

Synthesis of Dialkyl(1-allyl-3-arylprop-2-yn-1-yl)- and Dialkyl(1-allyl-3-alkenylprop-2-yn-1-yl)amines by the Stevens Rearrangement

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Abstract—The Stevens rearrangement of dialkyl(allyl)(3-arylprop-2-yn-1-yl)- and dialkyl(allyl)(3-alkenylprop-2-yn-1-yl)ammonium bromides gave dialkyl(1-allyl-3-arylprop-2-yn-1-yl)- and dialkyl(1-allyl-3-alkenylprop-2-yn-1-yl)amines. Here, the allyl group acts as migrating group, and 3-aryl- or 3-alkenylprop-2-yn-1-yl, as receiving one. The yields of the Stevens rearrangement products fall down as the alkyl chain becomes longer, as well as with introduction of a methyl group into the *meta* or *para* position of the aromatic ring.

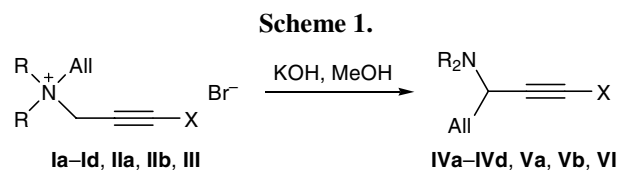
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We previously found that dimethyl(allyl)(prop-2-yn-1-yl or 3-phenylprop-2-yn-1-yl)ammonium bromides undergo base-catalyzed intramolecular cyclization to give mainly the corresponding 1-allyl-2,2-dimethyl-3a,4-dihydrobenzo[*f*]isoindolium and 1-allyl-2,2-dimethylbenzo[*f*]isoindolium bromides and that the cyclization is accompanied by β -elimination, the contribution of the latter process attaining 10–12% [1].

Pharmacological testing has shown that one of these salts exhibits pronounced cardiovascular activity. With a view to extend the number of ammonium salts having a 1-allyl-3-arylprop-2-yn-1-yl group and capable of undergoing cyclization, as well as to obtain new biologically active isoindolium salts, in the present work we have synthesized new dialkyl(1-allyl-3-arylprop-2-yn-1-yl)amines by the Stevens rearrangement of the corresponding ammonium salts.

It is known that the Stevens rearrangement smoothly occurs with dimethyl- and diethyl(allyl)(3-phenylprop-2-yn-1-yl)ammonium and 1-allyl-1-(3-phenylprop-2-yn-1-yl)piperidinium bromides by the action of powdered potassium hydroxide in the presence of a few drops of methanol [2]. To obtain the target amines, as starting compounds we used dipropyl- and dibutyl(allyl)(3-phenylprop-2-yn-1-yl)ammonium bromides **Ia** and **Ib**, 1-allyl-1-(3-phenylprop-2-yn-1-yl)pyrrolidinium bromide (**Ic**), 4-allyl-4-(3-phenylprop-2-yn-1-yl)morpholinium bromide (**Id**), diethyl-

(allyl)(3-*p*-tolylprop-2-yn-1-yl)ammonium bromide (**IIa**), 1-allyl-1-(3-*p*-tolylprop-2-yn-1-yl)piperidinium bromide (**IIb**), and diethyl(allyl)(3-*m*-tolylprop-2-yn-1-yl)ammonium bromide (**III**). In all cases, except for salt **IIa**, the rearrangement was accompanied by heat evolution. In the reaction with salt **III**, the heat evolution was weak; therefore, the reaction mixture was heated for several minutes at 60–70°C to complete the process. As a result, we obtained dipropyl- and dibutyl-(1-allyl-3-phenylprop-2-yn-1-yl)amines **IVa** and **IVb**, 1-(1-allyl-3-phenylprop-2-yn-1-yl)pyrrolidine (**IVc**), 4-(1-allyl-3-phenylprop-2-yn-1-yl)morpholine (**IVd**), diethyl(1-allyl-3-*p*-tolylprop-2-yn-1-yl)amine (**Va**), 1-(1-allyl-3-*p*-tolylprop-2-yn-1-yl)piperidine (**Vb**), and diethyl(1-allyl-3-*m*-tolylprop-2-yn-1-yl)amine (**VI**) in 64, 52, 60, 74, 55, 52, and 58% yield, respectively (Scheme 1). Comparison of the yields of amines **IVa–IVd**, **Va**, **Vb**, and **VI** with those of the dialkyl(1-allyl-3-arylprop-2-yn-1-yl)amines obtained previously [2]



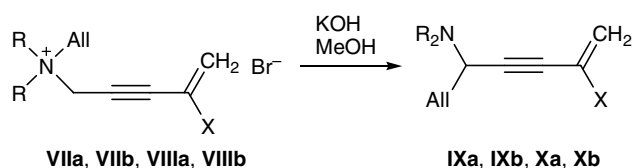
I, IV, X = Ph, **R** = Pr (**a**), Bu (**b**); **RR** = (CH₂)₄ (**c**), O(CH₂CH₂)₂ (**d**); **II, V, X** = *p*-MeC₆H₄, **R** = Et (**a**); **RR** = (CH₂)₅ (**b**); **III, VI, X** = *m*-MeC₆H₄, **R** = Et.

shows that extension of the alkyl group chain and the presence of a methyl group in the *meta* or *para* position of the aromatic rings reduces the yields of the Stevens rearrangement products, which may result from electronic and steric effects of the substituents.

It should be noted that amine **Va** was formed in a yield of 2% by reaction of ammonium salt **Ila** with an equimolar amount of potassium hydroxide in water and that piperidinium analog **Vb** was formed under analogous conditions together with the cyclization–cleavage product [3].

Like dimethyl- and diethylammonium analogs [4, 5], 1-allyl-1-(3-vinylprop-2-yn-1-yl)piperidinium and 4-allyl-4-(3-vinylprop-2-yn-1-yl)morpholinium bromides **VIIa** and **VIIIb** underwent Stevens rearrangement. The 3-isopropenylprop-2-yn-1-yl group in 1-allyl-1-(3-isopropenylprop-2-yn-1-yl)piperidinium and 4-allyl-4-(3-isopropenylprop-2-yn-1-yl)morpholinium bromides **VIIIa** and **VIIIb** was shown to act as receiving moiety in the Stevens rearrangement. In all cases, the rearrangement was accompanied by strong heat evolution, and the products were 1-(1-allyl-3-vinylprop-2-yn-1-yl)piperidine (**IXa**), 1-(1-allyl-3-vinylprop-2-yn-1-yl)morpholine (**IXb**), 1-(1-allyl-3-isopropenylprop-2-yn-1-yl)piperidine (**Xa**), and 1-(1-allyl-3-isopropenylprop-2-yn-1-yl)morpholine (**Xb**) (yield 60–62%; Scheme 2).

Scheme 2.



VII, IX, X = H, RR = (CH₂)₅ (**a**), O(CH₂CH₂)₂ (**b**);
VIII, X, X = Me, RR = (CH₂)₅ (**a**), O(CH₂CH₂)₂ (**b**).

The initial dialkyl(3-arylprop-2-yn-1-yl)amines, 1-(3-vinylprop-2-yn-1-yl)piperidine, and 4-(3-vinylprop-2-yn-1-yl)morpholine, as well as 1-(3-isopropenylprop-2-yn-1-yl)piperidine (**XIa**) and 1-(3-isopropenylprop-2-yn-1-yl)morpholine (**XIb**) were synthesized by the Mannich reaction [6–8]. The synthesis of dialkyl(3-isopropenylprop-2-yn-1-yl)amines, specifically of amine **Xb**, in the presence of excess morpholine was found to be accompanied by formation of dimorpholinomethane whose fraction attained 80% (according to the GLC and ¹H NMR data). Increase of the amount of isopropenylacetylene suppresses the formation of dimorpholinomethane. These findings

confirm our assumption that the Mannich reaction involves initial condensation of secondary amine with formaldehyde to give the corresponding amino alcohol; the latter, depending on the reaction conditions and the reactivity of acetylenic hydrogen atom, reacts either with the second amine molecule or with acetylenic compound or with both these [9].

The structure of salts **Ia–Ic**, **VIIIa**, and **VIIIb** and amines **IVa–IVd**, **Va**, **Vb**, **VI**, **IXa**, **IXb**, **Xa**, **Xb**, **XIa**, and **XIb** was proved by IR spectroscopy, amines **IVc**, **IVd**, **Xa**, **Xb**, **XIa**, and **XIb** were also characterized by ¹H NMR spectra, and UV spectra were recorded for ammonium salts **Ia–Ic**.

EXPERIMENTAL

The IR spectra were recorded in KBr or mineral oil on a UR-20 spectrometer. The UV spectra were measured on a Specord UV-Vis spectrophotometer from solutions in ethanol. The ¹H NMR spectra were obtained on a Varian Mercury-300 instrument at 300 MHz) using TMS as internal reference. The purity of the amines was checked by GLC on an LKhM-8MD chromatograph equipped with a 1.5-m × 4-mm column packed with Inetron AW-HMDS (stationary phase 10% of Carbovax-20M; carrier gas helium, flow rate 40 ml/min at 240°C).

Salts **Id**, **Ila**, **Ilb**, **III**, **VIIa**, and **VIIIb** were prepared by the procedures described in [3, 7], and dialkyl(3-arylprop-2-yn-1-yl)- and dialkyl(3-alkenylprop-2-yn-1-yl)amines were synthesized by the Mannich reaction [6–8].

Ammonium bromides Ia–Ic (general procedure). *Method A.* Allyl bromide, 30 mmol, was added to a solution of 15 mmol of dipropyl- or dibutyl(3-phenylprop-2-yn-1-yl)amine or 1-(3-phenylprop-2-yn-1-yl)pyrrolidine in a mixture of 15 ml of diethyl ether and 5 ml of acetonitrile. The mixture was left to stand overnight, the solvent was distilled off under reduced pressure, and the residue was washed with anhydrous diethyl ether (2 × 20 ml).

Allyl(3-phenylprop-2-yn-1-yl)dipropylammonium bromide (Ia). Yield 4.88 g (97%), hygroscopic substance. UV spectrum: λ_{max} 248 nm. IR spectrum, ν, cm⁻¹: 670, 730 (C₆H₅), 925, 995, 1640 (C=C), 1510, 1570 (C=C_{arom}), 2230 (C≡C), 3060 (C–H_{arom}). Found, %: Br 23.51; N 4.17. C₁₈H₂₆BrN. Calculated, %: Br 23.25; N 4.31.

Allyl(dibutyl)(3-phenylprop-2-yn-1-yl)ammonium bromide (Ib). Yield 5.35 g (98%), hygroscopic

substance. UV spectrum: λ_{\max} 250 nm. IR spectrum, ν , cm^{-1} : 670, 680, 730 (C_6H_5), 925, 995, 1640 ($\text{C}=\text{C}$), 1510, 1570 ($\text{C}=\text{C}_{\text{arom}}$), 2230 ($\text{C}\equiv\text{C}$), 3070 ($\text{C}-\text{H}_{\text{arom}}$). Found, %: Br 21.98; N 3.85. $\text{C}_{20}\text{H}_{30}\text{BrN}$. Calculated, %: Br 22.20; N 3.62.

1-Allyl-1-(3-phenylprop-2-yn-1-yl)pyrrolidinium bromide (Ic). Yield 4.50 g (98%), mp 131–132°C (from anhydrous ethanol). UV spectrum: λ_{\max} 250 nm. IR spectrum, ν , cm^{-1} : 680, 730 (C_6H_5), 925, 995, 1640 ($\text{C}=\text{C}$), 1510, 1570 ($\text{C}=\text{C}_{\text{arom}}$), 2210 ($\text{C}\equiv\text{C}$), 3080 ($\text{C}-\text{H}_{\text{arom}}$). Found, %: Br 26.14; N 4.58. $\text{C}_{16}\text{H}_{20}\text{BrN}$. Calculated, %: Br 26.40; N 4.35.

Amines IVa–IVg, Va, Vb, and VI (general procedure). Method B. A mixture of 13 mmol of salt **Ia–Id**, **IIa**, **IIb**, or **III** and 2 equiv of potassium hydroxide was thoroughly stirred and placed into a round-bottom flask equipped with a reflux condenser. Several drops of methanol were added, and the mixture warmed up. It was allowed to stand at room temperature and extracted with diethyl ether (2×40 ml), the extract was dried over MgSO_4 , the solvent was removed, and the residue was distilled under reduced pressure.

(1-Allyl-3-phenylprop-2-yn-1-yl)dipropylamine (IVa). Yield 2.12 g (64%), bp 145–146°C (1–2 mm), $n_{\text{D}}^{20} = 1.5020$; picrate: mp 95°C (from EtOH). IR spectrum, ν , cm^{-1} : 670, 680, 730 (C_6H_5), 925, 995, 1640 ($\text{C}=\text{C}$), 1510, 1600 ($\text{C}=\text{C}_{\text{arom}}$), 2230 ($\text{C}\equiv\text{C}$), 3060 ($\text{C}-\text{H}_{\text{arom}}$). Found, %: C 84.51; H 9.91; N 5.49. $\text{C}_{18}\text{H}_{25}\text{N}$. Calculated, %: C 85.0; H 9.65; N 5.32.

(1-Allyl-3-phenylprop-2-yn-1-yl)dibutylamine (IVb). Yield 1.91 g (52%), bp 150–151°C (1–2 mm), $n_{\text{D}}^{20} = 1.5110$ (amine **IVb** did not form picrate). IR spectrum, ν , cm^{-1} : 680, 730 (C_6H_5), 925, 995, 1640 ($\text{C}=\text{C}$), 1510, 1570 ($\text{C}=\text{C}_{\text{arom}}$), 2210, 2230 ($\text{C}\equiv\text{C}$), 3060 ($\text{C}-\text{H}_{\text{arom}}$). Found, %: C 85.00; H 10.18; N 4.95. $\text{C}_{20}\text{H}_{29}\text{N}$. Calculated, %: C 84.56; H 10.36; N 5.24.

1-(1-Allyl-3-phenylprop-2-yn-1-yl)pyrrolidine (IVc). Yield 1.75 g (60%), bp 137°C (1–2 mm), $n_{\text{D}}^{20} = 1.5510$; picrate: mp 120°C (from EtOH). IR spectrum, ν , cm^{-1} : 670, 730 (C_6H_5), 925, 995, 1640 ($\text{C}=\text{C}$), 1570 ($\text{C}=\text{C}_{\text{arom}}$), 2210, 2230 ($\text{C}\equiv\text{C}$), 3060 ($\text{C}-\text{H}_{\text{arom}}$). ^1H NMR spectrum ($\text{DMSO}-d_6$ - CCl_4 , 1:3), δ , ppm: 1.76 m (4H, 2 CH_2), 2.43 m (2H, CH_2), 2.66 m (4H, 2 CH_2), 3.70 d.d (1H, NCH, $J = 7.8, 7.0$ Hz), 5.04 d.q (1H, CH_2 , $J = 10.2, 1.6$ Hz), 5.12 d.q (1H, CH_2 , $J = 17.0, 1.8$ Hz), 5.91 d.d.t (1H, CH, $J = 17.0, 10.2, 6.9$ Hz), 7.25–7.37 (5H, C_6H_5). Found, %: C 85.73; H 8.63; N 6.17. $\text{C}_{16}\text{H}_{19}\text{N}$. Calculated, %: C 85.30; H 8.84; N 5.85.

4-(1-Allyl-3-phenylprop-2-yn-1-yl)morpholine (IVd). Yield 2.32 g (74%), bp 153°C (1–2 mm), $n_{\text{D}}^{20} = 1.5630$; picrate: mp 136°C (from EtOH). IR spectrum, ν , cm^{-1} : 670, 680, 730 (C_6H_5), 925, 995, 1640 ($\text{C}=\text{C}$), 1510, 1570 ($\text{C}=\text{C}_{\text{arom}}$), 2210, 2230 ($\text{C}\equiv\text{C}$), 3060 ($\text{C}-\text{H}_{\text{arom}}$). ^1H NMR spectrum ($\text{DMSO}-d_6$ - CCl_4 , 1:3), δ , ppm: 2.46 m (2H, CH_2), 2.50 m (2H, CH_2), 2.66 m (2H, CH_2), 3.53 d.d (1H, NCH, $J = 7.8, 7.0$ Hz), 3.64 m (4H, 2 CH_2), 5.07 d.q (1H, CH_2 , $J = 10.1, 1.5$ Hz), 5.14 d.q (1H, CH_2 , $J = 17.1, 1.8$ Hz), 5.91 d.d.t (1H, CH, $J = 17.1, 10.1, 6.9$ Hz), 7.25–7.37 (5H, C_6H_5). Found, %: C 79.15; H 7.72; N 5.51. $\text{C}_{16}\text{H}_{19}\text{NO}$. Calculated, %: C 79.63; H 7.94; N 5.80.

(1-Allyl-3-p-tolylprop-2-yn-1-yl)diethylamine (Va). Yield 1.72 g (55%), bp 133°C (1 mm), $n_{\text{D}}^{20} = 1.5350$; picrate: mp 104–105°C (from EtOH). IR spectrum, ν , cm^{-1} : 810, 840 (C_6H_4), 925, 995, 1640 ($\text{C}=\text{C}$), 1570, 1600 ($\text{C}=\text{C}_{\text{arom}}$), 2210, 2230 ($\text{C}\equiv\text{C}$), 3060 ($\text{C}-\text{H}_{\text{arom}}$). Found, %: C 84.95; H 9.34; N 6.07. $\text{C}_{17}\text{H}_{23}\text{N}$. Calculated, %: C 84.59; H 9.60; N 5.80.

1-(1-Allyl-3-p-tolylprop-2-yn-1-yl)piperidine (Vb). Yield 1.71 g (52%), bp 140–142°C (1 mm), $n_{\text{D}}^{20} = 1.5550$; picrate: mp 89–91°C (from EtOH). IR spectrum, ν , cm^{-1} : 810, 840 (C_6H_4), 925, 995, 1640 ($\text{C}=\text{C}$), 1510, 1570 ($\text{C}=\text{C}_{\text{arom}}$), 2210, 2230 ($\text{C}\equiv\text{C}$), 3060 ($\text{C}-\text{H}_{\text{arom}}$). Found, %: C 85.71; H 8.95; N 5.76. $\text{C}_{18}\text{H}_{23}\text{N}$. Calculated, %: C 85.32; H 9.15; N 5.53.

(1-Allyl-3-m-tolylprop-2-yn-1-yl)diethylamine (VI). Yield 1.82 g (58%), bp 120°C (1 mm), $n_{\text{D}}^{20} = 1.5310$; picrate: mp 112°C (from EtOH). IR spectrum, ν , cm^{-1} : 690, 610, 750 (C_6H_4), 925, 995, 1640 ($\text{C}=\text{C}$), 1510, 1570 ($\text{C}=\text{C}_{\text{arom}}$), 2210, 2230 ($\text{C}\equiv\text{C}$), 3060 ($\text{C}-\text{H}_{\text{arom}}$). Found, %: C 84.95; H 9.42; N 5.52. $\text{C}_{17}\text{H}_{23}\text{N}$. Calculated, %: C 84.59; H 9.60; N 5.80.

Amines XIa and XIb (general procedure). A metal vessel was charged with 33 g (500 mmol) of isopropenylacetylene, 1.35 g (450 mmol) of paraformaldehyde, 400 mmol of piperidine (34 g) or morpholine (34.8 g), 0.2 g of iron(III) chloride, and 150 ml of dioxane. The mixture was heated for 70 h at 90–95°C and acidified with hydrochloric acid. The solvent was distilled off under reduced pressure, and the residue was made alkaline and extracted with diethyl ether to isolate amine **XIa** or **XIb**.

1-(3-Isopropenylprop-2-yn-1-yl)piperidine (XIa). Yield 40.4 g (62%), bp 82–83°C (2–3 mm), $n_{\text{D}}^{20} = 1.4930$; picrate: mp 103°C (from EtOH). IR spectrum, ν , cm^{-1} : 810, 830 [$\text{C}(\text{CH}_3)=\text{CH}_2$], 1580 ($\text{C}=\text{C}$), 2230 ($\text{C}\equiv\text{C}$). ^1H NMR spectrum ($\text{DMSO}-d_6$ - CCl_4 , 1:3), δ , ppm: 1.88 d.d (3H, CH_3 , $J = 1.7, 1.1$ Hz), 2.47 m (6H,

3CH₂), 3.33 m (2H, CH₂), 3.61 m (4H, 2CH₂), 5.18 quint (1H, CH₂, *J* = 1.7 Hz), 5.21 br.s (1H, CH₂). Found, %: C 81.31; H 10.22; N 8.31. C₁₁H₁₇N. Calculated, %: C 80.98; H 10.43; N 8.59.

4-(3-Isopropenylprop-2-yn-1-yl)morpholine (XIb). Yield 36.3 g (55%), bp 93°C (2 mm), *n*_D²⁰ = 1.5250; picrate: mp 154°C (from EtOH). IR spectrum, *v*, cm⁻¹: 810, 830 [C(CH₃)=CH₂], 1580 (C=C), 2230 (C≡C). ¹H NMR spectrum (DMSO-*d*₆-CCl₄, 1:3), *δ*, ppm: 1.56 m (4H, 2CH₂), 1.87 d.d (3H, CH₃, *J* = 1.8, 1.0 Hz), 2.42 m (4H, 2CH₂), 3.43 m (2H, CH₂), 5.15 quint (1H, CH₂, *J* = 1.7 Hz), 5.18 br.s (1H, CH₂). Found, %: C 72.99; H 8.90; N 8.17. C₁₀H₁₅NO. Calculated, %: C 72.69; H 9.15; N 8.48.

Salts **VIIIa** and **VIIIb** were synthesized from amines **IXa** and **IXb**, respectively, according to method A.

1-Allyl-1-(3-isopropenylprop-2-yn-1-yl)piperidinium bromide (VIIIa). Yield 4.17 g (98%), mp 100°C (from anhydrous EtOH). IR spectrum, *v*, cm⁻¹: 810, 830 [C(CH₃)=CH₂], 925, 995, 1640 (C=C), 1580 (C=C), 2210, 2230 (C≡C). Found, %: Br 27.83; N 5.21. C₁₄H₂₂BrN. Calculated, %: Br 28.11; N 4.93.

4-Allyl-4-(3-isopropenylprop-2-yn-1-yl)morpholinium bromide (VIIIb). Yield 4.16 g (97%), mp 138°C (from anhydrous EtOH). IR spectrum, *v*, cm⁻¹: 810, 830 [C(CH₃)=CH₂], 925, 995, 1640 (C=C), 1580 (C=C), 2210, 2230 (C≡C). Found, %: Br 27.61; N 5.12. C₁₃H₂₀BrNO. Calculated, %: Br 27.92; N 4.89.

Amines **IXa**, **IXb**, **Xa**, and **Xb** were synthesized from salts **VIIa**, **VIIb**, **VIIIa**, and **VIIIb**, respectively, according to method B.

1-(1-Allyl-3-vinylprop-2-yn-1-yl)piperidine (IXa). Yield 1.47 g (60%), bp 99°C (2–3 mm), *n*_D²⁰ = 1.5110; picrate: mp 178°C (from EtOH). IR spectrum, *v*, cm⁻¹: 920, 930, 950, 1640 (C=C), 1580 (C=C), 2210, 2230 (C≡C). Found, %: C 82.12; H 9.87; N 7.61. C₁₃H₁₉N. Calculated, %: C 82.54; H 10.05; N 7.41.

4-(1-Allyl-3-vinylprop-2-yn-1-yl)morpholine (IXb). Yield 1.54 g (62%), bp 99–100°C (1–2 mm), *n*_D²⁰ = 1.5120; picrate: mp 115°C (from EtOH). IR spectrum, *v*, cm⁻¹: 920, 950, 1640 (C=C), 1580 (C=C), 2230 (C≡C). Found, %: C 75.78; H 8.63; N 7.11. C₁₂H₁₇NO. Calculated, %: C 75.39; H 8.90; N 7.33.

1-(1-Allyl-3-isopropenylprop-2-yn-1-yl)piperidine (Xa). Yield 1.72 g (65%), bp 116°C (1–2 mm),

*n*_D²⁰ = 1.4950; picrate: mp 81°C (from EtOH). IR spectrum, *v*, cm⁻¹: 810, 830 [C(CH₃)=CH₂], 925, 995, 1640 (C=C), 1580 (C=C), 2210, 2230 (C≡C). ¹H NMR spectrum (DMSO-*d*₆-CCl₄, 1:3), *δ*, ppm: 1.39–1.60 m (6H, 3CH₂), 1.87 d.d (3H, CH₃, *J* = 1.6, 1.2 Hz), 2.34 m (4H, 2CH₂), 2.54 m (2H, CH₂), 3.35 d.d (1H, NCH, *J* = 8.0, 7.2 Hz), 4.99 d.q (1H, CH₂, *J* = 10.2, 1.6 Hz), 5.05 d.q (1H, CH₂, *J* = 17.1, 1.7 Hz), 5.14 quint (1H, CH₂, *J* = 1.6 Hz), 5.17 br.s (1H, CH₂), 5.82 d.d.t (1H, CH, *J* = 17.1, 10.2, 6.9 Hz). Found, %: C 82.96; H 10.05; N 7.21. C₁₄H₂₁N. Calculated, %: C 82.76; H 10.34; N 6.90.

4-(1-Allyl-3-isopropenylprop-2-yn-1-yl)morpholine (Xb). Yield 1.68 g (63%), bp 95°C (3 mm), *n*_D²⁰ = 1.4960; picrate: mp 117–119°C (from EtOH). IR spectrum, *v*, cm⁻¹: 810, 830 [C(CH₃)=CH₂], 925, 995, 1640 (C=C), 1580 (C=C), 2210, 2230 (C≡C). ¹H NMR spectrum (DMSO-*d*₆-CCl₄, 1:3), *δ*, ppm: 1.89 d.d (3H, CH₃, *J* = 1.6, 1.2 Hz), 2.37 m (2H, CH₂), 2.40 m (2H, CH₂), 2.58 m (2H, CH₂), 3.40 d.d (1H, NCH); 3.60 m (4H, 2CH₂), 5.02 d.q (1H, CH₂, *J* = 10.1, 1.5 Hz), 5.08 d.q (1H, CH₂, *J* = 17.1, 1.8 Hz), 5.18 quint (1H, CH₂, *J* = 1.6 Hz), 5.21 br.s (1H, CH₂), 5.84 d.d.t (1H, CH, *J* = 17.1, 10.1, 6.9 Hz). Found, %: C 76.38; H 9.08; N 6.52. C₁₃H₁₉NO. Calculated, %: C 76.06; H 9.33; N 6.82.

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