SYNTHESIS AND TRANSFORMATIONS OF 6,6,7,7-TETRAMETHYL-2-QUINUCLIDONE

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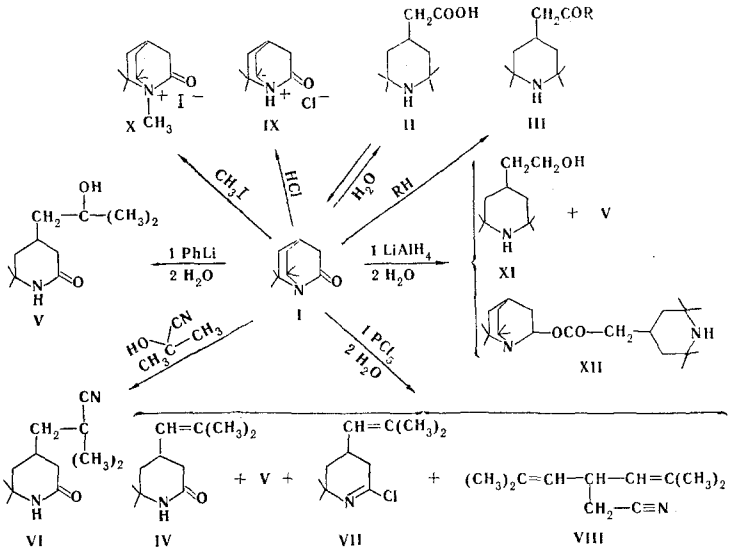
6,6,7,7-Tetramethyl-2-quinuclidone was synthesized, and its differences from the usual amides were shown. It enters into three types of chemical reactions: 1) the N–CO bond is broken under the influence of protic nucleophilic agents (water, alcohols, amines, hydroxylamine, and hydrazines), and nucleophilic agents are acylated by the (2,2,6,6-tetramethyl-4-piperidyl)-acetic acid residue; 2) the $N-C(CH_3)_2$ bond is broken by reaction with nucleophilic agents in aprotic media (phenyllithium in ether, PCl_5 in benzene, acetone cyanohydrin, and $LiAlH_4$ in ether) to form 4-substituted 6,6-dimethyl-2-piperidones; 3) reactions with the preservation of the quinuclidine ring occur on treatment with electrophilic reagents (hydrogen chloride and methyl iodide) in aprotic solvents and during reduction.

Bicyclic amides with an angular nitrogen atom of the 2-quinuclidone type are characterized by a rigidly fixed structure in which it is sterically impossible for the free p electrons of nitrogen and the π-electron cloud of the carbonyl group to be parallel. The absence of conjugation of the $\rangle N-C=O$ type, observed in the usual amides, provided a basis for Lukes and Fawcett [1, 2] to express doubt in the possibility of the real existence of such compounds. However, in 1956 we and M. V. Rubtsov [3] and in 1959-1965 Pracejus and co-workers [4, 5] reported the preparation of 2-quinuclidones by cyclization of acid chlorides of the corresponding 4-piperidylacetic acids in the presence of hydrogen chloride acceptors.



III: a R=OCH_3; b R=N⟨CH2CH2⟩O; c R=NHOH; d R=$NHNH_2$; e R=$NHNHC_6H_5$

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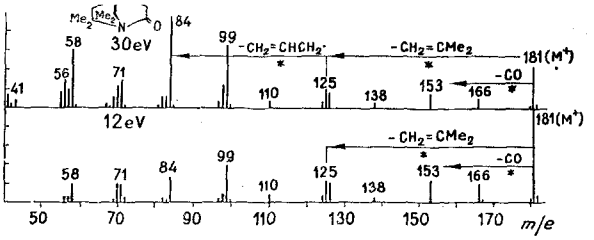
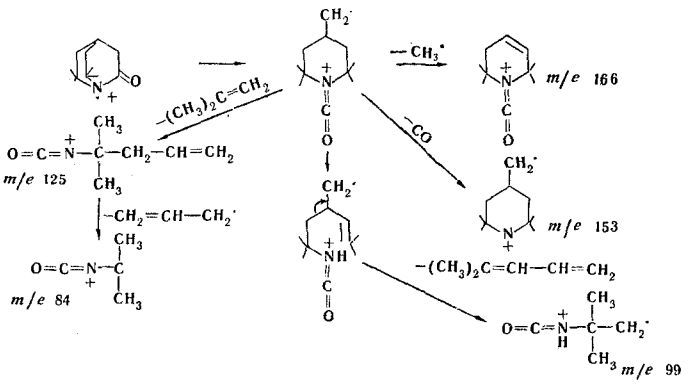


Fig. 1. Mass spectrum of 6,6,7,7-tetramethyl-2-quinuclidone.

In this paper we report the synthesis of a new representative of this series, viz., 6,6,7,7-tetramethyl-2-quinuclidone (I), and several peculiarities of its chemical behavior.

Compound I was synthesized from (2,2,6,6-tetramethyl-4-piperidyl) acetic acid (II) by converting it to the acid chloride with subsequent treatment with triethylamine. While the yields of the corresponding 2-quinuclidones were 40-72% for compounds that do not contain substituents or have two or three methyl groups in the α positions relative to the nitrogen atom [4, 5], steric hindrance due to the presence of four methyl groups led to a decrease in the yield in the step involving formation of I to 27%. The synthesized 6,6,7,7-tetramethyl-2-quinuclidone (I) has λ_{max} 230 nm ($\log \varepsilon$ 2.28) (in alcohol), pK_a 6.37 ± 0.05 (in water, determined potentiometrically), and a dipole moment of 3.95 D (determined in benzene), which agree with the characteristics of di- and trimethyl-2-quinuclidones [5]. The data of mass and PMR spectroscopy also are in agreement with structure I.

The fragmentation of amide I under electron impact at 30 and 12 eV (Fig. 1 [6]) is expressed by the following scheme:



Since amide conjugation in quinuclidone I is hindered, the high intensity of the molecular ion in the mass spectrum of this compound can be explained by an open form of M^+ as an amide fragment.

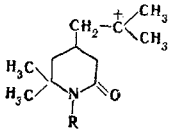
The PMR spectrum of I contains two singlets of six proton units (δ 1.03 and 1.41 ppm) which correspond to the CH_3 groups and attest to the presence of a plane of symmetry in the molecule. The H_4 proton gives a multiplet at 2.19 ppm (J = 2.9 Hz) which is characteristic for quinuclidine compounds [7]. The multiplet at 2.08 ppm (two proton units) is assigned to the C_3 protons, which interact with H_4, and the C_5 and C_8 protons, the signals of which form a group of lines from 1.25 to 1.55 ppm.

Since the effect of nitrogen on the carbonyl group in quinuclidone I is not characterized by combination of +M and −I effects as in the usual amides, but is determined only by the electron-accepting inductive interaction, an increase in the deficit of electrons on the carbon of the carbonyl group is observed in this compound and is manifested in the IR spectrum by an increase in the wave number of the valence vibrations of the C=O group. It is well known [8] that the valence vibrations of the carbonyl group of N,N-disubstituted amides are found at 1630-1670 cm^{-1}. This band is shifted to 1755 cm^{-1} in the IR spectrum of quinuclidone I, and in nitrogen-protonated compounds [hydrochloride and methiodide (IX and X)], where the −I effect is even more reinforced, the band is shifted to 1811 and 1820 cm^{-1}, respectively, i.e., to the region of the valence vibrations of the C=O group of the acid halides.

The increase in the electron deficit on the carbon of the carbonyl group leads to a considerable increase in the reactivity of the compound.

Quinuclidone I reacts with nucleophilic reagents considerably more readily than other amides and, in this respect, occupies an intermediate position between ketones and acid halides. The stabilization of the adducts formed occurs most frequently with breaking of the $N-C_2$ bond, and I thus acts as an acylating agent. Opening of the quinuclidine ring at the N–CO bond during the reaction of di- and trimethyl-2-quinucleidones with water and methanol has been previously described by Pracejus and co-workers [5] who, however, did not isolate the final products of the reaction but followed the course of the processes from the change in the pH of the medium, the angles of rotation, and the IR spectra of solutions. We made a systematic study of this type of reaction and showed that breaking of the N–CO bond to form (2,2,6,6-tetramethyl-4-piperidyl)acetic acid (II) or its derivatives (esters, hydrazides, amides, etc.) (IIIa-e) occurs in all cases during the reaction of compound I with various protic nucleophilic agents (water, alcohols, amines, hydroxylamines, and hydrazines). All of the final products of acylation of protic nucleophilic agents by substituted 2-quinuclidones were isolated in pure form, and their structures were confirmed by alternative synthesis from (2,2,6,6-tetramethyl-4-piperidyl)acetic acid.

Peculiar transformations are observed on treatment of amide I with nucleophilic agents and aprotic media (phenyllithium in ether, phosphorus pentachloride in benzene, acetone cyanohydrin, and lithium aluminum hydride in ether). All of these reagents also bring about breaking of the C–N bond in the quinuclidine ring. However, in contrast to the reactions described above, the process takes place at the $N-C(CH_3)_2$ bond rather than at the N–CO bond. Unstable carbonium ion A apparently forms initially in the process; A is stabilized either by cleavage of a proton to form 4-(2-methyl-1-propenyl)-2-piperidone (IV) or due to addition at the electrophilic center of the OH group (in the course of treatment of the reaction mass) or CN group to form, respectively, 4-(2-hydroxy-2-methylpropyl)-6,6-dimethyl-2-piperidone (V) or 4-(2-cyano-2-methylpropyl)-6,6-dimethyl-2-piperidone (VI). Thus, for example, 2-piperidone derivative V was obtained by the reaction of quinuclidone I with phenyllithium and was also isolated from the products of reaction with $LiAlH_4$ and PCl_5.



Carbonium ion A (R is a nucleophilic agent residue.)

The reaction of I with phosphorus pentachloride proceeds ambiguously: in addition to the two products of stabilization of carbonium ion A (IV and V), due to further interaction of the amide carbonyl with PCl_5 one also obtains a mixture of unstable mono- and dichloro derivatives, from which 2-chloro-4-(2-methyl-1-propenyl)-6,6-dimethyl-Δ^2-dehydropiperidine (VII) could be isolated. In addition, on treatment of amide I with PCl_4, the process does not terminate after breaking of only one $N-C(CH_3)_2$ bond. A part of the monocyclic reaction product undergoes breaking of a second $N-C(CH_3)_2$ bond and is converted to aliphatic nitrile VIII. The reaction of I with acetone cyanohydrin proceeds more unambiguously to form chiefly 4-(2-cyano-2-methylpropyl)-6,6-dimethyl-2-piperidone (VI).

The transformations of I with breaking of the $N-C(CH_3)_2$ bond have a certain analogy with the cleavage of sterically hindered amides described in [9]. However, in the latter case the process takes place under absolutely different conditions: on thermal treatment (with conversion to unsaturated compounds and nitriles) rather than during acid hydrolysis (with the formation of an unsaturated compound, ammonia, and acid). The formation of unsaturated amides from N-acylated, sterically hindered amides was also noted by a number of other investigators [4, 10-12], but the literature still does not contain any information regarding the cleavage of the $N-C(CH_3)_2$ bond under the influence of nucleophilic agents in aprotic media.

The above-described two types of conversions of amide I – with breaking of the N–CO or $N-C(CH_3)_2$ bonds – in all cases lead to structures which do not contain a quinuclidine ring. In addition, it turned out that 6,6,7,7-tetramethyl-2-quinuclidone, despite its high lability, is capable of reactions which occur with preservation of the quinuclidine structure. One such process is the reaction of quinuclidone I with electrophilic agents such as hydrogen chloride or methyl iodide in aprotic solvents, which leads to the formation

of salts IX and X, as well as its reduction with lithium aluminum hydride. The latter reaction occurs ambiguously. Above we noted the isolation from the products of this reaction of V, which is obtained by breaking of the $N-C(CH_3)_2$ bond and stabilization of the carbonium ion formed due to the addition of an OH group during treatment of the reaction mass with water. The second compound isolated from the products of this reaction is 2,2,6,6-tetramethyl-4-(2-hydroxyethyl)piperidine (XI), which is formed as a result of breaking of the N–CO bond in I. The structure of XI was confirmed by alternative synthesis from methyl (2,2,6,6-tetramethyl-4-piperidyl)acetate (IIIa). However, the most interesting product of the reduction of quinuclidone I with $LiAlH_4$ is 2-hydroxyquinuclidine derivative XII. It turned out that, in contrast to the usual amides and lactams, the carbonyl group in quinuclidone I is reduced to CHOH rather than to CH_2. The amino alcohol formed in the process is acylated under the reaction conditions by excess I to 2-[(2,2,6,6-tetramethyl-4-piperidyl)acetoxy]-6,6,7,7-tetramethylquinuclidine (XII). A triplet of the C_2 proton of the quinuclidone portion of the molecule with δ 4.12 ppm is clearly manifested in the PMR spectrum of XII, and a group of signals constituting 36 proton units from the remaining protons is observed at 0.73-2.17 ppm.

The results of the reduction of quinuclidone I depend to a considerable degree on the conditions used to carry out the reaction. The maximum quantity of XII is formed when the molar ratio of I and $LiAlH_4$ is 1:2, and the process is carried out for 3 h in refluxing ether. Increasing the amount of $LiAlH_4$ or raising the temperature (refluxing in tetrahydrofuran) shifts the process to favor substituted (2-hydroxyethyl)-piperidine (XI).

EXPERIMENTAL*

6,6,7,7-Tetramethyl-2-quinuclidone (I). Boiling thionyl chloride (150 ml) was added to 15 g (64 mmole) of the hydrochloride of (2,2,6,6-tetramethyl-4-piperidyl)acetic acid heated to 60°, and the mixture was refluxed for 3 h. The reaction mass was evaporated in vacuo, 30 ml of triethylamine and 100 ml of ether were added to the residue, and the mixture was refluxed for 20 h. The precipitate was removed by filtration, the solvent was removed by distillation, and the residue was vacuum distilled. The fraction with bp 100-101° (5 mm) was collected to give 3.14 g (27%) of I as a clear liquid with n_D^{20} 1.4930. Found %: C 72.8; H 10.7; N 7.7. $C_{11}H_{19}NO$. Calculated %: C 72.9; H 10.5; N 7.7. Hydrochloride IX was obtained as colorless crystals with mp 97-98°. Found %: Cl 16.3; N 6.4. $C_{11}H_{19}NO \cdot HCl$. Calculated %: Cl 16.3; N 6.4. Methiodide X was obtained as colorless crystals with mp 173-175° (decomp.). Found %: I 38.8; N 4.0. $C_{12}H_{22}INO$. Calculated %: I 39.3; N 4.3.

(2,2,6,6-Tetramethyl-4-piperidyl)acetic Acid (II). A mixture of 0.27 g (15 mmole) of I and 10 ml of water was heated for 30 min on a boiling-water bath, the mixture was evaporated to dryness, and the residue was triturated with acetone to give 0.28 g (94%) of acid II as a colorless powder with mp 280° (decomp.) which was soluble in water, only slightly soluble in alcohol, and insoluble in ether, acetone, and chloroform. Found %: C 66.4; H 10.3; N 7.2. $C_{11}H_{21}NO_2$. Calculated %: C 66.3; H 10.6; N 7.0.

Methyl (2,2,6,6-Tetramethyl-4-piperidyl)acetate (IIIa). A. Compound I [2 g (11 mmole)] was refluxed for 5 h with 20 ml of methanol, the solution was evaporated, and the residue was vacuum distilled to give 1.78 g (76%) of IIIa as a clear mobile liquid with bp 79-81° (1 mm) and n_D^{20} 1.4562. IR spectrum (ν, cm^{-1}): 1746 (CO). Found %: C 67.7; H 10.9; N 6.7. $C_{12}H_{23}NO_2$. Calculated %: C 67.6; H 10.9; N 6.6.

B. The hydrochloride of II [5 g (21 mmole)] was refluxed for 6 h in 50 ml of a 10% methanol solution of hydrogen chloride, and the solution was evaporated to dryness. The residue was made alkaline with a 50% solution of potassium carbonate, and IIIa was extracted with chloroform. The chloroform was removed, and the residue was distilled to give 3.3 g (73%) of IIIa. The hydrochloride was obtained as colorless crystals with mp 205-206°. Found %: Cl 14.5; N 5.7. $C_{12}H_{23}NO_2 \cdot HCl$. Calculated %: Cl 14.2; N 5.6. A mixture of the hydrochlorides of IIIa obtained by methods A and B did not give a melting-point depression.

(2,2,6,6-Tetramethyl-4-piperidyl)acetic Acid Morpholide (IIIb). A. A mixture of 1.35 g (7.5 mmole) of I and 13.5 ml of morpholine was refluxed for 12 h. The excess morpholine was removed by vacuum dis-

*All of the IR spectra of the compounds presented in this paper were obtained from mineral oil suspensions with a UR-10 spectrometer; the PMR spectra were obtained from CCl_4 solutions with a JNM 4H-100 spectrometer (100 MHz) with $Si(CH_3)_4$ as the internal standard; the mass spectra were obtained with an MKh-1303 mass spectrometer with a cylinder admission system at 20-50° with ionizing voltages of 30 and 12 eV; the UV spectra were obtained with an EPS-30 spectrometer. We thank Yu. N. Sheinker, K. F. Turchin, L. M. Alekseeva, and Yu. I. Pomerantsev for conducting the spectral investigations.

tillation, and the residue was recrystallized from ethyl acetate to give 1.95 g (97%) of IIIb as colorless crystals with mp 87-89°, which was quite soluble in the usual organic solvents. Found %: C 66.9; H 10.4; N 10.2. $C_{15}H_{28}N_2O_2$. Calculated %: C 67.1; H 10.5; N 10.4.

B. The hydrochloride of II [5 g (21 mmole)] was heated for 3 h with 50 ml of thionyl chloride at 80°, the thionyl chloride was removed by distillation, 20 ml of benzene and 50 ml of morpholine were added, and the mixture was stirred at 50° for 4 h. The precipitated morpholine hydrochloride was removed by filtration, the benzene was distilled, and the residue was recrystallized from ethyl acetate to give 6.35 g (67%) of IIIb with mp 88-89°. A mixture of IIIb obtained via methods A and B did not give a melting-point depression.

Hydrochloride of (2,2,6,6-Tetramethyl-4-piperidyl)acetohydroxamic Acid (IIIc). A mixture of 0.5 g (2.7 mmole) of I and 0.19 g (2.7 mmole) of hydroxylamine hydrochloride was refluxed for 2 h in 6 ml of alcohol. The precipitated colorless crystals were filtered and washed with ether to give 0.4 g (62%) of IIIc with mp 248-249°, which was quite soluble in water and aqueous alcohol and slightly soluble in ether, chloroform, and absolute alcohol. IR spectrum (ν, cm^{-1}): 1665 (C=O). Found %: C 52.6; H 9.3; Cl 14.3; N 11.3. $C_{11}H_{22}N_2O_2 \cdot HCl$. Calculated %: C 52.7; H 9.2; Cl 14.1; N 11.2.

(2,2,6,6-Tetramethyl-4-piperidyl)acetic Acid Hydrazide (IIId). A. A mixture of 2.5 g (14 mmole) of I, 12.5 ml of hydrazine hydrate, and 25 ml of xylene was refluxed in a Dean–Stark apparatus for 5 h. The precipitate was filtered, washed with ether, and dried to give 1.9 g (63%) of IIId as colorless crystals with mp 104-105°, which was quite soluble in alcohol and chloroform and slightly soluble in ether and benzene. IR spectrum (ν, cm^{-1}): 1640-1650 (C=O); 3290, 3450 (N–H). Found %: C 61.8; H 11.0; N 19.9. $C_{11}H_{23}N_3O$. Calculated %: C 61.9; H 10.9; N 19.7.

B. A mixture of 2.13 g (10 mmole) of IIIa and 0.75 ml of hydrazine hydrate in 10 ml of alcohol was refluxed for 6 h, evaporated to dryness in vacuo, and the residue was washed with ether to give 1.2 g (56%) of IIId. A mixture of IIId obtained via methods A and B did not give a melting-point depression.

(2,2,6,6-Tetramethyl-4-piperidyl)acetic Acid Phenylhydrazide (IIIe). A. A mixture of 2.4 g (13 mmole) of I, 2.15 g (20 mmole) of phenylhydrazine, and 20 ml of xylene was refluxed for 12 h in a Dean–Stark apparatus. The xylene was removed by vacuum distillation, and the residue was triturated with ether and dried to give 2.31 g (64%) of colorless crystals of IIIe with mp 123° which were quite soluble in alcohol and chloroform and slightly soluble in ether. Found %: C 70.6; H 9.2; N 14.6. $C_{17}H_{27}N_3O$. Calculated %: C 70.8; H 9.0; N 14.6.

B. A mixture of 5 g (21 mmole) of the hydrochloride of II and 50 ml of thionyl chloride was refluxed for 3 h, evaporated to dryness in vacuo, and the residue was dissolved in 50 ml of chloroform. A solution of 5.96 g (55 mmole) of phenylhydrazine in 50 ml of chloroform was added to this chloroform solution, and the mixture was stirred for 5 h at 80°. The cooled reaction mass was made alkaline with 50% aqueous potassium carbonate, and IIIe was extracted with chloroform. The chloroform was removed, and the residue was washed with ether to give 5.8 g (95%) of IIIe. A mixture of IIIe obtained via methods A and B did not give a melting-point depression.

Reaction of 6,6,7,7-Tetramethyl-2-quinuclidone with Phenyllithium. Compound I [1.3 g (7.2 mmole)] was refluxed for 6 h with phenyllithium [from 0.25 g (36 mg-atom) of lithium and 2.82 g (18 mmole) of bromobenzene] in 20 ml of ether. The reaction mass was treated with 10 ml of water and extracted with chloroform. The residue (0.45 g) after removal of the chloroform was triturated with ether and filtered to give 0.15 g (10%) of 4-(2-hydroxy-2-methylpropyl)-6,6-dimethyl-2-piperidone (V) as colorless crystals with mp 139-140° (from ethyl acetate) which were quite soluble in the usual organic solvents. IR spectrum (ν, cm^{-1}): 1650 (C=O); 3210, 3285, 3380 (N–H, O–H). Found %: C 66.3; H 10.6; N 7.2. $C_{11}H_{21}NO_2$. Calculated %: C 66.3; H 10.6; N 7.0. The filtrate was evaporated, and the residue was vacuum distilled to give 0.3 g (23%) of I with bp 100-101° (5 mm).

Reaction of 6,6,7,7-Tetramethyl-2-quinuclidone with Phosphorus Pentachloride. A mixture of 3.35 g (18.5 mmole) of I and 5.0 g (24 mmole) of phosphorus pentachloride was refluxed for 5 h in 50 ml of benzene. The benzene solution was washed with water, dried, and evaporated in vacuo. The residue (1.9 g) was chromatographed with a column filled with Al_2O_3 (50 g) to give 0.95 g of a fraction (eluted with petroleum ether) which was vacuum distilled. The first fraction was 0.51 g (16%) of β,β-bis(2-methyl-1-propenyl)propionitrile

(VIII), which was a colorless liquid with bp 90-92° (3 mm) and n_D^{20} 1.4616; IR spectrum (ν, cm^{-1}): 2255 (C ≡ N), 1657 (C=C). Found %: C 80.2; H 10.6; N 8.6. $C_{11}H_{17}N$. Calculated %: C 80.9; H 10.5; N 8.6. The second fraction was 0.25 g (7.2%) of 2-chloro-4-(2-methyl-1-propenyl)-6,6-dimethyl-Δ^2-dehydropiperidine (VII), which was a colorless liquid with bp 116-118° (3 mm) and n_D^{20} 1.4635. Found %: C 66.3; H 9.0; Cl 18.0; N 6.8. $C_{11}H_{18}ClN$. Calculated %: C 66.1; H 9.1; Cl 17.7; N 7.1. The third fraction was 0.1 g of a product with bp 130-140° (3 mm) which, according to elementary analysis, was a mixture of VII with a dihalo derivative of 6,6-dimethyl-4-(2-methyl-1-propenyl)piperidine. Further elution with a mixture of petroleum ether and chloroform (1:1) washed out first 0.35 g (9.5%) of V and then 0.49 g (15%) of 4-(2-methyl-1-propenyl)-6,6-dimethyl-2-piperidone (IV) as colorless crystals with mp 96-98° (from cyclohexane), which were quite soluble in the usual organic solvents; IR spectrum (ν, cm^{-1}): 1640 (C=O). Found %: C 72.7; H 10.4; N 7.5. $C_{11}H_{19}NO$. Calculated %: C 72.9; H 10.5; N 7.7.

4-(2-Cyano-2-dimethylpropyl)-6,6-dimethyl-2-piperidone (VI). A mixture of 0.6 g (3.3 mmole) of I and 2 g (24 mmole) of acetone cyanohydrin was allowed to stand at room temperature for 96 h, after which it was diluted with 10 ml of ether, and the resulting precipitate of the monohydrate of VI was filtered to give 0.38 g (51%) of a yellowish powder with mp 167-168° (decomp.). IR spectrum (ν, cm^{-1}): 3200-3450 (N–H), 2185 (C ≡ N), 1668 (C=O). Found %: C 63.5; H 9.4; N 12.9. $C_{12}H_{20}N_2O \cdot H_2O$. Calculated %: C 63.7; H 9.8; N 12.8.

2,2,6,6-Tetramethyl-4-(2-hydroxyethyl)piperidine (XI). Compound IIIa [2.13 g (10 mmole)] was reduced by 0.4 g (10 mmole) of lithium aluminum hydride in 40 ml of ether for 3 h at 35° to give 1.73 g (93%) of colorless crystals of XI with mp 63-64° (from alcohol) which were quite soluble in the usual organic solvents and slightly soluble in ether. Found %: C 71.4; H 12.2; N 7.6. $C_{11}H_{23}NO$. Calculated %: C 71.3; H 12.5; N 7.6. The hydrochloride was obtained as colorless crystals with mp 228-230° which were quite soluble in alcohol and water and insoluble in ether. Found %: Cl 16.2; N 6.4. $C_{11}H_{23}NO \cdot HCl$. Calculated %: Cl 16.0; N 6.3.

Reaction of 6,6,7,7-Tetramethyl-2-quinuclidone with Lithium Aluminum Hydride. Compound I [2.8 g (15 mmole)] was reduced with 1.6 g (30 mmole) of lithium aluminum hydride in 70 ml of ether for 5 h at 35°. The usual workup gave 0.06 g of V, which was only slightly soluble in ether, and the remaining compounds were vacuum distilled. The first fraction was 0.9 g (32%) of starting I with bp 100-101° (5 mm). The second fraction was 0.35 g (12%) of XI with bp 116-118° (5 mm), which crystallized on standing to give colorless needles with mp 63-64°. The third fraction was 0.31 g (11%) of 2-[(2,2,6,6-tetramethyl-4-piperidyl)acetoxy]-6,6,7,7-tetramethylquinuclidine (XII) as a viscous, yellow liquid with bp 126-128° (5 mm); IR spectrum (ν, cm^{-1}): 3200-3300 (N–H), 1750 (C=O). Found %: C 72.3; H 11.2; N 7.7. $C_{22}H_{40}N_2O_2$. Calculated %: C 72.4; H 11.1; N 7.7.

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