RESEARCH ON 1-AZABICYCLIC COMPOUNDS

XII.* SYNTHESIS, SEPARATION, AND STEREOCHEMISTRY OF EPIMERIC 3-TERT-BUTYLPYRROLIZIDINES

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1-(2-Furyl)-3-amino-4,4-dimethylpentane was used to obtain 3-tert-butyl-1,2-dihydropyrrolizine, the catalytic hydrogenation of which over Rh/Al_2O_3 at room temperature gives a mixture of cis- and trans-3,8-H-3-ter-butylpyrrolizidines with predominance of the cis isomer, whereas hydrogenation at 90-100°C gives a mixture containing the trans isomer as the principal component. The three-dimensional structures of the isomers follow from data on the catalytic hydrogenation and isomerization and the IR, Raman, and PMR spectra. A considerable percentage of the trans-fused form is characteristic for cis-3,8-H-3-tert-butylpyrrolizidine.

It has previously been shown [2, 3] that pyrrolizidine exists practically completely in cis-fused conformations.

The introduction of a methyl group in the 3 position of a bicyclic compound in place of a hydrogen atom in the trans orientation with respect to 8-H‡ leads to the appearance of nonbonded interactions between the hydrogen atoms of the methyl group and the hydrogen atoms of the ring, and 5-H^t and 5-H^c are involved in the strongest interactions. A decrease in the interactions may occur on passing to the trans-

TABLE 1. Characteristics of the Synthesized Compounds

Compound	bp, °C (mm)*	d_4^{20}	n_D^{20}	MR_D found	MR_D calc.	Empirical formula	Found, % C	Found, % H	Found, % N	Calc., % C	Calc., % H	Calc., % N
IV	118—120 (19)	0,9380	1,4745	54,36	54,93	$C_{11}H_{19}NO$	73,0	10,7	7.6	72,9	10,6	7,7
V	114—115 (21)	0,9438	1,5049	51,30	51,60	$C_{11}H_{17}N$	81,4	10,6	8,5	80,9	10,5	8,6
VI	123,6 (80)	0,8982	1,4726	52,21	52,53	$C_{11}H_{21}N$	78,7	12.4	8,7	79,0	12,7	8,4
VII	122,8 (80)	0,8885	1,4690	52,44	52,53	$C_{11}H_{21}N$	78,9	12,5	8,5	79,0	12,7	8,4

*For a comparison of the boiling points, isomers VI and VII were distilled with a manostat at identical pressures.

*See [1] for communication XI.

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‡Here and subsequently, the numeral indicates the number of the carbon atom in the bicyclic compound. The superscripts c and t for H denote the spatial orientation of the hydrogen atom under consideration with respect to the 8-H atom. The character of the fusion of the ring (cis or trans) is determined by the relative spatial orientation of 8-H and the unshared electron pair of the nitrogen atom.

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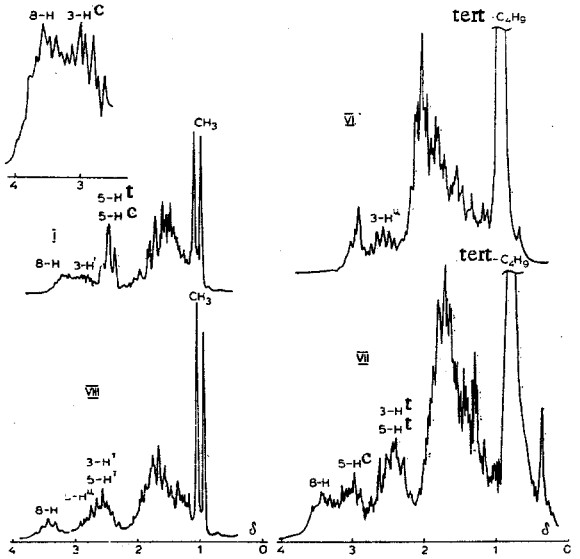
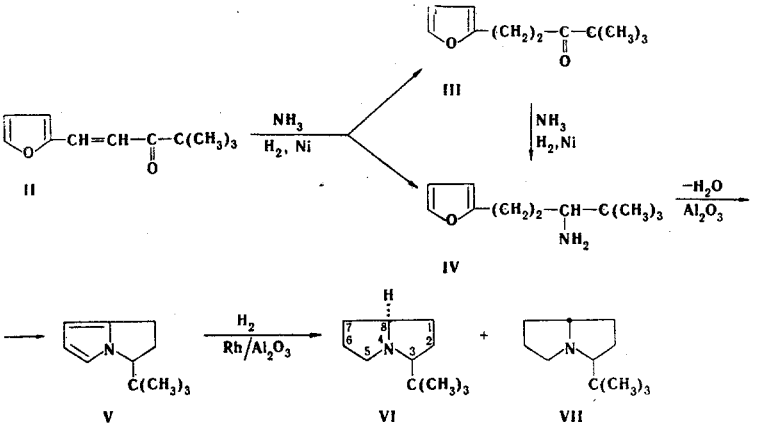


Fig. 1. PMR spectra of I, VIII, VI, and VII (pure liquids).

fused form in which the methyl group will occupy the preferred pseudoequatorial position. It is likely that the difference in energies between the cis- and trans-fused form of cis-3,8-H-3-methylpyrrolizidine (I) is less than the corresponding difference between the cis- and trans-fused conformations of pyrrolizidine, and the fraction of the trans-fused form of I may reach a definite level. A change in the equilibrium of the conformers of I was noted when the temperature was varied; however, the cis-fused forms are the predominant forms from −85 to 171°C [2].

The aim of the present research was to obtain stereoisomers of 3-tert-butylpyrrolizidines and to investigate their stereochemistry. The markedly strained epimer with a cis configuration, in which the fraction of the transfused form should be substantially higher than in I, seems of greatest interest.

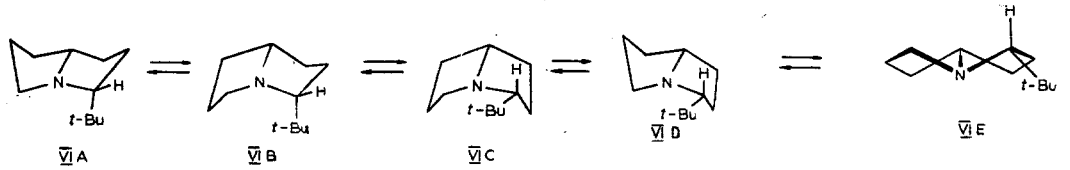
The 3-tert-butylpyrrolizidines were synthesized via the following scheme*:



Compounds VI and VII were isolated by fractional crystallization of mixtures of the perchlorates from mixtures of the bases, in one of which isomer VI predominated, whereas isomer VII predominated in the other. The physical properties of the compounds obtained are presented in Table 1.

On the basis of the known principles of the spatial relationships of the substrate and catalyst in the reactions of aromatic compounds with hydrogen [6] and the stereochemistry of the conversion of 3-methyl-1,2-dihydropyrrolizidine to epimeric 3-methylpyrrolizidines [7], it might be assumed that the catalytic hydrogenation of V under mild conditions would lead primarily to isomer VI.

Let us examine the principal conformations of VI and VII:†



*Furfurylidenepinacolone (II) [4] is a ketone with a markedly shielded carbonyl group. The reductive amination of II by the method in [5] proceeds slowly and gives, in addition to 1-(2-furyl)-3-amino-4,4-dimethylpentane (IV), 1-(2-furyl)-4,4-dimethyl-3-pentanone (III). The latter was subjected to reductive amination under the same conditions as II, and the overall yield of IV based on II reached 32%.

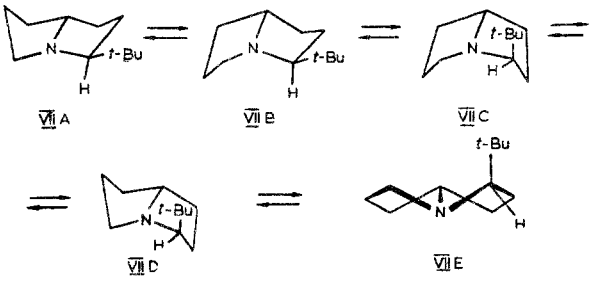
†The VIA, VIB (VID), and VIC conformations are analogous to the W, S, and U conformations of the cis-bicyclo[3.3.0]octane skeleton [8, 9]. We did not consider the other possible conformations of the cis-fused pyrrolizidine system of the T−S, T−W, and H−C type [9], inasmuch as their contribution is evidently small [8, 9].

TABLE 2. Chemical Shifts of the 8-H Proton in the PMR Spectra of 3-Methyl- and 3-Tert-Butylpyrrolizidines and Difference in the Chemical Shifts of the 8-H Protons of the Epimers at 25-34°C

Compound	Pure liquid		Solution					
			in cyclopentane			in benzene		
	δ, ppm	Δδ, ppm	concn., mole/liter	δ, ppm	Δδ, ppm	concn., mole/liter	δ, ppm	Δδ, ppm
I	3,22	0,25	1,30	3,22[2]	0.23	0,23	3,44[2]	0,08
VIII	3,47		1,20	3,45[2]		0,26	3,52[2]	
VI	⩽2,98	⩾0,43	0,93	⩽2,95	⩾0,37	0,20	⩽2,79	⩾0,51
VII	3,41		0,93	3,32		0,20	3,30	

The open (VIA) and semifolded (VIB) conformations with a pseudoaxial tert-butyl group are markedly strained because of considerable nonbonded interactions of tert-C_4H_9 with the hydrogen atoms trans-oriented with respect to 8-H. Repulsion of the closely situated 2-H^t and 6-H^t hydrogen atoms in folded conformation VIC is added to the strong interaction of tert-C_4H_9 with 5-H^t and 6-H^t. Of the cis-fused conformations, semifolded conformation VID with a pseudoequatorial orientation of the substituent should be considered preferable from the point of view of energy. However, in this case also very intimate steric contact between tert-C_4H_9 and 5-CH_2 is observed. The nonbonded interactions of the cis-fused conformations decrease in trans-fused form VIE with a pseudoequatorial tert-C_4H_9, which, however, is distinguished by considerable angular strain.

In cis-fused conformations of isomer VII the number of strong nombonded interactions with the participation of tert-C_4H_9 is lower than in the case of VI.



Conformations VIIA and VIIB with a pseudoequatorial orientation of the substituent will be thermodynamically preferable here. The other forms (VIIC, VIID, and VIIE) with a pseudoaxial tert-butyl group are strained.

It follows from the above that isomer VII has a smaller store of energy than VI. In fact, a mixture of 62% VI and 38% VII was converted at 95-100° on 15% Rh/Al_2O_3 to a mixture containing 5% VI and 95% VII.

A comparison of the PMR spectra of I and trans-3,8-H-3-methylpyrrolizidine (VIII) [2] with the PMR spectra of derivatives VI and VII (Fig. 1) reveals a similarity in the spectra of VII and VIII [2], which have a trans configuration at the 3-C atom. The multiplet at 3.41 ppm (IH) in the spectrum of isomer VII was assigned to 8-H. The complex signal at 3.09 ppm (IH) was assigned to $5H^c$. The signals of the two remaining protons attached to the α-carbon atoms – $3H^t$ and 5-H^t (2.14-2.82 ppm) – lie at stronger field and are overlapped with one another. The 1-C, 2-C, 6-C, and 7-C ring protons form a set of signals at 1.07-2.14 ppm.

Whereas the PMR spectra of VII and VIII are similar, the spectra of bases I and VI differ markedly (Fig. 1), and this indicates a difference in the conformational equilibria of the latter compounds. The shift of the lower boundary of the spectrum to stronger field is substantial in the spectrum of isomer VI: two signals, each of which corresponds to one proton, are found at 3.0 and 2.6 ppm. A group of markedly overlapped multiplets(10H) is located at 1.1-2.4 ppm. Whereas VI exists only in cis-fused conformations, the 5-H^c and 5-H^t hydrogen atoms undergo pronounced steric compression [10] on the tert-butyl group side, and the signals of these protons will therefore be found at lower field [10, 11] than in the case of isomer

VII. The opposite of this is observed experimentally. A comparison of the 8-H chemical shifts in the spectra of pyrrolizidine (3.37 ppm) [2], VIII, and VII (Table 2) shows that successive replacement of a hydrogen by CH_3 and tert-C_4H_9 changes the chemical shift of the indicated proton only slightly. In the case of isomer VI, the substituent is at a greater distance from 8-H than in the case of VII, and its effect on the chemical shift is consequently expressed even more weakly than in the case of VII. For this reason the chemical shifts of the 8-H proton in VI and VII should be approximately close in magnitude as in the case of epimers I and VIII; this is not in agreement with the experimental results.

Let us examine the interpretation of the PMR spectrum of isomer VI under the assumption that it exists in the VIE form. In this conformation the 8-H proton, existing in an antiparallel orientation with respect to the unshared electron pair of the nitrogen atom, experiences shielding similar to the shielding of the 10-H proton in quinolizidine [12] and should resonate at stronger field as compared with the other three α protons. The 5-H^t hydrogen atom occupies the pseudoequatorial position and, with respect to the given models, exists in a state of steric contact with the tert-butyl group, which entails additional deshielding of the proton [11]. The above-indicated information may serve as a basis for the assignment of the signal centered at 3.0 ppm to the 5-H^t proton. The pseudoaxial 3-H^c proton can be considered to be the X proton of an ABX system, where A and B are protons of the 2-CH_2 group. The signal at 2.6 ppm probably corresponds to the X portion of the three-spin system. The signal of the 5-H^c proton then lies at stronger field in the zone of overlapped multiplets above 2.4 ppm.

The relatively low values of the chemical shifts of the pseudoaxial α protons of the VIE conformation are not unusual, inasmuch as the environment of the 8-H and 5-H^c protons of the VIE conformer is similar to the environment of the axial 10-H and 4-H^c (6H^c) protons in quinolizidine, which have chemical shifts of 1.7 ppm* [13] (2.0 ppm [12] and 2.0 ppm [13], respectively. The position of the pseudoequatorial 5-H^t proton in VIE is similar to the position of the equatorial 6-H^t proton in cis-4, 10-H-4-methylquinolizidine [12, 14] (IX), which leads to the closeness of the chemical shifts of the 5-H^t proton in VIE (3.0 ppm) and the 6-H^t proton in IX (3.3 [12] or 3.15 ppm [14]).

Under real conditions of rapid exchange between conformations (probably with predominance of VIE), an averaged spectrum is observed. It is important that the general form of the X portion of the ABX system is retained in this case, and the signal with a chemical shift of 2.6 ppm pertains to 3-H^c.

The chemical shift of the 8-H proton is shifted to stronger field as the contribution of the trans-fused form to the equilibrium mixture of conformations increases [2]. From a comparison of the chemical shifts of the 8-H protons in the spectra of I, VIII, VI and VII, recorded for the pure liquids and solutions in cyclopentane and benzene (Table 2), it follows that the concentration of the trans-fused form in base VI is considerably higher than in the case of I, even if the multiplet at 3.0 ppm is assumed to be the signal of the 8-H proton.

The concept of the increased percentage of the VIE conformation is also confirmed by data from the vibrational (IR and Raman) spectra of VI and VII. A total of nine frequencies is observed in the Raman spectrum of VI below 650 cm^{-1}, whereas the Raman spectrum of VII has +13 frequencies in the same region (Table 3). The difference in the number of frequencies may be explained as follows. Although on the whole these two molecules do not have symmetry elements, the pyrrolizidine skeleton in conformer VIE is geometrically close to transbicyclo[3.3.0]octane, which has a center of symmetry (point group C_{2h}). The alternative prohibition is therefore possibly partially retained for some of these skeletal vibrations of the pyrrolizidine system in VIE. A comparison with the spectra of pyrrolizidine and trans- and cis-bicyclo [3,3.0]octane [15, 16] (Table 3) makes it possible to conclude that VII exists in cis-fused conformations, whereas isomer VI exists primarily in trans-fused form VIE. The bands of medium intensity at 540 cm^{-1} are apparently characteristic for the vibrations of the skeleton of the cis-fused conformations as a whole. This conclusion is confirmed by the spectra of various methyl- and dimethylpyrrolizidines that have primarily cis-fused conformations and absorption at 540 cm^{-1}. On the other hand, as in the spectrum of VI, this band is absent in the spectrum of cis-3,8-H-3-methyl-cis-5,8-H-5-methylpyrrolizidine, which exists primarily in the transfused conformation [17].

EXPERIMENTAL METHOD

The PMR spectra of pure liquid I and VI-VIII and of solutions of them in cyclopentane and benzene were recorded with BS-477 (60 MHz), Perkin-Elmer R-12 (60 MHz), and Hitachi-Perkin-Elmer R-20 (60

*For comparison, the values presented in [2, 12-14] on the τ scale were converted to the δ scale.

TABLE 3. Experimental Frequencies (cm^{-1}) of the Skeletal-Deformation Vibrations of Isomeric Bicyclo[3,3,0]octanes [15, 16], Pyrrolizidine, and tert-Butylpyrrolizidines in the Liquid Phase

trans-Bicyclo[3.3.0]-octane [15]			cis-Bicyclo[3.3.0]octane			Pyrrolizidine			cis-3,8-H-3-tert-butyl-pyrrolizidine (VI)			trans-3,8-H-3-tert-butyl-pyrrolidine (VII)		
IR	Raman	ρ	IR	Raman	ρ	IR	Raman	ρ	IR	Raman	ρ	IR	Raman	ρ
				197 m *	0,45		192 w	0,78		184 w	*dP*		150—180 vw	
													220 w	
				287 w	0,35		284 vw			287 m			294 m	
										324 sh				
				356 vw	*dP*		347 m	0,45		335 s	*P*		335 m	*P*
													388 vw	
372 w	—		390 w	390 m	0,14		386 m	0,51		397 m	*dP*		401 w	
—	436 vw								443 m	448 m	*P*	447 m	447 m	*dP*
465 w	—		465 w											
—	486 vs	*P*												
			510 w	514 w	0,18								496 sh	
				525 vw	*dP*				528 m	523 vs	*P*	515 vw	520 s	*P*
			532 m	532 m	0,25	532 m	536 m							
			545 sh	542 sh	*dP*	540 m	545 sh					542 m	545 m	
						552 m	559 sh							
—	571 m	*P*							572 m	576 m	*dP*	568 m	571 w	*dP*
	590 m	*dP*	584 m	585 m	<0,1									
610 w	—		612 m	613 vw	0,22	602 w	606 w	0,40		616 w	*dP*	605 w	608 w	
			665 vw			642 m	646 w	0,66	658 w			637 w	644 vw	

*Abbreviations: sh is shoulder, vw is very weak, w is weak, m is medium, s is strong, vs is very strong, P is polarized, and dP is depolarized.

MHz) spectrometers at 25-34° with hexamethyldisiloxane as the internal standard.* The chemical shifts are given on the δ scale.

The IR spectra of thin layers of the liquids were recorded with a UR-20 spectrometer. The Raman spectra were recorded with a Coderg PHO spectrometer at room temperature (excitation with the 6328 Å red line of an He–Ne laser).

The chromatographic analysis of mixtures of VI and VII was carried out with an LKhM-BM chromatograph with a thermal conductivity detector. The column (2.9 m long with an inner diameter of 3 mm) was filled with 12% triethanolamine on Spherochrome-1; the column temperature was 100° and the carrier gas (hydrogen) flow rate was 68 ml/min.

1-(2-Furyl)-3-amino-4,4-dimethylpentane (IV). A) A 780-ml rotating autoclave was charged with 98 g (0.55 mole) of ketone II [4] 400 ml of methanol saturated with ammonia, and 10 g of Raney nickel [18]. The initial hydrogen pressure was 128 atm. Reductive amination was carried out at 100-110° for 8 h, after which the catalyst was separated by filtration, the methanol was removed from the filtrate by distillation, and the residue was cooled and acidified with 18% HCl. Amine V went into solution as the hydrochloride salt. The layer of oil was separated, and the aqueous solution was extracted with benzene. The organic portion was washed with water and dried with Na_2SO_4. The benzene was removed by distilation, and the residue was fractionated at reduced pressure to give 54.5 g (54%) of ketone III with bp 116-118° (17 mm), d_4^{20} 0.9719, n_D^{20} 1.4662, and MR_D 51.39 (calculated 51.52) [bp 94-95° (8 mm), d_4^{20} 0.9757, and n_D^{20} 1.4639].

The aqueous layer (containing the hydrochloride of amine IV) was cooled and saturated with NaOH, and the resulting oil was separated. The aqueous solution was extracted with benzene, and the benzene extracts were combined with the oil. The resulting solution was dried with KOH, the benzene was removed by distillation, and the residue was distilled at reduced pressure to give 10 g (10%) of amine IV.

B) A 780-ml autoclave was charged with 107 g (0.95 mole) of ketone III, 400 ml of methanol saturated with ammonia, and 10 g of Raney nickel. The initial hydrogen pressure was 120 atm. The reaction was carried out at 100-110° for 25 h. The process was interupted every 8-9 h, a fresh portion of catalyst (10 g) was added to the autoclave, and hydrogen was again pumped in up to 120 atm. The catalyzate was worked up as in experiment A to give 44 g (41%) of amine IV.

1-(2-Furyl)-3-acetamido-4,4-dimethylpentane (IX). A 6.4-g (0.06 mole) sample of acetic anhydride was added to 3.75 g (0.02 mole) of amine IV, and the mixture was heated on a boiling-water bath for 1 h. It was then vacuum-fractionated to give 3 g (64%) of IX as a colorless liquid that began to crystallize rapidly at room temperature. The product had bp 173-174° (9-10 mm) and mp 82-83° (from 50% alcohol). Found: C 70.4; H 9.8; N 6.4% . $C_{13}H_{21}NO_2$. Calculated: C 69.9; H 9.5; N 6.3%.

3-tert-Butyl-1,2-dihydropyrrolizine (V). A quartz reactor with an inner diameter of 15 mm filled with an $Al_2O_3-ZrO_2$ catalyst [20] (bulk volume 60 ml) was placed in a cylindrical furnace with an adjustable heater. A stream of nitrogen was passed through the reactor constantly at 900-1000 ml / min, and the temperature on the surface of the reactor was maintained at 330-340°. A 43.6-g (0.24 mole) sample of amine IV was passed above the catalyst in the course of 3 h (the space velocity based on the liquid phase was 0.26 h^{-1}). Ether (80 ml) was added to the catalyst, and the ether solution was treated with 5% H_2SO_4 until the aqueous layer was acidic (pH 1-2). After this, the ether solution was washed with water and concentrated alkali solution and dried with KOH. It was then fractionated to give 26 g (66%) of V. The yields of V were 43-61% at dehydration temperatures of 340-380° when the catalyst was Al_2O_3, prepared by the method in [21] and thoroughly calcined at 700° for 3 h.

cis-3,8-H-3-tert-Butylpyrrolizidine (VI). A 610-ml autoclave was charged with 20.8 g (27 mmole) of freshly distilled V, 70 ml of ethanol, and 2.5 g of 15% Rh/Al_2O_3.† The initial hydrogen pressure was 120 atm. Hydrogenation was carried out at room temperature for 2 h and 20 min. In order to remove V residues, 18% HCl was added to the filtered catalyzate until the resulting solution was acidic (pH 3-4). The alcohol was then removed by distillation, and the unchanged V was extracted with ether. The aqueous solution was saturated with solid KOH, the organic layer was separated, and the aqueous solution was extracted with ether. The ether extracts were combined with the organic layer, and the ether solution was dried with KOH and distilled to give 14.7 g (69%) of a mixture consisting of 90% VI and 10% VII with bp 104-115°

*With the participation of K. Sh. Ovchinskii.

† In the preparation of the catalyst by the method in [22], the amount of rhodium chloride was selected in such a way as to have 15% of the metal on Al_2O_3.

(50 mm). The experiment was repeated four times with different batches of the catalyst. Depending on the activity of the catalyst used and the random deviation in the standard method of hydrogenation, the fraction of isomer VI in the mixture ranged from 95 to 82%. A 10-g sample of 60% $HClO_4$ (59 mmole) was added gradually to a solution of 9.8 g (58 mmole) of a mixture of VI (94%) and VII (6%) in 100 ml of ether, and the resulting white precipitate of the perchlorate was recrystallized twice from alcohol–ether (ethanol was added to a suspension of the perchlorate in ether until the salt dissolved completely in the boiling mixture) to give fine pinkish crystals with mp 227-228° (dec.). Found: C 49.4; H 7.9; N 5.3%. $C_{11}H_{21}N \cdot HClO_4$. Calculated: C 49.3; H 8.3; N 5.2%. A 2.85-g sample of the perchlorate of isomer VI was dissolved in water, and the free base was displaced by alkalization with solid KOH. Extraction with ether, drying, and distillation yielded 1.3 g of pure VI.

trans-3,8-H-3-tert-Butylpyrrolizidine (VII). A 150-ml autoclave was charged with 5.4 g (33 mmole) of V, 20 ml of ethanol, and 0.8 g of 15% Rh/Al_2O_3. The initial hydrogen pressure was 153 atm. Hydrogenation was carried out at 90-100° for 7 h. The catalyzate was worked up as described above for VI to give 4.7 g (85 %) of a mixture of isomers (5% VI and 95% VII) with b p 116-120° (55 mm). A 3.5-g sample of 60% $HClO_4$ (21 mmole) was added gradually to a solution of 3.5 g (21 mmole) of a mixture of the isomers (3% VI and 97% VII) in 30-40 ml of ether, and the resulting crystals (5.2 g) were recrystallized from water to give the perchlorate of isomer VII as fine pinkish crystals with mp 209-211°. Found: C 49.7; H 7.9; N 5.3% $C_{11}H_{21}N \cdot HClO_4$. Calculated: C 49.3; H. 8.3; N 5.2%; A 3.9-g sample of the perchlorate of isomer VII was treated in the usual way to give 2 g of pure isomer VII.

Catalytic Isomerization. A 3.4-g sample of a mixture of isomers consisting of 62% VI and 38% VII was heated in a scaled glass ampul with 0.5 g of 15% Rh/Al_2O_3 at 95-100° for 23 h. The resulting mixture of isomers contained 5% VI and 95% VII.

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