

To the memory of A.A. Petrov

Carbazoles Stannylation

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Abstract—Stannylation of 3,6-dibromocarbazoles with lithium trimethylstannanide in THF was investigated. At treating 3,6-dibromo-9-alkylcarbazoles with lithium trimethylstannanide arise 3,6-bis(trimethylstannyl)-9-alkylcarbazoles. The 3,6-dibromocarbazole unsubstituted at nitrogen affords lithium 3,6-bis(trimethylstannyl)-carbazol-9-yl. The conditions of its generation, alkylation, and conversion into 3,6-bis(trimethylstannyl)carbazole are considered.

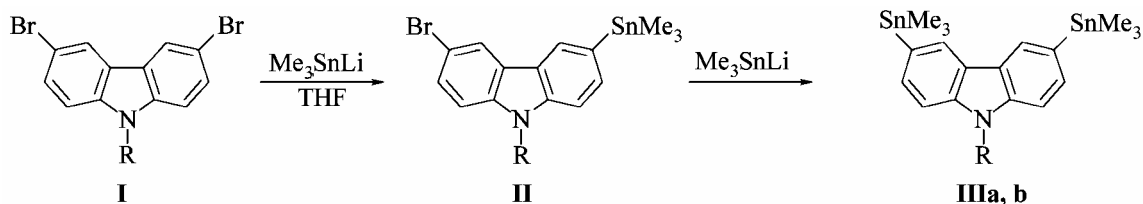
Organotin compounds are widely used in the organic synthesis, especially in the cross-coupling processes [1, 2]. However although the procedures for preparation these compounds are numerous, the stannylation of aromatic compounds is still poorly understood, especially regarding aryl derivatives with such electron-donor substituents as groups containing oxygen and nitrogen [3, 4]. Carbazoles may be also considered among such compounds, and reports on their stannylation are scanty [5].

This study was aimed at investigation of 3,6-dibromocarbazoles stannylation. We showed before that the best way of aminobenzenes stannylation was a reaction of alkali metal trialkylstannanide with halogenated anilines [6]. We selected as objects of the study 9-H and 9-alkyl-substituted 3,6-dibromocarbazoles, and as stannyating agent was chosen lithium trimethylstannanide.

The stability and reactivity of stannanides is considerably affected by solvents. The best solvent for preparation of stable stannanides is liquid ammonia. In this solvent sodium and lithium stannanides are sufficiently easily obtained from virtually equimolar amount of the alkali metal with respect to the trialkylstannyl rest. However it was demonstrated in

[4] by an example of *p*-haloanisoles that under common conditions the stanides in liquid ammonia did not stannylate the haloaryls possessing electron-donor substituents because of reduction processes occurring in this case. Therefore we chose as solvent ethers, namely, THF, diglyme, and tetraglyme. In a short communication [5] reporting on the synthesis of bis(trialkylstannyl) monomers as a substrate for stannylation with sodium stannanide a 9-benzyl-3,6-dibromocarbazole was mentioned, but no data on obtaining the corresponding stannylated carbazole nor on characteristics thereof were given (also in the later publications of Moore *et al.* [7, 8].

Our attempts to reproduce the procedure from [5] for preparation of 9-methyl-3,6-(trimethylstannyl)-carbazole were unsuccessful, although this product was detected by TLC. However its yield was low, and isolation from tetraglyme solution was laborious, and we failed to isolate the product in a pure state. No advantages provided the replacement of diglyme for tetraglyme. We established that lithium trimethylstannanide stannylated the N-alkylated 3,6-dibromocarbazoles in THF solution.



R = Me (a), Et (b).

The reaction takes place at room temperature within several hours. It does not stop at the monostannylation step but for complete conversion of the intermediate monostannylated derivative a considerable excess of lithium stannanide is required (more than 1.5-fold excess calculated on each bromine atom). The unreacted lithium stannanide was deactivated with methyl iodide. The yield of reaction products depended on content in the solution of lithium trimethylstannanide of hexamethylditin and polyalkylpolystannanes which catalyzed disproportionation of the lithium trimethylstannanide.

For further application most promising are the stannylated carbazoles with no substituents at the nitrogen atom. Kuivila and Wursthorn [9] showed that the reaction of sodium trimethylstannanide with *p*-bromoaniline in tetraglyme furnished *p*-trimethylstannylaniline in 40% yield. The formation of this product was ascribed to the nucleophilic attack of the stannanide on the halogen (S_N2) followed by reaction between arising aryl anion and trimethyltin bromide in the solvent cage where the reacting species were held by viscosity of solvent [9, 10].

It is known that in solutions of trialkylstannanides exists an equilibrium $R_3SnLi \rightleftharpoons R_2Sn + RLi$. Blake *et al.* [11] found that Bu_3SnLi sometimes reacted so as if it preliminary suffered a dissociation: $Bu_3SnLi \rightarrow Bu_2Sn + BuLi$. It was shown in [12] that a partial dissociation $R_3SnLi \rightarrow [(RLi)(R_2Sn)]$ occurred at the initial stage in the solvent cage, and alkyllithium reacted with an appropriate reagent. It was shown in particular that in the presence of R_3SnSnR_3 occurred fast decomposition of the stannanide due to reaction of hexaalkylditin both with alkyllithium giving tetraalkyltin and stannanide and with dialkyltin R_2Sn yielding R_3SnSnR_2Li . In the case of 3,6-dibromo-carbazole the presence of alkyllithium in the stannanide can result in lithiation of both N-H and C-Br bonds providing the corresponding N- and C-lithium derivatives of carbazole that on hydrolysis would afford products of dibromocarbazole reduction to monobromo-carbazole or carbazole.

It was reported [13] that in decomposition products of stannanides was found metallic lithium. Apparently alongside the stannanide dissociation into dialkyltin and alkyllithium another type of dissociation is probable: $2R_3SnLi \rightleftharpoons R_3SnSnR_3 + 2Li$. This was indirectly confirmed by the quantitative determination of active lithium in the stannanide solution in THF

(~120%); the excess was ascribed to the presence of free lithium [13]. It might exist in the solution in colloidal form. The dissociation with liberation of lithium should be favored by lithium chloride formation. The latter with the metallic lithium furnishes complexes of black color well observed in such reactions. These processes may be the reason of lower yield of reaction products with stannanides obtained from chlorides as compared with reaction products prepared with stannanides obtained from hexaalkylditin. This also may explain why even at a large excess of lithium commonly used in stannanides preparation in THF it is not possible to get rid of residual hexaalkylditin (TLC data). The presence of colloidal or finely divided lithium should prevent stannanide dissociation.

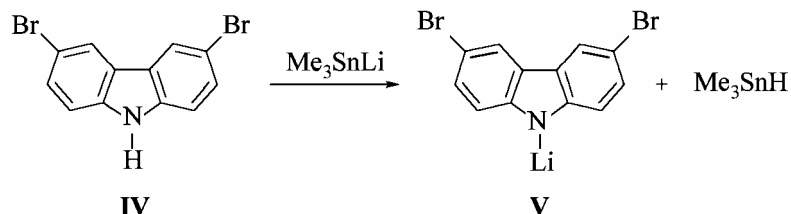
Thus several concurrent processes may occur during reaction between stannanide and 3,6-dibromocarbazole: stannanide attack on the C-Br bond of dibromocarbazole and attack of alkyllithium, if it formed, on C-Br and N-H bonds. The prevalence of one among these directions depends on many factors where the most important is the stability of stannanide under the reaction conditions.

At operations with stannanides in ether solvents the metallic lithium is always filtered off before experiments. We studied the reaction between 3,6-dibromo-carbazole with lithium trimethylstannanide in THF monitoring the reaction by TLC. Before applying the test sample to the plates for TLC it was treated with methyl iodide for conversion of active lithium compounds into methyl derivatives. The reaction was performed at the ratio Me_3SnLi -3,6-dibromocarbazole 3.5:1. A stannanide solution of 0.1 M concentration was slowly added to a vigorously stirred 0.15 M solution of dibromocarbazole in THF at room temperature. The TLC analysis showed that at adding stannanide to a solution of dibromocarbazole in THF first of all lithiation occurs at nitrogen yielding lithium 3,6-di-bromocarbazol-9-ide. The reaction is relatively fast and notably exothermic. No gas evolution means that the lithiation apparently occurs as acid-base exchange in keeping with the acid properties of 3,6-dibromocarbazole (pK_a in acetone 15.20) [14] and Me_3SnH (pK_a in DME 23.5) [15].

The arising trimethyltin hydride reacts with lithium trimethylstannanide affording hexamethylditin and apparently lithium hydride as shows the

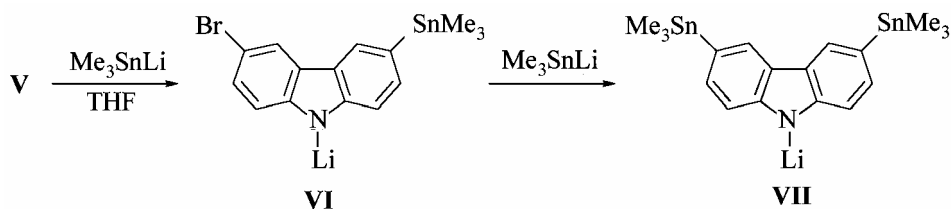
gas evolution at quenching the reaction mixture with water. The TLC analysis demonstrated that at this

stage of the reaction the amount of hexamethylditin in the reaction mixture increased.



Further addition of lithium trimethylstannanide to the generated lithium 3,6-dibromo-carbazol-9-ide results in successive replacement of bromine by stannyl groups. The reaction proceeds at room temperature and completes within 2 h. At the use of 3.5 equiv. of lithium per 1 equiv. of dibromocarbazole in the reaction mixture

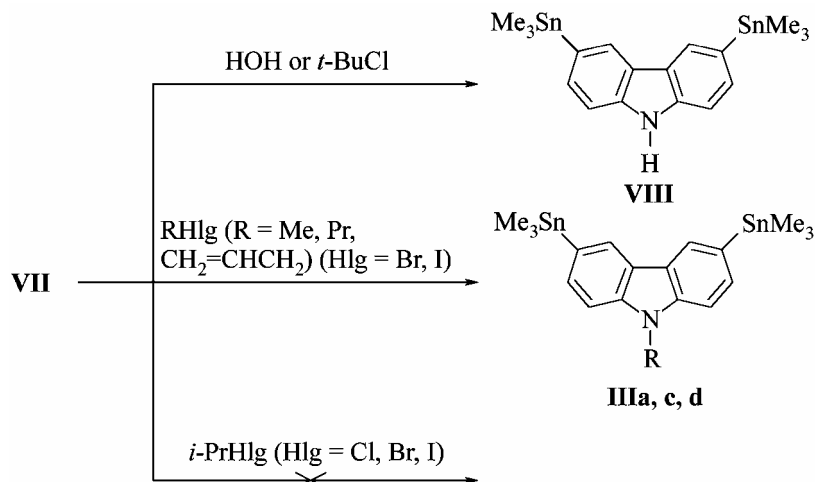
remain unreacted bromocarbazoles. However if metallic lithium is added to the reaction mixture the hexamethylditin formed in the course of reaction is converted into the lithium tri-methylstannanide, and the stannylation of bromocarbazoles is fulfilled even at the above indicated reagents ratio.



R = Me (a), Pr (c), CH₂=CHCH₂ (d).

The lithium 3,6-bis(trimethylstannyl)carbazol-9-ide is hydrolyzed by water to 3,6-bis(trimethylstannyl)carbazole. The latter forms also be treating the lithium 3,6-bis(trimethylstannyl)carbazol-9-ide with *tert*-butyl chloride. Primary haloalkanes as MeHlg,

PrHlg, CH₂=CH-CH₂Hlg (Hlg = Br, I) readily replace the lithium atom at nitrogen by the corresponding alkyl group. On the contrary isopropyl chloride, bromide, and iodide do not enter into this reaction.



It should be noted that in no case among the products of reaction between 3,6-dibromocarbazoles and lithium trimethylstannanide were found compounds originating from bromine reduction in the carbazole system. It means first of all that under conditions of our reactions no process of the type $\text{Me}_3\text{SnLi} \rightarrow \text{Me}_2\text{Sn} + \text{MeLi}$ occurred with lithium trimethylstannanide. Besides the readiness and completeness of dibromocarbazoles stannylation is not consistent with any of the previously suggested mechanisms of halogens substitution effected by stannanides.

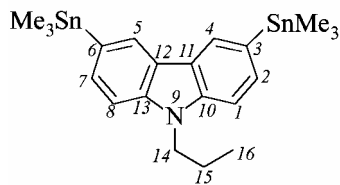
Taking into account the above described and also the fact that metallic lithium in contrast to alkyl lithium is fairly inert both toward dihalocarbazoles [16] and unsubstituted carbazole [17] we further used a procedure of addition of dibromocarbazole to lithium trimethylstannanide containing excess lithium. To the end of the process the reaction mixture was separated from lithium and treated with alkyl halide. The results obtained were similar to previously discussed.

The method of preparation of lithium 3,6-bis(trimethylstannyl)carbazol-9-ide we developed is very promising for preparation of versatile carbazole derivatives including monomers both for polymerization and polycondensation.

The structures of stannylated carbazoles were proved by ^1H and ^{13}C NMR spectroscopy, and their composition by elemental analysis.

EXPERIMENTAL

^1H and ^{13}C NMR spectra of compounds with the following general structure



were registered on spectrometer Bruker AM-200 at operating frequencies 200.132 (^1H) and 50.323 (^{13}C) MHz and Bruker AC-500 at operating frequencies 500.13 (^1H), 125.77 (^{13}C) MHz. All experiments were carried out under argon atom-sphere in glassware preliminary heated in a vacuum and cooled in an argon stream. Common devices for treating air-sensitive compounds were used.

3,6-Bis(trimethylstannyl)carbazole (VIII). Into a reaction flask just before experiment 30 ml of THF was distilled from LiAlH_4 and to check the absence of moisture there was put several milligrams of metallic sodium. No gas evolution was observed. Then into the flask was charged 1 g (0.005 mol) of trimethyltin chloride. Therewith the metallic sodium totally disappeared. The reaction mixture was cooled to -10°C , and thereto was charged within 30 min by small portions 0.14 g (0.02 mol) of metal lithium in the form of thin strips. As a result of vigorous stirring for 2 h the reaction mixture turned green-gray. TLC test showed a presence of some hexamethylditin that we failed to remove completely by prolonging the reaction. In 2 h after the start of reaction the cooling was removed, and when the reaction mixture warmed to room temperature within 10 min was added dropwise a solution of 0.65 g (0.002 mol) of 3,6-dibromocarbazole in 5 ml of THF. Slight self-heating was observed. The reaction mixture was stirred for 30 min more at room temperature and then 1.5 h at 66°C . After cooling the reaction mixture to room temperature the solution was decanted from unreacted lithium into the flask for distillation. The unreacted lithium was washed with several portions of anhydrous THF, and the washings were added to the reaction mixture in the distillation flask. Thereto was then added wet ethyl ether. The solvents were distilled off first at atmospheric, then at reduced pressure. The residue from the still was extracted with hot hexane or dichloromethane, and then with ether. The dry colorless residue of inorganic salts weighed 0.500 g (90% with respect to LiCl and LiBr). According to TLC data the main quantity of organic products contained the hexane extract, and it was a mixture of hexamethylditin and the target product. Hexane was distilled off, and from the residue at $80-90^\circ\text{C}$ (10 mm Hg) was removed the main part of hexamethylditin. The remaining 0.585 g of substance was recrystallized from petroleum ether (bp $40-70^\circ\text{C}$). We obtained 0.5 g (51%) of 3,6-bis(trimethylstannyl)carbazole. ^1H NMR spectrum (CDCl_3), δ , ppm: 0.42 s [18H, $(\text{CH}_3)_3\text{Sn}$, $^2J(^{117/119}\text{Sn}-^1\text{H})$ 54 Hz], 7.38 d [2H, C^4H , $^4J(^1\text{H}-^{117/119}\text{Sn})$ 165 Hz], 7.54 d [2H, C^2H , $^3J(^1\text{H}-^{117/119}\text{Sn})$ 43 Hz], 7.85 s [1H, NH], 8.28 s [2H, C^4H , $^3J(^1\text{H}-^{117/119}\text{Sn})$ 48 Hz]. ^{13}C NMR spectrum, δ , ppm.: 6.64 q [C^{17} , $^1J(^{13}\text{C}-^1\text{H})$ 3 Hz], $^1J(^{13}\text{C}_{sp^3}-^{117/119}\text{Sn})$ 332.84 Hz, $^1J(^{13}\text{C}_{sp^3}^{117/119}\text{Sn})$ 347.62 Hz], 110.55 s [$\text{C}^{1(8)}$, $^3J(^{13}\text{C}_{sp^2}-^{117/119}\text{Sn})$ 51 Hz], 123.24 t [$\text{C}^{11(12)}$, $^3J(^{13}\text{C}_{sp^2}-^{117/119}\text{Sn})$ 54 Hz], 127.63 d [$\text{C}^{d(5)}$,

$^3J(^{13}\text{C}_{sp2}-^1\text{H})$ 4 Hz, $^2J(^{13}\text{C}_{sp2}-^{117/119}\text{Sn})$ 41.26 Hz], 130.82 s [$\text{C}^{3(6)}$], 132.71 s [$\text{C}^{2(7)}$, $^2J(^{13}\text{C}_{sp2}-^{117/119}\text{Sn})$ 43.4 Hz], 139.42 s [$\text{C}^{10(13)}$, $^4J(^{13}\text{C}_{sp2}-^{117/119}\text{Sn})$ 10 Hz]. Found C 44.05; H 5.03; Sn 47.55. $\text{C}_{18}\text{H}_{25}\text{NSn}_2$. Calculated, %: C 43.86; H 5.07; Sn 48.21.

9-Methyl-3,6-bis(trimethylstannyl)carbazole (IIIa). (a) Into a two-neck flask equipped with a magnetic stirrer and a reflux condenser connected to an argon line was charged a solution of 2.034 g (0.006 mol) of 3,6-dibromo-9-methylcarbazole in 10 ml of anhydrous THF. The solution was cooled to 0°C, and thereto was added dropwise within 1 h a solution of lithium trimethylstannanide prepared from 6.6 g (0.033 mol) of trimethyltin chloride and 2.31 g (0.33 mol) of lithium in 50 ml of THF. The rate of stannanide addition was so adjusted that the temperature of the reaction mixture was below 5°C. The reaction mixture was stirred for 4 h at room temperature and then left standing for 12 h. Then 3 ml of methyl iodide was added thereto, and the solvents were distilled off in a vacuum. The residue was treated with water. The precipitated solid was filtered off and dried. The product was twice recrystallized from a benzene-ethanol mixture. We isolated 1.83 g (34%) of colorless crystals., mp 141-142°C. ^1H NMR spectrum, δ , ppm: 0.40 s [18H, $(\text{CH}_3)_3\text{Sn}$, $^2J(^{117/119}\text{Sn}-^1\text{H})$ 54 Hz], 3.83 s [3H, C^{14}H], 7.42 d [2H, C^1H , $^3J(^1\text{H}^1-\text{H}^2)$ 8 Hz], 7.59 d [2H, C^2H , $^3J(^1\text{H}^1-\text{H}^2)$ 8 Hz, $^3J(^{117/119}\text{Sn}-^1\text{H}^2)$ 43 Hz], 8.27 s [2H, C^4H , $^3J(^{117/119}\text{Sn}-^1\text{H}^4)$ 42 Hz]. ^{13}C NMR spectrum, δ , ppm: -9.17 d.d [$\text{C}^{17}-\text{H}$, $^1J(^{13}\text{C}_{sp3}-^{117/119}\text{Sn})$ 335 Hz, $^1J(^{13}\text{C}_{sp2}-^{117/119}\text{Sn})$ 347 Hz], 28.9 s [C^{14}], 108.4 d [$\text{C}^{1(8)}$, $^3J(^{13}\text{C}_{sp2}-^{117/119}\text{Sn})$ 51 Hz], 122.86 d [$\text{C}^{11(12)}$, $^3J(^{13}\text{C}_{sp2}-^{117/119}\text{Sn})$ 54 Hz], 127.63 d.d [$\text{C}^{4(5)}$, $^4J(^{13}\text{C}_{sp2}-^1\text{H})$ 4 Hz, $^2J(^{13}\text{C}_{sp2}-^{117/119}\text{Sn})$ 41 Hz], 132.6 d [$\text{C}^{2(7)}$, $^2J(^{13}\text{C}_{sp2}-^{117/119}\text{Sn})$ 44 Hz], 141.07 d [$\text{C}^{10(13)}$, $^4J(^{13}\text{C}_{sp2}-^{117/119}\text{Sn})$ 10 Hz]. Found, %: C 45.61, 46.17; H 5.96, 5.13; Sn 46.14, 45.14. $\text{C}_{19}\text{H}_{27}\text{NSn}_2$. Calculated, %: C 45.04; H 5.32; Sn 46.04.

(b) To a solution of 1 mmol of lithium 3,6-bis(trimethylstannyl)carbazol-9-ide (**VII**) was added 1 ml (0.01 mol) of methyl iodide in 15 ml of anhydrous ether. The solution turned light-gray. After 2 h of standing the solvents were distilled off, the residue was treated with water solution of ammonium chloride. The precipitate was filtered off and dried in a vacuum. The product obtained was recrystallized from hexane, mp 142-143°C. ^1H and ^{13}C NMR spectra of the product were identical to those described above.

9-Ethyl-3,6-bis(trimethylstannyl)carbazole (IIIb). A solution of lithium trimethylstannanide prepared from

4.5 g (0.023 mol) of trimethyltin chloride and 0.93 g (0.133 mol) of lithium in 15 ml of THF transferred through a siphon equipped with a glass frit into a dropping funnel was added dropwise within 1 h to a solution of 1.4 g (0.004 mol) of 3,6-dibromo-9-ethylcarbazole in 5 ml of THF. At the start of reaction a slight self-heating was observed. The reaction mixture was stirred at room temperature for 4 h and left standing for 12 h. The 2 ml of methyl iodide was added, and this also was accompanied by slight heat evolution. The solvent was removed in a vacuum, to the residue was added water. The precipitate was filtered off, washed with water, dried, dissolved in hot hexane, and after filtering of the hot solution the solvent was removed in a vacuum of a water-jet pump connected through a drying system. The substance obtained was recrystallized from ethol. We obtained 1.13 g of compound **IIIb**, mp 133°C. From the mother liquor was isolated 0.153 g more of compound **IIIb**, overall yield 62%.

^1H NMR spectrum (CCl_4), δ , ppm: 0.94 d [18H, $(\text{CH}_3)_3\text{Sn}$, $^2J(^{117/119}\text{Sn}-^1\text{H})$ 52 Hz], 2.0 t [3H, CH_3 , $^3J(^1\text{H}^1-\text{H}^2)$ 7 Hz], 4.9 q [2H, CH_2], 7.8-8.2 m [4H, C^1H , C^2H], 8.8 s [2H, C^4H]. Found, %: C 46.90, 47.52; H 6.01, 5.98; Sn 44.87, 44.04. $\text{C}_{20}\text{H}_{29}\text{NSn}_2$. Calculated, %: C 46.13; H 5.61; Sn 45.57.

9-Propyl-3,6-bis(trimethylstannyl)carbazole (IIIc). To a solution of 1 mmol of compound **VII** was added propyl iodide in excess. The solution turned light-gray. After standing for 2 h the solvents and excess propyl iodide were distilled off. The residue was diluted with 50 ml of dichloromethane, and the reaction mixture was washed with water solution of ammonium chloride. All solvents were distilled off at heating on a water bath, the residue was dried in a vacuum. The compound obtained was recrystallized from hexane, mp 126-126.5°C. Yield of reaction product 0.42 g (80%). ^1H NMR spectrum (CDCl_3), δ , ppm: 0.34 s [18H, $(\text{CH}_3)_3\text{Sn}$, $^2J(^1\text{H}-^{117/119}\text{Sn})$ 54 Hz], 0.93 t [3H, C^{16}H , $^2J(\text{C}^{16}\text{H}-\text{C}^{15}\text{H})$ 7.1 Hz], 1.87 m [2H, C^{15}H], 4.22 t [2H, C^{14}H , $^2J(\text{C}^{14}\text{H}-\text{C}^{15}\text{H})$ 7.1 Hz], 7.38 d [2H, $^3J(\text{C}^2\text{H}-\text{C}^1\text{H})$ 8 Hz], 7.51 d [2H, C^1H , $^3J(\text{C}^1\text{H}-\text{C}^2\text{H})$ 8 Hz], 8.20 d [2H, C^4H , $^3J(^1\text{H}-^{117/119}\text{Sn})$ 46.8 Hz]. Found, %: C 47.04, 46.97; H 5.92, 5.89; Sn 42.18, 41.04. $\text{C}_{16}\text{H}_{31}\text{NSn}_2$. Calculated, %: C 47.17; H 5.8; Sn 44.4.

9-Allyl-3,6-bis(trimethylstannyl)carbazole (IIIId). To a solution of 0.9 mmol of compound **VII** was added allyl iodide in excess. The solution turned light-gray. After standing for 2 h the solvents and

excess allyl iodide were distilled off under reduced pressure. The residue was diluted with 50 ml of dichloromethane, and the reaction mixture was washed with water solution of ammonium chloride. All solvents were distilled off at heating on a water bath, the residue was dried in a vacuum. The yield of the crude product 0.335 g (64%). The product obtained was recrystallized from hexane, mp 122–123°C. ^1H NMR spectrum (DMSO- d_6), δ , ppm: 0.4 s [18H, $(\text{CH}_3)_3\text{Sn}^2J(^{117/119}\text{Sn}-^1\text{H})$ 54 Hz], 4.95 m (4H), 5.95 m [1H, C^{10}H], 7.47 s [4H, C^3H], 8.21 s [2H, $\text{C}^4\text{H}^3J(^1\text{H}-^{117/119}\text{Sn})$ 47.6 Hz]. Found, %: C 47.18; H 5.35; Sn 43.25. $\text{C}_{16}\text{H}_{29}\text{NSn}_2$. Calcd., %: C 47.33; H 5.49; Sn 44.55.

Lithium 3,6-bis(trimethylstannyl)carbazol-9-ide (VII). In a flask equipped with a mechanical stirrer, a reflux condenser connected to an argon line, and a dropping funnel was prepared along the above described procedure lithium trimethylstannanide from 7.5 g (0.037 mol of trimethyltin chloride and 1.4 g (0.2 mol) of lithium in 100 ml of THF. Into the prepared solution of lithium stannanide was added by portions 0.325 g (0.001 mol) of crystalline dibromocarbazole. A slight heat evolution was observed. The reaction was monitored by TLC for disappearance of the initial carbazole. The reaction completed within 2–2.5 h, the initial and intermediate carbazoles were totally lacking. The solution of lithium 3,6-bis(trimethylstannyl)carbazol-9-ide was used for synthesis of compounds **IIIa**, **c**, **d**. The solution can be stored under argon no longer than 3 days.

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