Acid Tartrate of 3-(1-Methyl-2-octahydroindolyl)-1-propanol (X). A hot solution of 0.77 g (5 mmoles) (+)-tartaric acid in 8 ml abs. ethanol was added to a solution of 1 g (5 mmoles) aminoalcohol V in 8 ml absolute ethanol. Cooling and partial evaporation gave tartrate X as a crystalline precipitate with mp 132-133°C (from abs. ethanol) in 64% yield. Found: C, 55.3; H 8.6; N, 4.0%. Calculated for C_{16}H_{29}NO_{7}: C, 55.3; H, 8.3; N, 4.0%.

LITERATURE CITED

SATURATED NITROGEN-CONTAINING HETEROCYCLES.

12.* STRUCTURAL STUDIES OF CYCLOPENTA(b)PYRROLIDINYLALKANOLS


U13C NMR spectroscopy and x-ray diffraction structural analysis were used to establish the stereochemistry of 3-[N-methyl-2-cyclopenta(b)pyrrolinyl]-1-propanol and its acetyl derivative. The absolute configuration was determined for 3-[N-methyl-2-cyclopenta(b)pyrrolidinyl]-1-propanol acid tartrate dihydrate and the conformational aspects of its cation and tartrate anion were studied.

The present communication is devoted to a study of the stereochemistry of the isomers of cyclopenta(b)pyrrolidinylalkanols obtained in the hydrogenation of furfurylcyclopentylamines in aqueous acid solutions [2] and in the catalytic hydroamination of furfurylidene Cyclopentane- one in acidic water–ethanol [3].

Gas-liquid chromatographic analysis indicated that both these reactions proceed with steric specificity and the cyclopentapyrrolidine alcohols I–III are formed, independently of their method of preparation and nature of the catalyst used, as one of the possible geometric isomers with 97% chromatographic purity. 13C NMR spectroscopy, x-ray diffraction structural analysis

*For Communication 11, see [1].

and comparison with the structure of isomeric pentalanes which are carbocyclic analogs were used to establish the structure of these heterocyclic compounds.

\[
\begin{align*}
\text{I} & \quad \text{II} & \quad \text{III} & \quad \text{IV} & \quad \text{V}
\end{align*}
\]

\[
\begin{align*}
\text{I} & \quad \text{II} & \quad \text{III} & \quad \text{IV} & \quad \text{V}
\end{align*}
\]

\[\text{13C \{^1H\} NMR spectroscopy was used for the complete assignment of the signals of amino-alcohol II and its acetyl derivative IV (Table I). The methine, methylene, and methyl carbon atoms were distinguished using the off-resonance spectra. Considering the deshielding effect of the nitrogen atom and the strain of the condensed five-membered ring, the signals at 73.01, 39.38, and 66.00 ppm were assigned to the methine carbon atoms C(2), C(8), and C(3), respectively. The signal at 62.88 ppm was assigned to the carbon atom bearing the hydroxyl group, C(6), while the signal at 39.34 ppm was assigned to the methyl group carbon at nitrogen. The \[\text{13C NMR spectra data for the cis and trans isomers of 3-(1-methyl-2-pyrrolidinyl)-1-propanol [4] permitted the establishment of the cis orientation of the hydrogen atoms at C(2) and C(7). The dependence of the chemical shift of the carbon atom containing the hydroxyl group on molecular geometry was taken into account: This signal in the cis isomer is at 61-63 ppm, while it appears at 66-68 ppm in the trans isomer. The hydrogen atoms at C(2) and C(7) also occupy a cis position as indicated by the good agreement of the signals of angular C(2) in aminoalcohol II and substituted cis-pentalanes [5]. The signal for C(6) in 3-[1-methyl-2-cyclopenta(b)pyrrolidinyl]propyl-1-acetate (IV) is shifted downfield by 1.4 ppm due to the magnetic anisotropy of the acetyl group. The chemical shifts of the remaining carbon atoms correlate well with the signals for the analogous atoms in aminoalcohol II.}

Thus, cis fusion for the carbocyclic and heterocyclic rings and cis,cis orientation of the hydrogen atoms at C(2), C(7), and C(8) were established for II.

In order to confirm the conclusions concerning the structure of cyclopentapyrrolidinyl-propanol II on the basis of \[\text{13C NMR spectroscopy and to elucidate the conformation of this compound, we carried out an x-ray diffraction structural study of the acid tartrate (V) obtained from this alcohol and (+)-tartaric acid. The geometry of the cation and anion of salt V is shown in Fig. 1. The atomic coordinates and their isotropic temperature factors are given in Table 2. The bond and torsion angles are given in Tables 3 and 4, respectively.}

The absolute configuration of the asymmetric carbon atoms of the cation was unequivocally determined on the basis of the known absolute configuration of the anion of (+)-tartaric acid. According to the Cahn–Ingold–Prelog nomenclature, the asymmetric centers at C(2), C(7), and C(8) in the cation have R, S, and S configuration, respectively.

Each of the cis-fused five-membered rings of the 2-azabicyclo[3.3.0]octane system has envelope conformation. The heterocycle has a greater departure from planarity than found for the carbocyclic analog. Thus, C(2) extrudes from the plane of the remaining atoms which is found with a precision of ±0.07 Å by 0.79(2) Å (the flexure along the N–C(3) line is 50(1)°). On the other hand, the deviation of C(6) from the almost planar C(2)C(8)C(6)C(7) system (which is planar to ±0.05 Å) is 0.39(2) Å and the flexure along the C(3)–C(7) line is 26.2(9)°.

The cis fusion of the five-membered rings results in the approximate orthogonality of their mean planes: the dihedral angle formed by the C(7)C(8)C(3) and C(5)C(6)C(6)C(7)C(8) planes is 75(1)°.

The bond lengths and angles in the anion and cation of salt V have ordinary values. A detailed consideration of these values is not worthwhile in view of the relatively high ex-