Supporting Information for

Silver(I)-Promoted Insertion into X-H (X = Si, Sn, and Ge) Bonds

with N-Nosylhydrazones

Zhaohong Liu,^a Qiangqiang Li^a, Yang Yang,^a and Xihe Bi*^{a,b}

^aDepartment of Chemistry, Northeast Normal University, Changchun 130024, China

^bState Key Laboratory of Elemento-Organic Chemistry, Nankai University, Tianjin 300071,

China

E-mail: bixh507@nenu.edu.cn

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1. General Information

All reagents were purchased from commercial sources and used without purification unless otherwise mentioned. The products were purified by column chromatography over silica gel (200-400 size). ¹H and ¹³C Nuclear Magnetic Resonance (NMR) spectra were recorded at 25 °C on a Varian 500 MHz and 125 MHz or on a Bruker 400 MHz and 100 MHz, and TMS was used as internal standard. Mass spectra were recorded on BRUKER AutoflexIII Smartbeam MS-spectrometer. High resolution mass spectra (HRMS) were recorded on Bruck microTof by using ESI method.

2. Synthesis and Analytical Data of N-Nosylhydrazones



N-nitrobenzenesulfonylhydrazide (NBSH) was prepared according to literature procedure.¹

General procedure for converting carbonyl compounds to *N***-nosylhydrazones:** To a stirred solution of NBSH (2.0 mmol, 1 equiv) in methanol (2 mL) were added carbonyl compounds (2.2 mmol, 1.1 equiv) and the mixture was stirred for 1-2 h at room temperature. The mixture was filtered and the resulting solid was washed with ice cold diethyl ether and dried under reduced pressure to give pure *N*-nosylhydrazones. The yields were around 80% in general.



(1a) White solid, m.p. 149-150 °C; ¹H-NMR (500 MHz, DMSO-d₆) δ 12.17 (s, 1H), 8.09-8.06 (m, 2H), 8.02-8.00 (m, 1H), 7.89-7.87 (m, 2H), 7.58-7.57 (m, 2H), 7.39-7.38 (m, 3H); ¹³C-NMR (125 MHz, DMSO-d₆) δ 148.4, 148.3, 135.3, 133.9, 133.1, 131.4, 131.0, 130.9, 129.3, 127.5, 125.1; HRMS (ESI) m/z calcd. for C₁₃H₁₁N₃O₄SNa [M+Na]⁺ 328.0362, found 328.0368.



(1b) White solid, m.p. 152-153 °C; ¹H-NMR (400 MHz, DMSO-d₆) δ 12.28 (s, 1H), 8.08-8.02 (m, 3H), 7.92-7.90 (m, 2H), 7.61 (d, J = 8.4 Hz, 2H), 7.47 (d, J = 8.4 Hz, 2H); ¹³C-NMR (125 MHz, DMSO-d₆) δ 148.4, 144.4, 143.0, 131.4, 129.2, 128.8, 127.4, 127.0, 125.5, 125.1, 121.1; HRMS (ESI) m/z calcd. for C₁₃H₁₀ClN₃O₄SNa [M+Na]⁺ 361.9973, found 361.9968.



(1c) White solid, m.p. 178-179 °C; ¹H-NMR (500 MHz, DMSO-d₆) δ 12.54 (s, 1H), 8.12 (s, 1H), 8.09-8.08 (m, 1H), 8.02-8.01 (m, 1H), 7.90-7.89 (m, 2H), 7.85 (d, *J* = 7.5 Hz, 2H), 7.76 (d, *J* = 7.5 Hz, 2H); ¹³C-NMR (125 MHz, DMSO-d₆) δ 148.3, 146.1, 138.3, 135.5, 133.2, 131.2, 131.1, 130.4, 128.0, 125.1, 119.0, 112.7; HRMS (ESI) m/z calcd. for C₁₄H₁₀N₄O₄SNa [M+Na]⁺ 353.0314, found 353.0321.



(1d) White solid, m.p. 107-108 °C; ¹H-NMR (600 MHz, DMSO-d₆) δ 12.39 (s, 1H), 8.14 (s, 1H), 8.10-8.07 (m, 1H), 8.03-8.00 (m, 1H), 7.96 (d, *J* = 8.4 Hz, 2H), 7.91-7.88 (m, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 3.84 (s, 3H); ¹³C-NMR (151 MHz, DMSO-d₆) δ 166.2, 148.3, 146.8, 138.2, 135.3, 133.1, 131.4, 131.2, 130.9, 130.0, 127.5, 125.1, 52.7; HRMS (ESI) m/z calcd. for C₁₅H₁₃N₃O₆SNa [M+Na]⁺ 386.0419, found 386.0438.



(1e) Yellow solid, m.p. 154-155 °C; ¹H-NMR (600 MHz, DMSO-d₆) δ 12.15 (s, 1H), 8.12 (s, 1H), 8.10-8.18 (m, 1H), 8.02-8.00 (m, 1H), 7.90-7.88 (m, 2H), 7.7-7.66 (m, 6H), 7.47 (t, *J* = 7.8 Hz, 2H), 7.38 (t, *J* = 7.8 Hz, 1H); ¹³C-NMR (151 MHz, DMSO-d₆) δ 148.3, 147.8, 142.3, 139.7, 135.2, 133.0, 132.9, 131.4, 131.0, 129.4, 128.4, 128.0, 127.5, 127.1, 125.0; HRMS (ESI) m/z calcd. for C₁₉H₁₅N₃O₄SNa [M+Na]⁺ 404.0677, found 404.0657.



(1f) White solid, m.p. 166-167 °C; ¹H-NMR (500 MHz, DMSO-d₆) δ 12.41 (s, 1H), 8.44 (s, 1H), 8.10-8.08 (m, 1H), 8.03-8.01 (m, 1H), 7.91-7.89 (m, 2H), 7.74 (d, *J* = 7.2 Hz, 1H), 7.47 (d, *J* = 7.2 Hz, 1H), 7.41 (t, *J* = 7.2 Hz, 1H), 7.35 (t, *J* = 7.2 Hz, 1H); ¹³C-NMR (125 MHz, DMSO-d₆) δ 148.3, 144.1, 135.5, 133.6, 133.3, 132.4, 131.3, 131.2, 131.1, 130.5, 128.2, 127.1, 125.2; HRMS (ESI) m/z calcd. for C₁₃H₁₀ClN₃O₄SNa [M+Na]⁺ 361.9972, found 361.9976.



(1g) White solid, m.p. 172-173 °C; ¹H-NMR (600 MHz, DMSO-d₆) δ 12.39 (s, 1H), 8.31 (s, 1H), 8.10-8.18 (m, 1H), 8.06-7.99 (m, 1H), 7.94-7.85 (m, 3H), 7.65 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.14 (td, *J* = 7.7, 1.5 Hz, 1H); ¹³C-NMR (151 MHz, DMSO-d₆) δ 150.9, 148.2, 140.1, 135.4, 135.3, 133.1, 132.4, 131.3, 131.1, 129.0, 127.2, 125.1, 100.2; HRMS (ESI) m/z calcd. for C₁₃H₁₀IN₃O₄SNa [M+Na]⁺ 453.9330, found 453.9360.



(1h) White solid, m.p. 152-153 °C; ¹H-NMR (400 MHz, DMSO-d₆) δ 12.15 (s, 1H), 8.08-8.00 (m, 3H), 7.90-7.88 (m, 2H), 7.30 (t, J = 8.0 Hz, 1H), 7.15-7.13 (m, 2H), 6.97 (d, J = 8.0 Hz, 1H), 3.75 (s, 3H; ¹³C-NMR (125 MHz, DMSO-d₆) δ 160.0, 148.4, 148.1, 135.4, 135.3, 133.1, 131.3, 131.1, 130.5, 125.0, 120.1, 116.8, 112.0, 55.7; HRMS (ESI) m/z calcd. for C₁₄H₁₃N₃O₅SNa [M+Na]⁺ 358.0468, found 358.0476.



(1i) White solid, m.p. 147-148 °C; ¹H-NMR (600 MHz, DMSO-d₆) δ 12.39 (s, 1H), 8.17 (s, 1H), 8.10-8.08 (m, 1H), 8.02-7.99 (m, 1H), 7.92 (s, 1H), 7.91-7.86 (m, 3H), 7.73 (d, J = 7.8 Hz, 1H), 7.62 (t, J = 7.8 Hz, 1H); ¹³C-NMR (151 MHz, DMSO-d₆) δ 148.4, 146.5, 135.3, 135.0, 133.0, 131.3, 131.0, 130.9, 130.4, 130.1 (q, J = 32.0 Hz), 127.0 (q, J = 3.0 Hz), 125.0, 124.3 (q, J = 272.0 Hz), 123.7 (q, J = 3.6 Hz).



(1j) White solid, m.p. 163-164 °C; ¹H-NMR (400 MHz, DMSO-d₆) δ 12.39 (s, 1H), 8.32 (s, 1H), 8.11-8.09 (m, 1H), 8.02-8.00 (m, 1H), 7.91-7.89 (m, 2H), 7.52 (d, *J* = 8.8 Hz, 1H), 7.22 (d, *J* = 2.8 Hz, 1H), 6.96-6.93 (m, 1H), 3.74 (s, 3H); ¹³C-NMR (125 MHz, DMSO-d₆) δ 159.1, 148.4, 146.2, 135.6, 134.5, 133.3, 133.2, 131.3, 131.0, 125.0, 119.1, 114.5, 111.6, 56.0; HRMS (ESI) m/z calcd. for C₁₄H₁₂BrN₃O₅SNa [M+Na]⁺ 435.9573, found 435.9560.



(1k) Brown solid, m.p. 148-149 °C; ¹H-NMR (500 MHz, DMSO-d₆) δ 11.96 (s, 1H), 8.08 (s, 1H), 8.05-8.02 (m, 2H), 8.01-7.99 (m, 1H), 7.89-7.87 (m, 2H), 7.68 (s, 1H), 6.62 (s, 1H); ¹³C-NMR (125 MHz, DMSO-d₆) δ 148.4, 146.2, 145.4, 141.4, 135.2, 133.1, 131.4, 131.0, 125.0, 122.3, 107.4; HRMS (ESI) m/z calcd. for C₁₁H₉N₃O₅SNa [M+Na]⁺ 318.0155, found 318.0150.



(11) Brown solid, m.p. 152-153 °C; ¹H-NMR (400 MHz, DMSO-d₆) δ 11.99 (s, 1H), 8.10 (s, 1H), 8.07-8.04 (m, 1H), 8.01-7.99 (m, 1H), 7.89-7.87 (m, 3H), 7.56-7.55 (m, 1H), 7.29 (d, *J* = 4.8 Hz, 1H);
¹³C-NMR (125 MHz, DMSO-d₆) δ 148.4, 144.0, 137.0, 135.2, 133.1, 131.4, 131.1, 129.3, 128.3, 125.0, 124.8; HRMS (ESI) m/z calcd. for C₁₁H₉N₃O₄S₂Na [M+Na]⁺ 333.9926, found 333.9935.



(1m) Yellow solid, m.p. 184-185 °C; ¹H-NMR (500 MHz, DMSO-d₆) δ 12.15 (s, 1H), 8.32 (s, 1H), 8.24 (s, 1H), 8.13-8.11 (m, 1H), 8.00-7.98 (m, 1H), 7.94 (d, *J* = 8.0 Hz, 1H), 7.90 (d, *J* = 8.0 Hz, 1H), 7.88-7.85 (m, 4H), 7.39-7.37 (m, 3H), 7.30 (t, *J* = 7.5 Hz, 1H), 2.29 (s, 3H); ¹³C-NMR (125 MHz, DMSO-d₆) δ 148.4, 146.5, 143.0, 135.4, 135.1, 134.1, 133.0, 131.3, 131.2, 131.1, 130.9, 127.4, 126.9, 126.3, 125.0, 124.8, 123.2, 117.6, 113.6, 21.6; HRMS (ESI) m/z calcd. for C₂₂H₁₈N₄O₆S₂Na [M+Na]⁺ 521.0559, found 521.0567.



(1n) White solid, m.p. 150-151 °C; ¹H-NMR (600 MHz, DMSO-d₆) δ 11.20 (s, 1H), 8.07-8.05 (m, 1H), 8.00-7.98 (m, 1H), 7.89-7.86 (m, 2H), 7.63-7.61 (m, 2H), 7.37-7.34 (m, 3H), 2.28 (s, 3H); ¹³C-NMR (151 MHz, DMSO-d₆) δ 154.8, 148.7, 137.6, 135.0, 132.8, 131.6, 130.7, 130.1, 128.8, 126.6, 124.8, 15.1; HRMS (ESI) m/z calcd. for C₁₄H₁₃N₃O₄SNa [M+Na]⁺ 342.0520, found 342.0521.



1p

(1p) Yellow solid, m.p. 128-129 °C; ¹H-NMR (600 MHz, DMSO-d₆) δ 11.29 (s, 1H), 8.18 (s, 1H), 8.13-8.10 (m, 1H), 8.02-7.99 (m, 1H), 7.95 (dd, J = 6.1, 3.4 Hz, 1H), 7.90-7.87 (m, 3H), 7.85-7.85 (m, 2H), 7.55-7.52 (m, 2H), 2.40 (s, 3H); ¹³C-NMR (151 MHz, DMSO-d₆) δ 154.5, 148.7, 135.1, 134.9, 133.8, 133.0, 132.8, 131.5, 130.8, 129.0, 128.2, 127.9, 127.5, 127.0, 126.9, 124.8, 123.6, 14.8; HRMS (ESI) m/z calcd. for C₁₈H₁₅N₃O4₅Na [M+Na]⁺ 392.0681, found 392.0676.



(1q) White solid, m.p. 158-159 °C; ¹H-NMR (600 MHz, DMSO-d₆) 10.70 (s, 1H), 8.13-8.08 (m, 1H), 8.03 (dt, J = 7.5, 3.7 Hz, 1H), 7.94-7.89 (m, 2H), 7.60-7.55 (m, 3H), 7.39 (t, J = 7.2 Hz, 1H), 7.33 (t, J = 7.6 Hz, 2H), 7.29 (dd, J = 6.1, 3.1 Hz, 4H); ¹³C-NMR (151 MHz, DMSO-d₆) δ 156.3, 148.8, 137.1, 135.2, 133.0, 132.6, 131.4, 130.8, 130.5, 130.2, 129.5, 129.1, 128.8, 127.8, 124.9; HRMS (ESI) m/z calcd. for C₁₉H₁₅N₃O₄SNa [M+Na]⁺404.41.0685, found 404.0671.



(1r) Yellow solid, m.p. 148-149 °C; ¹H-NMR (400 MHz, DMSO-d₆) δ 12.10 (s, 1H), 8.03-7.98 (m, 2H), 7.90-7.88 (m, 3H), 7.56 (d, J = 7.2 Hz, 2H), 7.37-7.28 (m, 3H), 7.02 (d, J = 16.0 Hz, 1H), 6.89-6.83 (m, 1H); ¹³C-NMR (125 MHz, DMSO-d₆) δ 150.6, 148.4, 140.5, 136.1, 135.2, 133.2, 131.8, 130.8, 129.5, 129.3, 127.7, 125.2, 125.0; HRMS (ESI) m/z calcd. for C₁₅H₁₃N₃O₄SNa [M+Na]⁺354.0519, found 354.0525.



(1s) Yellow solid, m.p. 104-105 °C; ¹H-NMR (500 MHz, CDCl₃) δ 9.29 (s, 1H), 8.24-8.22 (m, 1H), 7.85-7.83 (m, 1H), 7.77-7.73 (m, 2H), 6.70 (s, 1H), 1.09-1.08 (m, 18H), 1.02-1.01 (m, 3H),; ¹³C-NMR (125 MHz, CDCl₃) δ 148.1, 134.5, 132.8, 132.6, 131.6, 127.6, 125.3, 110.2, 93.5, 18.4, 10.9; HRMS (ESI) m/z calcd. for C₁₈H₂₇N₃O₄SSiNa [M+Na]⁺ 432.1384, found 432.1387.

3. Optimization of the Reaction Conditions

				NaH (1.5 equiv) cat., solv.		ŞiEt ₃	Ph.		Ph N A	
		1a', R = Ts 1a, R = Ns	2a	temp.,	N ₂ , 24 h	Ph ^A H 3aa	4aa	Ph +	5aa	
Entry	R	Cat.	Sc	olvent	Temp		Yield (%) ^b			
		<mark>(30 mol %)</mark>			<mark>. (°C)</mark>	<mark>3aa</mark>	<mark>la/la'</mark>	<mark>4aa</mark>	<mark>5aa</mark>	
<mark>1</mark>	Ts	a AgOTf	C	H ₂ Cl ₂	<mark>40</mark>	<mark>15</mark>	<mark>68</mark>	<mark>6</mark>	<mark>4</mark>	
<mark>2</mark>	<mark>N:</mark>	s AgOTf	C	H ₂ Cl ₂	<mark>40</mark>	<mark>57</mark>	<mark>22</mark>	<mark>10</mark>	<mark>2</mark>	

STable 1 Optimization of the Reaction Conditions^{*a*}

<mark>4</mark>	<mark>Ns</mark>	AgOAc	CH ₂ Cl ₂	<mark>80</mark>	<mark>55</mark>	<mark>0</mark>	22	<mark>14</mark>
<mark>5</mark>	<mark>Ns</mark>	AgOTAF	CH ₂ Cl ₂	<mark>80</mark>	<mark>60</mark>	<mark>0</mark>	<mark>16</mark>	<mark>4</mark>
<mark>6</mark>	<mark>Ns</mark>	AgF	CH ₂ Cl ₂	<mark>80</mark>	<mark>28</mark>	<mark>0</mark>	<mark>60</mark>	2
7	<mark>Ns</mark>	Ag ₂ CO ₃	CH ₂ Cl ₂	<mark>80</mark>	<mark>34</mark>	<mark>0</mark>	<mark>48</mark>	<mark>6</mark>
<mark>8</mark>	<mark>Ns</mark>	AgOTf	1,4-dioxane	<mark>80</mark>	<mark>20</mark>	<mark>0</mark>	<mark>26</mark>	<mark>18</mark>
<mark>9</mark>	<mark>Ns</mark>	AgOTf	PhCl	<mark>80</mark>	<mark>6</mark>	<mark>32</mark>	<mark>10</mark>	<mark>16</mark>
<mark>10</mark>	<mark>Ns</mark>	AgOTf	MeCN	<mark>80</mark>	<mark><5</mark>	<mark>20</mark>	<mark>6</mark>	<mark>40</mark>
<mark>11</mark>	<mark>Ns</mark>	AgOTf	ClCH ₂ CH ₂ Cl	<mark>80</mark>	<mark>32</mark>	<mark>0</mark>	<mark>42</mark>	<mark>18</mark>
<mark>12</mark>	<mark>Ns</mark>	Cu(OTf) ₂	CH ₂ Cl ₂	<mark>80</mark>	<mark>7</mark>	<mark>0</mark>	<mark>26</mark>	<mark>44</mark>
<mark>13</mark>	<mark>Ns</mark>	Cu(MeCN) ₄ PF ₆	CH ₂ Cl ₂	<mark>80</mark>	<mark>10</mark>	<mark>0</mark>	<mark>26</mark>	<mark>20</mark>
14 ^c	<mark>Ns</mark>	Rh ₂ (OAc) ₄	CH ₂ Cl ₂	<mark>80</mark>	<mark>25</mark>	<mark>0</mark>	<mark>60</mark>	2

^{*a*} Reaction conditions: **1a** (0.3 mmol), **2a** (1.5 mmol). NaH (0.45 mmol), and the catalyst (30 mol %) in solvent (6.0 mL) for 24 h under N₂-atmosphere. ^{*b*} Yield calculated from ¹H-NMR spectroscopy with CH_2Br_2 as the internal standard. ^{*c*} $Rh_2(OAc)_4$ (5 mol %) was used. ^{*d*} Isolated yield.

4. Synthesis and Analytical Data of compounds 3aa to 3sa, and 3bb-3bs

The synthesis of compounds **3aa-3sa** and **3bb-3bs** is performed according to the below given procedure for the synthesis of compound **3aa**.



General procedure (with **3aa** as an example): To a flame-dried sealed tube were added *N*-nosylhydrazone **1a** (91.5 mg, 0.3 mmol, 1.0 equiv), NaH (18 mg, **60 wt%**, 0.45 mmol, 1.5 equiv) and dry CH_2Cl_2 (6.0 mL, 0.05 M) inside a glove box. The resulting mixture was stirred at room temperature for 1 h. Then, triethylsilane **2a** (240 µl, 1.5 mmol, 5.0 equiv) and AgOTf (23.1 mg, 0.09 mmol, 30 mol %) were added and the tube was sealed and heated at 80 °C for additional 24

h.The reaction was monitored by TLC. When the reaction was completed, the crude reaction mixture was allowed to reach room temperature, and filtered through a short pad of silica gel with EtOAc as an eluent. The filtrate was evaporated under reduced pressure to leave a crude mixture, which was purified by column chromatography on silica gel (eluting with petroleum ether) to afford **3aa** as a colorless oil (50.1 mg, 81% yield).



(3aa) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.19 (t, *J* = 7.5 Hz, 2H), 7.05 (t, *J* = 7.5 Hz, 1H), 7.01 (d, *J* = 7.5 Hz, 2H), 2.09 (s, 2H), 0.91 (t, *J* = 8.0 Hz, 9H), 0.51 (q, *J* = 8.0 Hz, 6H); ¹³C-NMR (CDCl₃, 125 MHz) δ 140.6, 128.11, 128.07, 123.7, 21.6, 7.3, 3.0; HRMS (ESI) m/z calcd. for C₂₆H₄₅Si₂ [2M+H]⁺: 413.3058, found: 413.3054.



(3ba) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.18 (d, J = 8.5 Hz, 2H), 6.95 (d, J = 8.5 Hz, 2H),
2.08 (s, 2H), 0.93 (t, J = 8.0 Hz, 9H), 0.52 (q, J = 8.0 Hz, 6H); ¹³C-NMR (125 MHz, CDCl₃) δ 139.2,
129.4, 129.3, 128.2, 21.1, 7.3, 2.9.



(3ca) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.48 (d, J = 8.0 Hz, 2H), 7.09 (d, J = 8.0 Hz, 2H),
2.19 (s, 2H), 0.91 (t, J = 8.0 Hz, 9H), 0.51 (q, J = 8.0 Hz, 6H); ¹³C-NMR (125 MHz, CDCl₃) δ 147.4,
132.0, 128.6, 119.4, 107.4, 23.1, 7.2, 2.9; HRMS (ESI) m/z calcd. for C₁₄H₂₁NNaSi [M+Na]⁺:
254.1335, found: 254.1337.



(3da) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.88 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 3.88 (s, 3H), 2.18 (s, 2H), 0.91 (t, *J* = 8.0 Hz, 9H), 0.51 (q, *J* = 8.0 Hz, 6H); ¹³C-NMR (125 MHz, CDCl₃) δ 167.3, 147.1, 129.6, 127.9, 125.8, 51.8, 22.6, 7.2, 2.9; HRMS (ESI) m/z calculated for C₁₅H₂₄NaO₂Si [M+Na]⁺: 287.1438, found: 287.1424.



(**3ea**) Colourless oil; ¹**H-NMR** (500 MHz, CDCl₃) δ 7.58 (d, *J* = 8.5 Hz, 2H), 7.44 (d, *J* = 8.5 Hz, 2H), 7.41 (t, *J* = 8.0 Hz, 2H), 7.29 (t, *J* = 8.0 Hz, 1H), 7.08 (d, *J* = 8.0 Hz, 2H), 2.14 (s, 2H), 0.94 (t, *J* = 8.0 Hz, 9H), 0.54 (q, *J* = 8.0 Hz, 6H); ¹³**C-NMR** (125 MHz, CDCl₃) δ 141.2, 139.9, 136.6, 128.6, 128.5, 126.8, 126.74, 126.70, 21.3, 7.3, 3.0; **HRMS** (ESI) m/z calculated for C₁₉H₂₆NaSi [M+Na]+: 282.1877, found: 282.1884.



(3fa) Colourless oil; ¹**H-NMR** (500 MHz, CDCl₃) δ 7.28 (d, *J* = 8.0 Hz, 1H), 7.15-7.03 (m, 2H), 6.98-7.00 (m, 1H), 2.28 (s, 2H), 0.91 (t, *J* = 8.0 Hz, 9H), 0.56 (q, *J* = 8.0 Hz, 6H); ¹³**C-NMR** (125 MHz, CDCl₃) δ 139.1, 132.7, 129.9, 129.3, 126.4, 125.2, 19.4, 7.2, 3.4.



(3ga) Colourless oil; ¹**H-NMR** (500 MHz, CDCl₃) δ 7.76 (dd, *J* = 8.0 Hz, *J* = 1.0 Hz, 1H), 7.18 (td, *J* = 7.5, 1.0 Hz, 1H), 7.08-7.06 (m, 1H), 6.75-6.72 (m, 1H), 2.37 (s, 2H), 0.91 (t, *J* = 8.0 Hz, 9H), 0.59 (q,

J = 8.0 Hz, 6H); ¹³**C-NMR** (125 MHz, CDCl₃) δ 144.6, 139.4, 128.4, 127.9, 125.6, 100.4, 27.1, 7.3, 3.6.





(3ha) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.11 (t, J = 7.5 Hz, 1H), 6.61 (d, J = 7.5 Hz, 2H),
6.57 (s, 1H), 3.77 (s, 3H), 2.08 (s, 2H), 0.92 (t, J = 8.0 Hz, 9H), 0.51 (q, J = 8.0 Hz, 6H); ¹³C-NMR
(125 MHz, CDCl₃) δ 159.5, 142.3, 129.0, 120.8, 113.9, 109.0, 55.0, 21.8, 7.3, 3.0.



(3ia) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.32-7.28 (m, 2H), 7.26-7.24 (m, 1H), 7.19-7.17 (m, 1H), 2.16 (s, 2H), 0.91 (t, J = 8.0 Hz, 9H), 0.51 (q, J = 8.0 Hz, 6H); ¹³C-NMR (125 MHz, CDCl₃) δ 141.8, 131.3 (d, J = 1 Hz), 130.4 (d, J = 31.6 Hz), 128.5, 124.5 (q, J = 3.8 Hz), 124.0 (q, J = 270.0 Hz), 120.6 (q, J = 3.8 Hz), 21.9, 7.2, 2.9.





(3ja) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.28 (d, J = 8.5 Hz, 1H), 6.55 (d, J = 3.0 Hz, 1H), 6.43 (dd, J = 9.0, 3.0 Hz, 1H), 3.68 (s, 3H), 2.21 (s, 2H), 0.84 (t, J = 8.0 Hz, 9H), 0.51 (q, J = 8.0 Hz, 6H); ¹³C-NMR (125 MHz, CDCl₃) δ 158.6, 142.0, 133.1, 115.0, 114.4, 111.3, 55.3, 22.4, 7.3, 3.5.





(3n) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.30-7.29 (m, 1H), 7.09 (s, 1H), 6.15-6.14 (m, 1H),
1.79 (s, 2H), 0.93 (t, J = 8.0 Hz, 9H), 0.52 (q, J = 8.0 Hz, 6H); ¹³C-NMR (125 MHz, CDCl₃) δ 142.3,
137.8, 121.6, 112.2, 8.5, 7.3, 3.1.



(30) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.19-7.17 (m, 1H), 6.79-6.78 (m, 1H), 6.69-6.68 (m, 1H), 2.11 (s, 2H), 0.92 (t, J = 8.0 Hz, 9H), 0.52 (q, J = 7.5 Hz, 6H); ¹³CNMR (125 MHz, CDCl₃) δ 139.6, 129.0, 124.7, 117.6, 15.5, 7.3, 3.1.





(3ma) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 8.00 (d, *J* = 8.0 Hz, 1H), 7.68 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 7.5 Hz, 1H), 7.28 (t, *J* = 7.5 Hz, 1H), 7.21 (t, *J* = 7.5 Hz, 1H), 7.15-7.13 (m, 3H), 2.28 (s, 3H), 2.02 (s, 2H), 0.85 (t, *J* = 8.0 Hz, 9H), 0.45 (q, *J* = 8.0 Hz, 6H); ¹³C-NMR (125 MHz, CDCl₃) δ 144.4, 135.3, 135.1, 132.0, 129.5, 126.6, 124.4, 122.9, 121.4, 121.2, 119.5, 113.9, 21.4, 8.6, 7.2, 3.2; HRMS (ESI) m/z calculated for C₂₂H₂₉NNaO₂SSi [M+Na]⁺: 422.1578, found: 422.1582.



(3na) Colourless oil; ¹**H-NMR** (500 MHz, CDCl₃) δ 7.24-7.21 (m, 2H), 7.08-7.06 (m, 3H), 2.30 (q, *J* = 7.5 Hz, 1H), 1.37 (d, *J* = 7.5 Hz, 3H), 0.89 (t, *J* = 8.0 Hz, 9H), 0.51 (q, *J* = 8.0 Hz, 6H); ¹³**C-NMR** (CDCl₃, 125 MHz) δ 146.3, 128.0, 127.1, 124.2, 26.8, 15.4, 7.5, 2.0.



(30a) Colourless oil; ¹**H-NMR** (500 MHz, CDCl₃) δ 7.32 (t, *J* = 7.5 Hz, 2H), 7.25 (t, *J* = 7.5 Hz, 1H), 7.21 (d, *J* = 7.5 Hz, 2H), 3.07-3.00 (m, 1H), 0.93 (t, *J* = 8.0 Hz, 9H), 0.69-0.60 (m, 6H); ¹³**C-NMR** (125 MHz, CDCl₃) δ 134.3 (q, *J* = 3.4 Hz), 129.0, 128.5, 128.4 (q, *J* = 276.0 Hz), 126.6, 40.9 (q, *J* = 11 Hz), 7.0, 2.9.



3ра

(**3pa**) Colourless oil; ¹**H-NMR** (500 MHz, CDCl₃) δ 7.80-7.66 (m, 3H), 7.48 (s, 1H), 7.41 (t, *J* = 7.5 Hz, 1H), 7.35 (t, *J* = 7.5 Hz, 1H), 7.24 (dd, *J* = 8.0, 1.0 Hz, 1H), 2.47 (q, *J* = 7.5 Hz, 1H), 1.47 (d, *J* = 7.5 Hz, 3H), 0.90 (t, *J* = 8.0 Hz, 9H), 0.54 (q, *J* = 8.0 Hz, 6H); ¹³**C-NMR** (125 MHz, CDCl₃) δ 144.1, 133.7, 131.2, 127.5, 127.23, 127.17, 127.0, 125.7, 124.4, 124.3, 27.1, 15.5, 7.5, 2.1; **HRMS** (ESI) m/z calculated for C₁₈H₂₆NaSi [M+Na]⁺: 293.1797, found: 293.1784.



(**3qa**) Colourless oil; ¹**H-NMR** (500 MHz, CDCl₃) δ 7.27-7.21 (m, 8H), 7.14-7.09 (m, 2H), 3.65 (s, 1H), 0.84 (t, *J* = 8.0 Hz, 9H), 0.60 (q, *J* = 8.0 Hz, 6H); ¹³**C-NMR** (125 MHz, CDCl₃) δ 142.9, 128.8, 128.2, 125.0, 43.0, 7.5, 3.4; **HRMS** (ESI) m/z calculated for C₁₉H₂₆NaSi [M+Na]⁺: 305.1697, found: 305.1684.



3ra

(3ra) Colourless oil; ¹**H-NMR** (500 MHz, CDCl₃) δ 7.31-7.23 (m, 4H), 7.18-7.10 (m, 1H), 6.28-6.20 (m, 2H), 1.70 (dd, *J* = 5.0, *J* = 2.0 Hz, 2H), 0.96 (t, *J* = 8.0 Hz, 9H), 0.57 (q, *J* = 8.0 Hz, 6H); ¹³**C-NMR** (125 MHz, CDCl₃) δ 138.5, 128.4, 128.1, 128.0, 126.1, 125.4, 18.8, 7.4, 3.3.



(3sa) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 1.59 (s, 2H), 1.05-1.03 (m, 18H), 1.02-1.00 (m, 3H), 0.96 (t, J = 8.0 Hz, 9H), 0.63 (q, J = 8.0 Hz, 6H); ¹³C-NMR (125 MHz, CDCl₃) δ 106.6, 78.4, 18.6, 11.5, 7.3, 3.4, 3.1, 1.0.





(3bb) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.15 (d, J = 8.5 Hz, 2H), 6.91 (d, J = 8.5 Hz, 2H),
2.05 (s, 2H), 1.32-1.20 (m, 12H), 0.87 (t, J = 7.5 Hz, 9H), 0.50-0.44 (m, 6H); ¹³C-NMR (125 MHz,
CDCl₃) δ 139.3, 129.4, 129.3, 128.1, 26.7, 25.9, 22.2, 13.7, 11.6;



(**3bc**) Colourless oil; ¹**H-NMR** (500 MHz, CDCl₃) δ 7.15 (d, *J* = 8.5 Hz, 2H), 7.01 (d, *J* = 8.5 Hz, 2H), 2.15 (s, 2H), 1.08-1.03 (m, 3H), 1.01-1.00 (m, 18H); ¹³**C-NMR** (125 MHz, CDCl₃) δ 139.6, 129.8, 129.4, 128.1, 18.6, 18.5, 10.9; **HRMS** (ESI) m/z calculated for C₁₆H₂₇ClNaSi [M+Na]⁺:305.1464, found: 305.1456.



(3bd) Colourless oil; ¹H NMR (500 MHz, CDCl₃) δ 7.16 (d, J = 8.5 Hz, 2H), 6.91 (d, J = 8.5 Hz, 2H),
2.05 (s, 2H), 0.92 (t, J = 8.0 Hz, 3H), 049 (q, J = 8.0 Hz, 2H), 0.05 (s, 6H); ¹³C-NMR (125 MHz, CDCl₃)
δ 139.1, 129.5, 129.3, 128.2, 24.7, 7.2, 6.4, -4.2.



(3be) Colourless oil; ¹H NMR (500 MHz, CDCl₃) δ 7.17 (d, J = 8.0 Hz, 2H), 6.90 (d, J = 8.5 Hz, 2H),
2.07 (s, 2H), 0.92 (s, 9H), 0.10 (s, 6H); ¹³C-NMR (125 MHz, CDCl₃) δ 139.2, 129.5, 128.2, 26.5, 22.1,
16.7, -6.6.





(3bf) Colourless oil; ¹H NMR (500 MHz, CDCl₃) δ 7.87 (s, 1H), 7.77 (s, 2H), 7.16 (d, J = 8.0 Hz, 2H),
6.81 (d, J = 8.0 Hz, 2H), 2.31 (s, 2H), 0.35 (s, 6H); ¹³C-NMR (125 MHz, CDCl₃) δ 141.5, 136.8, 133.4,
130.7 (q, J = 33.4 Hz), 130.4, 129.4, 128.5, 123.9 (q, J = 272.0 Hz), 123.0 (q, J = 3.6 Hz), 25.2, -3.8.



(**3bg**) Colourless oil; ¹**H-NMR** (500 MHz, CDCl₃) δ 7.45-7.43 (m, 4H), 7.40-7.36 (m, 2H), 7.35-7.31 (m, 4H), 7.07 (d, *J* = 8.5 Hz, 2H), 6.77 (d, *J* = 8.5 Hz, 2H), 2.58 (s, 2H), 0.46 (s, 3H); ¹³**C-NMR** (125 MHz, CDCl₃) δ 137.4, 135.9, 134.6, 129.9, 129.8, 129.4, 128.1, 127.8, 24.0, -4.9; **HRMS** (ESI) m/z calculated for C₂₀H₁₉ClNaSi [M+Na]+: 345.0838, found: 345.0867.



(3bh) White solid, m.p. 166-167 °C; ¹H-NMR (500 MHz, CDCl₃) δ 7.41-7.38 (m, 9H), 7.33-7.31 (m, 6H), 7.01 (d, *J* = 8.5 Hz, 2H), 6.74 (d, *J* = 8.5 Hz, 2H), 2.87 (s, 2H); ¹³C-NMR (125 MHz, CDCl₃) δ 136.8, 135.9, 133.8, 130.4, 130.1, 129.7, 128.0, 127.8, 23.0; HRMS (ESI) m/z calculated for C₂₅H₂₁ClNaSi [M+Na]+: 407.0995, found: 407.0972.



(3bi) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.16 (d, J = 8.0 Hz, 2H), 6.99 (d, J = 8.0 Hz, 2H),
2.30 (s, 2H), 0.12 (s, 27H); ¹³C-NMR (125 MHz, CDCl₃,) δ 141.8, 129.6, 129.4, 128.3, 16.1, 1.1;
HRMS (ESI) m/z calculated for C₁₆H₃₃ClNa Si₄ [M+Na]+: 395.1246, found: 395.1254.



(3bj) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.11 (d, *J* = 8.5 Hz, 2H), 6.89 (d, *J* = 8.5 Hz, 2H), 2.26 (t, *J*_{Sn-H} = 56 Hz, 2H), 1.47-1.35 (m, 6H), 1.29-1.22 (m, 6H), 0.87 (t, *J* = 8.0 Hz, 9H), 0.80 (t, *J* = 8.0 Hz, 6H); ¹³C-NMR (125 MHz, CDCl3) δ 142.4, 128.22 (128.27, 128.18), 128.13, 128.05, 29.00 (29.08, 29.02), 27.30 (27.52, 27.09), 17.7, 13.7, 9.3 (10.60, 10.54, 8.11, 8.06); (The spliting peaks generated from J-coupling of ¹¹⁵Sn/¹¹⁹Sn with ¹³C).



(3bk) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.41-7.33 (m, 15H), 7.06 (d, J = 8.5 Hz, 2H), 6.93 (d, J = 8.5 Hz, 2H), 2.91 (t, J_{Sn-H} = 66 Hz, 2H); ¹³C-NMR (125 MHz, CDCl₃) δ 139.5, 139.3, 137.0 (137.1, 136.9), 129.11 (129.15, 129.06), 129.0, 128.7, 128.6, 128.37 (128.42, 128.31),

19.5(20.78,20.72, 18.25, 18.19). (The spliting peaks generated from J-coupling of $^{115}Sn/^{119}Sn$ with ^{13}C).



(3bl) Colourless oil; ¹**H-NMR** (600 MHz, CDCl₃) δ 6.96 (d, J = 7.8 Hz, 2H), 6.89 (d, J = 7.8 Hz, 2H), 2.26 (s, 3H), 2.25 (t, $J_{Sn-H} = 28$ Hz, 2H), 1.44-1.39 (m, 6H), 1.29-1.23 (m, 6H), 0.86 (t, J = 7.8 Hz, 9H), 0.79 (t, J = 7.8 Hz, 6H); ¹³**C-NMR** (150 MHz, CDCl3) δ 140.3, 132.0, 128.90 (128.93, 128.86), 126.87 (126.94, 126.80), 29.03 (29.10, 28.97), 27.33 (27.50, 27.15), 20.8, 17.5, 13.7, 9.23 (10.28, 10.23, 8.23, 8.18); (The spliting peaks generated from J-coupling of ¹¹⁵Sn/¹¹⁹Sn with ¹³C).



(3bm) Colourless oil; ¹H-NMR (600 MHz, CDCl₃) δ 7.56 (d, *J* = 7.8 Hz, 2H), 7.42-7.39 (m, 4H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.05 (d, *J* = 7.8 Hz, 2H), 2.34 (t, *J*_{Sn-H} = 28 Hz, 2H), 1.46-1.40 (m, 6H), 1.29-1.24 (m, 6H), 0.88-0.81 (m, 15H); ¹³C-NMR (150 MHz, CDCl3) δ 143.1, 141.3, 135.7, 128.6, 127.33 (127.40, 127.26), 126.92 (126.96, 126.88), 126.7, 126.5, 29.03 (29.10, 28.96), 27.32 (27.50, 27.14), 17.9, 13.7, 9.38 (10.43, 10.39, 9.38, 8.38, 8.33); (The spliting peaks generated from J-coupling of ¹¹⁵Sn/¹¹⁹Sn with ¹³C).



(3bn) Colourless oil; ¹H-NMR (600 MHz, CDCl₃) δ 7.26-7.24 (m, 1H), 7.07-7.04 (m, 2H), 6.92-6.90 (m, 1H), 2.40 (t, $J_{\text{Sn-H}}$ = 28 Hz, 2H), 1.45-1.39 (m, 6H), 1.27-1.22 (m, 6H), 0.87-0.81 (m, 15H); ¹³C-NMR (150 MHz, CDCl3) δ 142.2, 131.6, 129.06 (129.10, 129.02), 128.57 (128.63, 128.50), 126.56 (126.60, 126.52), 124.21 (124, 124.17), 28.94 (29.01, 28.87), 27.30 (27.48, 27.12), 17.2, 13.7,

10.04 (11.09, 11.05, 9.02, 8.98); (The spliting peaks generated from J-coupling of ¹¹⁵Sn/¹¹⁹Sn with ¹³C).



(3bo) Colourless oil; ¹**H-NMR** (600 MHz, CDCl₃) δ 7.07 (t, *J* = 7.8 Hz, 1H), 6.57 (d, *J* = 7.8 Hz, 1H), 6.54-6.51 (m, 2H), 3.76 (s, 3H), 2.28 (t, *J*_{Sn-H} = 28 Hz, 2H), 1.45-1.40 (m, 6H), 1.29-1.23 (m, 6H), 0.86 (t, *J* = 7.8 Hz, 9H), 0.81 (t, *J* = 7.8 Hz, 6H); ¹³**C-NMR** (150 MHz, CDCl3) δ 159.6, 145.4, 129.1, 119.6, 112.4, 108.4, 55.0, 29.02 (29.09, 28.95), 27.31 (27.49, 27.13), 18.36 (19.14, 19.11, 17.61, 17.58), 13.7, 9.35 (10.41, 10.36, 8.35, 8.30); (The spliting peaks generated from *J*-coupling of ¹¹⁵Sn/¹¹⁹Sn with ¹³C).



(3bp) Colourless oil; ¹H-NMR (600 MHz, CDCl₃) δ 7.20 (t, J = 7.2 Hz, 2H), 7.02 (d, J = 7.2 Hz, 2H), 6.98 (t, J = 7.2 Hz, 1H), 2.70 (q, J = 7.8 Hz, 1H), 1.57 (d, J = 7.8 Hz, 3H), 1.40-1.35 (m, 6H), 1.27-1.22 (m, 6H), 0.85 (t, J = 7.8 Hz, 9H), 0.79-0.76 (m, 6H); ¹³C-NMR (150 MHz, CDCl3) δ 149.0, 128.16 (128.20, 128.12), 125.51 (125.58, 125.44), 123.2, 29.05 (29.11, 28.98), 27.45 (27.62, 27.27), 26.8, 17.4, 13.7, 8.72 (9.73, 9.68, 7.76, 7.71); (The spliting peaks generated from Jcoupling of ¹¹⁵Sn/¹¹⁹Sn with ¹³C).



(3bq) Colourless oil; ¹H-NMR (600 MHz, CDCl₃) δ 7.74-7.66 (m, 3H), 7.41-7.39 (m, 2H), 7.33-7.30 (m, 1H), 7.19-7.18 (m, 1H), 2.86 (q, J = 7.8 Hz, 1H), 1.67 (d, J = 7.8 Hz, 3H), 1.41-1.35 (m, 6H), 1.26-1.20 (m, 6H), 0.83-0.78 (m, 15H); ¹³C-NMR (150 MHz, CDCl3) δ 146.7, 134.1, 130.8, 127.5,

127.4, 127.0, 126.31 (126.36, 126.26), 125.7, 124.0, 121.71 (121.79, 121.62), 29.08 (29.04, 29.01), 27.44 (27.62, 27.28), 27.3, 17.31, 13.6, 8.88 (9.88, 9.84, 7.91, 7.87); (The spliting peaks generated from J-coupling of ¹¹⁵Sn/¹¹⁹Sn with ¹³C).



(3br) Colourless oil; ¹H-NMR (600 MHz, CDCl₃) δ 7.23 (t, J = 7.2 Hz, 4H), 7.17 (d, J = 7.2 Hz, 4H), 7.06 (t, J = 7.2 Hz, 2H), 4.04 (s, 1H), 1.35-1.30 (m, 6H), 1.23-1.17 (m, 6H), 0.82-0.79 (m, 15H); ¹³C-NMR (150 MHz, CDCl3) δ 144.5, 128.3, 127.83 (127.91, 127.75), 124.3, 42.4, 28.86 (28.92, 28.79), 27.30 (27.49, 27.12), 13.6, 10.49 (11.52, 11.47, 9.51, 9.47); (The spliting peaks generated from Jcoupling of ¹¹⁵Sn/¹¹⁹Sn with ¹³C).



(3bs) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.15 (d, J = 8.5 Hz, 2H), 6.91 (d, J = 8.5 Hz, 2H), 2.17 (s, 2H), 1.33-1.25 (m, 12H), 0.87 (t, J = 7.0 Hz, 9H), 0.70-0.66 (m, 6H); ¹³C-NMR (125 MHz, CDCl3) δ 140.4, 129.1, 128.9, 128.1, 27.2, 26.5, 21.6, 13.7, 12.2.



(4) Colourless oil; ¹H-NMR (500 MHz, CDCl₃) δ 7.81 (d, J = 7.5 Hz, 2H), 7.53 (t, J = 7.5 Hz, 1H), 7.47 (t, J = 7.5 Hz, 2H), 0.99 (t, J = 7.5 Hz, 9H), 0.93-0.89 (m, 6H); ¹³C-NMR (125 MHz, CDCl₃) δ 236.1, 142.4, 132.6, 128.6, 127.1, 7.4, 3.7; HRMS (ESI) m/z calculated for C₁₃H₂₁OSi [2M+H]⁺: 221.1358, found: 221.1354.



(5) Colourless oil; ¹H NMR (500 MHz, CDCl₃) δ 7.18 (t, *J* = 7.5 Hz, 2H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.97 (d, *J* = 7.5 Hz, 2H), 1.65 (s, 1H), 0.92 (t, *J* = 8.0 Hz, 9H), 0.61 (q, *J* = 8.0 Hz, 6H), 0.04 (s, 9H); ¹³C-NMR (125 MHz, CDCl₃) δ 142.8, 129.0, 127.9, 123.2, 25.8, 7.8, 4.8, 0.2; HRMS (ESI) m/z calculated for C₁₆H₃₁Si₂ [M+H]⁺: 279.1964, found: 279.1954.

5. ¹H and ¹³C NMR Spectral Copies





S23

























S35


































































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3bk





7.571 7.558 7.411 7.408 7.408 7.338 7.338 7.292 7.292 7.058 $\begin{array}{c} 2.344\\ 2.2297\\ 1.448\\ 1.448\\ 1.421\\ 1.408\\ 1.408\\ 0.878\\ 0.878\\ 0.847\\ 0.847\\ 0.847\\ 0.847\\ 0.847\\ 0.819\end{array}$ -2.391 -2.344 -2.297 $\begin{array}{c} 1.460\\ 1.448\\ 1.429\\ 1.425\\ 1.421\\ 1.421\\ 1.421\\ 1.421\\ 1.425\\ 1.259\\ 1.256\\ 1.255\\ 1.$ Sn(n-Bu)3 Ph 3bm ٨ 6.35 6.3 2.4 2.3 1.6 1.5 1.4 1.3 1.2 15.31H ٣ ٢ ٩ ۲ ч 74 2.00 1.00 1.99 6.35 2.11 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 9.0 8.5 8.0 -135.70 128.62 127.33 126.66 126.66 _143.06 _141.34 29.03 -17.92 -13.69 -9.38 727.40-127.33-127.33-127.26-126.92-126.88-29.10-29.03-29.03-27.50 -27.32 -27.14 10.43 -9.38 8.33 Sn(n-Bu)3 Ph 3bm 127.2 27.4 9.6 1 150 70 60 20 10 0 140 130 120 110 100 90 80 50 40 30




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S74



S75



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S78