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ORGANIC SYNTHESIS AND INDUSTRIAL ORGANIC CHEMISTRY

Alkenylation of Anilines with Dicyclopentadiene, Cyclopentadiene, and Piperylene

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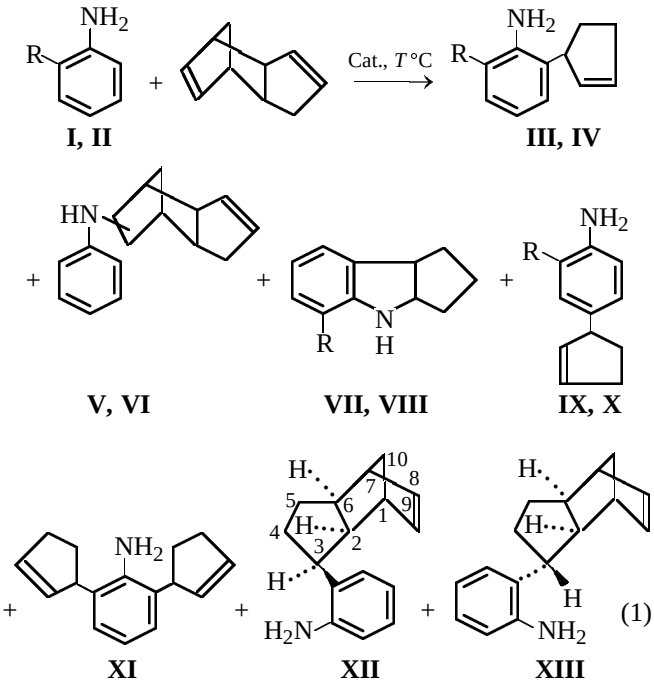
Abstract—Alkenylation of anilines with dicyclopentadiene, cyclopentadiene, and piperylene in the presence of mineral acids and Lewis acids was studied.

The discovery of the Claisen rearrangement of aromatic amines [1] provided access to 2-alkenyl-substituted arylamines, including cyclopentenyl- and pentenylanilines [2], which can be prepared from dicyclopentadiene (DCPD) or piperylene. A number of valuable compounds have been prepared from these amines, such as alkaloids [3], steel corrosion inhibitors, fungicides, plant growth stimulants, antiphytophthora agents, and local anesthetics [4–6]. The presence in allylaniline molecules of several reaction centers allows their transformations involving nitrogen atom, alkenyl fragment, or both centers simultaneously. The possibility of preparing new biologically active derivatives from 2-alkenylanilines by common reactions of amines or by certain specific reactions makes these compounds valuable synthons. To look for an alternative to the Claisen rearrangement of aromatic amines as a route to *o*-alkenylanilines, we studied alkenylation of anilines with DCPD, cyclopentadiene (CPD), and piperylene.

Reactions of DCPD with amines **I** and **II** in the presence of HCl at 130–220°C gave as main products the previously described [2] *o*-cyclopentenylanilines **III** and **IV** (yield of up to 45%) and also *N*-phenyl-(8*S*)- (**V**) and *N*-phenyl-(8*R*)-tricyclo[5.2.1.$0^{2,6}$]dec-3-en-8-amine (**VI**). Elevated temperatures (200°C) and longer reaction time favor cyclization of *o*-cyclopentenylanilines **III** and **IV** into hexahydrocyclopent[*b*]-indoles **VI** and **VIII** [7] and formation of *p*-cyclopentenylanilines **IX** and **X** [8] (Table 1). In the case of aniline (**I**), *o*,*o*-dicyclopentenylaniline (**XI**) [9] was also detected (<3%). When freshly distilled CPD is taken in reaction with aniline hydrochloride, the yield of a mixture of exo isomers **V** and **VI** decreases somewhat.



I, II **III, IV** **V, VI** **VII, VIII** **IX, X** **XI** **XII** **XIII** (1)

Here Cat. = HCl, $AlCl_3$, $FeCl_3$; R = H (**I**, **III**, **V**–**VII**, **IX**, **XI**); Me (**II**, **IV**, **VIII**, **X**).

With $AlCl_3$ used instead of HCl, the composition of the reaction products changes. The ratio of amines **I**, **IV** and **IX**, **X** is 1 : 1. The expected products **V** and

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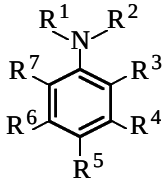
Table 1. Ratio of products of aniline (**I**) alkenylation with DCPD under various conditions (molar ratio aniline : DCPD 4 : 1)

Catalyst, mol	τ, h	*T*, °C	Product content, %					Total yield, %
			III	**VII**	**IX**	**V**, **VI**	**XII**, **XIII**	
HCl, 0.4	0.5	200	50	–	–	20	–	70
	2	150	45	–	–	15	–	60
	0.5	200	42	3	–	15	–	60
	5	200	29	14	2	14	–	57
$AlCl_3$:								
0.37	0.5	200	10	2	10	15	28	65
1.05	0.5	200	–	–	–	9	43	52
0.15	0.5	200	14	2	15	16	17	64
$FeCl_3 \cdot 6H_2O$, 0.15	0.5	200	20	–	21	14	13	68

Table 2. Substituted anilines in reaction with piperylene in the presence of KU-2 resin (reaction time 5 h, 180°C, molar ratio amine : piperylene 1 : 3)



Initial amine	Substituent							Yield of products,* %			Total yield, %
	R^1	R^2	R^3	R^4	R^5	R^6	R^7	2-AA	4-AA	2,4-diAA	
II	H	H	Me	H	H	H	H	13	28	24	65
XIV	H	H	H	Me	H	H	H	21	25	18	64
XV	H	H	H	H	Me	H	H	38	–	26	64
XVI	H	H	Me	H	Me	H	H	58	–	–	58
XVII	H	H	Me	H	H	H	Et	–	61	–	61
XVIII	H	H	Me	H	H	Me	H	–	52	–	52
XIX	H	H	OMe	H	OMe	H	H	–	38	–	38
XX	H	H	Cl	H	H	H	H	26	22	15	63
XXI	Bu	Bu	H	H	H	H	H	–	19	–	19

* (AA) Alkenylated aniline.

VI are formed, as well as the products of alkenylation of the aromatic ring, 2-[(3*S*)- (**XII**) and 2-[(3*R*)-tricyclo-[5.2.1.$0^{2,6}$]dec-8-en-3-yl]amines (**XIII**). With increasing amount of $AlCl_3$, the relative content of **III** and **IX** decreases. At a 1 : 1 ratio of DCPD and $AlCl_3$, only amines **XII** and **XIII** are formed. With the $FeCl_3 \cdot 6H_2O$ catalyst taken in the same amount as $AlCl_3$, the relative content of **III** and **IX** is somewhat higher than that of **V**, **VI**, **XII**, and **XIII**. In the case of *o*-toluidine (**II**) and $AlCl_3$ catalyst, the composition of products, according to GLC, is the same as with that in the case of aniline (**I**).

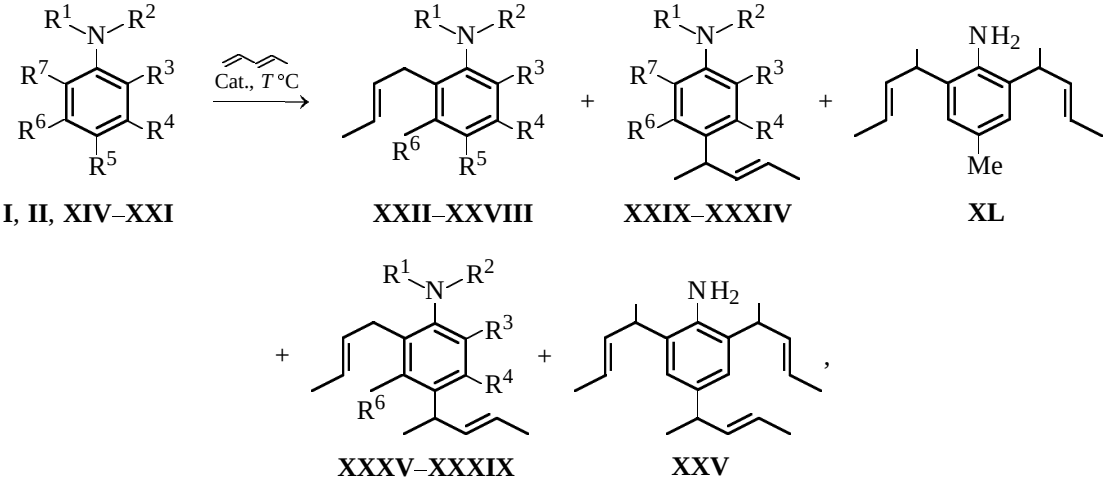
The structure of **V**–**XIII** was confirmed by spectroscopic methods [10] and elemental analysis. The IR spectra [11] of **V** and **VI** contain a characteristic band of the NH group at 3430 cm^{-1}, and the spectra of **XII** and **XIII**, the bands of the NH_2 group at 3390 and 3470 cm^{-1}. The aromatic protons in the ^1H NMR spectrum of **IX** appear as two doublets (2H each) at 6.12 and 7.00 ppm. The methine 1'-H signal is a multiplet (1H) at 3.85 ppm. The broadened singlet of the NH group is observed at 3.50 ppm. Four protons of two CH_2 groups give rise to multiplets at 1.60 (1H) and 2.40 ppm (3H).

In *N*-substituted anilines, the C^2 and C^6 atoms, as well as the C^3 and C^5 atoms, are mutually equivalent. Therefore, the corresponding region of the ^{13}C NMR spectrum contains only four aromatic carbon signals and two signals of the olefin carbon atoms of the tricyclodecene moiety.

The ^{13}C NMR spectrum of *o*-substituted anilines **XII** and **XIII** contains in the region of aromatic carbon signals two sets of signals (eight signals each). Twelve of these 16 signals were assigned to the carbon atoms of the phenyl ring using the pulse train of a *J*-modulated spin echo.

In the case of *p*-substitution, the ^{13}C NMR spectrum would be simpler owing to the symmetrical structure of the aromatic moiety. Exo addition of the *N*-phenyl substituent to DCPD in **V** and **VI** is confirmed by the upfield shift (to 39.6 and 39.4 ppm) of the triplet of the bridging C^{10} atom. The formation of isomeric products **V** and **VI** is confirmed by characteristic doublets at 52.95 (56.42) ppm in the ^{13}C NMR spectrum and by single-proton doublets of doublets of *exo* protons at 3.34 (3.33) ppm (3 : 1 ratio) in the ^1H NMR spectrum. The ^{13}C NMR spectrum of **V** and **VI** contains only one signal of the C^3 cyclopentene carbon atom (in **V**; in **VI** it is C^5), whereas the spectrum of the mixture of diastereomers **XII** and **XIII** contains in the range 26.6–33.4 ppm six signals: 29.6, 32.6, and 36.6 ppm for *anti* diastereomer **XIII** and 32.2, 33.4, and 33.5 ppm for *syn* diastereomer **XII**. The *o*-substitution of the aromatic ring is confirmed by calculations using additive parameters.

The results of alkenylation of anilines **I**, **II**, and **XIV**–**XXI** with piperylene in the presence of KU-2 are listed in Table 2. The reaction is selective and, in the case of 2,4-xylidine (**XVI**) and 2-methyl-6-ethylaniline (**XVII**), yields a single product. This is due to the availability in these amines of only one position capable of alkenylation:



I, **II**, **XIV**–**XXI** **XXII**–**XXVIII** **XXIX**–**XXXIV** **XL**

XXXV–**XXXIX** **XXV** (2)

where $R^1 = R^2 = R^3 = R^4 = R^5 = R^6 = R^7 = H$ (**I**, **XXII**, **XXIX**, **XXXV**); $R^1 = R^2 = R^4 = R^5 = R^6 = R^7 = H$, $R^3 = Me$ (**II**, **XXII**, **XXX**, **XXXV**); $R^1 = R^2 = R^3 = R^5 = R^6 = R^7 = H$, $R^4 = Me$ (**XIV**, **XXIV**, **XXXI**; $R^1 = R^2 = R^3 = R^4 = R^6 = R^7 = H$, $R^5 = Me$ (**XV**, **XXV**); $R^1 = R^2 = R^4 = R^6 = R^7 = H$, $R^3 = R^5 = Me$ (**XVI**, **XXVI**); $R^1 = R^2 = R^4 = R^5 = R^6 = H$, $R^3 = Me$, $R^7 = Et$ (**XVII**, **XXXII**); $R^1 = R^2 = R^4 = R^5 = R^7 = H$, $R^3 = R^6 = Me$ (**XVIII**, **XXXIII**); $R^1 = R^2 = R^4 = R^6 = R^7 = H$, $R^3 = R^5 = OMe$ (**XIX**, **XXVII**); $R^1 = R^2 = R^4 = R^5 = R^6 = R^7 = H$, $R^3 = Cl$ (**XX**, **XXVIII**, **XXXIV**, **XXXVII**); $R^1 = R^2 = Bu$, $R^4 = R^6 = R^7 = R^3 = R^5 = H$ (**XXI**, **XXXIX**).

In the case of 2,5-xylidine (**XVIII**), and also with *N*,*N*-dibutylaniline (**XXI**), the formation of a single product is probably due to the steric and electronic effects of substituents. Alkylation of aniline (**I**) with piperylene in the presence of supported H_3PO_4 gives a large set of products in low total yield (Table 3). With $AlCl_3$ used as catalyst for alkenylation of piperylene, the best result was achieved in synthesis of 2,4-tri(1-methylbut-2-en-1-yl)aniline. In other cases, it is impossible to selectively obtain a single product although the total yield is sometimes high, (Table 4).

The physicochemical characteristics of compounds alkenylated with piperylene were reported in [12–14].

Table 3. Alkenylation of aniline (**I**) with piperylene in the presence of supported H_3PO_4 (reaction time 5 h, 150°C)

Catalyst	Aniline : piperylene molar ratio	Yield of products, %			Total yield, %
		2-AA	4-AA	2,4-diAA	
10% H_3PO_4 on silica gel	1 : 1	6.5	4.5	–	11
	1 : 2	12	6	–	18
	1 : 3	9	8	–	17
10% H_3PO_4 on kieselguhr	1 : 1	11.5	10.5	–	22
	1 : 2	14	12	Traces	26
	1 : 3	22	20	2	44
18% H_3PO_4 on silica gel	1 : 1	12	9	–	21
	1 : 2	18	21	3	42
	1 : 3	34	29	6	69
SF-300, polyphosphoric acid	1 : 1	4.5	4	–	8.5
	1 : 2	19	16	–	35
	1 : 3	30	14	6	50
10% H_3PO_4 on silica gel*	1 : 1	7	5	–	12
	1 : 2	9	8	5	22
	1 : 3	14	8	7	29

* τ = 5 h, T = 200°C.

Table 4. Alkenylation of aniline with piperylene in the presence of $AlCl_3$

T, °C	τ, h	Solvent	Aniline : piperylene molar ratio	Yield, %				Total yield, %
				2-AA	4-AA	2,4-diAA	2,4,6-triAA	
80	5	Toluene	1 : 1	3	24	4	–	34
80	4	″	1 : 1	2	15	2	–	21
80	3	″	1 : 1	1	10	1	–	13
100	5	″	1 : 1	6	20	5	–	32
100	5	″	1 : 3	2	22	17	2	43
130	5	″	1 : 1	2	13	5	–	19
130	5	″	1 : 3	4	16	5	3	28
100	5	Benzene	1 : 1	5	10	5	4	24
130	5	″	1 : 1	24	27	14	2	67
130	5	″	1 : 3	9	7	36	34	86
130	5	″	2 : 1	9	19	4	–	32
130	5	″	1 : 5	–	–	1	98	99
150	5	″	1 : 1	2	18	6	3	29
100	5	Hexane	1 : 1	12	33	8	2	55
130	5	″	1 : 1	21	45	16	2	84
130	5	″	1 : 3	19	25	44	5	93
150	5	″	1 : 1	15	18	21	2	56

EXPERIMENTAL

The IR spectra were taken on a UR-20 spectrometer, and the ^1H and ^{13}C NMR spectra, on a Bruker AM 300 spectrometer (working frequencies 300 and 75 MHz, respectively). Internal reference TMS, solvent $CDCl_3$. Elemental analysis was performed with an M-185B C–H–N analyzer, and the GLC analysis (including purity check), on a Chrom-5 chromatograph (carrier gas helium, flame-ionization detector, 1200 × 3-mm column, stationary phase SE-30, 5% on Chromaton N-AW DMCS, working temperature 50–300°C). Column chromatography was performed on silica gel LS 40/100 μm and Silpearl (eluent hexane). Qualitative TLC analysis was made with Silufol UV 254 and UV 254/366 plates; the chromatograms were developed under UV (λ = 254 nm) or with iodine vapor.

The alkenylation of anilines with DCPD, CPD, and piperylene was performed in a sealed heat-resistant

ampule or a 17-ml metallic autoclave, charged with the reactants and catalyst. The component ratios, reaction times and temperatures, and product yields are given in Tables 1–4. After the reaction was complete, the ampule or autoclave was cooled and opened, and the reaction mixture was transferred with chloroform into a separatory funnel. The mixture was treated with 5% KOH (2 × 100 ml), extracted with chloroform (2 × 50 ml), and dried over crystalline KOH. Then the mixture was filtered, the solvent was evaporated, and the residue was fractionated in a vacuum. With HCl catalyst, the reaction performed at 140–180°C gave after vacuum distillation monoalkenylated amine **III** or **IV**. Using the same reaction at 200°C with aniline **I** or **II**, we isolated, after vacuum distillation, amine **III** or **IV** with an admixture of indoline **VII** or **VIII** and *p*-substituted product **IX** or **X**. Indolines and *p*-substituted products were identified by GLC upon addition of authentic samples [3, 8]. The reaction with $AlCl_3$ catalyst gave, after vacuum distillation, a mixture of **III** + **IX** or **IV** + **X**, chromatographed on silica gel (eluent benzene). The products were identified by comparison of their ^1H and ^{13}C NMR spectra with those of authentic samples [2, 8, 9]. The fraction containing anilines **V** and **VI**, prepared by alkenylation of **I** in the presence of HCl, was analyzed as a mixture. The fraction containing N- (**V**, **VI**) and C-substituted products (**X**, **XI**), obtained by the reaction with $AlCl_3$ catalyst, was separated by chromatography on silica gel (eluent hexane). The **V** + **VI** and **X** + **XI** pairs were obtained as several fractions with varied component ratios. The physicochemical characteristics of 2-(cyclopent-2-enyl)anilines **II** and **IV** [2] indolines **VII** and **VIII** [7], and 4-(cyclopent-2-enyl)anilines **IX** and **X** [8, 9] were consistent with the relevant published data. Alkenylated anilines **XXII**–**XL** were isolated by column chromatography (alumina, eluent hexane), and their physicochemical characteristics were compared with those of authentic samples [12–14].

Mixture of anilines V and VI. IR spectrum, ν, cm^{-1}: 3430 (NH). ^1H NMR spectrum, δ, ppm (*J*, Hz): 1.12–2.75 m (10H, $3CH_2$, 4CH), 3.33 d.d (1H, J_1 3.57, J_2 7.60, NCH), 3.34 d.d (1H, J_1 3.53, J_2 7.69, NCH), 3.62 br.s (1H, NH), 5.49–5.80 m (2H, CH=CH), 6.66 d (2H, 2-H and 6-H, *J* 8.25), 6.70 1 (1H, 4-H), 7.20 t (2H, 3-H and 5-H).

Found, %: C 6.00, H 6.87, N 5.23.

$C_{16}H_{19}N$.

Calculated, %: C 6.27, H 7.45, N 5.49.

^{13}C NMR spectrum of *N*-phenyl-(8*S*)-tricyclo-[5.2.1.$0^{2,6}$]dec-4-en-8-amine (**V**), δ_C, ppm: 29.2 (C^3), 39.4 (C^{10}), 40.6 (C^9), 43.2 (C^2), 48.1 (C^8), 55.1 (C^7), 55.9 (C^1), 56.42 (C^6), 113.00 ($C^{2',6'}$), 129.30 ($C^{3',5'}$), 116.80 ($C^{4'}$), 131.70 (C^5), 132.30 (C^4), 147.70 ($C^{1'}$).

^{13}C NMR spectrum of *N*-phenyl-(8R)-tricyclo-[5.2.1.$0^{2,6}$]dec-4-en-8-amine (**VI**), δ_C, ppm: 29.2 (C^5), 36.6 (C^{10}), 40.5 (C^9), 42.3 (C^6), 45.5 (C^8), 52.9 (C^2), 55.8 (C^7), 55.9 (C^1), 113.2 ($C^{2',6'}$), 116.90 ($C^{4'}$), 129.30 ($C^{3',5'}$), 131.60 (C^3), 132 (C^4), 147.60 ($C^{1'}$).

2-Methyl-4-(cyclopent-2-enyl)aniline (X). R_f 0.42 (hexane–MeOH, 99 : 1). IR spectrum, ν, cm^{-1}): 1295, 3390, 3460 (NH_2). ^1H NMR spectrum, δ, ppm (*J*, Hz): 1.50–2.50 m (4H, $2CH_2$), 2.32 s (3H, CH_3), 3.62 br.s (2H, NH_2), 3.95 (1H, CH), 5.90–6.05 m (2H, CH=CH), 6.74 d (1H, *J* 7.69, 6-H), 7.04 d (1H, 5-H), 7.10 s (1H, 3-H). ^{13}C NMR spectrum, δ, ppm: 50.56 ($C^{1'}$), 17.40 (CH_3), 131.20 ($C^{2'}$), 135.02 ($C^{3'}$), 32.48 ($C^{4'}$), 34.01 ($C^{5'}$), 142.85 (C^1), 132.36 (C^2), 129.21 (C^3), 136.62 (C^4), 125.57 (C^5), 115.06 (C^6).

Found, %: C 83.09, H 8.51, N 7.77.

$C_{12}H_{15}N$.

Calculated, %: C 83.24, H 8.67, N 8.09.

2,6-Di(cyclopent-2-enyl)aniline (XI). Yield 3%, bp 168–170°C (2 mm Hg). The physicochemical characteristics agree with published data [2].

Mixture of anilines XII and XIII. IR spectrum, ν, cm^{-1}: 3390, 3470 (NH_2). ^1H NMR spectrum, δ, ppm (*J*, Hz): 1.20–2.80 m (10H, $3CH_2$, 4CH); 3.10, 3.23 (unresolved multiplets, Ar–CH); 3.60 br.s (2H, NH_2); 5.60–5.85 m (2H, CH=CH); 6.70 d (1H, *J* 7.80, 6'-H); 6.76 t (1H, 5'-H, *J* 7.80); 7.03 t (1H, 4'-H); 7.18 d (1H, 3'-H, *J* 7.70).

Found, %: C 6.12, H 6.79, N 5.00.

$C_{16}H_{19}N$.

Calculated, %: C 6.27, H 7.45, N 5.49.

^{13}C NMR spectrum of 2-[(3*S*)-tricyclo-[5.2.1.$0^{2,6}$]-dec-8-en-3-yl]aniline (**XII**), δ_C, ppm: 29.6 (C^5), 32.6 (C^4), 36.6 (C^3), 39.1 (C^{10}), 40.3 (C^7), 42.1 (C^1), 44.2 (C^2), 53.6 (C^6), 115.4 ($C^{6'}$), 118.3 ($C^{4'}$), 125.6 ($C^{5'}$),

126.2 ($C^{3'}$), 130.7 (C^{9}), 131.8 ($C^{2'}$), 132.1 (C^{8}), 144.1 ($C^{1'}$).

^{13}C NMR spectrum of 2-[(3*R*)-tricyclo-[5.2.1.$0^{2,6}$]-dec-8-en-3-yl]aniline (**XIII**), δ_C, ppm: 32.2 (C^{5}), 33.4 (C^{3}), 33.5 (C^{4}), 39.3 (C^{10}), 41.4 (C^{7}), 42.6 (C^{1}), 43.2 (C^{2}), 52.5 (C^{6}), 115.5 ($C^{6'}$), 118.1 ($C^{4'}$), 125.3 ($C^{5'}$), 126.2 ($C^{3'}$), 131.3 ($C^{2'}$), 132.1 (C^{8}), 132.8 (C^{9}), 144.2 ($C^{1'}$).

CONCLUSION

Alkenylation of anilines with dicyclopentadiene or cyclopentadiene in the presence of HCl yields *o*-cyclopentenylanilines as the main products. At higher temperatures (200°C) and longer reaction times, perhydrocyclopent[b]indolines are formed. With $AlCl_3$ catalyst, both *o*- and *p*-cyclopentenyl derivatives are obtained. Also, significant amounts of dicyclopentadiene substitution products are formed in both cases. Alkenylation of anilines with piperylene gives 2-, 4-, 2,4-di-, and 2,4,6-trialkenylated products. If the 4- or 2,4-positions of the aromatic ring are substituted, the alkenyl group is introduced at the free *o*- or *p*-position, respectively.

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