

HYDROBORATION OF 1-ALLYL-1,2,3,4-TETRAHYDROQUINOLINE*

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Hydroboration of 1-allyl-1,2,3,4-tetrahydroquinoline (*I*) with triethylamine-borane in the molar ratio 1 : 1 afforded a product from which 6,7-benzo-5-aza-1-borasp[4,5]decane (*II*) was isolated. Ethanolysis of *II* gave diethyl 3-(1,2,3,4-tetrahydro-1-quinolyl)propylboronate (*III*). Acid hydrolysis of the crude hydroboration product and subsequent oxidation with alkaline hydrogen peroxide led to a mixture of 3-(1,2,3,4-tetrahydro-1-quinolyl)-1-propanol (*IV*), 1,2,3,4-tetrahydroquinoline (*V*) and 1-propyl-1,2,3,4-tetrahydroquinoline (*VI*). Hydroboration of *I* with triethylamine-borane in the molar ratio 3 : 1, followed by oxidation, gave a mixture of *IV*, *V*, *VI* and 1-(1,2,3,4-tetrahydro-1-quinolyl)-2-propanol (*VIII*). Hydroboration of *I* with diborane *in situ* in diethylene glycol dimethyl ether at 20°C and subsequent acid hydrolysis and oxidation with alkaline hydrogen peroxide afforded a mixture of *IV*, *V* and *VIII*; when the hydroboration product was first heated to 150°C and then subjected to the above-mentioned processing, only compounds *IV* and *V* were formed.

In our previous communications we reported on preparation of spirocyclic compounds, containing a quaternary nitrogen atom bonded coordinatively to an sp^3 -hybridized boron atom, by hydroboration of N-allyl derivatives of monocyclic unsaturated amines¹ or 2-allyl-1,2,3,4-tetrahydroisoquinoline² with triethylamine-borane.

In the present study, we treated 1-allyl-1,2,3,4-tetrahydroquinoline (*I*) with triethylamine-borane under the same reaction conditions as used previously (*i.e.* 1 : 1 molar ratio of the hydroborating agent to the substrate). However, the results of these experiments were at variance with those obtained previously with the above-mentioned systems^{1,2}. The reaction gave only a small amount of 6,7-benzo-5-aza-1-borasp[4,5]decane (*II*) which is analogous to the previously prepared spirocyclic amine-boranes^{1,2}. A major part of the product, however, was an undistillable material and the triethylamine-borane was partly recovered from the reaction mixture.

Unlike the previously obtained spirocyclic amine-boranes^{1,2}, the compound *II* was unstable in air and reacted vigorously with ethanol. This high reactivity can be

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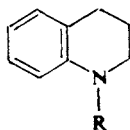
explained assuming that the nitrogen atom bonded to the benzene nucleus has a lower propensity to donate its electron pair to the dative bond than in aliphatic amines and that the boryl group is thus less stabilized by this bond. The existence of the B–N bond in the compound *II* was proved by the ^1H NMR spectrum in which (due to the positive charge at the nitrogen atom) the signals of benzene protons *ortho* and *para* to the nitrogen atom (11 and 13) are shifted downfield as compared with the spectra of 1,2,3,4-tetrahydroquinoline derivatives without a positive charge at the nitrogen atom.

Mass spectrum of the compound *II* contains no M^+ ion but displays an $(\text{M}-1)^+$ ion, typical for amine-boranes $^{1-4}$. As expected, the 1-methylene-1,2,3,4-tetrahydroquinolinium ion at m/z 146 is the most abundant.

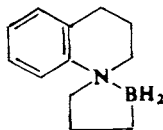
On the basis of our previous results 1,2,4 , we identified in the IR spectrum of the amine-borane *II* the B–H deformation vibration bands at 1 120 and 1 195 cm^{-1} and B–H stretching vibration bands in the region 2 220–2 380 cm^{-1} . We assigned also bands due to C–H deformation vibrations (at 1 490 cm^{-1}), C–C aromatic stretching vibrations (1 450, 1 502, 1 584 and 1 603 cm^{-1}) and C–H stretching vibrations (2 850–3 070 cm^{-1}).

Reaction of the compound *II* with ethanol afforded diethyl 3-(1,2,3,4-tetrahydro-1-quinolyl)propylboronate (*III*). Its ^1H NMR spectrum revealed no B–N bond.

The crude product of the 1 : 1 hydroboration of *I* with triethylamine-borane from which only the formed triethylamine and the unreacted hydroboration reagent had been distilled off, on boiling with hydrochloric acid in acetone and subsequent oxidation with alkaline hydrogen peroxide afforded a mixture of 3-(1,2,3,4-tetrahydro-1-quinolyl)-1-propanol (*IV*) and 1,2,3,4-tetrahydroquinoline (*V*) with small amount of 1-propyl-1,2,3,4-tetrahydroquinoline (*VI*) (Table I). The alcohol *IV* was obtained in 50% yield by distillation.

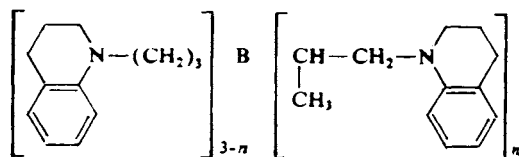


- I*, R = $\text{CH}_2\text{CH}=\text{CH}_2$
III, R = $(\text{CH}_2)_3\text{B}(\text{OC}_2\text{H}_5)_2$
IV, R = $(\text{CH}_2)_3\text{OH}$
V, R = H
VI, R = $\text{CH}_2\text{CH}_2\text{CH}_3$
VIII, R = $\text{CH}_2\text{CH}(\text{OH})\text{CH}_3$

*II*

The fact that a part of the employed triethylamine-borane was recovered unreacted whereas all the substrate *I* reacted indicates that one BH_3 species liberated from triethylamine-borane can react with more than one molecule of *I*. To confirm this assumption we performed the reaction of *I* with triethylamine-borane in the molar

ratio 3 : 1. As expected, neither of the starting compounds was detected in the reaction mixture and the product (unstable on exposure to air) was shown by ^1H NMR spectrum to be tris[3-(1,2,3,4-tetrahydro-1-quinolyl)-1-propyl]borane (*VIIa*) contaminated with compounds bearing 1-(1,2,3,4-tetrahydro-1-quinolyl)-2-propyl groups (*VIIb*–*VIIId*). Oxidation of this product with alkaline hydrogen peroxide afforded a mixture which contained, in addition to compounds *IV* and *V*, compound *VI* and 1-(1,2,3,4-tetrahydro-1-quinolyl)-2-propanol (*VIII*) (Table I).



VII

In formula *VII*: *a*, *n* = 0; *b*, *n* = 1; *c*, *n* = 2; *d*, *n* = 3.

It was also of interest to compare products of the above-described reaction with triethylamine-borane with those obtained by hydroboration with diborane at 20°C, followed by acid hydrolysis and oxidation with alkaline hydrogen peroxide, and to study the behaviour of the low-temperature hydroboration product on heating. Diborane was prepared *in situ* from boron trifluoride etherate and sodium borohydride in diethylene glycol dimethyl ether. A sample, taken from the product of hydroboration at 20°C, was hydrolyzed and oxidized in the usual manner. All the remaining product was heated to 150°C and samples, withdrawn at appropriate intervals, were processed in the same way and analyzed by gas-liquid chromatography. The sample

TABLE I
Products (% wt) of hydroboration of *I* followed by oxidation

Product	Method of hydroboration ^a			
	A	B	C	D
<i>V</i>	16.1	8.4	30	44.5
<i>VI</i>	1.6	4.8	0	0
<i>VIII</i>	0	2.9	13	0
<i>IV</i>	82.3	83.9	57	55.5

^a A, 1 mol of $\text{Et}_3\text{N}.\text{BH}_3$ /1 mol of *I*; B, 1 mol of $\text{Et}_3\text{N}.\text{BH}_3$ /3 mol of *I*; C, B_2H_6 *in situ* at 20°C; D, B_2H_6 *in situ* at 20°C and heating of the reaction mixture to 150°C.

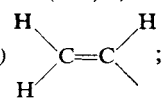
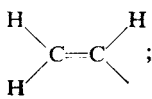
taken at 20°C contained compounds *IV*, *V* and *VIII* whereas in the samples taken at 150°C only compounds *IV* and *V* were found. All the samples taken at 150°C were of the same composition, and the content of the alcohol *IV* was practically identical with that found at 20°C (Table I). Apparently, the compound formed by hydroboration at 20°C, which on hydrolysis and oxidation affords the alcohol *VIII*, is on heating converted into a derivative which on the same processing gives the compound *V*.

In this paper we describe also new preparation of compound *I* and an independent synthesis of the hitherto undescribed alcohol *VIII*. Compound *I* was synthesized by reduction of 1-allylquinolinium bromide with triethylammonium formate in formic acid, the alcohol *VIII* was obtained by reduction of 1-(1,2,3,4-tetrahydro-1-quinoly)-2-propanone with lithium aluminium hydride.

EXPERIMENTAL

The temperature data are uncorrected. Gas-liquid chromatography (GLC) was carried out on a Chrom 2 (1 700 × 6 mm columns) or Chrom 5 (2 400 × 3 mm columns) chromatographs; carrier gas nitrogen, flame-ionization detector; stationary phases: Chrom 2: 15% silicone elastomer E 301 on Chromosorb N-AW, 15% poly(1,4-butanediol succinate) (BDS) on Chromaton N-AW or 10% Apiezon L on Chromosorb W; Chrom 5: 10% silicone elastomer E 301 on Chromaton N-AW-DMCS. The peak areas were measured by an IT-2 integrator. Molar responses were not determined and were assumed to be the same for all compounds. Thin-layer chromatography was performed on Silufol UV 254 sheets (silica gel according to Pitra, containing a luminiscent indicator, on aluminium foil, binder starch) or on Alufol sheets (neutral alumina on aluminium foil, binder starch). Infrared spectra were taken on a Perkin-Elmer 325 spectrophotometer. ¹H NMR Spectra were determined on Varian XL-100-15 (100.1 MHz) or on Varian XL-200 (200 MHz; stated with the spectra) instruments in deuteriochloroform at 35°C unless stated otherwise; internal standard tetramethylsilane. Mass spectra were measured on a Gas Chromatograph-Mass Spectrometer LKB 9000 (Produkter AB, Stockholm) instrument. The ionic species are given in *m/z* units with relative intensity per cents in parentheses.

1-Allyl-1,2,3,4-tetrahydroquinoline (*I*)

Triethylamine (79.0 g; 0.78 mol) was added to a solution of 1-allylquinolinium bromide⁵ (98.0 g; 0.39 mol) in 85% formic acid (145 ml; 3.1 m/l). After refluxing for 11 h, the mixture was made alkaline with 40% sodium hydroxide (phenolphthalein), the organic layer was separated and the aqueous one was extracted four times with ether (80 ml). The extracts were combined with the original organic layer, dried over potassium carbonate, and taken down affording 34.0 g (50%) of *I*, b.p. 140°C/2.4 kPa (reported⁶ b.p. 135°C/3.3 kPa). ¹H NMR Spectrum, ppm: 1.94 (2 H, m, $J_{2,3} = J_{3,4} = 6$ Hz), H₍₃₎; 2.75 (2 H, t, $J = 6$ Hz) H₍₄₎; 3.25 (2 H, t, $J = 6$ Hz) H₍₂₎; 3.83 (2 H, d, $J = 5$ Hz) CH₂-C=; 5.10 (1 H, d, $J_{cis} = 11$ Hz) ; 5.14 (1 H, d, $J_{trans} = 17$ Hz) ; 5.81 (1 H, m, $J_{\alpha,\beta} = 5$ Hz, $J_{cis} = 11$ Hz, $J_{trans} = 17$ Hz) —CH—; 6.43–6.65 (2 H, m) H₍₆₎ and H₍₈₎; 6.84–7.15 (2 H, m) H₍₅₎ and H₍₇₎.

1-(1,2,3,4-Tetrahydro-1-quinolyl)-2-propanol (*VIII*)

A solution of 1-(1,2,3,4-tetrahydro-1-quinolyl)-2-propanone⁷ (2.95 g; 0.016 mol) was added dropwise to a stirred solution of lithium aluminium hydride (0.3 g; 0.008 mol) in ether (40 ml). After reflux for 1 h under stirring, the mixture was decomposed with 4% sodium hydroxide (1.2 ml) and the solid was removed by filtration. The filtrate was dried over potassium carbonate and the ether was evaporated, leaving 2.6 g (87%) of the alcohol *VIII*, b.p. 165–166°C/2.5 kPa. For C₁₂H₁₇NO (191.3) calculated: 75.35% C, 8.96% H, 7.32% N; found: 75.26% C, 8.87% H, 7.10% N. ¹H NMR Spectrum (31°C), ppm: 1.19 (3 H, d, *J* = 6 Hz) CH₃; 1.93 (2 H, m, *J*_{2,3} = *J*_{3,4} = 6 Hz) H₍₃₎; 2.25 (1 H, s) OH; 2.75 (2 H, t, *J* = 6 Hz) H₍₄₎; 2.92–3.50 (4 H, m) CH₂—N—CH₂; 4.06 (1 H, m, *J*_{α,β} = *J*_{β,γ} = 6 Hz) CH—O; 6.46–6.72 (2 H, m) H₍₆₎ and H₍₈₎; 6.78–7.12 (2 H, m) H₍₅₎ and H₍₇₎. IR Spectrum (CCl₄), cm⁻¹: 3 600 (OH).

6,7-Benzo-5-aza-1-borospiro[4,5]decane (*II*)

A mixture of *I* (12.0 g; 0.069 mol) and triethylamine-borane⁸ (8.0 g; 0.069 mol) was heated under reflux condenser in an atmosphere of nitrogen. At 132°C (bath) the reaction commenced (boiling), the bath was removed, the mixture cooled and triethylamine distilled off (3.8 g; 54%; identified by GLC on Chrom 2, E 301, 70°C or BDS, 85°C). Distillation of the product gave 2.0 g (25%) of the starting triethylamine-borane, b.p. 90–94°C/1.7 kPa, identified by GLC (Chrom 2, E 301, 100°C) and thin-layer chromatography (Alufol, benzene, *R_F* 0.7), and 5.4 g of a liquid b.p. 50–132°C/7 Pa which on repeated distillation afforded 1.7 g (13%) of *II*, b.p. 118–119°C/3 Pa. For C₁₂H₁₈BN (187.1) calculated: 77.03% C, 9.70% H, 5.78% B, 7.49% N; found: 77.03% C, 9.93% H, 5.85% B, 7.45% N. Mass spectrum: 186 (11), 156 (34), 146 (100), 145 (56), 144 (36), 133 (86), 132 (100), 130 (36), 118 (36), 117 (34). ¹H NMR Spectrum, ppm: 0.96 (2 H, m, *J*_{1,2} = *J*_{2,3} = 6.5 Hz) H₍₂₎; 1.68–2.48 (4 H, m) H₍₃₎ and H₍₉₎; 2.84 (2 H, t, *J*_{8,9} = 6 Hz) H₍₈₎; 2.95–3.60 (4 H, m) H₍₄₎ and H₍₁₀₎; 6.82–7.27 (3 H, m) H₍₁₂₎, H₍₁₃₎ and H₍₁₄₎; 7.38–7.64 (1 H, m) H₍₁₁₎. IR Spectrum (CCl₄), cm⁻¹: γ(CH) 725 (m); δ(BH₂) 1 120 (s), 1 195 (i); δ(CH₂) 1 433 (m), 1 490 (s); ν(CC) 1 450 (i), 1 502 (m), 1 584 (w), 1 603 (m); ν(BH₂) 2 220 (w), 2 320 (s), 2 380 (s); ν(CH₂) 2 850 (m), 2 870 (m), 2 900 (i), 2 940 (s); ν(CH) 3 030 (w), 3 070 (w).

Diethyl 3-(1,2,3,4-tetrahydro-1-quinolyl)propylboronate (*III*)

Compound *II* (0.5 g; 0.0027 mol) was added to ethanol (2.0 ml). An instantaneous reaction took place, accompanied with evolution of hydrogen and heat. Removal of excess ethanol *in vacuo* gave 0.6 g (82%) of compound *III*, b.p. 119°C/2.0 Pa. For C₁₆H₂₆BNO₂ (275.2) calculated: 69.83% C, 9.52% H, 3.93% B, 5.09% N; found: 69.86% C, 9.65% H, 3.97% B, 5.34% N. ¹H NMR Spectrum, ppm: 0.74 (2 H, t, *J*_{β,γ} = 7 Hz) CH₂—B; 1.18 (6 H, t, *J* = 7 Hz) 2 CH₃; 1.66 (2 H, m, *J*_{α,β} = *J*_{β,γ} = 7 Hz) CH₂—C—B; 1.92 (2 H, m, *J*_{2,3} = *J*_{3,4} = 6 Hz) H₍₃₎; 2.71 (2 H, t, *J*_{3,4} = 6 Hz) H₍₄₎; 3.19 (2 H, t, *J*_{α,β} = 7 Hz) CH₂—C—C—B; 3.25 (2 H, t, *J*_{2,3} = 6 Hz) H₍₂₎; 3.82 (4 H, q, *J* = 7 Hz) 2 OCH₂; 6.36–6.72 (2 H, m) H₍₆₎ and H₍₈₎; 6.78–7.10 (2 H, m) H₍₅₎ and H₍₇₎.

Hydroboration of *I* with Triethylamine-borane in Molar Ratio 1 : 1

a) Compound *I* (17.3 g; 0.10 mol) was treated with triethylamine-borane⁸ (11.5 g; 0.10 mol) in the same manner as described for the preparation of the amine-borane *II*. Triethylamine (5.3 g; 52%) and the unreacted triethylamine-borane (3.0 g; 26%) were distilled off and the resi-

due was dissolved in acetone (100 ml). Dilute (15%) hydrochloric acid (60 ml) was added dropwise to the stirred solution and the mixture was refluxed for 15 min with stirring. The acid and acetone were distilled off *in vacuo* and the residue was suspended in tetrahydrofuran (170 ml). After addition of 40% sodium hydroxide (100 ml), 30% hydrogen peroxide (85 ml) was added dropwise with stirring (strong spontaneous warming). The stirred mixture was refluxed for 2 h, cooled, the organic layer separated and the aqueous layer extracted with ether (5 × 50 ml). The extracts were combined with the original organic layer, dried over potassium carbonate and taken down. Repeated distillation of the residue gave 9.5 g (50%) of a yellowish liquid, b.p. 101°C/0.7 Pa, R_F 0.2 (Silufol, cyclohexane-ethyl acetate 4 : 1), identified by its elemental analysis and ^1H NMR spectrum⁹ as 3-(1,2,3,4-tetrahydro-1-quinoly)-1-propanol⁹ (*IV*) (reported⁹ b.p. 115°C/1 Pa).

b) Compound *I* (12.1 g; 0.07 mol) was hydroborated with triethylamine-borane⁸ (8.1 g; 0.07 mol) in the same manner as described for the preparation of the amine-borane *II*. Triethylamine (1.8 g; 25%) and the unreacted triethylamine-borane (3.8 g; 47%) were distilled off, the residue was dissolved in acetone (70 ml) and 15% hydrochloric acid (42 ml) was added dropwise under stirring. The stirred mixture was refluxed for 15 min and the acid and acetone were removed by distillation *in vacuo*. The residue was dissolved in water (30 ml) and the solution mixed with tetrahydrofuran (70 ml) and 40% NaOH (20 ml). To the stirred mixture 30% hydrogen peroxide (16 ml) was added (spontaneous warming). After reflux with stirring for 4 h, the organic layer was separated and the aqueous one extracted with tetrahydrofuran (4 × 30 ml). The extracts were combined with the original organic layer, dried over potassium carbonate and taken down leaving 9.9 g (79%) of a liquid boiling at 60–122°C/3 Pa which, according to GLC (Chrom 5, E 301, 150–240°C, 5°C/min) and ^1H NMR spectrum, proved to be a mixture of *V*, *VI* and *IV* in the ratio 16:1 : 1.6 : 82:3.

Hydroboration of *I* with Triethylamine-borane in Molar Ratio 3 : 1

A mixture of compound *I* (13.0 g; 0.075 mol) and triethylamine-borane⁸ (2.9 g; 0.025 mol) was heated under nitrogen. At bath temperature 128°C a vigorous reaction occurred, the bath was removed and the mixture cooled. Triethylamine (1.6 g; 63%) was removed by distillation (maximum bath temperature 150°C). Heating at 2.1 kPa gave no distillate at bath temperatures up to 150°C and the product (13.5 g) represented a yellowish very viscous liquid which turned brown on exposure to air during several minutes.

A part of this product was dissolved in hexadeuterioacetone immediately after preparation. ^1H NMR Spectrum of this solution corresponded to the expected tris[3-(1,2,3,4-tetrahydro-1-quinoly)-1-propyl]borane (*VIIa*) containing compounds *VIIb*–*VIIc*. After storing for several weeks at –20°C, the hexadeuterioacetone solution deposited a white crystalline compound which immediately after exposure to air turned into a wine-red amorphous mass. Its ^1H NMR spectrum (200 MHz) (freshly made solution in hexadeuterioacetone) corresponded to compound *VIIa*, purer than the fresh hydroboration product. ^1H NMR Spectrum (hexadeuterioacetone, 200

MHz), ppm: 0.62–0.98 (6 H, m) $\text{CH}_2\text{—B}$ $\begin{array}{l} \text{CH}_2 \\ \text{CH}_2 \end{array}$; 1.45–1.96 (12 H, m) $\text{CH}_2\text{—C—B}$ $\begin{array}{l} \text{C—CH}_2 \\ \text{C—CH}_2 \end{array}$
and $\text{H}_{(3)}$ of the tetrahydroquinoline rings; 2.66 (6 H, t, $J = 6$ Hz) $\text{H}_{(4)}$ of the tetrahydroquinoline rings; 3.00–3.43 (12 H, m) 3 $\text{CH}_2\text{—N—CH}_2$; 6.35–6.66 (6 H, m) $\text{H}_{(6)}$ and $\text{H}_{(8)}$ of the tetrahydroquinoline rings; 6.76–7.04 (6 H, m) $\text{H}_{(5)}$ and $\text{H}_{(7)}$ of the tetrahydroquinoline rings.

The hydroboration product (10.7 g) in tetrahydrofuran (60 ml) was mixed with 3M-NaOH (6.0 ml) and 30% hydrogen peroxide was added dropwise stirring at 19–22°C (cooling). After

each addition of several drops the mixture was tested for peroxides (potassium iodide — starch in hydrochloric acid). After the positive reaction had occurred, the addition was stopped (4.2 ml of 30% H_2O_2). The mixture was saturated with potassium carbonate, the organic layer separated and the aqueous one extracted with tetrahydrofuran (2×10 ml). After drying over potassium carbonate and removal of the solvent the material was distilled, giving three fractions: a) b.p. $110-140^\circ C/2.1$ kPa (0.8 g); b) b.p. $140-175^\circ C/2.1$ kPa (1.6 g); c) b.p. $175-184^\circ C/2.1$ kPa (7.5 g). According to GLC (Chrom 5, E 301, $150-240^\circ C$, $5^\circ C/min$) and 1H NMR spectra, the fractions contained compounds *V*, *VI*, *VIII* and *IV* in the following ratios: a) 47.8 : 27.8 : 2.2 : 22.2, b) 20.2 : 16.1 : 4.8 : 58.9, and c) 1.7 : 0 : 2.6 : 95.7, which corresponded to total 9.9 g (91%) of the mixture of the mentioned compounds in the ratio 8.4 : 4.8 : 2.9 : 83.9.

Hydroboration of *I* with Diborane *in situ* in Diethylene Glycol Dimethyl Ether

A solution of boron trifluoride etherate (1.8 ml; 0.014 mol) in diethylene glycol dimethyl ether (3.5 ml) was added under nitrogen during 15 min at $20^\circ C$ to a stirred mixture of sodium borohydride (0.4 g; 0.01 mol), compound *I* (1.7 g; 0.01 mol) and diethylene glycol dimethyl ether (15 ml). After stirring at $20^\circ C$ for 1 h a sample (2.0 ml) was withdrawn with a syringe through a rubber septum (for processing of the sample *vide infra*). The bath temperature was then increased to $150^\circ C$, another sample was withdrawn and the mixture was stirred at this temperature. Other three samples were taken at 75 minutes' intervals. Each sample was decomposed with 15% hydrochloric acid (0.8 ml; evolution of hydrogen), diluted with tetrahydrofuran (2 ml), made alkaline with 40% sodium hydroxide (1.0 ml) and boiled under stirring with 30% hydrogen peroxide (3.5 ml) for 1 h. After cooling, the organic layer was separated and the aqueous one extracted with tetrahydrofuran (3×2 ml). The extracts were combined with the original organic layer and analyzed by GLC (Chrom 5, E 301, $150-240^\circ C$, $5^\circ C/min$). The first sample contained compounds *V*, *VIII* and *IV* in the ratio 30 : 13 : 57, all other samples consisted of *V* and *IV* in the ratio 44.5 : 55.5.

The elemental analyses were performed in the Analytical Laboratory of this Department (Dr L. Helešic, Head). The NMR spectra taken on Varian XL-100-15 were obtained in the Laboratory of NMR Spectroscopy of this Institute (Dr P. Trška, Head), the spectra determined on Varian XL-200 were measured by Dr D. Šaman, Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, Prague. Mass spectra were taken by Dr J. Mitera, IR spectra by Dr E. Janečková and Dr A. Kohoutová of the Central Laboratories of this Institute.

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