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Reaction of Vinylidenecyclopropanes with Aromatic Imines in the Presence of Lewis Acids

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Abstract—1-Methyl-2-(2-methylpropenylidene)-1-phenylcyclopropane, 7-(2-methylpropenylidene)bicyclo-[4.1.0]heptane, and (*Z*)-9-(2-methylpropenylidene)bicyclo[6.1.0]non-4-ene react with *N*-benzylideneanilines in the presence of boron trifluoride–ether complex to give pyrrolidine derivatives. Reactions of 1-methyl-1-phenyl-2-diphenylvinylidenecyclopropane with *N*-benzylideneanilines in the presence of $BF_3 \cdot Et_2O$, $Yb(OTf)_3$, or $Sc(OTf)_3$ lead to the formation of substituted 1,2,3,4-tetrahydroquinolines. 7-Diphenylvinylidenebicyclo-[4.1.0]heptane in the presence of $BF_3 \cdot Et_2O$ undergoes isomerization into 5-phenyl-8,9,10,11-tetrahydro-7*H*-cyclohepta[*a*]naphthalene.

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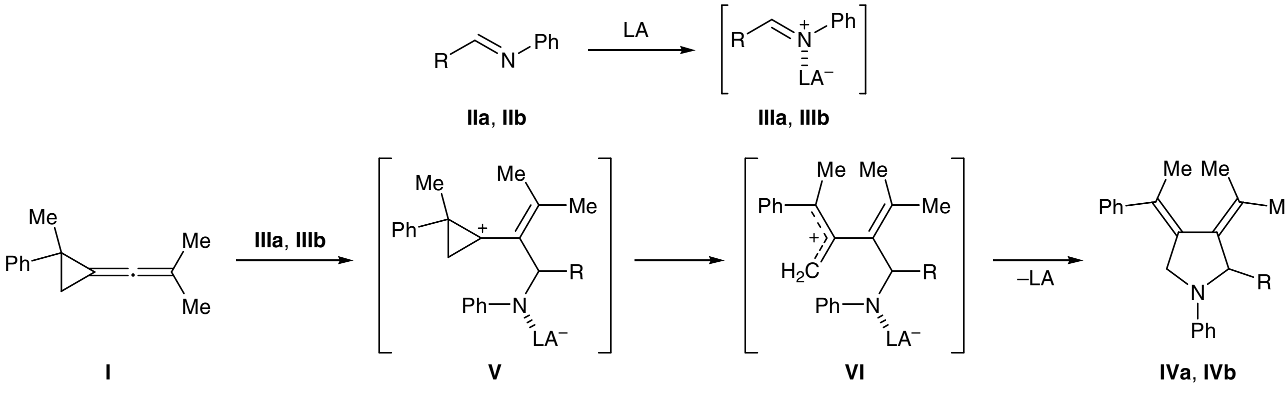
Reactions of alkenes with aromatic imines in the presence of Lewis acids can formally be regarded as aza-Diels–Alder reactions where the aromatic Schiff base acts as aza-diene. These reactions provide a convenient synthetic route to six-membered nitrogen-containing heterocycles, such as piperidines, tetrahydroquinolines, etc. [1]. Shi et al. recently showed that methylenecyclopropanes are capable of reacting with various imines to give tetrahydroquinoline derivatives spiro-fused to a cyclopropane ring [2]. Analogous reactions with compounds having a cumulated double bond system were not reported. The goal of the present work was to study reactions of substituted vinylidenecyclopropanes with imines in the presence of Lewis acids and elucidate the mechanism of the process and effect of substituent nature on the reaction direction.

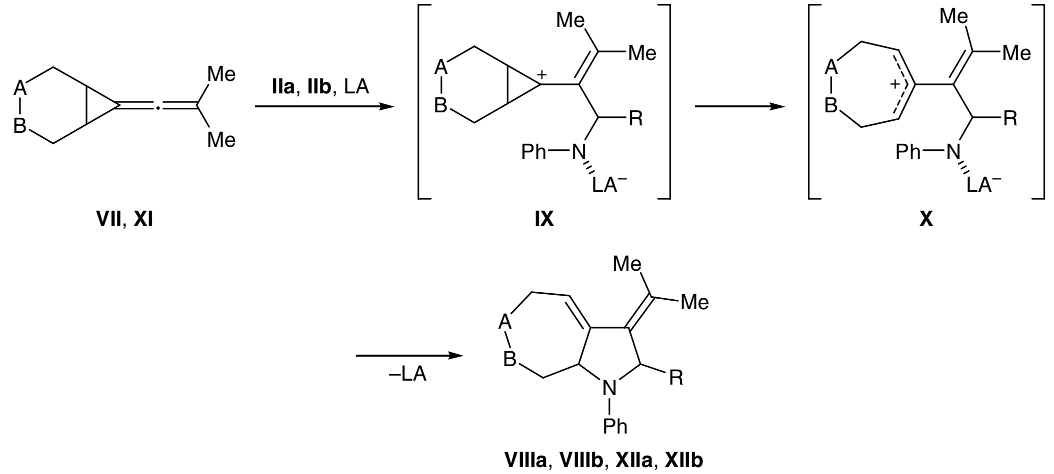
The reactions of 1-methyl-2-(2-methylpropenylidene)-1-phenylcyclopropane (**I**) with *N*-benzylidene- and *N*-(4-chlorobenzylidene)anilines **IIa** and **IIb** in methylene chloride in the presence of boron trifluoride–ether complex resulted in the formation of complex mixtures of products. We succeeded in isolating by column chromatography only the corresponding 2-aryl-3-(1-methylethylidene)-1-phenyl-4-[(*Z*)-1-phenylethylidene]pyrrolidines **IVa** and **IVb** in up to 10% yield. Their structure was determined on the basis of spectral and analytical data. The ^1H NMR spectra of **IVa** and **IVb** contained signals (δ, ppm) from methyl-

Scheme 1.



$LA = BF_3 \cdot Et_2O$; R = Ph (**a**), $4\text{-}ClC_6H_4$ (**b**).

Scheme 2.



VII, **VIII**, A–B = CH_2–CH_2; **XI**, **XII**, A–B = $CH_2CH{=}CHCH_2$; R = Ph (**a**), 4-ClC_6H_4 (**b**); LA = $BF_3 \cdot Et_2O$, $Yb(OTf)_3$.

ene protons (3.96 d and 4.11 d, J = 11 Hz) and CH proton (5.62 s) in the pyrrolidine ring; signals from the methyl protons appeared at δ 1.9–2.3 ppm, indicating that the methyl groups are attached to double-bonded carbon atoms.

As shown previously [3, 4], structures analogous to pyrrolidines **IVa** and **IVb** with *E* configuration of the phenylethylidene fragment are characterized by the methyl group signal located in the region δ 1.9–2.3 ppm, while in the spectra of the corresponding *Z* isomers this signal is displaced upfield (δ 1.4–1.5 ppm) due to shielding by the phenyl group. In the ^{13}C NMR spectra of **IVa** and **IVb**, signals at δ_C 52.6 and 65.9 ppm were assigned to C^2 and C^5, respectively; also, signals from methyl carbon atoms, olefinic fragments, and aromatic rings were present.

The mechanism of formation of pyrrolidines **IVa** and **IVb** may be interpreted as follows. In the first step, Schiff base **II** reacts with Lewis acid to give complex **III** which then acts as electrophile toward initial vinylidenecyclopropane. Here, the electrophilic attack is directed at the central carbon atom of the allene system with formation of cyclopropyl cation **V**; the latter undergoes cyclopropyl–allyl rearrangement, yielding cation **VI**, and cyclization of **VI** leads to substituted pyrrolidine **IV** (Scheme 1).

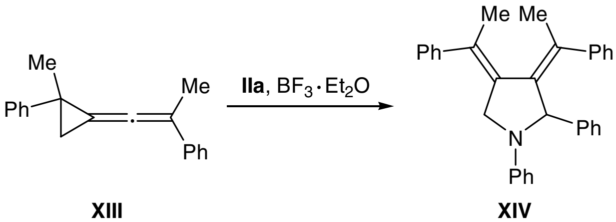
Likewise, 7-(2-methylpropenylidene)bicyclo[4.1.0]-heptane (**VII**) reacted with Schiff bases **IIa** and **IIb** in methylene chloride in the presence of $BF_3 \cdot Et_2O$ to produce a complex mixture of products, from which we isolated by column chromatography 2-aryl-3-(1-methylethylidene)-1-phenyl-1,2,3,5,6,7,8,8a-octahydrocyclohepta[*b*]pyrroles **VIIIa** and **VIIIb** in 23 and 37% yield, respectively. The structure of **VIIIa** and **VIIIb** was determined on the basis of their spectral parameters and elemental compositions. In the ^1H NMR spectra of compounds **VIIIa** and **VIIIb** we observed signals from protons on C^{8a} and C^2 at δ 4.7 (br.d, J = 10.2 Hz) and 5.6 ppm (s), respectively, and olefinic proton signal at δ 6.0 ppm (t, J = 6.5 Hz), as well as signals from aromatic protons and protons of methyl and methylene groups. The C^{8a} and C^2 atoms gave signals in the ^{13}C NMR spectra at δ_C 64 and 66 ppm, respectively; methylene carbon nuclei resonated at δ_C 27.5–31.1 ppm, and methyl carbon nuclei, at δ_C 23.1 and 23.9 ppm.

The mechanism of formation of cycloheptapyrroles **VIIIa** and **VIIIb** is analogous to that proposed above for pyrrolidines **IVa** and **IVb**, i.e., the process involves formation of cation **IX**, followed by cyclopropyl–allyl rearrangement leading to intermediate **X**. The latter undergoes cyclization to fused pyrrolidines **VIIIa** and **VIIIb** (Scheme 2). When the reaction was carried out in the presence of trifluoromethanesulfonic acid, the yield of **VIIIb** was about 10%.

A complex mixture of products was also formed in the reaction of (*Z*)-9-(2-methylpropenylidene)bicyclo-[6.1.0]non-4-ene (**XI**) with Schiff bases **IIa** and **IIb** in the presence of $BF_3 \cdot Et_2O$ (in methylene chloride) or $Yb(OTf)_3$ (in acetonitrile). By preparative thin-layer chromatography we isolated 2-aryl-3-(1-methylethylidene)-1-phenyl-1,2,3,5,6,9,10,10a-octahydrocyclonona[*b*]pyrroles **XIIa** and **XIIb** in 11.4 and 13.7% ($BF_3 \cdot Et_2O$) or 7.9 and 8.6% yield [$Yb(OTf)_3$], respectively. Pyrrolidines **XIIa** and **XIIb** were formed with participation of the allene fragment in **XI**. The structure of compounds **XIIa** and **XIIb** was determined on the basis of their spectral parameters and analytical data. The ^{1}H NMR spectra of **XIIa** and **XIIb** contained signals from protons on C^{10a} and C^2 at δ 4.8 (br.s) and 5.5 ppm (s), respectively; olefinic protons resonated at δ 5.2 (1H) and 5.4 ppm (2H); signals from aromatic protons and methyl (δ 1.9 and 2.0 ppm) and methylene groups were also present. The C^{10a} and C^2 signals appeared in the ^{13}C NMR spectra at δ_C 61.0 and 64.6 ppm, respectively, signals from the methylene carbon atoms were located at δ_C 25.4–34.1 ppm, and methyl carbon atoms were characterized by chemical shifts of δ_C 22.3 and 23.3 ppm.

1-Methyl-1-phenyl-2-(2-phenylpropenylidene)cyclopropane (**XIII**) reacted with *N*-benzylideneaniline (**IIa**) in the presence of $BF_3 \cdot Et_2O$ in CH_2Cl_2 to give a complex mixture of products; from this mixture we isolated by column chromatography 8% of pure 1,2-diphenyl-3,4-bis[(*Z*)-1-phenylethylidene]pyrrolidine (**XIV**) (Scheme 3). The structure of **XIV** was assigned on the basis of its spectral parameters. The ^{1}H NMR spectrum of **XIV** contained signals from methylene protons in the pyrrolidine ring at δ 3.97 (d) and 4.25 ppm (d, *J* = 12 Hz), CH proton at δ 5.48 ppm (s), and methyl protons at δ 2.1 and 2.3 ppm. The position of the CH_3 signals indicates that these groups are attached to double-bonded carbon atoms and is consistent with *Z* configuration of the 1-phenylethylidene fragments in positions *3* and *4* of the pyrrolidine ring.

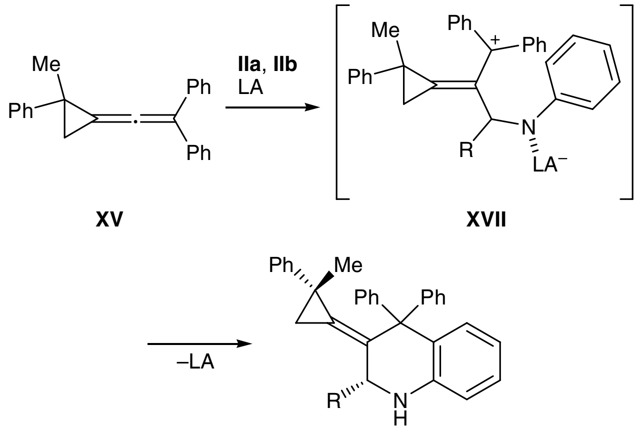
Scheme 3.



XIII **XIV**

In the $BF_3 \cdot Et_2O$-catalyzed reactions of Schiff bases **IIa** and **IIb** with vinylidenecyclopropane **XV** containing two phenyl groups at the double bond we obtained 2-aryl-3-(2-methyl-2-phenylcyclopropylidene)-4,4-diphenyl-1,2,3,4-tetrahydroquinolines **XVIa** and **XVIb** in 21 and 13% yield, respectively (Scheme 4). The use of $Yb(OTf)_3$ or $Sc(OTf)_3$ instead of $BF_3 \cdot Et_2O$ did not increase the yield (see Experimental). Compounds **XVIa** and **XVIb** showed in the IR spectra an absorption band at 3400 cm^{-1} due to stretching vibrations of the N–H bond. In the ^{1}H NMR spectra of these compounds we observed signals from protons in the cyclopropane ring (δ 0.6 ppm, d, *J* = 8.0 Hz; 0.8 ppm, d, *J* = 8.0 Hz), methyl protons (δ 0.90 ppm, s), NH proton (δ 4.1 ppm, s), 2-H (δ 5.2 ppm, s), and protons in the aromatic rings. The structure of tetrahydroquinolines **XVI** was unambiguously proved by the results of X-ray analysis of compound **XVIb** (Fig. 1). Presumably, tetrahydroquinolines **XVIa** and **XVIb** are formed through intramolecular Friedel–Crafts reaction in intermediate cation **XVII**.

Scheme 4.



XV **XVII**

XVIa, **XVIb**

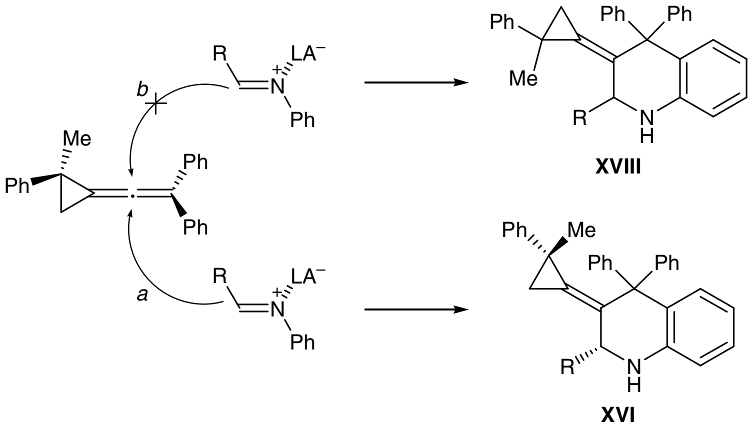
XVI, R = Ph (**a**), $4\text{-}ClC_6H_4$ (**b**); LA = $BF_3 \cdot Et_2O$, $Yb(OTf)_3$, $Sc(OTf)_3$.

It might be presumed that an electrophile approaches the cumulated bond system in vinylidenecyclopropane from the sterically less hindered side (approach *a* in Scheme 5). Alternative approach *b* would lead to the formation of isomeric tetrahydroquinolines **XVIII** which were not detected in the reaction mixture.

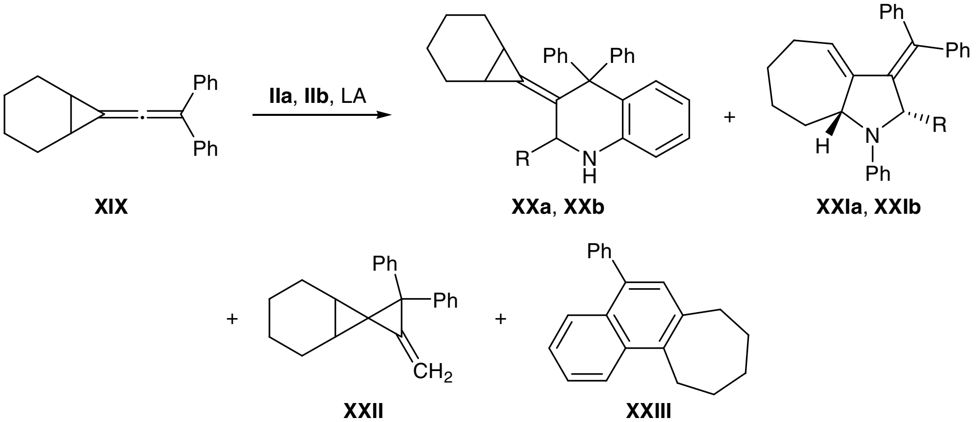
The reactions of 7-diphenylvinylidenebicyclo[4.1.0]heptane (**XIX**) with Schiff bases **IIa** and **IIb** (CH_2Cl_2, $BF_3 \cdot Et_2O$) gave complex mixtures of products. Separation of the product mixtures by column chromatography allowed us to isolate 14–16% of 2-aryl-3-(bicyclo[4.1.0]hept-7-ylidene)-4,4-diphenyl-1,2,3,4-tetrahydroquinolines **XXa** and **XXb**, 5–7% of 2-aryl-3-(diphenylmethylidene)-1-phenyl-1,2,3,5,-6,7,8,8a-octahydrocyclohepta[*b*]pyrroles **XXIa** and **XXIb**, 6% of 3'-methylidene-2',2'-diphenylspiro[bicyclo[4.1.0]heptane-7,1'-cyclopropane] (**XXII**), and 4% of 5-phenyl-8,9,10,11-tetrahydro-7*H*-cyclohepta[*a*]naphthalene (**XXIII**) (Scheme 6).

The structure of products **XX**–**XXIII** was determined on the basis of their spectral and analytical data.

Scheme 5.



Scheme 6.



LA = $BF_3 \cdot Et_2O$; R = Ph (**a**), 4-ClC_6H_4 (**b**).

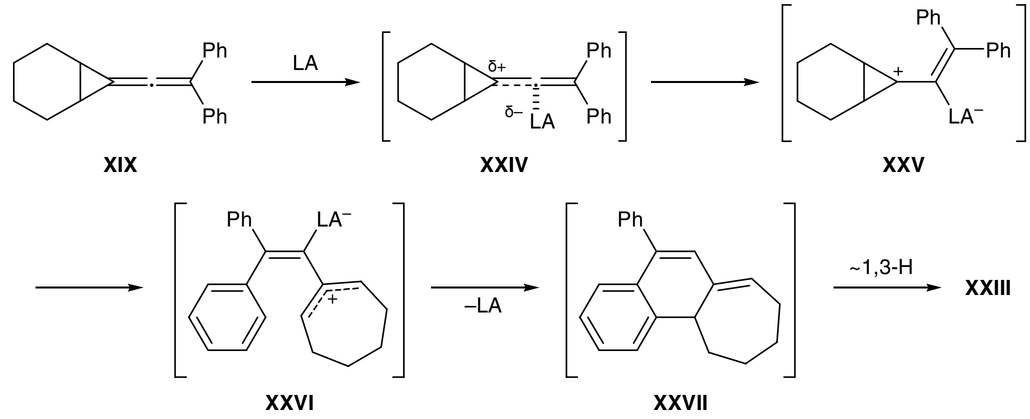
The IR spectra of **XXa** and **XXb** contained an absorption band at 3400 cm^{-1}, corresponding to stretching vibrations of the NH group. In the ^1H NMR spectra of **XXa** and **XXb** signals at δ 3.9 (br.s, NH), 5.1 (s, 2-H), and 0.5–1.3 ppm (10H, bicyclo[4.1.0]heptane fragment), as well as aromatic proton signals, were present. Compounds **XXIa** and **XXIb** displayed in the ^1H NMR spectra signals from protons on C^{8a} and C^2 at δ 4.5 (br.d, $J = 10.2$ Hz) and 5.6 ppm (s), respectively, olefinic proton at δ 5.5 ppm (t, $J = 6.5$ Hz), methylene groups in the region δ 1.3–2.3 ppm, and aromatic protons. Their ^{13}C NMR spectra contained signals from C^{8a} and C^2 at δ_C 64.3 and 68.2 ppm and methylene carbon atoms in the region δ_C 26.5–33.3 ppm. The structure of **XXIa** was finally proved by the X-ray diffraction data (Fig. 2).

In the IR spectrum of **XXII** we observed an absorption band at 1790 cm^{-1}, which arises from stretching vibrations of the exocyclic double bond in the methylidenecyclopropane fragment. The ^1H NMR spectrum of this compound contained singlets at δ 5.21 and 5.42 ppm from the protons at the double bond and signals from protons in the aromatic substituents and bicyclo[4.1.0]heptane fragment (δ 1.3–2.0 ppm). Carbon atoms at the exocyclic double bond resonated in the ^{13}C NMR spectrum at δ_C 95.8 ppm; the spiro[2.2]pentane fragment gave signals at δ_C 20.1 (C^1, C^6), 23.4 (C^7), 32.9 (C^8), and 126.3 (C^9) ppm; and signals from the C^2–C^6 methylene groups appeared in the region δ_C 20.8–22.0 ppm. The structure of **XXII** was finally proved by X-ray analysis (Fig. 3).

The formation of methylidenecyclopropane **XXII** was observed only when the reactions of **XIX** with **IIa** and **IIb** were carried out in methylene chloride. No compound **XXII** was detected in the reaction performed in dichloroethane. These findings suggest that solvent molecule is involved in the process. Compound **XXII** was not formed in the absence of Schiff base **II**.

Naphthalene derivative **XXIII** was formed as a result of isomerization of initial vinylidenecyclopropane

Scheme 7.



XIX by the action of $BF_3 \cdot Et_2O$. Isomerizations of some vinylidenecyclopropanes into naphthalene and methylidenecyclopropane derivatives in the presence of Lewis acids have been reported [5]. We have found that compound **XIX** in methylene chloride in the presence of $BF_3 \cdot Et_2O$ is quantitatively converted into naphthalene **XXIII**: the yield of the product isolated by column chromatography is 97%. The NMR spectra of the reaction mixture contained no signals assignable to other compounds. Scheme 7 shows a probable mechanism of formation of compound **XXIII**. Initially, vinylidenecyclopropane **XIX** reacts with the catalyst to give complex **XXIV** which is converted into zwitterionic species **XXV**; cyclopropyl–allyl rearrangement of the latter produces intermediate **XXVI**, and the subsequent intramolecular Friedel–Crafts reaction and 1,3-hydride shift in tricyclic compound **XXVII** lead to the final product. Compound **XXIII** showed in the ^1H NMR spectrum signals from methylene protons in the region δ 1.8–3.4 ppm and aromatic protons in the region δ 7.3–8.3 ppm. Methylene carbon atoms resonated in the ^{13}C NMR spectrum at δ_C 27.3–37.1 ppm.

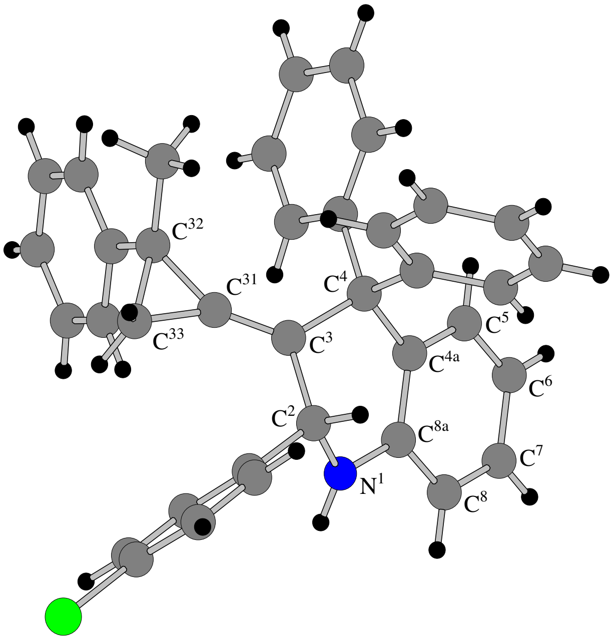


Fig. 1. Structure of the molecule of 2-(4-chlorophenyl)-3-(2-methyl-2-phenylcyclopropylidene)-4,4-diphenyl-1,2,3,4-tetrahydroquinoline (**XVIb**) according to the X-ray diffraction data.

Most probably, the main factor determining the direction of the examined reactions is the nature of substituents at the double bond in the initial vinylidenecyclopropane, i.e., their ability to participate in the stabilization of the carbocation formed in the first stage. Our results show that only products whose formation involves cyclopropyl–allyl rearrangement (substituted pyrrolidines) can be isolated from substrates having methyl groups at the double bond. If a phenyl group is present at the double bond in the substrate molecule, the process leads mainly to compounds formed via intramolecular Friedel–Crafts reaction, namely tetrahydroquinoline derivatives.

EXPERIMENTAL

The IR spectra were recorded from 2% solutions in chloroform on a UR-20 spectrometer. The ^1H and ^{13}C NMR spectra were measured on a Bruker DPX-300 instrument (300.13 MHz for ^1H) from 2% solutions in $CDCl_3$. The elemental analyses were obtained on a Hewlett-Packard 185-B CHN analyzer. The melting points were were determined on a Boetius melting point apparatus. The progress of reactions and the purity of products were monitored by thin-layer chromatography on Silufol UV-254 plates. Column chromatography was performed using silica gel L 100–160 (100–160 μm) and L 40–100 (40–100 μm). Intial vinylidenecyclopropanes **I**, **VII**, and **XI** were synthesized as described in [6], and compounds **XIII**, **XV**, and **XIX**, by the procedure reported in [7].

3-(1-Methylethylidene)-1,2-diphenyl-4-[(*Z*)-1-phenylethylidene]pyrrolidine (IVa). A 10-ml round-bottom flask was charged under argon with 0.5 g (2.7 mmol) of vinylidenecyclopropane **I**, 0.49 g (2.7 mmol) of Schiff base **IIa**, and 3 ml of anhydrous methylene chloride. Boron trifluoride–ether complex, 0.08 g (0.54 mmol), was added dropwise under stirring, and the mixture was stirred for 3 days at room temperature under argon. The solvent was evaporated, and the residue was subjected to column chromatography using hexane–ethyl acetate (50:1, by volume) as eluent. Yield 0.096 g (9.8%), amorphous substance. ^{1}H NMR spectrum, δ, ppm: 1.99 s (3H, Me), 2.02 s (3H, Me), 2.29 s (3H, Me), 3.94 d (1H, CH_2, J = 10.9 Hz), 4.09 d (1H, CH_2, J = 10.9 Hz), 5.41 s (1H, CH), 6.46–6.66 m (10H, H_{arom}), 6.97–7.05 m (5H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 21.3, 21.4, 23.6 (Me), 52.8 (C^5), 65.9 (C^2), 112.9, 116.8, 126.3, 129.7, 130.3, 131.1, 131.4, 131.7, 133.2, 133.6, 135.9, 136.3, 141.1, 142.8, 147.5. Found, %: C 88.58; H 7.32; N 3.69. $C_{27}H_{27}N$. Calculated, %: C 88.72; H 7.45; N 3.83.

2-(4-Chlorophenyl)-3-(1-methylethylidene)-1-phenyl-4-[(*Z*)-1-phenylethylidene]pyrrolidine (IVb) was synthesized in a similar way from 0.5 g (2.7 mmol) of vinylidenecyclopropane **I**, 0.58 g (2.7 mmol) of Schiff base **IIb**, and 0.08 g (0.27 mmol) of $BF_3 \cdot Et_2O$ in 3 ml of anhydrous methylene chloride. Yield 0.091 g (8.7%). Amorphous substance. ^{1}H NMR spectrum, δ, ppm: 1.97 s (3H, Me), 2.02 s (3H, Me), 2.26 s (3H, Me), 3.95 d (1H, CH_2, J = 10.9 Hz), 4.07 d (1H, CH_2, J = 10.9 Hz), 5.44 s (1H, CH), 6.58–6.89 m (9H, H_{arom}), 7.08–7.26 m (5H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 21.2, 21.5, 23.4 (Me), 52.6 (C^5), 65.9 (C^2), 112.8, 116.9, 127.8, 128.2, 129.7, 130.3, 131.5, 132.3, 133.1, 133.6, 134.1, 134.6, 136.2, 142.8, 143.4, 147.6. Found, %: C 80.89; H 6.47; N 3.41. $C_{27}H_{26}ClN$. Calculated, %: C 81.08; H 6.55; N 3.50.

3-(1-Methylethylidene)-1,2-diphenyl-1,2,3,5,6,7,-8,8a-octahydrocyclohepta[*b*]pyrrole (VIIIa). A 5-ml round-bottom flask was charged under argon with 0.25 g (1.8 mmol) of vinylidenecyclopropane **VII**, 0.32 g (1.8 mmol) of Schiff base **IIa**, and 3 ml of anhydrous methylene chloride, and 0.05 g (0.35 mmol) of $BF_3 \cdot Et_2O$ was added dropwise under stirring. The mixture was stirred for 3 days at room temperature under argon and evaporated, and the residue was subjected to column chromatography on silica gel (gradient elution with hexane–ethyl acetate, 45:1, 40:1, 35:1, by volume). Yield 0.13 g (23%), colorless crystals, mp 137–139°C (from MeOH). ^{1}H NMR spectrum, δ, ppm: 1.13–1.14 m (2H, CH_2), 1.78–2.10 m (4H, CH_2), 1.94 s (3H, Me), 1.98 s (3H, Me), 2.28–2.48 m (2H, CH_2), 4.73 d (1H, CH, J = 10.2 Hz), 5.65 s (1H, CH), 6.06 t (1H, =CH, J = 6.5 Hz), 6.57–

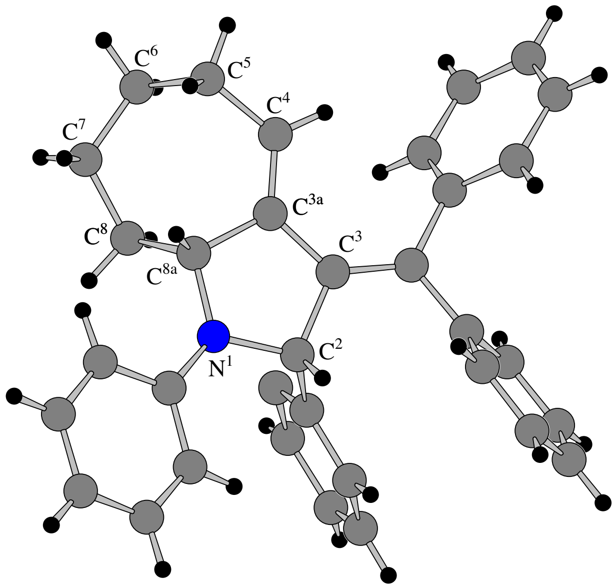


Fig. 2. Structure of the molecule of 3-(diphenylmethylidene)-1,2-diphenyl-1,2,3,5,6,7,8,8a-octahydrocyclohepta[*b*]-pyrrole (**XXIa**) according to the X-ray diffraction data.

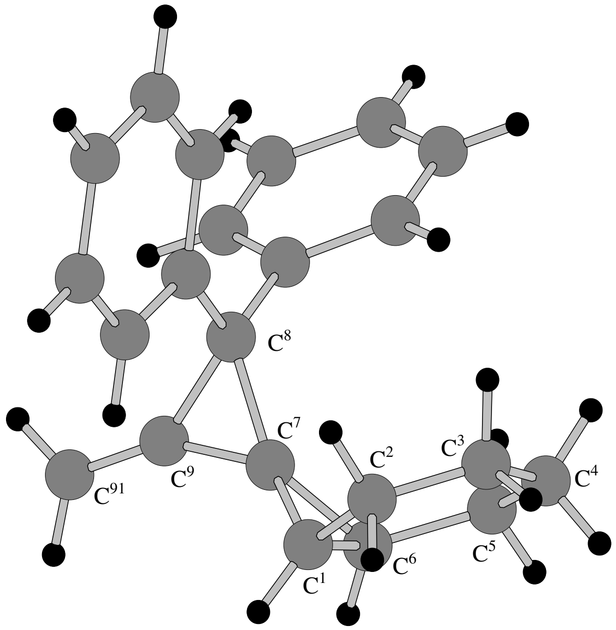


Fig. 3. Structure of the molecule of 3'-methylidene-2',2'-diphenylspiro[bicyclo[4.1.0]heptane-7,1'-cyclopropane] (**XXII**) according to the X-ray diffraction data.

6.64 m (3H, H_{arom}), 7.10–7.25 m (7H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 23.2, 24.0 (Me), 27.6, 29.5, 29.6, 31.0 (CH_2), 64.5 (C^5), 66.6 (C^2), 115.2, 116.5, 126.4, 126.8, 127.9, 128.5, 128.8, 129.1, 133.7, 143.6, 145.0. Found, %: C 87.46; H 8.29; N 4.35. $C_{24}H_{27}N$. Calculated, %: C 87.49; H 8.26; N 4.35.

2-(4-Chlorophenyl)-3-(1-methylethylidene)-1-phenyl-1,2,3,5,6,7,8,8a-octahydrocyclohepta[*b*]pyrrole (VIIIb). *a.* Compound **VIIIb** was synthesized as described above for **VIIIa** from 0.25 g (1.7 mmol) of vinylidenecyclopropane **VII**, 0.37 g (1.7 mmol) of Schiff base **IIb**, and 0.025 g (0.17 mmol) of $BF_3 \cdot Et_2O$ in 3 ml of anhydrous methylene chloride. Yield 0.23 g (37%), colorless crystals, mp 161–162°C (from MeOH). ^{1}H NMR spectrum, δ, ppm: 1.11–1.14 m (2H, CH_2), 1.79–2.11 m (4H, CH_2), 1.90 s (3H, Me), 1.97 s (3H, Me), 2.24–2.46 m (2H, CH_2), 4.68 d (1H, CH, J = 10.2 Hz), 5.62 s (1H, CH), 6.06 t (1H, =CH, J = 6.5 Hz), 6.58–6.63 m (3H, H_{arom}), 7.05–7.17 m (6H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 23.1, 24.0 (Me), 27.5, 29.4, 29.6, 31.0 (CH_2), 64.5 (C^5), 66.0 (C^2), 115.3, 116.8, 126.7, 128.7, 129.1, 129.2, 132.4, 133.4, 142.1, 144.7, 145.7. Found, %: C 79.38; H 7.24; N 3.85. $C_{24}H_{26}ClN$. Calculated, %: C 79.21; H 7.20; N 3.85.

b. Trifluoromethanesulfonic acid, 0.14 g (0.34 mmol), was added dropwise to a mixture of 0.25 g (1.7 mmol) of vinylidenecyclopropane **VII**, 0.39 g (1.7 mmol) of Schiff base **IIb**, and 3 ml of anhydrous methylene chloride under stirring in a stream of argon. The mixture was stirred for 3 days at room temperature under argon and evaporated, and the residue was subjected to column chromatography on silica gel using hexane–ethyl acetate (50:1, by volume) as eluent. Yield 0.062 g (10%).

3-(1-Methylethylidene)-1,2-diphenyl-1,2,3,5,-6,9,10,10a-octahydrocyclonona[*b*]pyrrole (XIIa). *a.* Boron trifluoride–ether complex, 0.082 g (0.58 mmol), was added dropwise to a mixture of 0.5 g (2.9 mmol) of vinylidenecyclopropane **XI**, 0.52 g (2.9 mmol) of Schiff base **IIa**, and 3 ml of anhydrous methylene chloride under stirring in a stream of argon. The mixture was stirred for 3 days at room temperature under argon and evaporated, and the residue was subjected to preparative thin-layer chromatography using hexane–ethyl acetate (50:1, by volume) as eluent. Yield 0.11 g (11.4%), light yellow oily substance. IR spectrum, ν, cm^{-1}: 1160, 1270, 1380, 1460, 1510, 1610, 2870, 2940, 3050. ^{1}H NMR spectrum, δ, ppm: 1.76–1.82 m (2H, CH_2), 1.85–2.04 m (2H, CH_2), 1.93 s (3H, Me), 2.01 s (3H, Me), 2.11–2.20 m (2H, CH_2), 2.39–2.47 m (2H, CH_2), 4.81 br.s (1H, CH), 5.12–5.21 m (1H, =CH), 5.35–5.45 m (2H, =CH), 5.47 s (1H, CH), 6.70–6.90 m (4H, H_{arom}), 7.19–7.28 m (6H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 22.2, 23.3 (Me), 25.4, 28.5, 29.8, 34.1 (CH_2), 61.0 (C^5), 64.6 (C^2), 113.5, 116.9, 125.8, 129.3, 130.1, 130.9, 131.3, 131.4, 131.5, 131.6, 135.7, 138.2, 147.5. Found, %: C 87.67; H 8.14; N 3.83. $C_{26}H_{29}N$. Calculated, %: C 87.84; H 8.22; N 3.94.

b. A mixture of 0.5 g (2.9 mmol) of vinylidenecyclopropane **XI**, 0.52 g (2.9 mmol) of Schiff base **IIa**, 120 mg of $Yb(OTf)_3$, and 3.5 ml of anhydrous acetonitrile was stirred for 4 days at room temperature under argon. The solvent was distilled off, and the residue was subjected to preparative thin-layer chromatography using hexane–ethyl acetate (50:1, by volume) as eluent. Yield 0.076 g (7.9%).

2-(4-Chlorophenyl)-3-(1-methylethylidene)-1-phenyl-1,2,3,5,6,9,10,10a-octahydrocyclonona[*b*]-pyrrole (XIIb) was synthesized in a similar way from 0.5 g (2.9 mmol) of vinylidenecyclopropane **XI** and 0.62 g (2.9 mmol) of Schiff base **IIb** in the presence of $BF_3 \cdot Et_2O$ or $Yb(OTf)_3$. Yield 0.15 g (13.7%, *a*), 0.094 g (8.6%, *b*), light yellow oily substance. IR spectrum, ν, cm^{-1}: 1030, 1110, 1370, 1460, 1510, 1610, 2870, 2940, 3040. ^{1}H NMR spectrum, δ, ppm: 1.73–1.79 m (2H, CH_2), 1.87–2.06 m (2H, CH_2), 1.95 s (3H, Me), 2.02 s (3H, Me), 2.12–2.22 m (2H, CH_2), 2.40–2.48 m (2H, CH_2), 4.83 br.s (1H, CH), 5.17–5.26 m (1H, =CH), 5.36–5.47 m (2H, =CH), 5.49 s (1H, CH), 6.73–6.90 m (4H, H_{arom}), 7.24–7.35 m (5H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 22.2, 23.3 (Me), 25.5, 28.5, 29.9, 34.2 (CH_2), 61.1 (C^5), 64.7 (C^2), 113.4, 117.1, 126.1, 127.9, 129.1, 129.6, 130.2, 130.8, 131.3, 131.5, 132.4, 134.7, 140.5, 147.4. Found, %: C 80.19; H 7.17; N 3.47. $C_{26}H_{28}ClN$. Calculated, %: C 80.08; H 7.24; N 3.59.

1,2-Diphenyl-3,4-bis[(*Z*)-1-phenylethylidene]-pyrrolidine (XIV). Boron trifluoride–ether complex, 0.029 g (0.2 mmol), was added dropwise to a mixture of 0.25 g (1.0 mmol) of vinylidenecyclopropane **XIII**, 0.18 g (1.0 mmol) of Schiff base **IIa**, and 2 ml of anhydrous methylene chloride under stirring in a stream of argon. The mixture was stirred for 3 days at room temperature under argon, the solvent was evaporated, and the residue was subjected to column chromatography on silica gel using hexane–ethyl acetate (40:1, by volume) as eluent. Yield 0.034 g (8.1%), amorphous substance. ^{1}H NMR spectrum, δ, ppm: 2.11 s

(3H, Me), 2.28 s (3H, Me), 3.97 d (1H, CH_2, J = 12.1 Hz), 4.25 d (1H, CH_2, J = 12.1 Hz), 5.48 s (1H, CH), 6.55–7.41 m (20H, H_{arom}). Found, %: C 89.73; H 6.65; N 3.14. $C_{32}H_{29}N$. Calculated, %: C 89.89; H 6.84; N 3.28.

3-(2-Methyl-2-phenylcyclopropylidene)-2,4,4-triphenyl-1,2,3,4-tetrahydroquinoline (XVIa). *a*. Boron trifluoride–ether complex, 0.058 g (0.4 mmol), was added dropwise to a mixture of 0.29 g (0.9 mmol) of vinylidenecyclopropane **XV**, 0.17 g (0.9 mmol) of Schiff base **IIa**, and 3 ml of anhydrous methylene chloride under stirring in a stream of argon. The mixture was stirred for 3 days at room temperature under argon, the solvent was evaporated, and the residue was subjected to column chromatography on silica gel using hexane–ethyl acetate (50:1, by volume) as eluent. Yield 0.097 g (21%), colorless crystals, mp 195–196°C (from EtOH). IR spectrum, ν, cm^{-1}: 1080, 1110, 1170, 1260, 1290, 1320, 1470, 1500, 1620, 1780, 2960, 3050, 3400. ^1H NMR spectrum, δ, ppm: 0.53 d (1H, CH_2, J = 8.5 Hz), 0.83 d (1H, CH_2, J = 8.5 Hz), 0.91 s (3H, Me), 4.11 br.s (1H, NH), 5.23 s (1H, CH), 6.52–7.38 m (24H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 21.5 (Me), 23.4 (CH_2), 35.1 (C, cyclopropane), 60.6 (C^4), 62.0 (C^2), 115.1, 117.6, 125.1, 125.3, 126.5, 126.8, 127.0, 127.8, 128.1, 128.7, 129.7, 130.1, 131.7, 131.9, 135.0, 144.4, 145.2, 145.3, 145.7, 146.0. Found, %: C 90.65; H 6.32; N 2.91. $C_{37}H_{31}N$. Calculated, %: C 90.76; H 6.38; N 2.86.

b. A mixture of 50 mg (0.16 mmol) of vinylidenecyclopropane **XV**, 29 mg (0.16 mmol) of Schiff base **IIa**, 27 mg of $Yb(OTf)_3$, and 0.5 ml of anhydrous methylene chloride was stirred for 5 days at room temperature under argon. The solvent was distilled off, and the residue was subjected to preparative thin-layer chromatography using hexane–ethyl acetate (50:1, by volume) as eluent. Yield 5.8 mg (7.6%). When the reaction time was prolonged to 10 days, the yield of **XVIa** was 9.8 mg (12.8%).

c. A mixture of 50 mg (0.16 mmol) of vinylidenecyclopropane **XV**, 29 mg (0.16 mmol) of Schiff base **IIa**, 22 mg of $Sc(OTf)_3$, and 0.5 ml of anhydrous methylene chloride was stirred for 5 days at room temperature under argon. The solvent was distilled off, and the residue was subjected to preparative thin-layer chromatography using hexane–ethyl acetate (50:1, by volume) as eluent. Yield 2.6 mg (3.4%). When the reaction time was prolonged to 10 days, the yield of **XVIa** was 4.4 mg (5.7%).

2-(4-Chlorophenyl)-3-(2-methyl-2-phenylcyclopropylidene)-4,4-diphenyl-1,2,3,4-tetrahydroquinoline (XVIb) was synthesized in a similar way from 0.21 g (0.7 mmol) of vinylidenecyclopropane **XV** and 0.15 g (0.7 mmol) of Schiff base **IIb** in the presence of $BF_3 \cdot Et_2O$. Yield 0.048 g (13%), colorless crystals, mp 231–232°C (from EtOH). IR spectrum, ν, cm^{-1}: 1050, 1090, 1110, 1180, 1270, 1290, 1320, 1450, 1510, 1620, 1790, 2960, 3050, 3400. ^1H NMR spectrum, δ, ppm: 0.62 d (1H, CH_2, J = 8.5 Hz), 0.85 d (1H, CH_2, J = 8.5 Hz), 0.90 s (3H, Me), 4.04 br.s (1H, NH), 5.22 s (1H, CH), 6.54–7.40 m (23H, H_{arom}). Found, %: C 84.82; H 5.85; N 2.61. $C_{37}H_{30}ClN$. Calculated, %: C 84.79; H 5.77; N 2.67.

Reaction of 7-diphenylvinylidenebicyclo[4.1.0]-heptane (XIX) with *N*-benzylideneaniline (IIa) in the presence of $BF_3 \cdot Et_2O$. Boron trifluoride–ether complex, 0.048 g (0.34 mmol), was added dropwise to a mixture of 0.48 g (1.7 mmol) of compound **XIX**, 0.306 g (1.7 mmol) of Schiff base **IIa**, and 3 ml of anhydrous methylene chloride under stirring in a stream of argon. The mixture was stirred for 3 days at room temperature under argon, the solvent was evaporated, and the residue was subjected to column chromatography on silica gel using hexane–ethyl acetate (40:1, by volume) as eluent to isolate 0.11 g (14%) of 3-(bicyclo[4.1.0]hept-7-yl)-2,4,4-triphenyl-1,2,3,4-tetrahydroquinoline (**XXa**), 0.055 g (7%) of 3-(diphenylmethylidene)-1,2-diphenyl-1,2,3,5,6,7,8,8a-octahydrocyclohepta[*b*]pyrrole (**XXIa**), 0.030 g (6%) of 3'-methylidene-2',2'-diphenylspiro[bicyclo[4.1.0]-heptane-7,1'-cyclopropane] (**XXII**), and 0.019 g (4%) of 5-phenyl-8,9,10,11-tetrahydro-7*H*-cyclohepta[*a*]-naphthalene (**XXIII**).

Compound **XXa**. Colorless crystals, mp 203–204°C (from EtOH). IR spectrum, ν, cm^{-1}: 1110, 1190, 1250, 1290, 1330, 1470, 1510, 1620, 1780, 2970, 3050, 3400. ^1H NMR spectrum, δ, ppm: 0.48–1.23 m (10H, 2CH, $4CH_2$), 3.98 br.s (1H, NH), 5.12 s (1H, CH), 6.50 d (1H, H_{arom}, J = 7.1 Hz), 6.75–7.36 m (18H, H_{arom}). Found, %: C 89.83; H 6.78; N 2.97. $C_{34}H_{31}N$. Calculated, %: C 90.02; H 6.89; N 3.09.

Compound **XXIa**. Colorless crystals, mp 182–183°C (from MeOH). IR spectrum, ν, cm^{-1}: 1040, 1090, 1170, 1360, 1450, 1510, 1610, 2870, 2940, 3050. ^1H NMR spectrum, δ, ppm: 1.34–1.83 m (4H, CH_2), 2.00–2.29 m (4H, CH_2), 4.57 br.d (1H, CH, J = 9.4 Hz), 5.42 s (1H, CH), 5.56 t (1H, =CH, J = 5.8 Hz), 6.62–6.70 m (3H, H_{arom}), 6.87–7.31 m (17H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 26.5, 28.7, 29.6, 33.3 (CH_2), 64.3 (C^5), 68.2 (C^2), 112.0, 116.4, 126.7, 127.2, 128.2, 128.5, 128.6, 128.8, 129.1, 129.3, 129.4, 129.5, 137.6, 138.5, 143.4, 143.6, 145.1, 146.4. Found, %: C 90.17; H 6.81; N 3.02. $C_{34}H_{31}N$. Calculated, %: C 90.02; H 6.89; N 3.09.

Compound **XXII**. Colorless crystals, mp 104–105°C (from MeOH). IR spectrum, ν, cm^{-1}: 910, 1050, 1090, 1110, 1290, 1340, 1450, 1510, 1590, 1780, 2840, 2950, 3040. ^1H NMR spectrum, δ, ppm: 1.29–1.96 m (10H), 5.22 s (1H, =CH), 5.42 s (1H, =CH), 7.23–7.44 m (10H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 20.1 (2CH), 20.8, 21.5, 21.8, 22.0 (CH_2), 23.4 (C^7), 32.9 (C^8), 95.8 (=CH_2), 126.3 (C^9), 128.2, 128.4, 128.5, 128.9, 129.3, 141.5, 144.8, 147.9. Found, %: C 92.25; H 7.63. $C_{22}H_{22}$. Calculated, %: C 92.26; H 7.74.

Compound **XXIII**. Colorless crystals, mp 50–51°C (from MeOH). ^1H NMR spectrum, δ, ppm: 1.80–2.02 m (6H, $3CH_2$), 3.10 t (2H, CH_2, J = 5.1 Hz), 3.39 t (2H, CH_2, J = 5.1 Hz), 7.34 s (1H, H_{arom}), 7.40–7.59 m (7H, H_{arom}), 7.99 d (1H, H_{arom}, J = 8.4 Hz), 8.28 d (1H, H_{arom}, J = 8.4 Hz). ^{13}C NMR spectrum, δ_C, ppm: 27.4, 28.1, 28.6, 32.9, 37.1 (CH_2), 124.0, 124.9, 126.0, 127.1, 127.4, 128.6, 130.0, 130.7, 131.1, 132.4, 138.4, 138.6, 140.8, 141.7. Found, %: C 92.49; H 7.44. $C_{21}H_{20}$. Calculated, %: C 92.60; H 7.40.

Reaction of 7-diphenylvinylidenebicyclo[4.1.0]-heptane (XIX) with *N*-(4-chlorobenzylidene)aniline in the presence of $BF_3 \cdot Et_2O$. Boron trifluoride–ether complex, 0.014 g (0.19 mmol), was added dropwise to a mixture of 0.27 g (0.96 mmol) of vinylidenecyclopropane **XIX**, 0.21 g (0.96 mmol) of Schiff base **IIb**, and 3 ml of anhydrous methylene chloride under stirring in a stream of argon. The mixture was stirred for 3 days at room temperature under argon, the solvent was evaporated, and the residue was subjected to column chromatography on silica gel using hexane–ethyl acetate (40:1, by volume) as eluent to isolate 0.076 g (16%) of 3-(bicyclo[4.1.0]hept-7-yl)-2-(4-chlorophenyl)-4,4-diphenyl-1,2,3,4-tetrahydroquinoline (**XXb**), 0.024 (5%) of 2-(4-chlorophenyl)-3-(diphenylmethylidene)-1-phenyl-1,2,3,5,6,7,8,8a-octahydrocyclohepta[*b*]pyrrole (**XXIb**), 0.016 g (5.7%) of compound **XXII**, and 0.011 (4%) of cycloheptanaphthalene (**XXIII**).

Compound **XXb**. Colorless crystals, mp 217–218°C (from EtOH). IR spectrum, ν, cm^{-1}: 1110, 1190, 1210, 1270, 1290, 1340, 1470, 1520, 1610, 1780, 2970, 3050, 3400. ^1H NMR spectrum, δ, ppm: 0.47–0.54 m (1H, CH), 0.64–0.69 m (1H, CH), 0.80–1.27 m (8H, CH_2), 3.93 br.s (1H, NH), 5.11 s (1H, CH), 6.53 d (1H, H_{arom}, J = 7.3 Hz), 6.71–6.92 m (4H, H_{arom}), 7.18–7.33 m (13H, H_{arom}). Found, %: C 83.63; H 6.12; N 2.95. $C_{34}H_{30}ClN$. Calculated, %: C 83.67; H 6.20; N 2.87.

Compound **XXIb**. Colorless crystals, mp 195–196°C (from EtOH). IR spectrum, ν, cm^{-1}: 1050, 1090, 1180, 1370, 1450, 1510, 1620, 2870, 2940, 3050. ^1H NMR spectrum, δ, ppm: 1.36–1.79 m (4H, CH_2), 2.05–2.34 m (4H, CH_2), 4.64 br.d (1H, CH, J = 9.4 Hz), 5.45 s (1H, CH), 5.55 t (1H, =CH, J = 5.8 Hz), 6.60–6.75 m (5H, H_{arom}), 6.89–7.30 m (14H, H_{arom}). Found, %: C 83.48; H 6.34; N 2.78. $C_{34}H_{30}ClN$. Calculated, %: C 83.67; H 6.20; N 2.87.

5-Phenyl-8,9,10,11-tetrahydro-7*H*-cyclohepta[*a*]-naphthalene (XXIII). Two drops of $BF_3 \cdot Et_2O$ were added to a mixture of 0.1 g (0.35 mmol) of vinylidenecyclopropane **XIX** and 1 ml of anhydrous methylene chloride under stirring in a stream of argon. The mixture was stirred for 4 h at room temperature under argon, the solvent was evaporated, and the product was isolated by flash chromatography using hexane as eluent. Yield 0.097 g (97%).

X-Ray diffraction data for compound XVIb. MoK_α irradiation, λ = 0.71069 Å, graphite monochromator. $C_{37}H_{30}ClN$. *M* 489.65. Monoclinic crystal system, space group *C*12/*c*1 (no. 15); unit cell parameters: a = 46.2930(69), b = 7.5992(7), c = 18.2125(30) Å; β = 112.67(1)°; V = 5911.97(2720) $Å^3$; d_{calc} = 1.191 g/cm^3. Selected bond lengths (Å) and bond angles (deg): $N^1–C^{8a}$ 1.399(6), $N^1–C^2$ 1.463(6), $C^2–C^3$ 1.525(6), $C^3–C^{31}$ 1.320(6), $C^3–C^4$ 1.544(6), $C^4–C^{4a}$ 1.538(6), $C^{4a}–C^{8a}$ 1.405(6), $C^{31}–C^{33}$ 1.466(6), $C^{31}–C^{32}$ 1.482(6), $C^{32}–C^{33}$ 1.532(6); $C^{8a}N^1C^2$ 118.4(4), $N^1C^2C^3$ 106.8(4), $C^{31}C^3C^2$ 123.0(4), $C^{31}C^3C^4$ 127.0(4), $C^2C^3C^4$ 109.9(4), $C^{4a}C^4C^3$ 109.2(3), $C^3C^{31}C^{33}$ 145.1(4), $C^3C^{31}C^{32}$ 151.8(4), $C^{33}C^{31}C^{32}$ 62.6(3), $C^{31}C^{32}C^{33}$ 58.2(3), $C^{31}C^{33}C^{32}$ 59.2(3).

X-Ray diffraction data for compound XXIa. MoK_α irradiation, λ = 0.71069 Å, graphite monochromator. $C_{34}H_{31}N$. *M* 453.62. Triclinic crystal system, space group *P*-1 (no. 2); unit cell parameters: a = 10.346(5), b = 10.555(5), c = 13.057(5) Å; α = 110.685(5), β = 100.326(5), γ = 96.187(5)°; V = 1289.4(10) $Å^3$; d_{calc} = 1.166 g/cm^3. Selected bond lengths (Å) and bond angles (deg): $N^1–C^{8a}$ 1.444(3), $N^1–C^2$ 1.482(3), $C^2–C^3$ 1.546(3), $C^3–C^{3a}$ 1.457(3), $C^{3a}–C^4$ 1.340(3), $C^{3a}–C^{8a}$ 1.499(3), $C^4–C^5$ 1.503(3), $C^5–C^6$ 1.510(4), $C^6–C^7$ 1.525(3), $C^7–C^8$ 1.525(3), $C^8–C^{8a}$ 1.528(4); $C^{8a}N^1C^2$ 114.26(16), $N^1C^2C^3$ 102.5(2), $C^{3a}C^3C^2$ 108.5(2), $C^4C^{3a}C^3$ 128.9(2), $C^4C^{3a}C^{8a}$ 120.8(2), $C^3C^{3a}C^{8a}$ 109.9(2), $C^{3a}C^4C^5$ 124.8(2), $C^4C^5C^6$ 117.0(3),

$C^5C^6C^7$ 116.3(2), $C^6C^7C^8$ 114.8(2), $C^7C^8C^{8a}$ 113.8(2), $N^1C^{8a}C^{3a}$ 104.8(2), $N^1C^{8a}C^8$ 112.7(2), $C^{3a}C^{8a}C^8$ 114.26(18).

X-Ray diffraction data for compound XXII. MoK_α irradiation, $\lambda = 0.71069$ Å, graphite monochromator. $C_{22}H_{22}$. M 286.41. Monoclinic crystal system $P12_1/n1$ (no. 14); unit cell parameters: a = 9.7840(14), b = 15.9160(19), c = 10.6322(17) Å; β = 102.63(2)°; V = 1615.64(65) Å^3; d_{calc} = 1.177 g/cm^3. Selected bond lengths (Å) and bond angles (deg): $C^7–C^9$ 1.424(4), $C^7–C^6$ 1.485(4), $C^7–C^1$ 1.493(4), $C^7–C^8$ 1.540(4), $C^6–C^1$ 1.510(4), $C^6–C^5$ 1.518(4), $C^5–C^4$ 1.480(5), $C^9–C^{91}$ 1.304(4), $C^9–C^8$ 1.495(3), $C^7–C^9$ 1.424(4); $C^9C^7C^6$ 135.8(2), $C^9C^7C^1$ 135.1(2), $C^6C^7C^1$ 60.95(19), $C^9C^7C^8$ 69.46(17), $C^6C^7C^8$ 143.0(2), $C^1C^7C^8$ 138.3(2), $C^7C^6C^1$ 59.80(18), $C^7C^6C^5$ 125.4(2), $C^1C^6C^5$ 120.0(3), $C^4C^5C^6$ 114.1(3), $C^7C^1C^2$ 121.8(2), $C^7C^1C^6$ 59.25(17), $C^{91}C^9C^7$ 151.7(3), $C^{91}C^9C^8$ 144.6(3), $C^7C^9C^8$ 63.61(18).

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