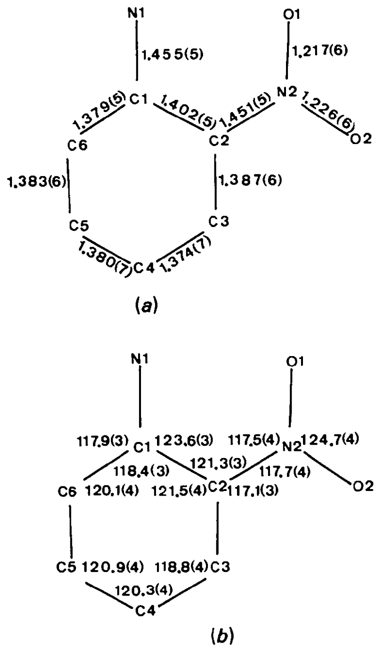

Fig. 2. The molecular structure of *o*-nitroaniline hydrochloride. (*a*) Bond lengths in Å. (*b*) Angles in degrees. (Values in parentheses are standard deviations.)

plane and the nitro group 26·1°). This is probably caused by overcrowding of the nitro and amino groups. The angle N(1)—C(1)—C(2) is 5·7° larger than the angle N(1)—C(1)—C(6) and the N(1) and N(2) atoms are bent out of the ring plane in opposite directions [N(1) by −0·08, N(2) by +0·09 Å]. The C(1)—C(2) bond is significantly longer than the other five C—C bonds. The latter bonds appear to be of equal length.

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Structures of 1-[(2*S*,3*S*)-2-(*N*,*N*-Dibenzylamino)-3-methyl-1-pentyl]pyridinium *p*-Toluenesulphonate, $C_{25}H_{31}N_2^+.C_7H_7O_3S^-$, and 1-[(2*S*)-2-(*N*,*N*-Dibenzylamino)-1-propyl]pyridinium *p*-Toluenesulphonate, $C_{22}H_{25}N_2^+.C_7H_7O_3S^-$

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Abstract. $C_{25}H_{31}N_2^+.C_7H_7O_3S^-$ (ILO), $M_r = 530·74$, monoclinic, $P2_1$, $a = 11·551$ (1), $b = 10·428$ (1), $c = 12·357$ (1) Å, $\beta = 94·72$ (1)°, $V = 1483·4$ Å^3, $Z = 2$, $F(000) = 568$, $D_x = 1·147$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 0·146 mm^{-1}, final $R = [\sum(|F_o| - |F_c|)/\sum F_o] = 0·042$ for 1733 unique reflections. $C_{22}H_{25}N_2^+.C_7H_7O_3S^-$ (ALO), $M_r = 488·64$, orthorhombic, $P2_12_12_1$, $a = 15·225$ (6), $b = 9·663$ (5), $c = 17·822$ (16) Å, $V = 2622$ (4) Å^3, $Z = 4$, $F(000) = 1040$, $D_x = 1·190$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 0·158 mm^{-1}, final $R = 0·045$ for 989 unique reflections. Both ILO and ALO are salts composed of a positively charged pyridinium moiety and a negatively charged tosyl group. With the exception of one of the phenyl rings, the structures of the pyridinium moieties of ILO and ALO are almost superimposable. The conformation about the C–C bond connecting the amino-group and pyridinium-ring N atoms is *gauche*, which results in crowded structures and short contacts between these N atoms, 2·926 (4) and 2·908 (11) Å for ILO and ALO respectively.

Introduction. Treatment of *N*,*N*-dibenzyl-L-isoleucinol with *p*-toluenesulphonyl chloride in pyridine did not result in the expected tosyl derivative, but yielded a crystalline solid of composition $C_{32}H_{38}N_2O_3S$ (within 0·1%) according to elemental analysis. Conductivity experiments and mass spectroscopy indicated the

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presence of a pyridinium fragment, but NMR spectroscopy did not support this observation. The tosylation of *N,N*-dibenzyl-L-alaninol also failed and yielded a product with a similar behaviour. In order to elucidate the structures of these unexpected products, X-ray analyses were undertaken.

Experimental. Rod-shaped crystals of ILO, pyramidal crystals of ALO, from chloroform–cyclohexane at room temperature. ILO: Nonius CAD-4 diffractometer, ω–2θ scan; ALO: Nonius CAD-3, ω scan; Zr-filtered Mo $K\alpha$, room-temperature lattice parameters from least squares for 25 reflections, angles measured by a centring routine associated with the diffractometer; standard reflections showed no significant changes; absorption corrected. ILO: 0·5 × 0·2 × 0·1 mm, 2771 independent reflections, $2\theta_{\max} = 50°$, 1773 with $I > 2{\cdot}5\sigma(I)$; ALO: 0·3 × 0·3 × 0·1 mm, 1326 independent reflections, $2\theta_{\max} = 39{\cdot}5°$, 989 with $I > 2{\cdot}5\sigma(I)$. Both structures: *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), Fourier methods, isotropic then anisotropic refinements, H by difference syntheses and included with fixed isotropic thermal parameters. ILO: $wR = 0{\cdot}049$, $w = [\sigma^2(F_o) + 0{\cdot}0005F_o^2]^{-1}$, $S = 1{\cdot}38$, average shift/error = 0·07, maximum shift/error = 0·54; ALO: $wR = 0{\cdot}053$, $w = [\sigma^2(F_o) + 0{\cdot}0008F_o^2]^{-1}$, $S = 1{\cdot}47$, average shift/error = 0·16, maximum shift/error 0·72; difference syntheses showed no densities outside ± 0·25 e Å^{-3}, scattering factors for non-H from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965); XRAY system (Stewart, 1976) used for all calculations.

Discussion. The atomic coordinates and equivalent isotropic temperature factors are listed in Table 1.*

Table 1. *Final atomic coordinates* (×10⁴) *and equivalent isotropic temperature factors* (Å² ×10²) *with e.s.d.'s in parentheses*

	ILO				ALO			
	x	*y*	*z*	U_{eq}^*	*x*	*y*	*z*	U_{eq}^*
S	2492 (1)	4531	5178 (1)	6·9 (1)	6040 (2)	6329 (3)	6128 (1)	6·8 (1)
O(1)	1257 (3)	4204 (5)	5079 (3)	10·6 (3)	6775 (4)	7272 (8)	6224 (4)	10·0 (4)
O(2)	2885 (3)	5093 (3)	4211 (3)	9·0 (3)	5493 (4)	6220 (8)	6783 (3)	9·5 (4)
O(3)	3239 (3)	3494 (3)	5572 (3)	9·1 (3)	6307 (4)	5008 (7)	5839 (3)	9·5 (4)
C(1)	2662 (4)	5761 (5)	6158 (3)	6·4 (3)	5368 (5)	7111 (10)	5433 (4)	5·2 (4)
C(2)	3753 (5)	6026 (6)	6636 (4)	7·8 (4)	4655 (6)	6448 (9)	5116 (6)	7·2 (5)
C(3)	3913 (5)	7060 (6)	7334 (4)	8·4 (4)	4135 (5)	7120 (11)	4593 (5)	6·1 (5)
C(4)	3024 (6)	7850 (5)	7560 (4)	8·3 (4)	4306 (6)	8426 (12)	4368 (5)	6·2 (5)
C(5)	1938 (5)	7544 (6)	7094 (4)	8·2 (4)	5017 (7)	9077 (10)	4648 (5)	8·2 (6)
C(6)	1742 (4)	6523 (5)	6396 (4)	7·1 (4)	5549 (6)	8424 (11)	5187 (6)	8·7 (6)
C(7)	3265 (7)	8988 (7)	8280 (5)	12·8 (7)	3730 (6)	9155 (11)	3777 (6)	9·9 (7)
C(8)	7227 (4)	5853 (5)	7671 (4)	6·5 (3)	6020 (6)	7972 (11)	2627 (5)	7·3 (5)
C(9)	7256 (6)	7134 (5)	7338 (5)	8·9 (5)	5279 (8)	7122 (11)	2567 (8)	8·8 (6)
C(10)	8063 (7)	7944 (5)	7827 (6)	10·6 (6)	4819 (6)	7048 (11)	1896 (8)	8·2 (6)
C(11)	8834 (6)	7515 (7)	8660 (5)	10·4 (5)	5120 (7)	7774 (12)	1303 (7)	8·5 (6)
C(12)	8798 (5)	6256 (5)	8997 (4)	7·5 (4)	5873 (7)	8630 (10)	1344 (5)	6·9 (5)
C(13)	8004 (4)	5406 (4)	8508 (4)	5·5 (3)	6332 (5)	8725 (10)	2016 (6)	5·8 (5)
C(14)	7977 (4)	4038 (5)	8848 (4)	5·9 (3)	7117 (6)	9672 (9)	2099 (5)	6·8 (5)
N(1)	7909 (2)	3132 (3)	7936 (3)	4·5 (2)	7870 (4)	9020 (7)	2499 (4)	5·6 (4)
C(15)	7511 (3)	1868 (4)	8237 (4)	5·5 (3)	8500 (5)	10082 (10)	2746 (4)	6·3 (5)
C(16)	6301 (3)	1905 (4)	8629 (3)	5·3 (3)	8227 (6)	10888 (10)	3446 (6)	6·2 (5)
C(17)	6047 (4)	1261 (5)	9547 (4)	7·5 (4)	8716 (6)	12016 (12)	3668 (7)	7·9 (6)
C(18)	4955 (5)	1324 (7)	9907 (4)	9·1 (5)	8528 (9)	12735 (12)	4313 (8)	10·1 (7)
C(19)	4101 (4)	2022 (6)	9368 (5)	8·9 (5)	7829 (9)	12345 (14)	4766 (6)	9·1 (7)
C(20)	4325 (4)	2658 (6)	8444 (5)	9·0 (5)	7323 (7)	11232 (14)	4545 (7)	8·7 (6)
C(21)	5419 (4)	2593 (5)	8066 (4)	7·2 (4)	7539 (7)	10496 (9)	3897 (6)	6·8 (5)
C(22)	9011 (3)	3139 (4)	7395 (3)	4·9 (3)	8287 (5)	7918 (10)	2041 (4)	5·3 (4)
C(23)	10030 (4)	2347 (5)	7982 (4)	6·3 (3)	8912 (6)	8418 (10)	1424 (5)	7·7 (6)
C(24)	8798 (3)	2736 (4)	6214 (3)	5·6 (3)	8769 (5)	6888 (9)	2531 (5)	6·0 (5)
N(2)	7985 (3)	3630 (3)	5612 (3)	5·0 (2)	8150 (4)	6237 (8)	3072 (4)	5·3 (3)
C(25)	6857 (4)	3321 (5)	5467 (3)	5·7 (3)	8050 (6)	6760 (9)	3764 (6)	5·8 (5)
C(26)	6066 (4)	4180 (6)	4948 (4)	7·7 (4)	7412 (7)	6243 (12)	4240 (5)	7·8 (6)
C(27)	6495 (6)	5346 (6)	4622 (4)	8·2 (4)	6868 (6)	5196 (12)	4006 (7)	7·9 (6)
C(28)	7640 (5)	5613 (5)	4770 (4)	8·2 (4)	6992 (7)	4644 (10)	3296 (6)	7·7 (6)
C(29)	8394 (4)	4762 (5)	5269 (4)	6·5 (3)	7633 (7)	5170 (11)	2840 (5)	6·5 (5)
C(30)	11196 (4)	3019 (6)	7855 (4)	7·7 (4)				
C(31)	11377 (4)	4220 (6)	8490 (5)	8·8 (5)				
C(32)	10096 (4)	970 (5)	7601 (5)	8·7 (4)				

$^*U_{eq} = (U_{11} + U_{22} + U_{33})/3$.

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* Tables of structure factors, anisotropic thermal parameters of the non-H atoms, coordinates of the H atoms and least-squares planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38157 (45 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The conformation of the title compounds and the numbering of the atoms are shown in Fig. 1. Both ILO and ALO are salt-like molecules composed of a positively charged pyridinium fragment and a negatively charged *p*-toluenesulphonate fragment. Bond distances and angles are given in Table 2, selected torsion angles in Table 3. Corresponding distances and angles in ILO and ALO show a good general agreement, the average deviation of these distances and angles being 0·014 (19) Å and 1·4 (12)° respectively. In the *p*-toluenesulphonate anion of ILO and ALO the tetrahedral surrounding of sulphur is distorted in an identical way, in that the tripod of S—O bonds is flattened with respect to the S—C bond. The average values of the O—S—O and O—S—C angles are 112·9 (2) and 105·7 (2)° for ILO and 112·9 (4) and 105·8 (4)° for ALO respectively. Both molecules have four phenyl rings and one pyridinium ring, which are all planar within 1–1·5 σ.

The conformations of the pyridinium fragments of ILO and ALO, consisting of common atoms, are almost identical. The torsion angles about five out of the seven bonds which determine the conformation of these fragments agree closely for the two compounds. The average difference of 12 pairs of common torsion angles about these five bonds, N(1)—C(14), N(1)—C(22), N(2)—C(24), C(13)—C(14) and C(22)—C(24), is 1·9 (11)° (Table 3), whereas the torsion angles about the two remaining bonds, N(1)—C(15) and C(15)—C(16), differ appreciably, 15·6 (12) and 35·7 (16)° respectively (Table 3). The conformational correspondence and difference of the pyridinium fragments is also illustrated by the angles between the three ring systems: the angles between the pyridinium ring (*a*) and phenyl ring (*b*) [C(8)···C(13)] differ by only 6·0 (8)°, whereas the angles between the pyridinium ring and phenyl ring (*c*) [C(16)···C(21)] and the angles between phenyl ring (*b*) and (*c*) differ by about 30·5 (8) and 18·4 (8)° respectively.

The torsion angles about C(22)—C(24), 60·7 (4) and 59·1 (9)° for ILO and ALO respectively, are of special interest in that these angles situate the amino group and pyridinium N atoms in an ideal *gauche* conformation at very short distances of 2·926 (4) and 2·908 (11) Å respectively. Together with the two substituent benzyl groups at the amino N, this *gauche* conformation causes the amino N to be completely encapsulated. This manifest preference for the folded *gauche* conformation over the stretched *trans* conformation may well be caused by the so-called '*gauche* effect'. This effect, which was introduced by Wolfe (1972), predicts that the conformation with the maximum

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

	ILO	ALO		ILO	ALO		ILO	ALO
S—O(1)	1·462 (4)	1·454 (7)	C(10)—C(11)	1·380 (10)	1·349 (18)	C(21)—C(16)	1·385 (6)	1·373 (14)
S—O(2)	1·437 (3)	1·437 (6)	C(11)—C(12)	1·379 (9)	1·415 (14)	C(22)—N(1)	1·486 (5)	1·484 (11)
S—O(3)	1·443 (4)	1·435 (7)	C(12)—C(13)	1·378 (7)	1·389 (13)	C(22)—C(23)	1·567 (6)	1·531 (12)
S—C(1)	1·763 (5)	1·774 (9)	C(13)—C(8)	1·393 (7)	1·393 (14)	C(22)—C(24)	1·519 (6)	1·514 (12)
C(1)—C(2)	1·376 (7)	1·382 (13)	C(13)—C(14)	1·489 (7)	1·512 (12)	C(24)—N(2)	1·479 (5)	1·488 (11)
C(2)—C(3)	1·384 (8)	1·385 (14)	C(14)—N(1)	1·468 (6)	1·490 (11)	C(25)—N(2)	1·340 (6)	1·341 (12)
C(3)—C(4)	1·364 (8)	1·350 (15)	C(15)—N(1)	1·455 (6)	1·473 (11)	C(25)—C(26)	1·397 (7)	1·383 (14)
C(4)—C(5)	1·373 (9)	1·347 (13)	C(15)—C(16)	1·518 (5)	1·528 (13)	C(26)—C(27)	1·386 (9)	1·374 (16)
C(4)—C(7)	1·496 (9)	1·540 (13)	C(16)—C(17)	1·371 (6)	1·378 (14)	C(27)—C(28)	1·350 (9)	1·386 (16)
C(5)—C(6)	1·378 (8)	1·406 (14)	C(17)—C(18)	1·373 (8)	1·374 (18)	C(28)—C(29)	1·356 (7)	1·368 (14)
C(6)—C(1)	1·378 (7)	1·371 (14)	C(18)—C(19)	1·356 (8)	1·389 (19)	C(29)—N(2)	1·353 (6)	1·361 (13)
C(8)—C(9)	1·399 (7)	1·401 (15)	C(19)—C(20)	1·363 (8)	1·380 (18)	C(23)—C(30)	1·537 (6)	
C(9)—C(10)	1·362 (9)	1·387 (19)	C(20)—C(21)	1·385 (6)	1·397 (16)	C(23)—C(32)	1·515 (7)	
						C(30)—C(31)	1·484 (8)	

	ILO	ALO		ILO	ALO		ILO	ALO
O(1)—S—C(1)	106·2 (2)	105·0 (4)	C(9)—C(10)—C(11)	120·4 (6)	118·5 (10)	C(16)—C(21)—C(20)	120·6 (5)	121·6 (9)
O(1)—S—O(2)	113·5 (2)	113·3 (4)	C(10)—C(11)—C(12)	120·0 (6)	122·5 (11)	N(1)—C(22)—C(23)	115·3 (3)	115·7 (7)
O(1)—S—O(3)	113·9 (2)	112·4 (4)	C(11)—C(12)—C(13)	121·0 (5)	119·5 (9)	N(1)—C(22)—C(24)	110·7 (3)	111·2 (7)
O(2)—S—C(1)	104·5 (2)	105·3 (4)	C(12)—C(13)—C(14)	121·4 (4)	121·5 (9)	C(23)—C(22)—C(24)	110·8 (3)	108·7 (7)
O(2)—S—O(3)	111·4 (2)	113·0 (4)	C(8)—C(13)—C(12)	118·4 (4)	117·8 (8)	C(22)—C(24)—N(2)	110·7 (3)	110·2 (6)
O(3)—S—C(1)	106·5 (2)	107·0 (4)	C(8)—C(13)—C(14)	120·2 (4)	120·6 (9)	C(24)—N(2)—C(25)	119·1 (3)	120·6 (7)
S—C(1)—C(2)	119·1 (4)	122·8 (7)	C(13)—C(14)—N(1)	113·6 (4)	113·5 (7)	C(24)—N(2)—C(29)	118·9 (3)	119·2 (7)
S—C(1)—C(6)	121·5 (4)	120·1 (7)	C(14)—N(1)—C(15)	112·6 (3)	110·5 (6)	C(25)—N(2)—C(29)	121·9 (4)	120·0 (7)
C(2)—C(1)—C(6)	119·2 (4)	117·1 (8)	C(14)—N(1)—C(22)	110·4 (3)	111·7 (6)	N(2)—C(25)—C(26)	120·0 (4)	120·5 (8)
C(1)—C(2)—C(3)	119·6 (5)	120·5 (9)	C(15)—N(1)—C(22)	114·8 (3)	112·7 (6)	C(25)—C(26)—C(27)	117·4 (5)	120·2 (9)
C(2)—C(3)—C(4)	122·3 (5)	121·9 (8)	N(1)—C(15)—C(16)	112·1 (3)	115·0 (7)	C(26)—C(27)—C(28)	120·8 (5)	118·6 (10)
C(3)—C(4)—C(5)	116·8 (5)	118·8 (9)	C(15)—C(16)—C(21)	120·7 (4)	123·0 (8)	C(27)—C(28)—C(29)	120·9 (5)	119·8 (9)
C(3)—C(4)—C(7)	119·7 (6)	121·4 (8)	C(15)—C(16)—C(17)	121·1 (4)	119·4 (9)	C(28)—C(29)—N(2)	119·1 (5)	120·8 (9)
C(5)—C(4)—C(7)	123·5 (6)	119·8 (9)	C(17)—C(16)—C(21)	118·1 (4)	117·5 (9)	C(22)—C(23)—C(30)	110·1 (4)	
C(4)—C(5)—C(6)	122·7 (5)	120·4 (9)	C(16)—C(17)—C(18)	120·6 (5)	121·9 (10)	C(22)—C(23)—C(32)	114·2 (4)	
C(1)—C(6)—C(5)	119·3 (5)	121·2 (9)	C(17)—C(18)—C(19)	121·2 (5)	120·6 (11)	C(30)—C(23)—C(32)	109·5 (4)	
C(9)—C(8)—C(13)	120·5 (5)	121·4 (9)	C(18)—C(19)—C(20)	119·4 (5)	118·3 (11)	C(23)—C(30)—C(31)	114·7 (4)	
C(8)—C(9)—C(10)	119·6 (5)	120·2 (11)	C(19)—C(20)—C(21)	120·0 (5)	120·1 (10)			

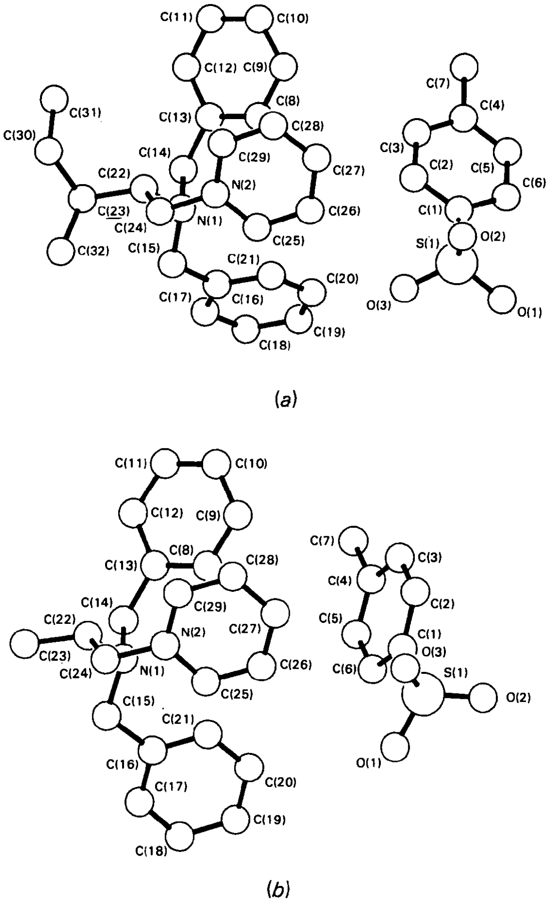

Fig. 1. Molecular structure and atomic labelling of (*a*) ILO and (*b*) ALO.

Table 3. *Selected torsion angles* (°) *with e.s.d's in parentheses*

	ILO	ALO
O(1)–S–C(1)–C(2)	162·6 (4)	171·7 (8)
O(1)–S–C(1)–C(6)	−22·3 (5)	−8·1 (9)
O(2)–S–C(1)–C(2)	−77·2 (4)	−68·3 (9)
O(2)–S–C(1)–C(6)	97·9 (4)	111·9 (8)
O(3)–S–C(1)–C(2)	40·8 (4)	52·1 (8)
O(3)–S–C(1)–C(6)	−144·1 (4)	−127·7 (8)
C(8)–C(13)–C(14)–N(1)	46·2 (6)	47·3 (11)
C(12)–C(13)–C(14)–N(1)	−133·0 (4)	−136·0 (9)
C(13)–C(14)–N(1)–C(15)	−161·4 (3)	−164·1 (7)
C(13)–C(14)–N(1)–C(22)	68·8 (4)	69·5 (9)
C(14)–N(1)–C(15)–C(16)	61·0 (4)	77·5 (9)
C(22)–N(1)–C(15)–C(16)	−171·5 (3)	−156·7 (7)
C(14)–N(1)–C(22)–C(23)	79·3 (4)	79·1 (8)
C(14)–N(1)–C(22)–C(24)	−153·9 (3)	−156·3 (7)
C(15)–N(1)–C(22)–C(23)	−49·3 (5)	−46·0 (9)
C(15)–N(1)–C(22)–C(24)	77·5 (4)	78·6 (8)
N(1)–C(15)–C(16)–C(17)	−134·4 (4)	−172·0 (9)
N(1)–C(15)–C(16)–C(21)	45·7 (6)	12·0 (13)
N(1)–C(22)–C(24)–N(2)	60·7 (4)	59·1 (9)
C(23)–C(22)–C(24)–N(2)	−170·0 (3)	−172·4 (7)
C(22)–C(24)–N(2)–C(25)	−97·2 (4)	−93·7 (9)
C(22)–C(24)–N(2)–C(29)	79·8 (4)	81·1 (10)
N(1)–C(22)–C(23)–C(30)	−144·5 (4)	
N(1)–C(22)–C(23)–C(32)	91·8 (5)	
C(24)–C(22)–C(23)–C(30)	88·7 (4)	
C(24)–C(22)–C(23)–C(32)	−34·9 (5)	
C(22)–C(23)–C(30)–C(31)	71·2 (5)	
C(32)–C(23)–C(30)–C(31)	−162·5 (5)	

number of *gauche* interactions between adjacent polar bonds is the preferred one. In spite of some ingenious theoretical treatments (Ponec & Chavalovský, 1974; Bingham, 1976; Whangbo & Wolfe, 1976; Exner, Jehlička, Trška & Engberts, 1977), the causes of the folding have not been defined clearly. Intramolecular dipole–dipole interactions, dipole–induced dipole interactions and dispersion forces have all been invoked to explain the conformational preferences. As was pointed out by Exner *et al.* (1977), this rule is still essentially empirical in character. A modified version of the *gauche* rule (Exner & Engberts, 1979) states that symmetrical positions of adjacent polar bonds and/or lone pairs are disfavoured. The configuration about C(22)–C(24) in ILO and ALO, with two polar bonds in the *gauche* conformation, follows the earlier concept of Wolfe, as well as the generalization of Exner & Engberts, that less-symmetrical forms are preferred for molecules with adjacent polar bonds and/or lone pairs.

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