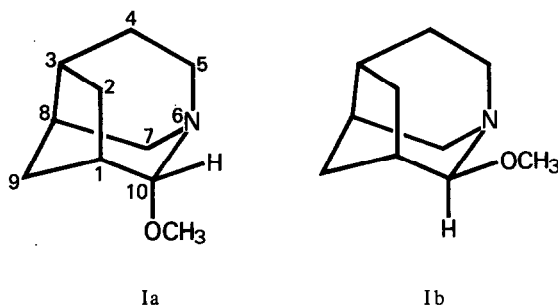


X-RAY STRUCTURE ANALYSIS OF (1RS, 10 SR)-10-METHOXY-6-AZATRICYCLO [4.3.1.0<sup>3,8</sup>] DECANE  
HYDROCHLORIDE

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The methanolysis of DL-erythro-5-chloro-1-azatricyclo [4.4.0.0<sup>3,8</sup>] decane leads to the formation of 10-methoxy-6-azatricyclo [4.3.1.0<sup>3,8</sup>] decane (I) <sup>1</sup>. Only one of the two possible isomers is obtained in this reaction, and the present X-ray analysis was performed in order to identify it unequivocally.



Crystals of the hydrochloride of I were obtained from chloroform solution and sealed in Lindemann capillary tubes to avoid decomposition by moisture. X-ray diffraction diagrams from these crystals are rather complex with the dominant reflexions displaying orthorhombic symmetry. We decided to neglect some very weak reflexions altogether and to try and solve the structure on the basis of an assumed orthorhombic unit cell of dimensions  $a = 13.681 \pm 0.013$  Å,  $b = 8.870 \pm 0.007$  Å,  $c = 17.103 \pm 0.021$  Å, containing 8 molecules of  $I \cdot HCl$ . We felt justified in this approach because the indexed reflexions account for more than 95 % of the total scattered intensity<sup>2</sup>. The systematic absences  $(0kl) : k=2n+1$ ,  $(h0l) : l=2n+1$  are compatible with either the centrosymmetric space-group  $Pbcm$  ( $Pbcm$ , # 57)<sup>3</sup>, or the non-centrosymmetric  $Pca2_1$  (# 29)<sup>3</sup>.

Table I.  
Fractional Atomic Coordinates, with Least Squares e.s.d.'s in parentheses.

Atom	Molecule A			Molecule B		
	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>
Cl	0.6623 ( 5)	-0.1082 ( 7)	0.6145 ( 5)	0.1337 ( 5)	0.6169 ( 7)	0.3928 ( 5)
O	0.4222 (14)	0.0126 (22)	0.4894 (14)	0.3796 (10)	0.4940 (22)	0.5055 (11)
N	0.5802 (14)	-0.0769 (18)	0.4480 (12)	0.2225 (14)	0.5755 (21)	0.5524 (12)
C (1)	0.4559 (14)	0.0207 (22)	0.3561 (15)	0.3582 (16)	0.4847 (27)	0.6413 (15)
C (2)	0.5321 (18)	0.0312 (28)	0.2898 (16)	0.2817 (17)	0.4612 (28)	0.7095 (16)
C (3)	0.5816 (17)	-0.1234 (27)	0.2940 (15)	0.2267 (17)	0.6142 (32)	0.7107 (16)
C (4)	0.6846 (15)	-0.1106 (29)	0.3346 (17)	0.1250 (18)	0.5964 (36)	0.6731 (19)
C (5)	0.6739 (15)	-0.0340 (25)	0.4084 (15)	0.1330 (19)	0.5247 (35)	0.5937 (18)
C (7)	0.5468 (23)	-0.2339 (25)	0.4269 (17)	0.2550 (20)	0.7319 (24)	0.5766 (14)
C (8)	0.5159 (17)	-0.2250 (25)	0.3415 (14)	0.2967 (18)	0.7229 (28)	0.6592 (15)
C (9)	0.4161 (18)	-0.1369 (29)	0.3402 (22)	0.3917 (22)	0.6426 (31)	0.6539 (20)
C (10)	0.4966 (16)	0.0410 (26)	0.4355 (13)	0.3097 (15)	0.4669 (23)	0.5616 (14)
C (O)	0.4376 (21)	0.0871 (34)	0.5604 (19)	0.3676 (18)	0.4208 (31)	0.4301 (17)

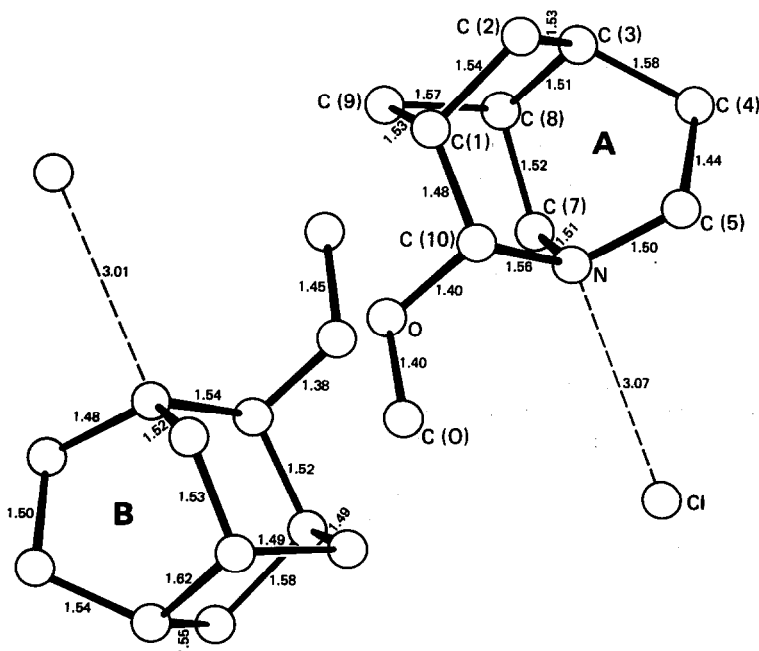


Fig. 1.

Numbering scheme and bond lengths (Å) in I. Molecules A and B are related by a pseudo inversion center.

Intensities of 1337 independent reflexions were measured on an automatic diffractometer using MoK $\alpha$  radiation with a graphite monochromator and corrected only for Lorentz-polarization factors. Direct methods were applied to solve the phase problem. Since E-value statistics were inconclusive with regard to the centrosymmetric or non-centrosymmetric nature of the crystals, it was first attempted to find a solution by using triple products in space group Pcam. When this failed, a multi-resolution weighted tangent formula procedure<sup>4)</sup> was applied on the basis of the non-centrosymmetric space-group Pca2<sub>1</sub>. Phases for the 200 highest E-values were developed from 5 starting phases, and 17 atoms could be located from the Fourier synthesis. One cycle of structure factor - Fourier calculation led to coordinates for all 26 non-hydrogen atoms in the asymmetric unit. Least squares refinement of coordinates and anisotropic temperature factors for all non-hydrogen atoms converged at  $R = \sum ||F_o| - |F_c|| / \sum |F_o| = 12.4\%$  for the 1064 reflexions with  $F_o > 4\sigma(F_o)$ , and  $R = 13.1\%$  for all 1252 'observed' reflexions<sup>5)</sup>. Obviously, these R values are unusually high by present day standards. They are, however, not surprising in the light of our rather arbitrary choice of space-group and the problems of measuring intensities in the presence of appreciable 'streaks' in reciprocal space.

Values of the refined fractional coordinates are reported in Table 1. A drawing of the contents of the asymmetric unit is shown in Figure 1<sup>8)</sup>, with the numbering scheme and values of the bond lengths ( $\sigma \sim 0.02 \text{ \AA}$ ). The geometry of the molecules is seen to agree, within the rather wide limits of accuracy of the analysis, with that of three-dimensional stick-and-ball models. The isomer is unequivocally identified as Ia, with the substituent at C(10) axial to the six-membered ring C(1)-C(10)-N-C(7)-C(8)-C(9). The four torsion angles which best illustrate this fact are listed in Table 2. A discussion of the chemical implications of our result is contained in the preceding paper by *Teufel, Heusler and Jenny*<sup>1)</sup>.

Table 2.

Torsion Angles Relevant to the Isomer Identification in Ia.

Angle	Molecule A	Molecule B
O - C(10) - C(1) - C(2)	174°	-178°
O - C(10) - C(1) - C(9)	60°	-64°
O - C(10) - N - C(5)	165°	-162°
O - C(10) - N - C(7)	-67°	71°

A crystallographic feature of some interest is evident from both the coordinate table and the drawing of the structure, namely the occurrence of an approximate inversion center relating the two molecules in the asymmetric unit. This pseudo center is located at  $x/a = 0.403 \pm 0.005$ ,  $y/b = 0.250 \pm 0.008$ ,  $z/c = 0.500 \pm 0.005$  in the orthorhombic cell chosen for our analysis. The true space-group may well contain this center of symmetry and lack some symmetry element of space-group Pca2<sub>1</sub>. We are presently trying to grow single crystals in order to find the true space-group.

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- 2) The neglected reflexions can probably be indexed in terms of the chosen unit cell with fractional index values for  $l$  ( $l = h \cdot n/4$ ).
- 3) International Tables for X-Ray Crystallography, Vol. I, The Kynoch Press, Birmingham (1952).
- 4) Our tangent formula program is based on formulae given by *M.M. Woolfson* in describing the MULTAN program <sup>6</sup>). The authors would like to thank *Dr W.E. Oberhänsli* for many helpful discussions and hints concerning multiresolution methods.
- 5) The quantity minimized was  $\sum w \left| |F_o| - |F_c| \right|^2$ , with a Hughes <sup>7</sup>) type weighting scheme. Weights based on  $\sigma(F_o)$  could not be used because of the dependence of this quantity on  $l$  due to the severe broadening of reflexions parallel to  $c^*$ .
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