3-Quinuclidylmethyl 2-Thienyl Ketone. This compound was obtained by reaction of ester IIb with 2-thienylmagnesium bromide. The precipitate that formed on acidification of the reaction mixture was removed by suction filtration and washed with water to give the hydrobromide, with mp 225-226° (from methanol), in 51% yield. Found: C 49.5; H 5.8; Br 25.4; S 10.2%. $C_{13}H_{17}NOS \cdot HBr$. Calculated: C 49.4; H 5.7; Br 25.3; S 10.1%.

3,3-Bis(4-chlorphenyl)methylenequinuclidine. A mixture of 5 g (0.014 mole) of (3-quinuclidyl)bis-(4-chlorophenyl)carbinol and 40 ml of thionyl chloride was refluxed for 20 h, after which it was vacuum evaporated, and the residue was made alkaline with potassium carbonate and extracted with chloroform.

1,1-Diphenyl-2-(3-quinuclidyl)ethylene. A solution of 3.07 g (0.01 mole) of (3-quinuclidylmethyl)diphenylcarbinol in 6 ml of 85% formic acid was refluxed for 30 min, after which the formic acid was removed by vacuum distillation, and the residue was made alkaline with potassium carbonate and extracted with benzene. The remaining IV were similarly obtained (see Table 2).

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REACTION OF DIMETHYLAMINES WITH EPIMERIC (AT THE 2 AND 4 POSITIONS) trans-2-METHYL- AND trans-1,2-DIMETHYL-4-VINYLETHYNYLDECAHYDRO-4-QUINOLOLS

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A mixture of the corresponding 4-(4-dimethylamino-1,2-butadienyl)- and 4-(4-dimethylamino-1-butynyl)decahydro-4-quinolols, with predominance of the allene components, is formed from each vinylacetylenic alcohol as a result of the addition of diethylamine to epimeric (at the 2 and 4 positions) trans-2-methyl- and trans-1,2-dimethyl-4-vinylethynyldecahydro-4-quinolols. On the basis of the PMR spectra and data on the stabilities of allenic and acetylenic diamino alcohols under the conditions of their formation, it was concluded that the addition of dimethylamine to 4-vinylethynyldecahydro-4-quinolols proceeds simultaneously via two pathways – at the 1,4 and 3,4 positions of the vinylethynyl substituent.

We have previously shown that some decahydroquinoline derivatives have high physiological activity [1]. Continuing our search for new physiologically active compounds in this series, in the present research we studied the reaction of epimeric (at the 2 and 4 positions) trans-2-methyl- and trans-1,2-dimethyl-4-vinylethynyldecahydro-4-quinolols [2] with dimethylamines.

It is known that lithium dimethylamide readily adds to conjugated vinylacetylenic hydrocarbons to give acetylenic or allenic amines [3]. The addition of lithium dimethylamide to methylvinylacetylene proceeds in

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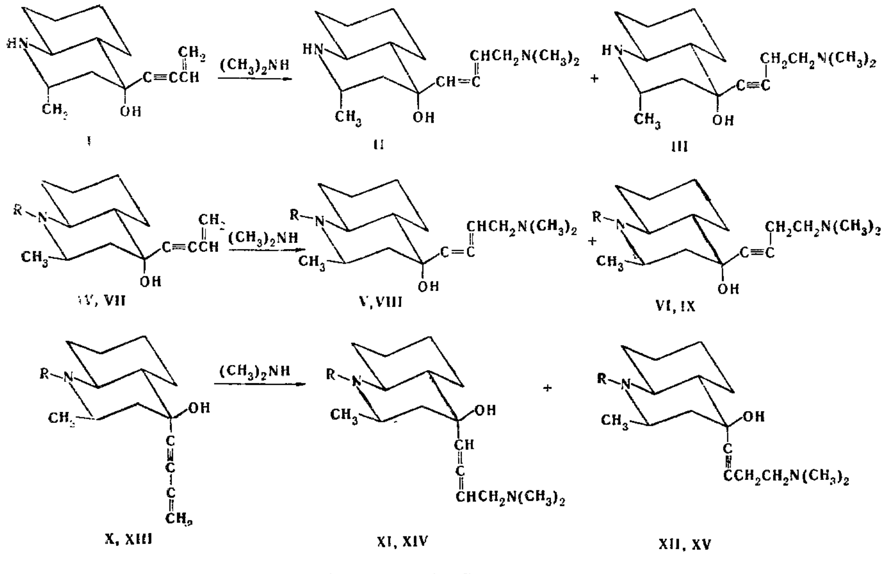
the 1,4 position to give an allenic amine, which, under the influence of excess lithium dimethylamide, is rearranged to an acetylenic amine. In the case of vinylacetylene, acetylenic amines can be obtained either as a result of 1,2 addition or as a result of rearrangement of the allenic adducts to acetylenic compounds at the instant of the reaction. In the case of ethyl- and butylvinylacetylenes only allenic amines are obtained. It is also known [4] that free dimethylamine adds to vinylacetylene only under pressure at elevated temperatures. If the reagents are taken in equimolar amounts, only the allenic amine is obtained; however, if excess dimethylamine is used, a mixture of allenic and acetylenic amines of the $CH_2=C=CHCH_2N(CH_3)_2$ and $CH_3C\equiv CCH_2N(CH_3)_2$ types is formed. Under the influence of excess reagent, the initially formed allenic amines are rearranged through the intermediate formation of a carbanion to acetylenic amines (Favorskii rearrangement). In this case, in contrast to the data in [3], the formation of an amine with a terminal acetylenic bond is not observed.

The addition of dimethylamine to stereoisomeric vinylacetylenic decahydroquinolols also proceeds at elevated temperatures under pressure. In all cases a mixture of allenic and acetylenic diamino alcohols, with predominance of the former, is formed. The composition of the mixture was determined from the integral curve of the PMR spectrum. We were unable to determine the percentage of allenic and acetylenic components in the mixture by means of the PMR spectrum only in the case of vinylacetylenic alcohol XIII; however, according to the thin-layer-chromatography (TLC) data and crystallization of the dimethiodides, the allenic amine also predominates here. The optimum reaction conditions (25% aqueous dimethylamine solution present in a tenfold excess, 120-125°C, 5-10 h) under which the reaction proceeded practically to completion were found. The PMR spectra show that the ratio of the allenic and acetylenic diamino alcohols formed affects the configuration of the vinylethynyl substituents in the 4 position: The ratio of allene to acetylene is 3 : 1 or 2 : 1 in the case of an equatorial vinylethynyl substituent, whereas the isomeric allene and acetylene are formed in approximately equal amounts in the case of an axially oriented vinylethynyl substituent.

An absorption band at 1965 cm^{-1}, which is characteristic for an allenic system of bonds and is absent in the IR spectra of acetylenic diamino alcohols, is observed in the IR spectra of all of the isolated allenic diamino alcohols. An intense line at 2230 cm^{-1}, which corresponds to a disubstituted triple bond, and a less intense line at 2250 cm^{-1} are observed in the Raman spectra of all the acetylenic diamino alcohols.

The mixtures of allenic and acetylenic diamino alcohols formed in the reaction of 2-methyl-4-vinylethynyldecahydro-4-quinolols I and IV (2a4e and 2e4e, respectively) with dimethylamine were separated by repeated crystallization from hexane. We were unable to separate the mixture of diamino alcohols XI and XII in the case of the reaction of vinylacetylenic alcohol X (2e4a) with dimethylamine. Only the corresponding allenic diamino alcohol VIII was isolated by crystallization of the mixture obtained from 1,2-dimethyl-4-vinylethynyldecahydro-4-quinolol VII (2e4e). We were unable to isolate isomeric acetylenic diamino alcohol IX from the mixture, and it was synthesized by methylation of the corresponding nor-analog (VI). Allenic and acetylenic diamino alcohols VIII and IX were also isolated from the mixture in the form of dimethiodides VIIIa and IXa. Dimethiodides XIVa and XVa were similarly isolated from a mixture of the methiodides of allenic and acetylenic diamino alcohols XIV and XV.



IV-VI, X R=H; VII-IX, XIII R=CH_3

In the case of dimethiodide XIVa it was shown by treatment of the dimethiodides with lithium in liquid ammonia that under these conditions, in addition to dequaternization, one observes hydrogenolysis of the nitrogen–carbon bond and reduction on the multiple bonds in the side chain to form 1,2-dimethyl-4-butyldecahydro-4-quinolone, identical to the compound previously obtained by hydrogenation of vinylacetylenic alcohol XIII [2].

Partial hydrogenolysis of the C–N bond in the side chain also occurs in the hydrogenation of allenic and acetylenic diamino alcohols in the presence of a palladium catalyst, and this markedly hinders isolation of the basic products of the reaction of 4-(4-dimethylaminobutyl)decahydro-4-quinolols. The addition of 2 moles of hydrogen in the presence of $Pd/CaCO_3$ to allenic (V) and acetylenic (VI) diamino alcohols leads to the formation of the same saturated diamino alcohol (XVIII); this constitutes one of the proofs for the structures of these compounds. The corresponding dimethylaminobutyl-substituted decahydroquinolols were also obtained by hydrogenation of other allenic and acetylenic diamino alcohols or mixtures of them.

Interconversions of the allenic and acetylenic diamino alcohols do not occur under the reaction conditions, since both diamino alcohols are recovered unchanged after heating allenic alcohol II or acetylenic alcohol III with dimethylamine in a ratio of 1 : 10 at 125° for 8 h. Diamino alcohols II and III were also found to be resistant to heating in a sealed ampul at 50° for 10 h with a 20% alcoholic potassium hydroxide solution. An increase in the temperature to 100° leads to destruction of diamino alcohols II and III. The stability of the allenic diamino alcohols observed in our research compels us to doubt that the acetylenic diamino alcohols in the reaction under investigation are formed as a result of rearrangement of the allenic diamino alcohols at the instant of the reaction under the influence of excess dimethylamine [5]. The assumption that the formation of both isomeric diamino alcohols proceeds simultaneously and independently of one another is more likely.

The spatial orientation of the methyl group attached to C_2 and of the vinylethynyl group attached to C_4 in the starting vinylacetylenic alcohols was previously established by mass spectrometery [6]. The transfusion of the piperidine and cyclohexane rings was established by PMR spectroscopy [7]. It therefore remains here to examine the structure of the side chain in the allenic and acetylenic alcohols. The $-CH=C=CHCH_2N(CH_3)_2$ fragment gives four groups of signals in the PMR spectrum: A singlet at 2.2 ppm $[N(CH_3)_2]$, a doublet of doublets at 2.8 ppm (CH_2), a doublet of triplets at 5.00 ppm $(-CH=)$, and a triplet of doublets at 5.26 ppm $(=CH-)$, i.e., the protons of the $-CH=C=CHCH_2-$ chain form an AMX_2 spin system ($J_{MX} = 6.75$ Hz, $J_{AX} = 2.25$ Hz, and $J_{AM} = 6.23$ Hz). The assignment of the protons to the multiplets of the spectrum and the determination of the constants were confirmed by double homonuclear resonance. There is a complex multiplet at 2.40 ppm in the PMR spectra of III, VI, and IX. The character of the lines of the indicated multiplets is in good agreement with the theoretically calculated values [8] for an AA'BB' proton system, and this makes it possible to assign them to the $RC\equiv CCH_2CH_2N(CH_3)_2$ grouping.

The set of results of a chemical and spectral study of the products of the reaction of dimethylamine with stereoisomeric 2-methyl- and 1,2-dimethyl-4-vinylethynyldecahydro-4-quinolols makes it possible to conclude that the latter proceeds simultanesouly via two pathways: In the 1,4 and 3,4 positions of the vinylethynyl substituents to give allenic diamino alcohols in the first case and acetylenic diamino alcohols in the second.

EXPERIMENTAL

The IR spectra of CCl_4 solutions and KBr pellets of the compounds were recorded with a UR-20 spectrometer. The Raman spectra were recorded with a Spex Ramalog Ramalog-4-spectrometer at $\Delta\nu$ 1900-2400 cm^{-1} during illumination of the capillary containing the substance to be analyzed with the monochromatic emission of an argon laser with a wavelength of 5145 Å. The PMR spectra of $CDCl_3$ solutions of the compounds were recorded with a JEOL-PS-100 spectrometer with tetramethylsilane as the standard. Thin-layer chromatography was carried out on Woelm neutral Al_2O_3 in ethanol–ammonium hydroxide (1 : 0.01). The melting points of the synthesized compounds were measured with a Kofler apparatus.

2a-Methyl-4e-(4-dimethylamino-1,2-butadienyl)decahydro-4-quinolol (II) and 2a-Methyl-4e-(4-dimethylamino-1-butynyl)decahydro-4-quinolol (III). A mixture of 6 g (27 mmole) of vinyl acetylenic alcohol I and 48 g (270 mmole) of 25% aqueous dimethylamine solution was heated in a sealed glass ampul at 125° for 8 h, after which the excess dimethylamine and water were removed by vacuum distillation (with a water aspirator) at 60°, and the residue was dissolved in ether. The ether solution was dried with magnesium sulfate and the ether was evaporated to give 6.9 g (96%) of a viscous oily substance (R_f 0.18; 0.32). The reaction product was dissolved in ether, and the ether solution was allowed to stand in the cold. The resulting mixture was filtered to give 3.5 g of crystals, fractional crystallization of which from hexane yielded 0.64 g (9%) of allenic diamino alcohol II with mp 103-104° and R_f 0.18. IR spectrum: 1965 (C=C=C) and 3620 cm^{-1} (OH). Found: C 72.6; H 10.7; N 10.7%. $C_{16}H_{28}N_2O$. Calculated: C 72.6; H 10.7; N 10.6%.

Acetylenic diamino alcohol III [0.6 g (8%)], with mp 112-113° and R_f 0.32, was isolated from the mother liquor. Raman spectrum: 2230 (intense) and 2250 cm^{-1} (weak) ($C\equiv C$). Found: C 72.5; H 10.6; N 10.5%. $C_{16}H_{28}N_2O$. Calculated: C 72.6; H 10.7; N 10.6%. A mixture of II and III melted at 84-98°. The ratio of addition products II and III in the crude mixture, determined by PMR spectroscopy, was 2 : 1.

2e-Methyl-4e-(4-dimethylamino-1,2-butadienyl)decahydro-4-quinolol (V) and 2e-Methyl-4e-(4-dimethylamino-1-butynyl)decahydro-4-quinolol (VI). A mixture of 6 g (27 mmole) of vinylacetylenic alcohol IV and 48 g (270 mmole) of 25% aqueous dimethylamine was heated at 120° for 5 h, after which it was worked up as described above to give 7 g (97%) of a mixture of amines (R_f 0.20 and 0.34). An ether solution of the mixture was allowed to stand, after which it was worked up to yield 4.2 g of crystals, fractional crystallization of which from hexane gave 0.58 g (8%) of allenic diamino alcohol V with mp 104-105° and R_f 0.20. IR spectrum: 1965 ($C=C=C$) and 3620 cm^{-1} (OH). Found: C 72.7; H 10.7; N 10.7%. $C_{16}H_{28}N_2O$. Calculated: C 72.6; H 10.7; N 10.6%.

Workup of the mother liquor yielded 0.52 g (7%) of acetylenic diamino alcohol VI with mp 120-121° and R_f 0.34. Raman spectrum: 2230 (intense) and 2250 cm^{-1} (weak) ($C\equiv C$). Found: C 72.5; H 10.6; N 10.7%. $C_{16}H_{28}N_2O$. Calculated: C 72.6; H 10.7; N 10.6%. A mixture of V and VI melted at 76-94°. The ratio of allene V and acetylene VI in the crude mixture (by PMR spectroscopy) was 3 : 1.

2e-Methyl-4a-(4-dimethylamino-1,2-butadienyl)decahydro-4-quinolol (XI) and 2e-Methyl-4a-(4-dimethylamino-1-butynyl)decahydro-4-quinolol (XII). A mixture of 3 g (13.5 mmole) of vinylacetylenic alcohol X and 24 g (135 mmole) of 25% aqueous dimethylamine was heated at 120° for 5 h, after which it was worked up to give 3.45 g (98%) of a viscous liquid mixture of amino alcohols XI (R_f 0.23) and XII (R_f 0.39). This mixture began to crystallize, and we were unable to isolate individual diamino alcohols XI and XII. The ratio of XI and XII in the mixture, determined by PMR spectroscopy, was 54 : 46.

1,2e-Dimethyl-4e-(4-dimethylamino-1,2-butadienyl)decahydro-4-quinolol (VIII) and 1,2e-Dimethyl-4e-(4-dimethylamino-1-butynyl)decahydro-4-quinolol (IX). A total of 3.4 g (96%) of a mixture of amino alcohols (R_f 0.27 and 0.40) was obtained from 3 g (10.8 mmole) of vinylacetylenic alcohol VII and 24 g (135 mmole) of 25% aqueous dimethylamine after heating at 120° for 6 h. Successive crystallization of the product from ether and hexane yielded 0.9 g (25%) of allenic diamino alcohol VIII with mp 98-99° and R_f 0.27. IR spectrum: 1965 and 3622 cm^{-1}. Found: C 73.1; H 10.9; N 10.2%. $C_{17}H_{30}N_2O$. Calculated: C 73.3; H 10.8; N 10.1%. Dimethiodide VIIIa and mp 208-210°. IR spectrum: 1965 cm^{-1}. Found: C 40.5; H 6.4; I 45.0; N 4.9%. $C_{19}H_{36}I_2N_2O$. Calculated: C 40.6; H 6.5; I 45.1; N 5.0%. The ratio of VIII and IX in the crude mixture, determined by PMR spectroscopy, was 62 : 38, respectively.

A 2-ml (30 mmole) sample of methyl iodide was added to a solution of 2 g (7 mmole) of the crude mixture of VIII and IX in ether. Crystallization of the product from ethanol yielded 1 g (25%) of the dimethiodide, with mp 208-210° (no melting-point depression was observed for a mixture of this product with dimethiodide VIIIa), and 0.35 g (9%) of dimethiodide IXa with mp 241-243°. Raman spectrum: 2240 (intense) and 2260 cm^{-1} (weak) ($C\equiv C$). Found: C 40.6; H 6.5; I 45.3; N 4.8%. $C_{19}H_{36}I_2N_2O$. Calculated: C 40.6; H 6.5; I 45.1; N 5.0%.

A 0.25-g (78%) sample of acetylenic diamino alcohol IX, with mp 78-79° and R_f 0.40, was obtained from 0.3 g (1 mmole) of VI, 0.6 g (8 mmole) of 40% formalin, and 0.5 g (9 mmole) of 85% formic acid. Raman spectrum: 2230 (intense) and 2250 cm^{-1} (weak) ($C\equiv C$). No melting-point depression was observed for a mixture of the dimethiodide (mp 241-243°) with dimethiodide IXa.

1,2e-Dimethyl-4a-(4-dimethylamino-1,2-butadienyl)decahydro-4-quinolol (XIV) and 1,2e-Dimethyl-4a-(4-dimethylamino-1-butynyl)decahydro-4-quinolol (XV). Reaction of 3 g (10.8 mmole) of vinylacetylenic alcohol XIII with 24 g (135 mmole) of 25% aqueous dimethylamine at 120° for 6 h yielded 3.4 g (96%) of a mixture of two substances (R_f 0.30 and 0.44), 2 g of which was converted to the dimethiodide. Repeated crystallization from absolute ethanol yielded 0.9 g (22%) of the dimethiodide (XIVa) of the allenic diamine with mp 219-221°. IR spectrum: 1965 ($C=C=C$) and 3620 cm^{-1} (OH). Found: C 40.5; H 6.7; I 45.2; N 4.8%. $C_{19}H_{36}I_2N_2O$. Calculated: C 40.6; H 6.5; I 45.1; N 5.0%. Subsequent crystallization of the mother liquor yielded 0.4 g (10%) of dimethiodide XVa with mp 233-235°. Raman spectrum: 2240 (intense) and 2260 cm^{-1} (weak) ($C\equiv C$). Found: C 40.8; H 6.6; I 45.0; N 5.1%. $C_{19}H_{36}I_2N_2O$. Calculated: C 40.6; H 6.5; I 45.1; N 5.0%.

Dequaternization of Dimethiodide XIVa. A 0.3-g (42 mg-atom) sample of lithium and 5 ml of water were added successively at −50° with vigorous stirring to a solution of 1 g (1.8 mmole) of dimethiodide XIVa in 200 ml of liquid ammonia, after which the excess ammonia was evaporated, 15 ml of water was added to the residue, and the solution was saturated with K_2CO_3. The liberated base was extracted with ether, the extract was dried with magnesium sulfate, and the ether was removed by distillation to give 0.36 g of alcohol XVI with mp

117-118° (from hexane). No melting-point depression was observed for a mixture of this product with a genuine sample of 1,2-dimethyl-4-butyldecahydro-4-quinolol obtained by hydrogenation of vinyl acetylenic alcohol XIII [2].

2-Methyl-4-(4-dimethylaminobutyl)decahydro-4-quinolols (XVII-XIX). A) Exhaustive hydrogenation of 1 g of a mixture of II and III in the presence of $Pd/CaCO_3$ in ethanol and subsequent repeated crystallization of the reaction products from hexane gave 0.48 g of amino alcohol XVII with mp 99-100° and R_f 0.09. Found: C 71.8; H 12.1; N 10.6%. $C_{16}H_{32}N_2O$. Calculated: C 71.6; H 12.0; N 10.4%.

B) Treatment of 0.2 g of V under similar conditions yielded 0.13 g of amino alcohol XVIII with mp 86-87° (from hexane) and R_f 0.12. Found: C 71.4; H 12.1; N 10.5%. $C_{16}H_{32}N_2O$. Calculated: C 71.6; H 12.0; N 10.4%.

Hydrogenation of 0.2 g of VI yielded 0.11 g of a substance with mp 86-87° and R_f 0.12. No melting-point depression was observed for a mixture of this product with XVIII.

C) Exhaustive hydrogenation of 1 g of a mixture of XI and XII in ethanol over $Pd/CaCO_3$ gave 0.56 g of amino alcohol XIX with mp 123-124° and R_f 0.13. Found: C 71.7; H 12.2; N 10.3%. $C_{16}H_{32}N_2O$. Calculated: C 71.6; H 12.0; N 10.4%.

1,2-Dimethyl-4-(4-dimethylaminobutyl)decahydro-4-quinolols (XX, XXI). A) Hydrogenation of 1 g of a mixture of amino alcohols XIV and XV in the presence of $Pd/CaCO_3$ in ethanol yielded 0.9 g of a mixture (R_f 0.14 and 0.53), chromatography of which with a column filled with a column filled with activity II Al_2O_3 in an ether—petroleum ether system (1 : 1) yielded 0.5 g of a first fraction (R_f 0.53) and 0.4 g of a second fraction (R_f 0.14). Crystallization of the first fraction from hexane yielded 0.15 g of amino alcohol XVI, which was identical to a genuine sample. Workup of the second fraction yielded 0.3 g of dimethiodide XXa with mp 228-230°. Found: C 40.4; H 7.2; I 44.7; N 4.8%. $C_{19}H_{40}I_2N_2O$. Calculated: C 40.3; H 7.1; I 44.8; N 4.9%.

B) Methylation of 0.5 g (1.8 mmole) of XIX with a mixture of 0.9 g (12 mmole) of 40% formalin and 0.8 g (14 mmole) of 85% formic acid yielded 0.45 g (86%) of alcohol XX with mp 68-69° and R_f 0.14. Found: C 72.1; H 12.2; N 10.0%. $C_{17}H_{34}N_2O$. Calculated: C 72.3; H 12.2; N 9.9%. The dimethiodide of alcohol XX had mp 228-230° and did not depress the melting point of dimethiodide XXa.

C) Hydrogenation of 1 g of a mixture of VIII and IX under similar conditions yielded a reaction product (R_f 0.13 and 0.51), chromatography of which with a column filled with activity II Al_2O_3 and elution with petroleum ether and subsequent crystallization from hexane yielded 0.25 g of amino alcohol XXI with mp 83-84° and R_f 0.13. Found: C 72.1; H 12.0; N 10.1%. $C_{17}H_{34}N_2O$. Calculated: C 72.3; H 12.1; N 9.9%. Dimethiodide XXIa had mp 258-260°. Found: C 40.1; H 7.3; I 44.5; N 5.0%. $C_{19}H_{40}I_2N_2O$. Calculated: C 40.3; H 7.1; I 44.8; N 4.9%.

B) Methylation of 0.2 g (0.7 mmole) of amino alcohol XVIII with a mixture of 0.35 g (4.6 mmole) of 40% formalin and 0.3 g (5 mmole) of 85% formic acid yielded 0.18 g of a substance with mp 83-84° and R_f 0.13. No melting-point depression was observed for a mixture of this product with XXI.

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