Stannylation as a Highly Regio- and Stereoselective route to 2-Substituted Tributylstannyl Allylamines

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Dedicated to Prof. H.J. Bestmann in recognition of seven years distinguished service as Executive Editor of Synthesis

N-Protected propargylamines react with Bu₃Sn(Bu)Cu(CN)₂Li₂ under very mild conditions, affording, in high regiospecific fashion after quench with electrophiles, the corresponding 2-substituted 3-(tributylstannyl)allylamines, in good to excellent yields. These, upon metatlation and further reaction with electrophiles, give stereodefined 1,2-disubstituted allylamines.

While providing a synthetically useful tool when applied to terminal alkynes where two stereo- and regiodefined vinyl organometallics with differential reactivity are simultaneously generated, metallometalation reactions have been studied less than hydrometalation and carbonmetalation processes. A serious drawback in metallometalation reactions stems from the variable regioselectivity: this leads often to mixtures of regioisomeric vinyl metal derivatives as in the case of the stannylation of 1-alkynes, which does, however, provide a widely used route to vinylstannanes.

In a preceding paper we reported that the silylation of protected propargylamine occurs with a complete control of the regiochemistry leading, after quenching of the intermediate organocuprate, to 2-substituted allylamines of relevant importance as ‘building blocks’ for naturally occurring and biologically active systems.

Our ongoing interest in the field of unsaturated nitrogen containing compounds as potential biologically active building blocks, coupled with the continuing popularity of organotin intermediates in organic synthesis and the recent discovery of new high order cuprates, prompted us to apply the stannylation reaction to the propargyl amino system, and the preliminary results are reported herein.

Key targets of this work, were the study of the regioselectivity of the addition reaction and the valuable synthetic potential of the intermediate substituted vinylstannanes.

To this end, we began to study the reaction of the high order cuprate Bu₃Sn(Bu)Cu(CN)₂Li₂ prepared according to the recent report by Lipshutz with NHBoe protected propargylamic amine. Remarkably the reaction occurred at appreciable rates even under very mild conditions (see experimental) leading, after quenching, with electrophiles, to a wide range of substituted vinylstannanes, in good to excellent yields (Schemes 1 and Table).

The Table shows the high regiospecificity and the dominance of isomer 2a which is the alternate isomer of that commonly found in the stannylation reactions and in many metallometalation reactions of terminal alkynes. The structure of the two regioisomers was assigned by ¹H-NMR analysis: the coupling constant of

\[ J = 18.9 \text{ Hz} \]

Observed for the vinylic protons in compound 2a is in fact in good agreement with those found by Kund. The high regiocontrol observed in this reaction, does not have any counterpart in the recent analogous reaction run on propargyl and homopropargyl alcohol in which, using the same reagent, the opposite isomer and a 1:1 mixture of the two regioisomers were obtained respectively. Moreover, the isomer ratio appears to be completely unaffected by the workup conditions.

While the choice of the Boc or lSiMe₃ protected amino group in 1 was unimportant as far as the yields of the reaction product was concerned, after quenching the stannylation intermediate with water, careful choice of the protecting group had to be made in other cases. Thus, for example, Boc protection, although more suitable for isolation of the reaction products, may be incompatible (see entry 5 in the Table) with several electrophiles used in the quenching step. The relatively low yield observed when iodine was used as the electrophile may, on the other hand, be due to an easy, α,β-elimination of tributyltin iodide to give a sizeable amount of the starting material 1.

Although the mechanistic interpretation of the stannylation and other metallometalation reactions is still under debate, a change in regiochemistry in the cuprous ion catalyzed stannylation of 1-alkynes can be reasonably accounted for by a change in regioselectivity during the formation of the bimetallic intermediate (Scheme 2). Accordingly it would be possible to envision a change in the regiochemistry for the stannylation in the presence of suitable functional groups on the alkyne and coordination between nitrogen and copper atoms might assist in stabilizing complex A in respect to B, thus leading to the observed regiospecificity. Results in Table 1, provide furthermore one of the few sets of structures in which a range of electrophiles other than proton are employed in a stannylation reaction, thus
### Table. Substituted Stannyl Allylamines Prepared

<table>
<thead>
<tr>
<th>Entry</th>
<th>Electrophile</th>
<th>Protecting Group</th>
<th>Products</th>
<th>Regioisomeric Ratio a/b</th>
<th>Yield (%)</th>
<th>Molecular Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>H&lt;sup&gt;+&lt;/sup&gt;</td>
<td>R&lt;sup&gt;1&lt;/sup&gt; = H&lt;br&gt;R&lt;sup&gt;2&lt;/sup&gt; = Boc</td>
<td>Bu&lt;sub&gt;3&lt;/sub&gt;Sn&lt;sup&gt;→&lt;/sup&gt;NH&lt;sub&gt;Boc&lt;/sub&gt;&lt;br&gt;2b&lt;br&gt;2b</td>
<td>95 : 5</td>
<td>&gt; 95 (74)&lt;sup&gt;c&lt;/sup&gt;</td>
<td>C&lt;sub&gt;20&lt;/sub&gt;H&lt;sub&gt;42&lt;/sub&gt;NO&lt;sub&gt;3&lt;/sub&gt;Sn (446.3)</td>
</tr>
<tr>
<td>2</td>
<td>H&lt;sup&gt;+&lt;/sup&gt;</td>
<td>R&lt;sup&gt;1&lt;/sup&gt; = SiMe&lt;sub&gt;3&lt;/sub&gt;,&lt;br&gt;R&lt;sup&gt;2&lt;/sup&gt; = SiMe&lt;sub&gt;3&lt;/sub&gt;</td>
<td>Bu&lt;sub&gt;3&lt;/sub&gt;Sn&lt;sup&gt;→&lt;/sup&gt;N(SiMe&lt;sub&gt;3&lt;/sub&gt;)&lt;sub&gt;2&lt;/sub&gt;&lt;br&gt;3a&lt;br&gt;3b</td>
<td>95 : 5</td>
<td>&gt; 95 (80)&lt;sup&gt;c&lt;/sup&gt;</td>
<td>C&lt;sub&gt;21&lt;/sub&gt;H&lt;sub&gt;49&lt;/sub&gt;NSi&lt;sub&gt;3&lt;/sub&gt;Sn (490.5)</td>
</tr>
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<td>3</td>
<td>acetyl chloride</td>
<td>R&lt;sup&gt;1&lt;/sup&gt; = SiMe&lt;sub&gt;3&lt;/sub&gt;,&lt;br&gt;R&lt;sup&gt;2&lt;/sup&gt; = SiMe&lt;sub&gt;3&lt;/sub&gt;</td>
<td>Bu&lt;sub&gt;3&lt;/sub&gt;Sn&lt;sup&gt;→&lt;/sup&gt;N(SiMe&lt;sub&gt;3&lt;/sub&gt;)&lt;sub&gt;2&lt;/sub&gt;&lt;br&gt;4a&lt;br&gt;4b</td>
<td>90 : 10</td>
<td>90</td>
<td>—&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>4</td>
<td>acetyl chloride</td>
<td>R&lt;sup&gt;1&lt;/sup&gt; = H&lt;br&gt;R&lt;sup&gt;2&lt;/sup&gt; = Boc</td>
<td>Bu&lt;sub&gt;3&lt;/sub&gt;Sn&lt;sup&gt;→&lt;/sup&gt;NH&lt;sub&gt;Boc&lt;/sub&gt;,&lt;br&gt;5a&lt;br&gt;5b</td>
<td>80 : 20</td>
<td>64&lt;sup&gt;c&lt;/sup&gt;</td>
<td>—&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>5</td>
<td>methyl iodide</td>
<td>R&lt;sup&gt;1&lt;/sup&gt; = H&lt;br&gt;R&lt;sup&gt;2&lt;/sup&gt; = Boc</td>
<td>Bu&lt;sub&gt;3&lt;/sub&gt;Sn&lt;sup&gt;→&lt;/sup&gt;NH&lt;sub&gt;Boc&lt;/sub&gt;&lt;br&gt;6a&lt;br&gt;6b</td>
<td>95 : 5</td>
<td>89 (68)&lt;sup&gt;c&lt;/sup&gt;</td>
<td>C&lt;sub&gt;21&lt;/sub&gt;H&lt;sub&gt;43&lt;/sub&gt;NO&lt;sub&gt;3&lt;/sub&gt;Sn (460.3)</td>
</tr>
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<td>6</td>
<td>allyl bromide</td>
<td>R&lt;sup&gt;1&lt;/sup&gt; = H&lt;br&gt;R&lt;sup&gt;2&lt;/sup&gt; = Boc</td>
<td>Bu&lt;sub&gt;3&lt;/sub&gt;Sn&lt;sup&gt;→&lt;/sup&gt;NH&lt;sub&gt;Boc&lt;/sub&gt;&lt;br&gt;7a&lt;br&gt;7b</td>
<td>90 : 10</td>
<td>84 (64)&lt;sup&gt;c&lt;/sup&gt;</td>
<td>C&lt;sub&gt;23&lt;/sub&gt;H&lt;sub&gt;45&lt;/sub&gt;NO&lt;sub&gt;3&lt;/sub&gt;Sn (486.3)</td>
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<tr>
<td>7</td>
<td>chlorotrimeethylsilane</td>
<td>R&lt;sup&gt;1&lt;/sup&gt; = H&lt;br&gt;R&lt;sup&gt;2&lt;/sup&gt; = Boc</td>
<td>Bu&lt;sub&gt;3&lt;/sub&gt;Sn&lt;sup&gt;→&lt;/sup&gt;NH&lt;sub&gt;Boc&lt;/sub&gt;,&lt;br&gt;8a&lt;br&gt;8b</td>
<td>90 : 10</td>
<td>95 (76)&lt;sup&gt;c&lt;/sup&gt;</td>
<td>—&lt;sup&gt;b&lt;/sup&gt;</td>
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<tr>
<td>8</td>
<td>carbon dioxide dimethyl sulfate</td>
<td>R&lt;sup&gt;1&lt;/sup&gt; = H&lt;br&gt;R&lt;sup&gt;2&lt;/sup&gt; = Boc</td>
<td>Bu&lt;sub&gt;3&lt;/sub&gt;Sn&lt;sup&gt;→&lt;/sup&gt;NH&lt;sub&gt;Boc&lt;/sub&gt;,&lt;br&gt;9a&lt;br&gt;9b</td>
<td>92 : 8</td>
<td>87 (74)&lt;sup&gt;c&lt;/sup&gt;</td>
<td>C&lt;sub&gt;22&lt;/sub&gt;H&lt;sub&gt;43&lt;/sub&gt;NO&lt;sub&gt;3&lt;/sub&gt;Sn (504.3)</td>
</tr>
<tr>
<td>9</td>
<td>iodine</td>
<td>R&lt;sup&gt;1&lt;/sup&gt; = H&lt;br&gt;R&lt;sup&gt;2&lt;/sup&gt; = Boc</td>
<td>Bu&lt;sub&gt;3&lt;/sub&gt;Sn&lt;sup&gt;→&lt;/sup&gt;NH&lt;sub&gt;Boc&lt;/sub&gt;&lt;br&gt;10a&lt;br&gt;10b</td>
<td>90 : 10</td>
<td>58 (49)&lt;sup&gt;d&lt;/sup&gt;</td>
<td>—&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

* As determined by integration of the vinyl protons in the ¹H-NMR spectra of the crude mixture. Isolated yields are given in parenthesis.
* Microanalyses are given only for pure isolated a isomers: C ± 0.38, H ± 0.32, N ± 0.37.
* Pure a isomer.
* Regioisomeric mixture.

Outlining a straightforward route to synthetically useful and versatile building blocks such as 2-substituted allylamines 6a–10a, whose importance is widely documented<sup>6</sup> and where the presence of the tributyltin moiety at position 1, provides a further site for selective functionalization.

Taking advantage of the conversion of the protected propargylamino into a vinyl stannane, compounds 2a and 6a in the Table were further reacted with electrophiles, under palladium(II) complexes catalysis, to afford in good yields trisubstituted olefins (Scheme 3). In each case the reaction resulted in cross-coupled products derived from retention of configuration with respect to the vinyl-tin bond. Due to the great difference in the reactivity of 2a and 3a, fairly clean compounds are obtained even from 90/10 mixtures of vinyltin precursors a/b; e.g. the stereocontrolled two carbon homologation of the vinylstannane 2a into the corresponding dienamine, a key fragment in the construction of antitumor antibiotics.<sup>11</sup> was easily achieved even in a one-pot reaction through the route depicted in Scheme 3. Major advantages with respect to the recent reported procedure based on the hydrostannylation reaction<sup>9</sup> are the higher chemical yields as well as the tolerance, by the stannylcupration procedure of simple and common protecting groups at nitrogen, such as Boc, which does not seem to be the case when the tributyltin hydride based procedure is employed.

![Scheme 2](image-url)
All reagents were of commercial quality. N,N-Bis(trimethylsilyl) propargylamine was prepared as previously reported. All reactions were carried out under an inert atmosphere and in anhydrous solvents. Petroleum ether used refers to boiling range 30–50°C.

'H-NMR spectra were recorded on a Varian Gemini 200 instrument; chemical shifts are given with respect to TMS. IR spectra were recorded on a Perkin-Elmer 283 spectrophotometer. Mass spectra were obtained on a QMD 1000 Carlo Erba Instrument using a direct insertion probe and a 70 eV EI source, organotin fragments are given for 12Sn only. Chromatographic mobile phase was achieved by flash chromatography technique on Merck Kieselgel 60 (230–400 mesh ASTM). Preparative TLC was performed using Merck Kieselgel 60 plates. Microanalyses were obtained using a Perkin Elmer Elemental Analyser 240 C.

N-(tert-Butyloxycarbonyl)propargylamine (1): Propargylamine (5 mL, 78 mmol) is dissolved in a mixture of MeOH (160 mL), H2O (80 mL) and 1 N NaOH solution (80 mL) and cooled to 0°C. Di-tert-butyl dicarbonate (19 g, 86 mmol) is added under stirring and allowed to react overnight at r.t. After evaporation the MeOH, the aqueous solution is extracted with EtOAc (2 × 90 mL) and the organic layer is washed with water and dried (Na2SO4). The solvent is removed to give 1; yield: 7.8 g (61%); mp 37–38°C.

C12H22NO2 calc. C 61.91 H 8.44 N 9.03 (155.2) found 61.63 8.72 9.21

IR (CCl4): ν = 3465, 3314, 2981, 2932, 1726 cm⁻¹.

'H-NMR (CDCl3): δ = 1.41 (s, 9 H), 2.19 (t, 1 H, J = 2.5 Hz), 3.88 (dd, 2 H, J = 2.5, 5.6 Hz), 4.85 (br s, 1 H).

MS: m/z (%) = 140 (1), 100 (21), 59 (100), 58 (17), 57 (99), 56 (42), 43 (73), 41 (98).

Stannyleneuronation; General Procedure: Tributylstannylcuprate is prepared according to the general route outlined by Lipshutz. CuCN (66 mg, 0.75 mmol) is suspended in THF (2 mL), cooled at −78°C and then treated with 1.6 M BuLi in hexane (1 mL, 1.6 mmol). The mixture is allowed to react for 15 min and then Bu3SnH (0.43 mL, 1.6 mmol) is added dropwise. After stirring for 10 min at this temperature N-protected propargylamine (0.75 mmol) is added and allowed to react for 10 min. This mixture is used for reaction with various electrophiles (see Table) as described below.

(E)-N-(tert-Butyloxy carbonyl)-3-tritylstannyl-2-propen-1-amine (2a): After workup by quenching with NH4Cl/NH4OH buffer solution at low temperature and evaporation of the solvent, 588 mg of the crude mixture is obtained. Purification by TLC using petroleum ether/EtOAc (15:1) as eluent affords 2a; yield: 248 mg (74%).

IR (CCl4): ν = 3460, 2963, 2927, 2872, 2875, 1723 cm⁻¹.

'H-NMR (CDCl3): δ = 0.82–0.97 (m, 15 H), 1.21–1.56 (m, 12 H), 1.44 (s, 9 H), 3.75–3.80 (br t, 2 H), 4.59 (br s, 1 H), 5.94 (dt, A part of an AB system, 1 H, J = 19.0, 4.3 Hz), 6.10 (dt, B part of an AB system, 1 H, J = 19.0, 1.0 Hz).

MS: m/z (%) = 390 (15, M – Bu⁻), 334 (36, Bu2SnHCH =CH2NHBOc), 316 (92, Bu2SnCH =CHCH =N-C =O⁻), 260 (94, Bu3SnHCH =CHCH =N-C =O⁻), 240 (100, H2SnCH =CHCH =N-C =O⁻), 202 (92, SnCH =CHCH =N-C =O⁻), 179 (24, BuSnH2), 177 (37, BuSn⁻), 162 (46, SnN =C =O⁻), 121 (59, SnH⁻), 59 (83).

(E)-N,N-Bis(trimethylsilyl)-3-tritylstannyl-2-propen-1-amine (3a): The reaction mixture is quenched with NH4Cl/NH4OH buffer solution at low temperature and diluted with Et2O. The organic layer is separated, washed with brine (10 mL) and dried (Na2SO4). After evaporation of the solvent an analytically pure sample of 3a is obtained as a colourless oil, by bulb-to-bulb distillation; yield: 294 mg (80%).

C12H22NSn2 calc. C 54.12 H 10.07 N 2.86 (490.5) found 51.39 10.26 3.11

'H-NMR (CDCl3): δ = 0.07 (s, 18 H), 0.82–0.99 (m, 15 H), 1.21–1.58 (m, 12 H), 3.44–3.52 (m, 2 H), 5.90 (dt, A part of an AB system, 1 H, J = 18.9, 3.3 Hz), 6.04 (dt, B part of an AB system, 1 H, J = 18.9, 0.9 Hz).

MS: m/z (%) = 434 (37, M – Si⁻), 249 (57, H2SnCH =CHCH =NSiMe2), 200 (20, CH =CHCH =N(SiMe2)2), 193 (98, SnSiMe2), 184 (100, C =CHCH =N(SiMe2)2), 174 (65, CH3N(SiMe2)2), 170 (56, CH =CHCH =N(SiMe2)2), 150 (128, SiMe2), 86 (22, Me2Si =SiMe2), 73 (98, SiMe2).

(Z)-3-Acetyl-N,N-bis(trimethylsilyl)-3-tritylstannyl-2-propen-1-amine (4a): The reaction mixture is allowed to warm to r.t., acetyl chloride (118 mg, 1.5 mmol) is then added and allowed to stand overnight. After the usual workup 630 mg of crude mixture is obtained. IR (CCl4): ν = 1868 cm⁻¹ (C=O).

'H-NMR (CDCl3): δ = 0.02 (s, 18 H), 0.83–1.02 (m, 15 H), 1.22–1.59 (m, 12 H), 2.31 (s, 3 H), 4.27–4.32 (m, 2 H), 6.77 (t), 1 (H, J = 1.2 Hz).

(E)-N-Acetyl-N-(tert-butylocarbonyl)-3-tritylstannyl-2-propen-1-amine (5a): The reaction mixture is allowed to warm to 0°C, acetyl chloride (129 mg, 1.5 mmol) is then added and allowed to react for 3 h. After the usual workup 464 mg of crude mixture is obtained.

'H-NMR: (CDCl3): δ = 0.81–0.96 (m, 15 H), 1.17–1.52 (m, 12 H), 1.49 (m, 9 H), 2.51 (s, 3 H), 4.29–4.34 (m, 2 H), 5.87 (dt, A part of an AB system, 1 H, J = 19.1 Hz, J = 4.4 Hz), 6.02 (dt, B part of an AB system, 1 H, J = 19.1, 1.1 Hz).

(E)-N-(tert-Butyloxy carbonyl)-2-methyl-3-tritylstannyl-2-propen-1-amine (6a): The reaction mixture is warmed to 0°C, MeI (112 mg, 0.80 mmol) is then added and allowed to react for 3 h. After the usual workup, 625 mg of a crude mixture is obtained which is purified by flash chromatography using petroleum ether/EtOAc (20:1) as an eluent, giving 6a; yield: 237 mg (68%).

IR (CCl4): 3463, 2961, 2873, 2875, 1723 cm⁻¹.

'H-NMR (CDCl3): δ = 0.82–0.85 (m, 15 H), 1.21–1.59 (m, 12 H), 1.45 (s, 9 H), 1.76 (br s, 3 H), 3.73 (br d, 2 H, J = 5.8 Hz), 4.48–4.71 (br s, 1 H), 5.63 (br s, 1 H).

MS: m/z (%) = 401 (11), 348 (14), 292 (18), 218 (18), 121 (41), 59 (42), 57 (80), 41 (100).

(E)-N-(tert-Butyloxy carbonyl)-2-propyl-3-tritylstannyl-2-propen-1-amine (7a): The reaction mixture is warmed to −30°C, allyl bromide (95 mg, 0.80 mmol) is then added and allowed to reach r.t. overnight. After the usual workup 612 mg of a crude mixture is obtained which is purified by flash chromatography using petroleum ether/EtOAc (10:1) as an eluent to afford 7a; yield: 234 mg (64%).
IR (CCl₄): ν = 3461, 2963, 2928, 2876, 2853, 1720, 1608 cm⁻¹.  

1H-NMR (CDCl₃): δ = 0.82–0.94 (m, 15 H), 1.21–1.58 (m, 12 H), 1.43–3.1H), 2.77–2.84 (m, 2H), 3.71–3.76 (br m, 2H), 4.52–4.66 (br s, 1 H), 5.00–5.14 (m, 2H), 5.6–5.85 (m, 1 H), 5.73 (br s, 1 H).

MS: m/z (%) = 430 (7), 374 (37), 356 (30), 300 (19), 177 (29), 162 (26), 121 (30), 79 (20), 59 (48), 57 (100).

(Z)-N-(tert-Butoxy carbonyl)-3-tritylstannyl-2-trimethylsilyl-2-propen-1-amine (8a):

The reaction mixture is warmed to −10 °C, chlorotrimethylsilane (122 mg, 1.1 mmol) is added and left at this temperature for 3 h. After cooling to −30 °C the mixture is hydrolyzed as usual and worked up to give a crude mixture (697 mg). This is purified by chromatography over a 20 cm column of Florisil (100–200 mesh) with a double elution, first using petroleum ether and then petroleum ether/EtOAc (5:1) as an eluent. A regioisomeric mixture of 8a-8b is obtained, yield: 297 mg (76%).

IR (film): ν = 3390, 2957, 2926, 2872, 2853, 1707, 1249, 900 cm⁻¹.

1H-NMR (CDCl₃): δ = 0.21 (s, 9 H), 0.82–0.91 (m, 15 H), 1.19–1.51 (m, 12 H), 1.46 (s, 9 H), 3.78 (br s, 2 H), 3.82–3.97 (br s, 11 H), 5.94 (t, 1 H, J = 0.9 Hz).

MS: m/z (%) = 462 (14), 406 (29), 316 (17), 179 (26), 177 (42), 121 (35), 73 (56), 57 (100).

(Z)-N-(tert-Butoxy carbonyl)-2-methoxy carbonyl-3-tritylstannyl-2-propen-1-amine (9a):

The vinylepoxide is added at −78 °C to a CO₂ saturated solution in THF, left at this temperature for 1 h and then warmed to room temperature. Dimethyl sulfate (95 mg, 0.75 mmol) is added and the mixture is left for 2 h. After the usual workup 701 mg of crude mixture is obtained. TLC purification using petroleum ether/EtOAc (5:1) as an eluent, gives 9a; yield: 278 mg (74%).

IR (CCl₄): 3465, 2985, 2925, 2872, 1724, 1710 cm⁻¹.

1H-NMR (CDCl₃): δ = 0.81–0.95 (m, 15 H), 1.18–1.52 (m, 12 H), 1.42 (s, 9 H), 3.75 (s, 3 H), 4.01 (bd, 2H, J = 6.1 Hz), 4.79 (bt, 1H, J = 6.1 Hz), 6.93 (br s, 1 H).

MS: m/z (%) = 448 (55), 392 (53), 374 (100), 260 (39), 151 (48), 59 (66).

(Z)-N-(tert-Butoxy carbonyl)-2-iodo-3-tritylstannyl-2-propen-1-amine (10a):

I₂ (205 mg, 0.80 mmol) dissolved THF (2 mL) is added to the reaction mixture and left to warm to r.t. in 2 h. After the usual workup 650 mg of crude product is obtained and purified by flash chromatography using hexane/EtOAc (5:1) as eluent. A regioisomeric mixture of 10a+10b is obtained; overall yield: 206 mg (48%).

10a: 1H-NMR (CDCl₃): δ = 0.79–1.07 (m, 15 H), 1.20–1.62 (m, 12 H), 1.44 (s, 9 H), 3.94 (br d, 2H, J = 5.5 Hz), 4.91 (br s, 1 H), 7.08 (t, 1 H, J = 1.2 Hz).

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