technique (*SHELX*76; Sheldrick, 1976). Final R = 0·049, wR = 0·058, $\sum w(\Delta F)^2$ minimized, $w = [\sigma^2(F) + 0·005002F^2]^{-1}$, 529 parameters refined, S = 1·05, $(\Delta/\sigma)_{\max}$ (for non-H atoms) = 0·63, max. and min. electron densities in final difference synthesis 0·29 and −0·20 e Å^{-3}. All non-H atoms refined with anisotropic thermal parameters; H atoms included using riding model [C—H 1·08 Å] with a fixed overall isotropic temperature factor U = 0·083 Å^2, except those attached to O(29) atoms, which were found from ΔF synthesis and refined isotropically. Atomic scattering factors as incorporated in *SHELX*76; for torsion angles and other geometrical calculations the programs *PARST* (Nardelli, 1983) and *XANADU* (Roberts & Sheldrick, 1979) were used.

Discussion. Table 1* lists the final atomic coordinates with equivalent isotropic thermal parameters, following the numbering scheme of Fig. 1 (*PLUTO*; Motherwell & Clegg, 1978). Bond lengths, angles and selected torsion angles for the two independent molecules are given in Table 2. A view of the crystal packing is shown in Fig. 2. The shapes of the two crystallographically independent molecules are similar but not indentical. Specifically, the pyrrolidine rings of molecules *A* and *B* both adopt the envelope conformation but each has a different atom out of the plane defined by the other four atoms of the ring. Thus, in molecule *A*, atoms N(20*A*), C(21*A*), C(22*A*) and C(23*A*) are planar to within ± 0·019 Å and C(24*A*) lies out of this plane by 0·402 Å, whereas, in molecule *B*, atom C(21*B*) is displaced by 0·458 Å

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and all torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52753 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2$) *with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
Molecule *A*				
C(1*A*)	10588 (2)	1388	7768 (2)	3·27 (9)
C(2*A*)	10156 (2)	794 (4)	8376 (2)	3·32 (10)
C(3*A*)	10349 (3)	−681 (5)	8716 (2)	4·06 (10)
C(4*A*)	9854 (3)	−1260 (5)	9172 (2)	4·77 (12)
C(5*A*)	9137 (3)	−400 (5)	9281 (2)	4·96 (13)
C(6*A*)	8933 (3)	1055 (5)	8960 (2)	4·53 (12)
C(7*A*)	9442 (3)	1648 (4)	8520 (2)	4·01 (10)
C(8*A*)	11572 (3)	571 (4)	7893 (2)	3·44 (10)
C(9*A*)	12323 (3)	328 (4)	8699 (2)	4·10 (12)
C(10*A*)	13235 (3)	−275 (5)	8804 (3)	4·77 (14)
C(11*A*)	13419 (3)	−660 (5)	8133 (3)	5·38 (14)
C(12*A*)	12693 (3)	−404 (5)	7341 (3)	5·30 (16)
C(13*A*)	11776 (3)	202 (5)	7223 (3)	4·47 (12)
C(14*A*)	10871 (2)	3071 (4)	7882 (2)	3·51 (10)
C(15*A*)	10958 (3)	3876 (4)	7239 (3)	4·11 (12)
C(16*A*)	11277 (3)	5365 (5)	7346 (3)	5·16 (16)
C(17*A*)	11548 (3)	6080 (5)	8091 (3)	5·36 (16)
C(18*A*)	11488 (3)	5308 (5)	8752 (3)	5·50 (15)
C(19*A*)	11152 (3)	3793 (4)	8656 (3)	4·51 (12)
N(20*A*)	9853 (2)	1095 (4)	6892 (2)	3·61 (8)
C(21*A*)	8947 (3)	2069 (5)	6578 (2)	4·15 (10)
C(22*A*)	8044 (3)	1031 (6)	6438 (3)	5·56 (16)
C(23*A*)	8452 (3)	−580 (6)	6633 (3)	5·49 (13)
C(24*A*)	9501 (3)	−481 (5)	6669 (2)	4·48 (12)
C(25*A*)	8834 (3)	2806 (6)	5759 (3)	5·12 (13)
O(26*A*)	8698 (4)	4268 (4)	5768 (2)	8·15 (16)
C(27*A*)	8629 (6)	5130 (8)	5039 (4)	9·14 (22)
O(28*A*)	8841 (3)	2114 (5)	5157 (2)	7·90 (15)
O(29*A*)	7844 (3)	−1706 (5)	6076 (2)	7·51 (13)
H(29*A*)	7797 (35)	−1172 (69)	5590 (33)	6·55 (24)
Molecule *B*				
C(1*B*)	5507 (2)	3715 (4)	7705 (2)	3·02 (9)
C(2*B*)	6491 (2)	4487 (4)	7822 (2)	3·26 (10)
C(3*B*)	7269 (3)	4608 (4)	8618 (2)	4·37 (11)
C(4*B*)	8186 (3)	5192 (5)	8717 (3)	5·18 (14)
C(5*B*)	8338 (3)	5618 (5)	8022 (3)	5·11 (14)
C(6*B*)	7579 (3)	5493 (5)	7222 (3)	4·59 (13)
C(7*B*)	6658 (3)	4929 (4)	7127 (2)	4·00 (12)
C(8*B*)	5121 (2)	4224 (4)	8369 (2)	3·19 (9)
C(9*B*)	5363 (2)	5649 (4)	8737 (2)	3·52 (10)
C(10*B*)	4877 (3)	6199 (5)	9225 (2)	4·32 (11)
C(11*B*)	4172 (3)	5328 (6)	9332 (2)	5·00 (13)
C(12*B*)	3940 (3)	3894 (5)	8974 (2)	4·53 (13)
C(13*B*)	4419 (3)	3346 (4)	8501 (2)	4·03 (10)
C(14*B*)	5763 (2)	1993 (4)	7764 (2)	3·45 (10)
C(15*B*)	5802 (2)	1261 (5)	7065 (2)	4·14 (11)
C(16*B*)	6158 (3)	−244 (5)	7149 (3)	4·68 (13)
C(17*B*)	6452 (3)	−1001 (5)	7895 (3)	5·38 (16)
C(18*B*)	6410 (3)	−283 (4)	8591 (3)	4·91 (12)
C(19*B*)	6070 (3)	1199 (4)	8518 (2)	3·98 (11)
N(20*B*)	4729 (2)	4069 (3)	6842 (2)	3·40 (7)
C(21*B*)	4386 (2)	5655 (4)	6713 (2)	3·44 (10)
C(22*B*)	3347 (3)	5657 (5)	6736 (2)	4·02 (11)
C(23*B*)	2937 (2)	4100 (5)	6402 (2)	4·47 (12)
C(24*B*)	3824 (3)	3110 (5)	6496 (2)	4·13 (10)
C(25*B*)	4330 (3)	6291 (5)	5894 (2)	4·58 (11)
O(26*B*)	4085 (3)	7788 (3)	5854 (2)	6·19 (10)
C(27*B*)	4075 (5)	8633 (7)	5142 (3)	7·95 (20)
O(28*B*)	4457 (3)	5645 (4)	5357 (2)	7·15 (14)
O(29*B*)	2290 (3)	4194 (5)	5525 (2)	8·43 (13)
H(29*B*)	1704 (39)	4712 (70)	5442 (32)	6·55 (27)

Table 2. *Bond lengths* (Å), *bond angles and selected torsion angles* (°).

	A	*B*		*A*	*B*
C(1)—C(2)	1·547 (5)	1·538 (5)	C(14)—C(19)	1·394 (6)	1·392 (5)
C(1)—C(8)	1·553 (5)	1·562 (5)	C(15)—C(16)	1·374 (6)	1·405 (6)
C(1)—C(14)	1·524 (3)	1·550 (5)	C(16)—C(17)	1·351 (7)	1·366 (7)
C(1)—N(20)	1·485 (4)	1·500 (3)	C(17)—C(18)	1·378 (8)	1·400 (8)
C(2)—C(3)	1·403 (5)	1·392 (4)	C(18)—C(19)	1·403 (6)	1·380 (5)
C(2)—C(7)	1·402 (6)	1·399 (6)	N(20)—C(21)	1·485 (5)	1·465 (5)
C(3)—C(4)	1·388 (7)	1·390 (6)	N(20)—C(24)	1·472 (5)	1·479 (4)
C(4)—C(5)	1·379 (7)	1·382 (8)	C(21)—C(22)	1·549 (6)	1·554 (6)
C(5)—C(6)	1·367 (6)	1·387 (5)	C(21)—C(25)	1·520 (6)	1·511 (6)
C(6)—C(7)	1·386 (7)	1·391 (6)	C(22)—C(23)	1·518 (7)	1·509 (6)
C(8)—C(9)	1·400 (4)	1·384 (5)	C(23)—C(24)	1·528 (7)	1·522 (6)
C(8)—C(13)	1·370 (7)	1·388 (6)	C(23)—O(29)	1·415 (6)	1·433 (4)
C(9)—C(10)	1·386 (6)	1·413 (6)	C(25)—O(26)	1·300 (6)	1·356 (6)
C(10)—C(11)	1·359 (8)	1·367 (7)	C(25)—O(28)	1·223 (7)	1·180 (6)
C(11)—C(12)	1·372 (6)	1·383 (6)	O(26)—C(27)	1·453 (9)	1·448 (7)
C(12)—C(13)	1·387 (7)	1·385 (6)	O(29)—H(29)	0·950 (60)	0·934 (60)
C(14)—C(15)	1·383 (6)	1·409 (6)			

	A	*B*
C(8)—C(1)—C(2)	112·4 (2)	113·3 (3)
C(14)—C(1)—C(2)	113·9 (3)	103·3 (3)
C(14)—C(1)—C(8)	103·8 (3)	112·6 (3)
N(20)—C(1)—C(2)	108·7 (2)	108·9 (3)
N(20)—C(1)—C(8)	107·6 (3)	109·3 (3)
N(20)—C(1)—C(14)	110·2 (2)	109·4 (2)
C(3)—C(2)—C(1)	122·4 (3)	120·3 (3)
C(7)—C(2)—C(1)	120·4 (3)	120·6 (3)
C(7)—C(2)—C(3)	116·7 (4)	118·7 (3)
C(4)—C(3)—C(2)	121·4 (4)	120·4 (4)
C(5)—C(4)—C(3)	120·2 (4)	120·1 (3)
C(6)—C(5)—C(4)	120·0 (5)	120·7 (4)
C(7)—C(6)—C(5)	119·9 (4)	119·0 (4)
C(6)—C(7)—C(2)	121·8 (4)	121·1 (3)
C(9)—C(8)—C(1)	120·7 (4)	120·3 (3)
C(13)—C(8)—C(1)	121·2 (3)	119·9 (3)
C(13)—C(8)—C(9)	117·7 (4)	119·1 (4)
C(10)—C(9)—C(8)	120·4 (4)	119·6 (4)
C(11)—C(10)—C(9)	121·1 (3)	120·2 (4)
C(12)—C(11)—C(10)	118·8 (4)	120·2 (5)
C(13)—C(12)—C(11)	120·9 (5)	119·7 (4)
C(12)—C(13)—C(8)	121·0 (3)	121·0 (4)
C(15)—C(14)—C(1)	120·2 (3)	119·0 (3)
C(19)—C(14)—C(1)	121·4 (3)	121·7 (3)
C(19)—C(14)—C(15)	118·1 (3)	118·9 (3)
C(16)—C(15)—C(14)	121·3 (4)	119·2 (4)
C(17)—C(16)—C(15)	121·2 (5)	120·7 (5)
C(18)—C(17)—C(16)	119·3 (4)	120·4 (4)
C(19)—C(18)—C(17)	120·5 (5)	119·3 (4)
C(18)—C(19)—C(14)	119·6 (4)	121·4 (4)
C(21)—N(20)—C(1)	116·1 (3)	114·9 (2)
C(24)—N(20)—C(1)	117·5 (2)	118·3 (3)
C(24)—N(20)—C(21)	106·2 (3)	106·3 (3)
C(22)—C(21)—N(20)	107·7 (3)	106·2 (3)
C(25)—C(21)—N(20)	108·7 (4)	112·0 (3)
C(25)—C(21)—C(22)	110·8 (3)	111·4 (3)
C(23)—C(22)—C(21)	106·1 (3)	103·3 (3)
C(24)—C(23)—C(22)	104·9 (4)	106·7 (3)
O(29)—C(23)—C(22)	114·6 (3)	110·1 (4)
O(29)—C(23)—C(24)	114·0 (4)	107·8 (4)
C(23)—C(24)—N(20)	107·7 (4)	108·1 (3)
O(26)—C(25)—C(21)	111·3 (4)	108·3 (4)
O(28)—C(25)—C(21)	124·7 (4)	128·2 (4)
O(28)—C(25)—O(26)	124·0 (5)	123·4 (4)
C(27)—O(26)—C(25)	117·1 (5)	116·6 (4)

	A	*B*
C(21)—N(20)—C(24)—C(23)	−28·0 (4)	−17·0 (4)
C(24)—N(20)—C(21)—C(25)	−100·9 (4)	−92·3 (4)
C(24)—N(20)—C(21)—C(22)	19·2 (4)	29·4 (4)
N(20)—C(21)—C(25)—O(26)	−129·9 (4)	−175·2 (3)
N(20)—C(21)—C(25)—O(28)	−51·9 (6)	5·7 (6)
N(20)—C(21)—C(22)—C(23)	−3·3 (5)	−30·3 (4)
C(22)—C(21)—C(25)—O(26)	112·0 (4)	66·0 (4)
C(22)—C(21)—C(25)—O(28)	−66·2 (6)	−113·0 (5)
C(25)—C(21)—C(22)—C(23)	115·5 (4)	91·8 (4)
C(21)—C(22)—C(23)—C(24)	−13·2 (5)	19·4 (4)
C(21)—C(22)—C(23)—O(29)	−139·0 (4)	−97·2 (4)
C(22)—C(23)—C(24)—N(20)	25·7 (4)	−2·4 (4)
O(29)—C(23)—C(24)—N(20)	151·9 (4)	115·8 (4)
C(21)—C(25)—O(26)—C(27)	176·9 (5)	175·1 (4)
O(28)—C(25)—O(26)—C(27)	−4·9 (8)	−5·8 (7)

from the plane defined by the other four ring atoms which are coplanar within ± 0·013 Å. It is thus apparent that two different conformers, owing to pseudorotation, have been frozen out during crystallization.

The methoxycarbonyl and hydroxyl substituents on atoms C(21) and C(23) are *cis* to each other in both molecules. The bulky triphenylmethyl group on atom N(20) orients itself *trans* to these substituents,

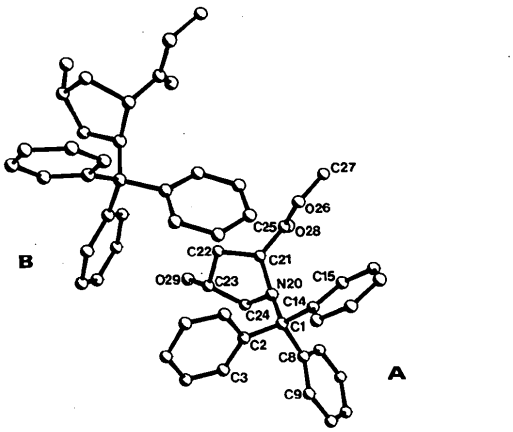

Fig. 1. Views of the two independent molecules, showing the atom-numbering scheme. The sequence of the phenyl ring C atoms is C(2)···C(7), C(8)···C(13) and C(14)···C(19).

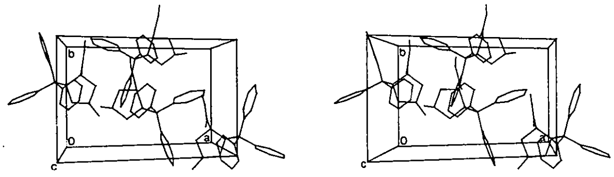

Fig. 2. Stereoscopic drawing of the packing of the title compound.

thereby avoiding steric congestion. By adopting this energetically favoured conformation the triphenylmethyl group alone does not hinder the carbonyl function from nucleophilic attack. Thus, the stability of (3) to ammonolysis must be related to the geometry of the ring portion of the molecule.

Both molecules show normal values of bond lengths and angles. Each *A* molecule forms an intermolecular O—H···O hydrogen bond with a neighbouring *B* molecule: O(29*A*)—H(29*A*)···O(29*B*^{i}) [(i): $1-x$, $-\frac{1}{2}+y$, $1-z$; O(29*A*)···O(29*B*) = 2·844 (6), H(29*A*)···O(29*B*) = 1·933 (61) Å and O(29*A*)—H(29*A*)···O(29B) = 160 (5)°]. There are no significant intramolecular contacts.

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11,28-Bis(chloroacetyl)-6,7,9,10,12,13,15,16,23,24,26,27,29,30,32,33-hexadecahydrodibenzo[*b*,*q*][1,4,7,10,16,19,22,25,13,28]octaoxadiazacyclotriacontine*

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Abstract. $C_{32}H_{44}Cl_2N_2O_{10}$, $M_r = 687·6$, monoclinic, $P2_1/c$, $a = 10·659$ (1), $b = 7·109$ (1), $c = 22·105$ (2) Å, $\beta = 90·80$ (1)°, $V = 1674·9$ (3) Å^3, $Z = 2$ (the molecule possesses a centre of symmetry), $D_x = 1·36$ Mg m^{-3}, Cu $K\alpha$ radiation, $\lambda = 1·54178$ Å, $\mu = 2·25$ mm^{-1}, $F(000) = 728$, $T = 293$ K, $R = 0·043$ for 2049 observed reflections with $|F_o| > 3\sigma(|F_o|)$. The structure of (9) has an extended shape with the azapolyether chains, which contain an equal number of *gauche* and *anti* C—C bonds, filling the potential

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* 2,5,11,14,21,24,30,33-Octaoxa-8,27-diazatricyclo[32.4.0.0^{15,20}]-octatriaconta-15,17,19,34,36,38(1)-hexaene.

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