# SYNTHESIS OF UNSATURATED TERTIARY AMINES AND α-ALLYL SUBSTITUTED KETONES FROM AZOMETHINES USING METAL COMPLEX CATALYSTS

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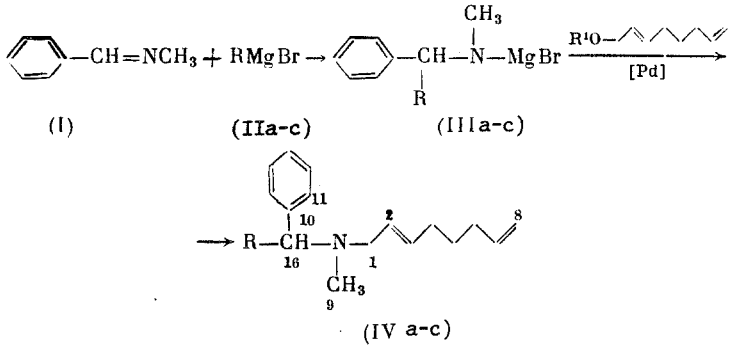
A method has been developed for the regioselective synthesis of unsaturated tertiary amines via the reaction of magnesium amides, derived from Schiff bases, with allylic electrophiles in the presence of Pd and Cu complexes. The reaction of ketimines which have been metallated using magnesium amide with functionalized allylic compounds is catalyzed by Pd complexes and leads to the formation of α-allyl substituted ketones with high regioselectivity.

Depending on the structure of the starting imine, the metallation of azomethines using Grignard reagents leads to either dialkylaminomagnesium halides [1, 2] or azaallyl anions [3, 4]; treatment of the latter with simple organic halides makes it possible to prepare α-substituted aldehydes and ketones [3, 4]. Dialkylaminomagnesium halides are used primarily for the preparation of secondary amines [5].

In continuation of our studies [6, 7] of the development of preparative methods for the synthesis of higher-order unsaturated amines and α-substituted ketones, we have investigated the transition metal complex-catalyzed reactions of metallated azomethines with functionalized allylic compounds. In these reactions, Pd and Cu phosphine complexes were used as the catalysts since they displayed the highest catalytic activity.

Reaction of benzalmethylamine (I) with ethyl- (IIa), n-propyl- (IIb), or n-butylmagnesium bromide (IIc) in ether solution results in the in situ [8] formation of the corresponding magnesium amides (IIIa-c). Subsequent reaction of (IIIa) with 1-methoxy-2,7-octadiene at 40°C for 5 h in the presence of 5 mole % $Pd(acac)_2$ + $2Ph_3P$ catalyst gives N-(2E, 7-octadienyl)-N-methyl-α-phenyl(n-propyl)amine (IVa) in ~95% yield. In the absence of catalyst (IVa) is not formed.



(I) (IIa-c) (IIIa-c) (IV a-c)

R = $C_2H_5$ (a); n-$C_3H_7$ (b); n-$C_4H_9$ (c); $R^1$ = $CH_3$, Ph, Ac.

The reaction of (IIIa) with 1-phenoxy- or 1-acetoxy-2,7-octadiene proceeds analogously [yield of (IVa) 90-96%]. As the size of the alkyl substituent in the alkylmagnesium halide (II) is increased, the yield of tertiary amines (IV) is reduced. In the case of the reactions of phenyl allyl or diallyl ether, allyl acetate, N-methyltriallylammonium iodide, diallyl sulfide, and phenyl allyl sulfone with the magnesium derivative (IIIa), the yield

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TABLE 1. Reaction of Magnesium Amide (IIIa) with Allylic Electrophiles in the Presence of $Pd(acac)_2$ (36-40°C in ether, 48-50°C in THF, 5 h)

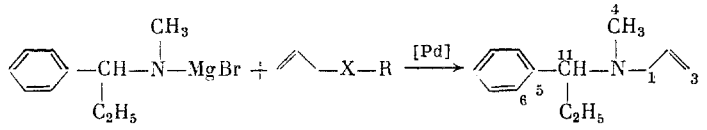
| No. | Allylic compound | Solvent | Yield, (V), % | No. | Allylic compound | Solvent | Yield (V), % |
|---|---|---|---|---|---|---|---|
| 1 | PhO— | THF | 94 | 4 | $(\quad)_3\overset{+}{N}(CH_3)\cdot\bar{I}$ | THF | 32 |
| 2 | —O— | THF | 90 | 5 | —S— | $Et_2O$ | 26 |
| 3 | —OAc | THF | 78 | 6 | $—SO_2Ph$ | $Et_2O$ | 18 |

TABLE 2. Reaction of Magnesium Amides with Organic Halides

| R | $R^1$ | $R^2$ | [M]* | Solvent | Reaction products | Product(s) yield, % |
|---|---|---|---|---|---|---|
| $CH_2=CHCH_2$ | $C_2H_5$ | $CH_2=CH—CH_2$ | [Cu] | THF | (VI) | 98 |
| $CH_3$ | $C_2H_5$ | $CH_3CH=CH—CH_2$ | [Cu] | THF | (VII) | 95 |
| Ph | $C_2H_5$ | $CH_2=CH—CH_2$ | [Cu] | THF | (VIII) | 68 |
| $CH_3$ | $n$-$C_3H_7$ | $CH_2=CH(CH_2)_3CH=CHCH_2$ | [Cu] | THF | (IV6) | 64 |
| $CH_3$ | $C_2H_5$ |  | [Pd] | THF | (IX) | 52 |
| $CH_3$ | $C_2H_5$ | $CH_2CO_2C_2H_5$ | [Cu] | THF | (X) | 75 |
| $CH_3$ | $C_2H_5$ | $(CH_2)_2OC_2H_5$ | [Pd] | $Et_2O$ | (XI) | 66 |
| $CH_3$ | $n$-$C_4H_9$ | $CH_2=CH(CH_2)_3CH=CHCH_2$ | [Cu] | THF | (IVb) | 54 |
| $CH_3$ | $C_2H_5$ | $CH_2—C\equiv CH$ | [Pd] | $Et_2O$ | (XII) | 70 |

*[Cu] = $Cu(acac)_2$ + $Ph_3P$, 25°C, 3 h; [Pd] = $Pd(acac)_2$ + $2Ph_3P$, 5 h, 36-40°C in ether, or 48-50°C in THF.

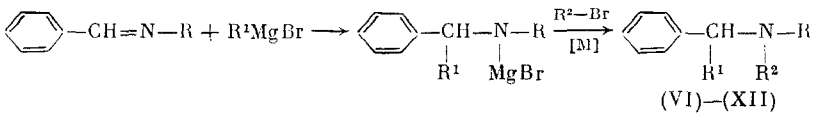
of N-allyl-N-methyl-α-phenyl(n-propyl)amine (V) depends on the nature of the leaving group in the allyl substrate (Table 1).



(IIIa) (V)

R—X = OPh, $OCH_2CH=CH_2$, OAc, $(CH_2=CH—CH_2)_2\overset{+}{N}(CH_3)\bar{I}$, $CH_2=CH—CH_2S$, $SO_2Ph$.

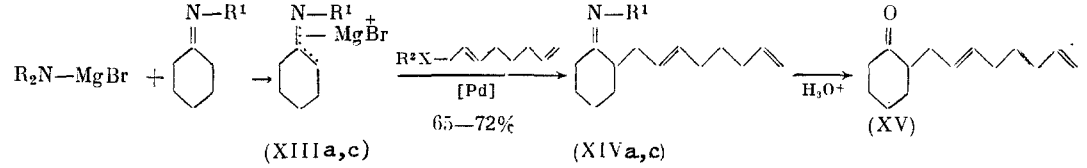
In order to extend the boundaries of this newly developed method we introduced into this reaction sequence other Schiff bases and organic halides of different structures, among them ones with functional substituents.



(VI)–(XII)

The results of these experiments are summarized in Table 2.

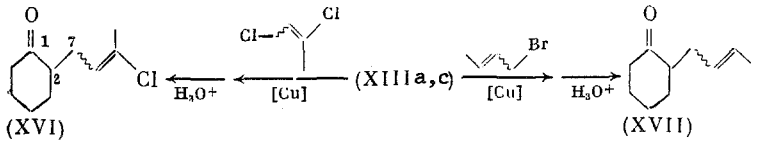
Metallation of cyclohexanone phenyl- or cyclohexylimine using diisopropyl or dicyclohexylaminomagnesium bromide (0°C, 1 h) gives magnesium 1-azaenolates (XIIIa, c), which surpass the corresponding alkali metal enolates [9] in terms of their reactivity with respect to palladium complex-catalyzed reactions with functionalized allylic electrophiles, leading to the highly regioselective formation of α-allyl substituted imines (XIV). Acidic workup of (XIV) gives (XV). Analogous results were obtained in experiments with 1-methoxy- and 1-acetoxy-2,7-octadiene (cf. [7]).



(XIIIa,c) (XIVa,c)

R = *i*-Pr, cyclo-$C_6H_{11}$; $R^2X$ = $PhSO_2$, $CH_3O$, AcO; $R^1$ = Ph (a); cyclo-$C_6H_{11}$ (c).

Crotyl bromide and 1,3-dichloro-2-butene react with (XIIIa, c) in the presence of CuI or CuCl catalyst to form α-allyl substituted cyclohexanones (XVI) and (XVII) in 65-70% yield.



(XVI) (XVII)

Based on these experimental results, we conclude that the reaction of magnesium amides or azaenolates with allylic electrophiles promises a regioselective method for the one-step synthesis of higher-order allyl amines of α-allyl substituted ketones.

## EXPERIMENTAL

2,7-Octadienyl esters were synthesized by the cotelomerization of butadiene with MeOH, PhOH, and AcOH [10, 11]. The reactions were carried out under an atmosphere of dry Ar. Ether and THF were distilled from $LiAlH_4$ prior to use. GLC analyses were performed on a Khrom-41 chromatograph in an He stream, with a 1.2 m × 3 mm column filled with 5% SE-30 on N-AW chromatone. IR spectra were recorded on a UR-20 spectrophotometer (for thin films), mass spectra were measured on an MX-1306 spectrometer at 70 eV and 130°C. PMR spectra were obtained using $CCl_4$ solutions on a Tesla BS-467 spectrometer at 60 MHz, $^{13}C$ NMR spectra on a Jeol FX-90Q spectrometer using $CDCl_3$ solutions versus TMS. Compounds (XV) and (XVII) were identified by comparison with known authentic samples [7, 12].

Reaction of Magnesium Amides with Allylic Substrates. To a solution of 1.19 g (10 mmoles) benzalmethylamine in 10 ml ether under Ar was added 8.5 ml of 1.2 M ethereal EtMgBr solution; the mixture was stirred for 2 h at 36-40°C, cooled to −5°C, and 0.15 g $Pd(acac)_2$ (0.5 mmole) and 0.262 g (1 mmole) $Ph_3P$ were added, along with 1.12 g (8 mmoles) 1-methoxy-2,7-octadiene. The mixture was stirred for 5 h at 36-40°C. At the completion of reaction 30 ml of saturated $NH_4Cl$ solution was added, and the mixture was extracted with ether (3 × 15 ml). The organic layer was dried over $MgSO_4$ and concentrated. Reactions of magnesium amides with organic halides (Table 2) were carried out in the presence of 3 mole % $Cu(acac)_2$ + $Ph_3P$ catalyst at room temperature for 3 h.

Reaction of Metallated Cyclohexanone Imines with Allylic Substrates. To a solution of 1.0 g (10 mmoles) i-$Pr_2NH$ in 3 ml ether under an Ar stream at 0°C was added 6.3 ml of 1.6 M ethereal EtMgBr solution; the mixture was then stirred for 1 h at 0°C, 1.73 g (10 mmoles) of cyclohexanone phenylimine was added, and the mixture was stirred again for 1 h

at 0°C, cooled to −5°C, and the catalyst, prepared by mixing 0.35 g (0.5 mmole) $Pd(acac)_2$ with 0.15 g (1 mmole) diisobutylaluminum hydride (DIBALH) in 2 ml toluene, was added, followed by 1.5 g (6 mmoles) phenyl 2,7-octadienyl sulfone. The mixture was stirred an additional 2 h at ~25°C, then worked up with 2 N HCl (2 h, 50°C), and extracted with ether. The reactions of metallated cyclohexanone imines with organic halides were carried out in the presence of monovalent copper salts CuI or CuCl at 0°C for 2 h. The reaction products were isolated by fractional distillation or column chromatography (silica gel L 40/100μ, ChSSR, hexane–ethyl acetate, 7:3). The purities of the isolated products (GLC) were ≥97%.

N-(2E,7-Octadienyl)-N-methyl-α-phenyl(n-propyl)amine (IVa). $R_f$ 0.66, $n_D^{20}$ 1.5114. IR spectrum (ν, $cm^{-1}$): 3085, 3030, 2980, 2940, 2790, 1645, 1605, 1500, 1460, 1360, 1000, 975, 915, 760, 705. PMR spectrum (δ, ppm): 0.70 t (3H, $CH_3$, J = 7 Hz), 1.13-1.75 m (4H, $CH_2$), 1.78-2.25 m (4H, $CH_2$), 2.05 s (3H, $CH_3$–N), 2.67-2.92 m (2H, $CH_2$–N), 3.06-3.38 m (1H, CH–Ph), 4.68-5.53 m (5H, olefinic), 7.17 s (5H, Ph). $^{13}C$ NMR spectrum (δ, ppm): 56.78 t ($C^1$), 133.24 d ($C^2$), 127.86 ($C^3$), 31.81 t ($C^4$), 28.56 t ($C^5$), 33.20 t ($C^6$), 138.66 d ($C^7$), 114.47 t ($C^8$), 38.27 q ($C^9$), 140.31 s ($C^{10}$), 127.86 d ($C^{11}$), 128.73 d ($C^{12}$), 126.78 d ($C^{13}$), 128.73 d ($C^{14}$), 127.86 d ($C^{15}$), 69.79 d ($C^{16}$), 25.66 t ($C^{17}$), 11.10 q ($C^{18}$). Mass spectrum, m/z 257 ($M^+$, 3.3%).

N-(2E,7-Octadienyl)-N-methyl-α-phenyl(n-butyl)amine (IVb). $R_f$ 0.70. IR spectrum (ν, $cm^{-1}$): 3080, 3040, 2970, 2950, 2800, 1650, 1610, 1500, 1460, 1020, 990, 930, 750, 720. PMR spectrum (δ, ppm): 0.81 t (3H, $CH_3$, J = 7 Hz), 1.13-1.70 m (6H, $CH_2$), 1.76-2.20 m (4H, $CH_2$), 2.13 s (3H, $CH_3$–N), 2.66-2.95 m (2H, $CH_2$–N), 3.13-3.46 m (1H, CH–Ph), 4.60-5.58 m (5H, olefinic), 7.13 s (5H, Ph). Mass spectrum, m/z 271 ($M^+$, 1.6%).

N-(2E-7-Octadienyl)-N-methyl-α-phenyl(n-pentyl)amine (IVc). $R_f$ 0.74. IR spectrum (ν, $cm^{-1}$): 3085, 3040, 2950, 2805, 1640, 1605, 1500, 1460, 1005, 980, 920. PMR spectrum (δ, ppm): 0.72 t (3H, $CH_3$, J = 7 Hz), 1.00-1.73 m (8H, $CH_2$), 1.83-2.35 m (4H, $CH_2$), 2.12 s (3H, $CH_3$–N), 2.67-3.00 m (2H, $CH_2$–N), 3.22-3.58 (1H, CH–Ph), 4.70-5.80 m (5H, olefinic), 7.23 s (5H, Ph). $M^+$ 285.

N-Allyl-N-methyl-α-phenyl(n-propyl)amine (V). Bp 55-56°C (9 mm Hg), $n_D^{20}$ 1.5056. IR spectrum (ν, $cm^{-1}$): 3085, 3030, 2970, 2940, 2790, 1640, 1600, 1495, 1460, 1360, 1000, 920, 770, 710. PMR spectrum (δ, ppm): 0.75 t (3H, $CH_3$, J = 7 Hz), 1.32-2.00 m (2H, $CH_2$), 2.13 s (3H, $CH_3$–N), 2.75-3.06 m (2H, $CH_2$–N), 3.06-3.60 m (1H, CH–Ph), 4.83-6.08 m (3H, olefinic), 7.18 s (5H, Ph). $^{13}C$ NMR spectrum (δ, ppm): 57.71 t ($C^1$), 136.38 d ($C^2$), 116.92 d ($C^3$), 38.36 q ($C^4$), 140.22 s ($C^5$), 127.89 ($C^6$), 128.71 d ($C^7$), 126.84 d ($C^8$), 128.71 d ($C^9$), 127.89 d ($C^{10}$), 69.85 d ($C^{11}$), 25.68 t ($C^{12}$), 11.05 q ($C^{13}$). Mass spectrum, m/z 189 ($M^+$, 3.15%).

N,N-Diallyl-α-phenyl(n-propyl)amine (VI). $R_f$ 0.72, $n_D^{20}$ 1.5155. IR spectrum (ν, $cm^{-1}$): 3070, 3050, 2970, 2935, 2880, 1655, 1600, 1480, 1455, 1365, 1130, 1005, 930, 770, 715. PMR spectrum (δ, ppm): 0.78 t (3H, $CH_3$, J = 7 Hz), 1.37-2.00 m (2H, $CH_2$), 2.77-3.83 m (5H, CH–Ph, $CH_2$–N), 4.72-6.08 m (6H, $CH_2$=CH), 7.23 s (5H, Ph). $^{13}C$ NMR spectrum (δ, ppm): 52.62 t ($C^1$, $C^4$), 136.92 d ($C^2$, $C^5$), 116.55 t ($C^3$, $C^6$), 140.26 s ($C^7$), 127.82 d ($C^8$), 128.64 d ($C^9$), 126.74 d ($C^{10}$), 128.64 d ($C^{11}$), 127.82 d ($C^{12}$), 65.32 d ($C^{13}$), 25.23 t ($C^{14}$), 11.31 q ($C^{14}$). Mass spectrum, m/z 215 ($M^+$, 1.9%).

N-(2-Butenyl)-N-methyl-α-phenyl(n-propyl)amine (VII), E/Z = 9:1. Bp 79-80°C (1 mm), $n_D^{20}$ 1.5161. IR spectrum (ν, $cm^{-1}$): 3080, 3020, 2960, 2930, 2870, 2780, 1600, 1450, 975, 770, 705. PMR spectrum (δ, ppm): 0.92 t (3H, $CH_3$), 2.08 s (3H, $CH_3$–N), 2.67-3.00 m (2H, $CH_2$–N), 3.10-3.40 m (1H, CH–Ph), 5.33-5.58 m (2H, CH=CH), 7.17 s (5H, Ph). $^{13}C$ NMR spectrum E-(VII) (δ, ppm): 56.82 t ($C^1$), 129.08 d ($C^2$), 127.65 d ($C^3$), 17.77 q ($C^4$), 38.23 q ($C^5$), 140.52 s ($C^6$), 127.86 d ($C^7$), 128.65 d ($C^8$), 126.78 d ($C^9$), 128.65 d ($C^{10}$), 127.86 d ($C^{11}$), 69.87 d ($C^{12}$), 25.83 t ($C^{13}$), 11.01 q ($C^{14}$). $^{13}C$ NMR spectrum Z-(VII) (δ, ppm): 50.97 t ($C^1$), 128.30 d ($C^2$), 126.07 d ($C^3$), 13.09 q ($C^4$), 38.23 q ($C^5$), 140.52 s ($C^6$), 127.86 d ($C^7$), 128.65 d ($C^8$), 126.78 d ($C^9$), 128.65 d ($C^{10}$), 127.86 d ($C^{11}$), 69.87 d ($C^{12}$), 25.83 t ($C^{13}$), 11.01 q ($C^{14}$). Mass spectrum, m/z 203 ($M^+$, 6.0%).

N-Allyl-N-phenyl-α-phenyl(n-propyl)amine (VIII). $R_f$ 0.70. IR spectrum (ν, $cm^{-1}$): 3080, 3060, 3020, 2960, 2870, 1600, 1000, 920, 750, 700. PMR spectrum (δ, ppm): 1.00 t (3H, $CH_3$, J = 7 Hz), 1.53-2.17 m (2H, $CH_2$), 3.62-3.83 m (2H, $CH_2$–N), 4.03-4.35 m (1H, CH–Ph), 4.78-5.67 m (3H, $CH_2$=CH), 6.42-7.42 m (10H, Ph). $^{13}C$ NMR spectrum (δ, ppm): 48.55 t ($C^1$), 136.23 d ($C^2$), 115.86 t ($C^3$), 149.36 s ($C^4$), 114.13 d ($C^5$, $C^9$), 129.03 d ($C^6$, $C^8$), 116.76 d ($C^7$), 141.43 s ($C^{10}$), 127.65 d ($C^{11}$, $C^{15}$), 128.25 d ($C^{12}$, $C^{14}$), 126.91 d ($C^{13}$), 63.50 d ($C^{16}$), 24.71 t ($C^{17}$), 11.88 q ($C^{18}$). Mass spectrum, m/z 251 ($M^+$, 7.4%).

N-(2-Pyridyl)-N-methyl-α-phenyl(n-propyl)amine (IX). $R_f$ 0.69, $n_D^{20}$ 1.5867. PMR spectrum (δ, ppm): 0.92 t (3H, $CH_3$, J = 7 Hz), 1.63-2.27 m (2H, $CH_2$), 2.63 s (3H, $CH_3$–N), 5.90-6.57 m, 8.00-8.20 m, 8.33-8.67 m (4H, pyridyl), 7.20 s (5H, Ph). $^{13}C$ NMR spectrum (δ, ppm): 159.40 s ($C^1$), 105.45 d ($C^2$), 137.36 d ($C^3$), 111.52 d ($C^4$), 147.63 d ($C^5$), 29.82 q ($C^6$), 141.60 s ($C^7$), 127.26 d ($C^8$), 128.21 d ($C^9$), 126.74 d ($C^{10}$), 128.21 d ($C^{11}$), 127.26 d ($C^{12}$), 57.95 ($C^{13}$), 24.01 t ($C^{14}$), 11.27 q ($C^{15}$). Mass spectrum, m/z 226 ($M^+$, 27.8%).

N-Methyl-N-[α-phenyl(n-propyl)]glycine Ethyl Ester (X). $R_f$ 0.70, $n_D^{20}$ 1.5037. IR spectrum (ν, $cm^{-1}$): 2960, 1725, 1600, 1640, 730, 705. PMR spectrum (δ, ppm): 0.86 t (3H, $CH_3$, J = 7 Hz), 1.20 t (3H, $CH_3$, J = 7 Hz), 1.42-2.17 m (2H, $CH_2$), 2.33 s (3H, $CH_3$), 3.17 d (2H, $CH_2$–N, J = 4 Hz), 3.42-3.75 m (1H, CH–Ph), 4.05 q (2H, $CH_2$, J = 7 Hz), 7.20 s (5H, Ph). Mass spectrum, m/z 235 ($M^+$, 7.0%).

N-(2-Ethoxyethyl)-N-methyl-α-phenyl(n-propyl)amine (XI). $R_f$ 0.67. IR spectrum (ν, $cm^{-1}$): 2980, 2880, 1505, 775, 730. PMR spectrum (δ, ppm): 0.77 t (3H, $CH_3$, J = 7 Hz), 1.17 t (3H, $CH_3$, J = 7 Hz), 1.25-2.13 m (2H, $CH_2$), 2.25 s (3H, $CH_3$), 2.58 q (2H, O–$CH_2$, J = 6 Hz), 3.17-3.67 m (1H, Ph–CH, 4H, N–$(CH_2)_2$–O), 7.25 s (5H, Ph). Mass spectrum, m/z 221 ($M^+$, 4.5%).

N-Propargyl-N-methyl-α-phenyl(n-propyl)amine (XII). $R_f$ 0.72, $n_D^{20}$ 1.5254. IR spectrum (ν, $cm^{-1}$): 3305, 3090, 3065, 3030, 2970, 2880, 2795, 1495, 1450, 760, 705. PMR spectrum (δ, ppm): 0.58 t (3H, $CH_3$, J = 7 Hz), 1.43-2.28 m (2H, $CH_2$), 2.17 s (3H, $CH_3$–N), 3.02-3.28 m (3H, $CH_2$–N, Ph–CH), 3.37 d (1H, ≡CH, J = 2 Hz), 7.13 s (5H, Ph). $^{13}C$ NMR spectrum (δ, ppm): 43.91 t ($C^1$), 78.93 ($C^2$), 72.99 d ($C^3$), 39.66 q ($C^4$), 141.35 s ($C^5$), 128.08 d ($C^6$, $C^{10}$), 128.38 d ($C^7$, $C^9$), 127.04 d ($C^8$), 68.61 d ($C^{11}$), 26.40 t ($C^{12}$), 10.62 q ($C^{13}$). Mass spectrum, m/z 187 ($M^+$, 1.6%).

2-($3^1$-Chloro-$2^1$-butenyl)-1-cyclohexanone (XVI, Z/E = 3:1). Bp 95-98°C (3 mm). IR spectrum (ν, $cm^{-1}$): 3030, 2950, 2870, 1710, 1670, 1140, 1080, 830. PMR spectrum (δ, ppm): 1.25-2.57 m (11H, CH, $CH_2$), 2.00 s (3H, $CH_3$), 5.38 t (1H, –CH=, J = 6.5 Hz). $^{13}C$ NMR spectrum E-(XVI) (δ, ppm): 211.23 s ($C^1$), 50.44 d ($C^2$), 33.73 t ($C^3$), 25.11 t ($C^4$), 27.98 t ($C^5$), 42.02 t ($C^6$), 28.76 t ($C^7$), 125.51 d ($C^8$), 129.98 ($C^9$), 20.87 q ($C^{10}$). $^{13}C$ NMR spectrum Z-(XVI) (δ, ppm): 211.73 s ($C^1$), 50.24 d ($C^2$), 33.73 t ($C^3$), 25.11 t ($C^4$), 27.98 t ($C^5$), 42.02 t ($C^6$), 28.96 t ($C^7$), 123.68 d ($C^8$), 131.18 s ($C^9$), 26.22 q ($C^{10}$). $M^+$ 186.

2-($2^1$-Butenyl)-1-cyclohexanone (XVII, Z/E = 1:4). Bp 71-72°C (3 mm), $n_D^{20}$ 1.4688. IR spectrum (ν, $cm^{-1}$): 2930, 2860, 1705, 1430, 1130, 975, 690. PMR spectrum (δ, ppm): 1.33-2.50 m (11H, CH, $CH_2$), 2.11 d (3H, $CH_3$, J = 3.5 Hz), 5.13-5.42 m (2H, CH=CH). $^{13}C$ NMR spectrum E-(XVII) (δ, ppm): 211.67 s ($C^1$), 50.85 d ($C^2$), 33.42 t ($C^3$), 25.00 t ($C^4$), 28.07 t ($C^5$), 42.10 t ($C^6$), 32.64 t ($C^7$), 128.86 d ($C^8$), 126.70 d ($C^9$), 17.95 q ($C^{10}$). $^{13}C$ NMR spectrum Z-(XVII) (δ, ppm): 212.67 s ($C^1$), 50.85 d ($C^2$), 33.42 t ($C^3$), 25.13 t ($C^4$), 28.07 t ($C^5$), 42.10 t ($C^6$), 26.83 ($C^7$), 128.14 d ($C^8$), 125.33 d ($C^9$), 12.86 q ($C^{10}$). $M^+$ 152.

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