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Approach to Preparative Synthesis of *ortho*-(1-Methylbut-2-en-1-yl)anilines, Precursors of New Cytotoxic Heterocycles

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Abstract—The reaction of aromatic Claisen rearrangement of *N*-(1-methylbut-2-en-1-yl)anilines in the presence of *p*-toluenesulfonic acid was investigated. *N*-Tosyl-2-(1-iodoethyl)-3-methylindoline derivatives were obtained; one of them exhibited a cytotoxic activity.

Keywords: halocyclization, indoles, alkenylanilines, cytotoxic activity, acid-catalyzed rearrangement

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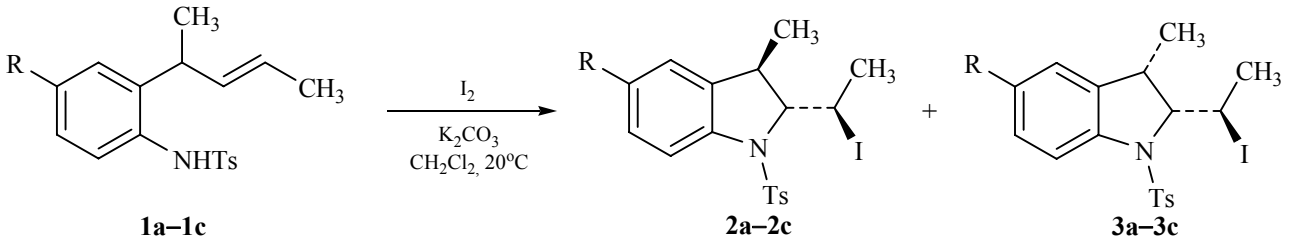
Some indole derivatives have been known to possess high biological activity [1–5]. Benzopyrrole derivatives may be modified by introduction of substituents into different positions of the heterocycle that allows investigation of the structure–activity relationship in several regioisomers. Therefore the synthesis of new specimens of the series and investigation of their biological activity attracted much attention of researchers [6–10]. Among the numerous approaches toward the synthesis of indole heterocycles [11–13] the methods based on electrophilic intermolecular cyclization of *ortho*-alkenylanilines [14] are of particular interest due to the possibility of preparation of a broad spectrum of substituted benzopyrroles.

In the present work we report on the results of the search for preparative method of the synthesis of 2-(1-methylbut-2-en-1-yl)anilines; the target compounds were the precursors of indoline derivatives (Scheme 1). Biochemical investigations of the products of halocyclization of tosylated pentenyl aniline **1a**, namely (2*R**,3*S**)-isomer of **3a**, revealed a cytotoxic activity towards HEK293 line (IC_{50} 10.72 μM), HepG2 (IC_{50} 40.83 μM), and Jurkat cells (IC_{50} 14.96 μM). In this regard, synthesis and investigation of cytotoxic activity of such indole derivatives is a promising direction of the search for new biologically important compounds.

The absence of a preparative synthesis of *ortho*-pentenylaniline **4a** (tosylate **1a** precursor) was a drawback of this approach. The compound might be prepared by direct alkenylation of aniline with 1,3-pentadiene in the presence of Lewis acids [15] or acid-catalyzed aromatic Claisen amino rearrangement of *N*-

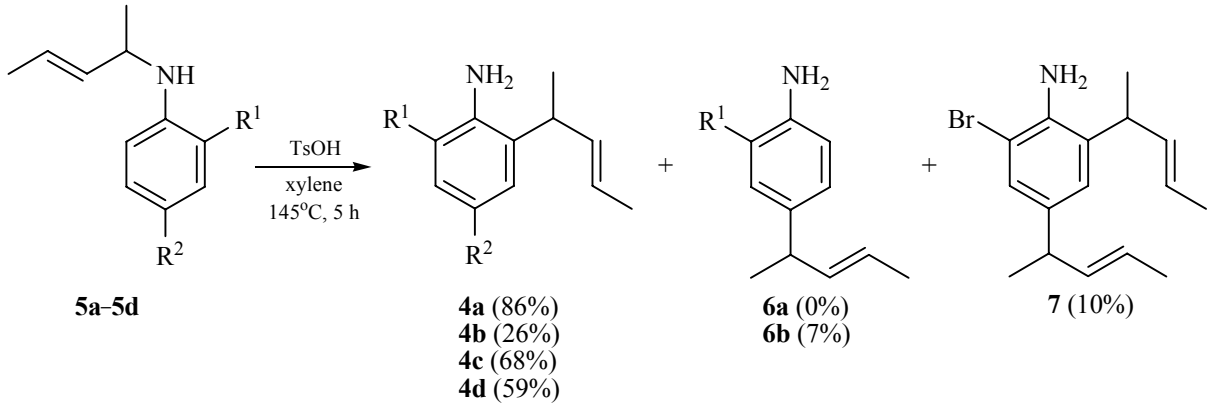
Scheme 1.



1a–1c **2a–2c** **3a–3c**

R = H (**a**), CH_3 (**b**), Cl (**c**).

Scheme 2.



5a–**5d**

4a (86%)
4b (26%)
4c (68%)
4d (59%)

6a (0%)
6b (7%)

7 (10%)

$R^1 = R^2 = H$ (**a**); $R^1 = Br$, $R^2 = H$ (**b**); $R^1 = H$, $R^2 = CH_3$ (**c**); $R^2 = Cl$ (**d**).

pentenylaniline **5a** in the presence of HCl [16] or 0.2 N H_2SO_4 [17]. Selectivity of compound **4a** formation in the acid-catalyzed rearrangements was higher [16, 17] than in the case of direct alkenylation of aniline with piperylene [15]. The catalyzed rearrangement of compound **5a** in the presence of HCl or H_2SO_4 proceeded at a temperature about 145°C and led to the formation of two isomeric products of rearrangement, **4a** and **6a**, in overall yield about 82% in an approximate ratio of 6 : 1 (Scheme 2). Isolation of product **4a** from the reaction mixture was done by prolonged vacuum distillation using highly effective rectification column or by the column chromatography on aluminum oxide that of course reduced the preparative value of the reaction.

To eliminate this drawback we used as acid an equivalent amount of *para*-toluenesulfonic acid. The reaction proceeded at reflux of the mixture of the reagents in xylene and resulted in the formation of *ortho*-isomer **4a** in a high yield.

In the case of amine **5a** the reaction proceeded regioselectively. In contrast, *ortho*-bromosubstituted analog **5b** (prepared by the reaction of bromoaniline with 3-chloropentene in Et_3N) afforded *ortho*-**4b** and *para*-**6b** isomers in a ratio 3.4 : 1 (Scheme 2). Moreover, 2-bromoaniline and dipentenylated product **7** were detected in the reaction mixture (**4b** : **7** = 3.4 : 1.9).

When using *para*-toluidine **5c** [18], the reaction led to the formation of *ortho*-isomer **4c** [16] (Scheme 2) as well as to a significant amount of *p*-toluidine generated due to decomposition of *N*-alkenyl tolylamine **5c**.

It has been known that in some cases chlorine atom in *para*-position of indole heterocycle favored a significant increase in the biological activity of the compound [19]. Aiming at further preparation of 5-chlorosubstituted indolines **2c** and **3c**, we synthesized compound **4d**. Unlike alkenyl arylamine **5c**, the rearrangement of amine **5d** in the presence of HCl or *p*-TsOH did not proceed regioselectively. Along with the formation of the target *ortho*-pentenylaniline **4d** the side-products of unidentified structure were formed. Nevertheless, the acid-catalyzed rearrangement of alkenyl arylamine followed by purification of the reaction mixture by column chromatography on silica gel proved to be the only approach to the synthesis of compound **4d**.

Tosylation of {4-chloro-2-[(2*E*)-1-methylbut-2-en-1-yl]phenyl}amine **4d** in pyridine led to the formation of sulfonyl amide **1c**. The reaction of compounds **1a**–**1c** with molecular iodine allowed the preparation of (2*R**,3*R**)-indolines **2a** [20], **2b** [21], **2c**, and (2*R**,3*S**)-indolines **3a** [19], **3b** [20], and **3c** (Scheme 1). It should be noted that in the course of the reaction the change in the ratio of the iodocyclization products was observed depending on the nature of the substituent in the *para*-position of the starting alkenylaniline. It was found that in the case of $R = CH_3$ the ratio of isomers **2b** : **3b** was ≈ 4 : 1 [21]. The absence of the substituent (R = H) led to reducing the ratio (**2a** : **3a** ≈ 2 : 1) [20]. Chlorine atom (R = Cl) possessing a negative inductive (–*I*) and positive mesomeric (+*M*) effects also promoted the change in the isomers ratio (**2c** : **3c** ≈ 2 : 3). Most probably it might be due to a significant effect of variably activated aromatic ring on the primary formation of two iodonium complexes.

In ^{1}H NMR spectra of isomers **2** and **3** a distinct differentiation of the proton signals at the key carbon

atoms of the heterocyclic fragment of the molecule was observed. Basing on the earlier obtained spectral characteristics of analogs **2a, 2b** and **3a, 3b** [20, 21], we attributed the signals of protons H^2, H^3, $H^{1'}$ and methyl groups in the spectra of compounds **2c** and **3c**. The data occurred to be slightly different as compared with the earlier prepared compounds.

The results of investigation of biological activity of newly prepared heterocycles **2c** and **3c** based on halo-substituted *ortho*-pentenylanilines will be reported elsewhere.

In summary, the rearrangement of *N*-(1-methylbut-2-en-1-yl)aniline catalyzed with *p*-TsOH proceeded regioselectively with the formation of 2-(1-methylbut-2-en-1-yl)aniline in a high yield while in the case of *N*-(1-methylbut-2-en-1-yl)-2-bromo- or -4-chloroanilines no regioselectivity was observed.

EXPERIMENTAL

IR spectra were recorded on a IRPresstige-21 spectrometer (Shimadzu). ^1H and ^{13}C NMR ($CDCl_3$) spectra were registered on a Bruker AM 300 instrument operating at 300.13 and 75.73 MHz, internal reference TMS. The product purity was monitored by GLC on a Chrom-5 chromatograph; carrier gas helium (50 mL/min), flame ionization detector, column 1200 × 3 mm, stationary phase liquid silicon SE-30 (5%) on a carrier Chromaton N-AW DMCS, working temperature 50–300°C. Elemental analysis was carried out on a CHNS Elemental Analyzer EURO EA-3000 instrument. Halogen content was determined by burning the sample by Schoeniger method followed with potentiometric titration. Column chromatography was performed on MN Kisielgel 60 silica gel (40–100 μM). TLC was done on Sorbfil plates (Sorbpolimer, Krasnodar) with iodine vapors as a spots developer.

***N*-{4-Chloro-2-[(2*E*)-1-methylbut-2-en-1-yl]phenyl}-4-methylbenzenesulfonamide (1c).** A mixture of compound **4d** (1 g, 5.11 mmol) and TsCl (1.1 g, 5.77 mmol) in 5 mL of anhydrous pyridine was stirred at 20°C for 17 h. Then it was diluted with 30 mL of water, stirred for 20 min, and evaporated in a vacuum. Methylene chloride (100 mL) and water (50 mL) were added to the residue. The organic layer was washed with HCl solution (50 mL, 5%) and water (20 mL), dried over $MgSO_4$, and concentrated in a vacuum. The residue (1.724 g) was chromatographed eluting with C_6H_6. Yield 1.47 g (85%). IR spectrum, ν, cm^{-1}: 3276 (N–H). ^1H NMR spectrum, δ, ppm: 1.08 d (3H, CH_3, J = 7.0 Hz), 1.65 d.d (3H, CH_3, J = 4.8, 0.9 Hz), 2.39 s (3H, CH_3), 3.05 q (1H, $H^{1'}$, J = 7.0 Hz), 5.23–5.37 m (2H, HC=CH), 6.61 s (1H, NH), 7.08 d (1H, H^3, J = 2.4 Hz), 7.14 d.d (1H, H^5, J = 2.4, 8.4 Hz), 7.23 d (2H, $H^{3'',5''}$, J = 8.4 Hz), 7.35 d (1H, H^6, J = 8.4 Hz), 7.58 d (2H, $H^{2'',6''}$, J = 8.4 Hz).

(2*R,3*R**)-5-Chloro-2-[(1*R**)-1-iodoethyl]-3-methyl-1-[(4-methylphenyl)silfonyl]indoline (2c) and (2*R**,3*S**)-5-chloro-2-[(1*R**)-1-iodoethyl]-3-methyl-1-[(4-methylphenyl)sulfonyl]indoline (3c).** A mixture of compound **1c** (1.12 g, 3 mmol as a solution in 15 mL of methylene chloride), iodine (0.9 g, 3.6 mmol), and K_2CO_3 (0.33 g) was stirred for 3 h at room temperature. The reaction progress was monitored by TLC eluting with benzene. After the reaction completion methylene chloride (25 mL), water (10 mL), and 5% solution of $Na_2S_2O_3$ (15 mL) were added. The organic layer was washed with water (10 mL), and dried over $MgSO_4$. After removing the solvent in a vacuum the residue was crystallized from ethanol (15 mL). **Compound 2c**. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.64 d (3H, CH_3, J = 7.2 Hz), 2.01 d (3H, CH_3, J = 7.0 Hz), 2.37 s (3H, CH_3), 3.07 d.q (1H, H^3, J = 3.5, 7.2 Hz), 3.34 d.d (1H, H^2, J = 3.0, 5.5 Hz), 4.48 d.q (1H, $H^{1'}$, J = 5.5, 7.2 Hz), 6.99 d.d ($1H_{Ar}$, J = 1.6, 8.0 Hz), 7.22 d.d ($1H_{Ar}$, J = 1.6, 8.0 Hz), 7.23 d.d ($2H_{Ar}$, J = 1.6, 8.0 Hz), 7.54 d ($2H_{Ar}$, J = 8.5 Hz), 7.62 d ($1H_{Ar}$, J = 8.5 Hz). **Compound 3c.** ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.35 d (3H, CH_3, J = 7.4 Hz), 1.78 d (3H, CH_3, J = 7.2 Hz), 2.37 s (3H, CH_3), 2.76 quintet (1H, H^3, J = 7.3 Hz), 4.34 d.q (1H, $H^{1'}$, J = 4.4, 7.2 Hz), 4.55 d.d (1H, H^2, J = 4.4, 8.4 Hz), 6.89 t ($1H_{Ar}$, J = 1.6 Hz), 7.17 d ($1H_{Ar}$, J = 7.9 Hz), 7.22 d.d ($1H_{Ar}$, J = 1.6, 8.4 Hz), 7.50 d ($2H_{Ar}$, J = 8.4 Hz), 7.55 d ($1H_{Ar}$, J = 8.4 Hz).

{2-[(2*E*)-1-Methylbut-2-en-1-yl]phenyl}amine (4a). A mixture of amine **5a** (19 g, 0.118 mol), xylene (70 mL), and *p*-TsOH (20.0 g, 0.118 mol) was stirred at 145°C for 7 h. After cooling NaOH (7 g, 0.175 mol) in water (70 mL) was added to the reaction mixture, the mixture was thoroughly stirred and extracted with *tert*-butyl methyl ether (200 mL). The organic layer was dried over KOH and concentrated in a vacuum. To separate aniline the residue was distilled at a temperature of heating oil bath of 100°C then the bath temperature was increased up to 145°C and amine **4a** was distilled off. Yield 16.5 g (86%), bp 102–104°C (2 mmHg). Physicochemical characteristics were the same as described earlier [17].

(2-Bromophenyl)-[(2*E*)-1-methylbut-2-en-1-yl] amine (5b). A mixture of 2-bromoaniline hydrobromide (49 g, 0.19 mol) with equivalent amount of NaOH in 100 mL of water was stirred for 30 min. The formed dark brown 2-bromoaniline was separated and dissolved in 70 mL of Et_3N. 2-Chloro-3-pentene (21 g, 0.2 mol) was added to the solution. The reaction mixture was heated at 90°C for 3 h on a water bath. An excess of Et_3N was distilled off at a reduced pressure. A solution of NaOH (20 g, 0.5 mol) in 100 mL of water was added; the mixture was stirred and then extracted with *tert*-butyl methyl ether (200 mL). The organic layer was dried over KOH, filtered, and concentrated at a reduced pressure. The residue was distilled in a vacuum. Yield 30 g (65%), bp 111°C (2 mmHg). IR spectrum, ν, cm^{-1}: 3409 (N–H), 669 (C–Br). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.34 d (3H, CH_3, J = 6.6 Hz), 1.69 d (3H, CH_3, J = 6.4 Hz), 3.91–4.01 m (1H, $H^{1'}$), 4.27 d (1H, NH, J = 5.0 Hz), 5.38–5.49 m and 5.58–5.72 m (2H, $H^{2',3'}$), 6.54 d.t (1H, H_{Ar}, J = 1.3, 7.5 Hz), 6.64 d.d (1H, H_{Ar}, J = 1.3, 8.2 Hz), 7.14 d.t (1H, H_{Ar}, J = 1.3, 8.0 Hz), 7.41 d.d (1H, H_{Ar}, J = 1.4, 7.9 Hz).

***N*-(1-Methylbut-2-en-1-yl)-4-chloroaniline (5d)** was prepared similarly from 4-chloroaniline (6.3 g, 49 mol) and chloropentene (7.35 g). Yield 8.05 g (84%), colorless liquid, bp 123–124°C (2 mmHg).

Rearrangement of alkenylaniline 5b. A mixture of amine **5b** (30 g, 0.125 mol), xylene (70 mL), and *p*-TsOH (21.5 g, 0.125 mol) was stirred at heating (140°C) on oil bath for 12 h. After cooling to room temperature a solution of NaOH (7 g, 0.175 mol) in 70 mL of H_2O was added, the mixture was thoroughly stirred, and then extracted with *tert*-butyl methyl ether (200 mL). The organic layer was separated, dried over KOH, and concentrated at a reduced pressure. The residue was distilled in a vacuum to get 2-bromoaniline (6 g, 28%), bp 60°C (2 mmHg), about 2 g of a mixture of 2-bromoaniline, amine **4b**, and *para*-isomer of compound **6b** in a ratio of 1 : 25 : 9, and approximately 10 g of a mixture of compounds **4b** and **6b**. The still bottoms contained compound **7**. Purification of 10 g of the mixture of compounds **4b** and **6b** by chromatography on a column filled with 280 g of silica gel provided amines **4b** (7.8 g, 26%, eluent – petroleum ether) and **6b** (2 g, 7%, eluent – benzene). Compound **7** (3.8 g, 10%) was isolated by chromatographic purification of the still bottoms on silica gel (eluent benzene).

{2-Bromo-6-[(2*E*)-1-methylbut-2-en-1-yl]phenyl}-amine (4b). Yield 7.8 g (26%). IR spectrum, ν, cm^{-1}: 3455, 3378 (NH_2), 678 (C–Br). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.37 d (3H, CH_3, J = 7.0 Hz), 1.69 t.d (3H, CH_3, J = 1.0, 3.9 Hz), 3.38–3.47 m (1H, $H^{1'}$), 4.25 br.s (2H, NH_2), 5.51–5.57 m (2H, $H^{2',3'}$), 6.64 t (1H, H^4, J = 7.9 Hz), 7.06 d.d (1H, H_{Ar}, J = 1.1, 7.5 Hz), 7.33 d.d (1H, H_{Ar}, J = 1.4, 7.9 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 17.83 and 19.44 (CH_3), 38.17 ($C^{1'}$); 110.73, 131.02, 142.01 ($C^{1,2,6}$); 119.06, 125.14, 126.01, 130.36, 134.40 ($C^{3–5,2',3'}$). Mass spectrum, m/z (I_{rel}, %): 239.0 (30) $[M]^+$, 224.0 (15), 210.0 (20), 145.1 (100), 130.1 (50).

{2-Bromo-4-[(2*E*)-1-methylbut-2-en-1-yl]phenyl}-amine (6b). Yield 2.0 g (7%). IR spectrum, ν, cm^{-1}: 3471, 3380 (NH_2), 678 (C–Br). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.27 d (3H, CH_3, J = 7.0 Hz), 1.67 t.d (3H, CH_3, J = 1.1, 6.1 Hz), 3.28 m (1H, $H^{1'}$), 3.96 br.s (2H, NH_2), 5.37–5.58 m (2H, $H^{2',3'}$), 6.70 d (1H, H^6, J = 8.2 Hz), 6.95 d.d (1H, H^5, J = 2.0, 8.2 Hz), 7.24 d (1H, H^3, J = 2.0 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 17.92 and 21.45 (CH_3), 41.17 ($C^{1'}$); 109.41, 137.97, 142.01 ($C^{1,2,6}$); 133.17, 123.70, 127.21, 130.99, 133.17 ($C^{3–5,2',3'}$). Mass spectrum, m/z (I_{rel}, %): 239.03 (20) $[M]^+$, 224.0 (12), 145.1 (100), 130.1 (30).

{2-Bromo-4,6-bis[(2*E*)-1-methylbut-2-en-1-yl]-phenyl}amine (7). Yield 3.8 g (10%). IR spectrum, ν, cm^{-1}: 3445, 3375 (NH_2), 678 (C–Br). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.28 d (3H, CH_3, J = 7.0 Hz), 1.37 d (3H, CH_3, J = 7.0 Hz), 1.68 d (3H, CH_3, J = 5.9 Hz), 1.70 d (3H, CH_3, J = 6.0 Hz), 3.24–3.44 m (2H, $H^{1',1''}$), 4.11 br.s (2H, NH_2), 5.38–5.60 m (4H, $H^{2',3',2'',3''}$), 6.88 d (1H, H^3, J = 1.8 Hz), 7.16 d (1H, H^5, J = 1.8 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 17.80, 19.35, 17.80, 21.51 (CH_3); 38.41, 41.43 ($C^{1',1''}$); 110.82, 130.83, 137.34, 139.88 ($C^{1,2,4,6}$); 123.46, 124.93, 125.11, 128.53, 134.49, 136.23 ($C^{3,5,2',3',2'',3''}$). Mass spectrum, m/z (I_{rel}, %): 307.1 (40) $[M]^+$, 224.0 (36), 144.1 (30), 69.1 (100).

{4-Methyl-2-[(2*E*)-1-methylbut-2-en-1-yl]phenyl}-amine (4c) was prepared similarly from amine **5c** (3.5 g, 20 mmol) and *p*-TsOH (3.4 g, 20 mmol). Yield 2.4 g (68%). Physicochemical characteristics were the same as described in [16].

{4-Chloro-2-[(2*E*)-1-methylbut-2-en-1-yl]phenyl}-amine (4d). *a*. Gaseous HCl was bubbled through a solution of compound **5d** (4.89 g, 25 mmol) in 20 mL of xylene for 30 min. Then the reaction mixture was heated under reflux for 5 h, cooled to room tem-

perature, and treated with a solution of NaOH (4 g, 100 mmol) in 50 mL of water. The product of the reaction was extracted with *tert*-butyl methyl ether. The organic layer was dried over $MgSO_4$ and concentrated in a vacuum. The residue was purified by chromatography eluting with petroleum ether. Yield 2.9 g (59%), bp 121–126°C (2 mmHg). IR spectrum, ν, cm^{-1}: 3416, 3374 (NH_2), 490 (C–Cl). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.35 d (3H, CH_3, J = 7.0 Hz), 1.69 d (3H, CH_3, J = 4.0 Hz), 3.30–3.40 m (1H, $H^{1'}$), 3.69 br.s (2H, NH_2), 5.49–5.52 m (2H, $H^{2',3'}$), 6.59 d (1H, H^6, J = 8.4 Hz), 6.99 d.d (1H, H^5, J = 2.4, 8.4 Hz), 7.06 d (1H, H^3, J = 2.4 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 17.77 and 19.24 (CH_3), 37.00 ($C^{1'}$); 117.02, 124.93, 126.58, 126.73, 134.10 ($C^{3,5,6,2',3'}$); 123.37, 131.37, 142.78 ($C^{1,2,4}$).

b. The reaction of amine **5d** (3 g, 15.3 mmol) with TsOH (2.64 g, 15.3 mmol) was performed according to the method for preparation of compound **4a**. Yield 1.7 g (57%).

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