Supporting Information

Employment of Palladium Pincer-Complexes in Phenylselenenylation of Organohalides

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Contents:

Experimental procedures and spectroscopic data for compounds **4a-m**: S2-S5

Computational details of DFT studies: S5-S12

¹³C-NMR spectra of compounds **4a-m**: S13-S25

References: S26

Experimental Procedures and Spectroscopic Data for Compounds 4a-m

All solvents used in the reactions were freshly distilled prior to use. The NMR spectra were recorded in CDCl₃ with ¹H at 300 or 400 MHz, ¹³C at 75.4 or 100.5 MHz and ⁷⁷Se at 76.3 MHz with CDCl₃ (δ[¹H] = 7.26 ppm, δ[¹³C] = 77.36 ppm) as internal standards and (PhSe)₂ (δ[⁷⁷Se] = 463.0 ppm) as external standard respectively.

General Procedure for the Palladium Pincer-Complex Catalyzed Phenylselenenylation.

Trimethylstannylphenylselenide **2** (88 mg, 0.275 mmol) and the corresponding electrophile (0.25 mmol) in THF (1.0 mL) was added to catalyst **1a** (2.8 mg, 0.005 mmol) at room temperature. Then this reaction mixture was stirred for the allotted temperatures and times given in Table 1. Thereafter the solvent was removed and the product was purified by silica gel chromatography.

Phenyl-(2-propynyl)-selenide (4a). This product was prepared by the above general procedure. For purification silica gel chromatography was employed using pentane as eluent. The obtained ¹H-NMR data is in agreement with the published literature values.¹ ¹H-NMR: δ 2.25 (t, ⁴J = 2.7 Hz, 1H), 3.49 (d, ⁴J = 2.7 Hz, ²J (Se, H) = 13.0 Hz, 2H), 7.30 (m, 3H), 7.61 (m, 2H). ¹³C-NMR: 12.7, 72.1, 81.2, 128.1, 129.5, 129.8, 133.6.

Phenyl-(3-phenyl-2-propynyl)-selenide (4b). This product was prepared by the above general procedure. For purification silica gel chromatography was employed using pentane as eluent. ¹H-NMR: δ 3.74 (s, ²J (Se, H) = 13.3 Hz, 2H), 7.30 (m, 8H), 7.65 (m, 2H). ¹³C-NMR: 14.0, 84.3, 86.6, 123.5, 128.1, 128.4, 128.5, 129.4, 130.0, 131.9, 134.1.

1-Phenyl-4-(phenylselanyl)-2-butyne-1-ol (4c). This product was prepared by the above general procedure. For purification silica gel chromatography was employed using pentane:ether 2:1 as eluent. ¹H-NMR: δ 2.23 (d, ³J = 6.2 Hz, 1H), 3.59 (d, ⁵J = 2.1 Hz, ²J (Se, H) = 13.6 Hz, 2H), 5.43 (dt, ³J = 6.2 Hz, ⁵J = 2.1 Hz, 1H), 7.32 (m, 6H), 7.43 (m, 2H), 7.59 (m, 2H). ¹³C-NMR: δ 13.1, 65.0, 83.8, 84.1, 126.9, 128.1, 128.6, 128.9, 129.4, 129.7, 134.0, 140.9.

N1-[1-Phenyl-4-(phenylselanyl)-2-butyryl]-4-methyl-1-benzenesulfonamide (4d). This product was prepared by the above general procedure. For purification silica gel chromatography was employed using pentane:CH₂Cl₂ 1:2 as eluent. ¹H-NMR: δ 2.41 (s, 3H), 3.28 (t, ⁵J = 2.1 Hz, 2H), 4.66 (d, ³J = 8.8 Hz, 1H), 5.28 (dt, ³J = 8.8 Hz, ⁵J = 2.1 Hz, 1H), 7.29 (m, 10H), 7.49 (m, 2H), 7.71 (d, ³J = 8.2 Hz, 2H). ¹³C-NMR: δ 12.8, 21.9, 49.7, 80.6, 84.1, 127.6, 127.8, 128.3, 128.7, 128.9, 129.5, 129.6, 129.8, 134.1, 137.7, 137.8, 143.8.

Phenyl-(2-propenyl)-selenide (4e). This product was prepared by the above general procedure. For purification silica gel chromatography was employed using pentane as eluent followed by bulb-to-bulb distillation. The obtained NMR data is in agreement with the published literature values.² ¹H-NMR: δ 3.53 (d, ³J = 7.6 Hz, ²J (Se, H) = 12.4 Hz, 2H), 4.94 (d, ³J = 10.0 Hz, 1H), 4.97 (d, ³J = 16.8 Hz, 1H), 5.95 (ddt, ³J = 7.6 Hz, 10.0 Hz, 16.8 Hz, 1H), 7.27 (m, 3H), 7.51 (m, 2H). ¹³C-NMR: δ 31.0, 117.2, 127.4, 129.3, 130.3, 133.7, 134.7.

Phenyl [(E)-3-phenyl-2-propenyl]-selenide (4f). This product was prepared by the above general procedure. For purification silica gel chromatography was employed using pentane as eluent. The obtained NMR data is in agreement with the published literature values.³ ¹H-NMR: δ 3.69 (d, ³J = 6.9 Hz, ²J (Se, H) = 12.5 Hz, 2H), 6.24 (d, ³J = 15.7 Hz, 1H), 6.33 (dt, ³J = 6.9 Hz, 15.7 Hz, 1H), 7.25 (m, 8H), 7.53 (m, 2H). ¹³C-NMR: δ 31.0, 126.2, 126.6, 127.6, 127.7, 128.8, 129.3, 130.2, 132.4, 134.2, 137.2.

Methyl-(E)-3-methyl-4-(phenylselanyl)-2-butenolate (4g). This product was prepared by the above general procedure. For purification silica gel chromatography was employed using pentane:ether 10:1 as eluent. ¹H-NMR: δ 2.28 (d, ⁴J = 1.2 Hz, 3H), 3.51 (d, ⁴J = 0.7 Hz, 2H), 3.63 (s, 3H), 5.45 (d, ⁴J = 1.2 Hz, 1H), 7.27 (m, 3H), 7.50 (m, 2H). ¹³C-NMR: δ 18.5, 38.7, 51.2, 117.2, 128.2, 129.4, 129.6, 134.7, 155.3, 166.9.

3-Phenylselenylcyclohex-1-ene (4h). This product was prepared by the above general procedure. For purification silica gel chromatography was employed using pentane as eluent

followed by bulb-to-bulb distillation. The obtained NMR data is in agreement with the published literature values.⁴ ¹H-NMR: δ 1.64 (m, 1H), 2.00 (m, 5H), 3.98 (m, 1H), 5.77 (m, 1H), 5.86 (m, 1H), 7.25 (m, 3H), 7.58 (m, 2H). ¹³C-NMR: δ 20.0, 25.3, 29.8, 41.5, 127.6, 128.1, 129.3, 130.1, 131.0, 134.5.

Benzyl-phenylselenide (4i). This product was prepared by the above general procedure. For purification silica gel chromatography was employed using pentane as eluent. The obtained NMR data is in agreement with the published literature values.² ¹H-NMR: δ 4.11 (s, ²*J* (Se, H) = 11.0 Hz, 2H), 7.23 (m, 8H), 7.46 (m, 2H). ¹³C-NMR: δ 32.6, 127.2, 127.6, 128.8, 129.2, 129.3, 130.7, 133.9, 139.0.

4-Bromobenzyl-phenyl-selenide (4j). This product was prepared by the above general procedure. For purification silica gel chromatography was employed using pentane as eluent. The obtained ¹H-NMR data is in agreement with the published literature values.⁵ ¹H-NMR: δ 4.02 (s, ²*J* (Se, H) = 11.2 Hz, 2H), 7.03 (d, ³*J* = 8.2 Hz, 2H), 7.24 (m, 3H), 7.38 (d, ³*J* = 8.2 Hz, 2H), 7.43 (m, 2H). ¹³C-NMR: 31.8, 121.0, 127.9, 129.4, 130.1, 130.8, 131.8, 134.2, 138.2.

4-Nitrobenzyl-phenyl-selenide (4k). This product was prepared by the above general procedure. For purification silica gel chromatography was employed using pentane:ether 15:1 as eluent. The obtained NMR data is in agreement with the published literature values.^{5, 6} ¹H-NMR: δ 4.09 (s, ²*J* (Se, H) = 12.5 Hz, 2H), 7.25 (m, 3H), 7.41 (m, 2H), 7.40 (m, 2H) 8.06 (d, ³*J* = 8.7 Hz, 2H). ¹³C-NMR: 31.7, 123.9, 128.5, 129.0, 129.5, 129.8, 134.9, 147.0, 147.2.

Phenyl-1-benzenecarboselenoate (4l). This product was prepared by the above general procedure. For purification silica gel chromatography was employed using pentane:ether 15:1 as eluent. The obtained NMR data is in agreement with the published literature values.⁷ ¹H-NMR: δ 7.48 (m, 5H), 7.61 (m, 3H), 7.95 (m, 2H). ¹³C-NMR: δ 126.1, 127.7, 129.3, 129.4, 129.7, 134.2, 136.7, 138.9, 193.7.

Phenyl-4-methoxy-1-benzenecarboselenoate (4m). This product was prepared by the above general procedure. For purification silica gel chromatography was employed using pentane:ether 10:1 as eluent. The obtained NMR data is in agreement with the published literature values.⁷ ¹H-NMR: δ 3.88 (s, 3H), 6.96 (d, $^3J = 9.0$ Hz, 2H), 7.42 (m, 3H), 7.61 (m, 2H), 7.91 (d, $^3J = 9.0$ Hz, 2H). ¹³C-NMR: δ 55.9, 114.4, 126.3, 129.3, 129.6, 130.0, 131.7, 136.8, 164.5, 191.6.

Stoichiometric Reactions of 1a with 2. Complex **1a** (30 mg, 0.054 mmol) was mixed with **2** (19 mg, 0.059 mmol) in CDCl₃ (0.7 mL) in an NMR-tube. The ⁷⁷Se NMR spectrum of this reaction mixture was recorded at -9 °C; thereafter the reaction mixture was treated with **3m** (11 mg, 0.064 mmol). Subsequently, the solvent was removed and **1a** was recovered by silica-gel chromatography using CH₂Cl₂:ether 20:1 as eluent. ⁷⁷Se NMR spectrum at -9 °C of the pure substances: 424.9 ppm and 427.1 ppm (**1a**); 0.5 ppm (**2**).

Molecular Geometries Obtained in the DFT Calculations

E(RB+HF-PW91) = -549.156303 Hartree

C1	-1.183795	2.133191	0.125241
C2	-1.173571	3.536101	0.067964
C3	0.032631	4.229223	-0.077667
C4	1.232421	3.518765	-0.188568
C5	1.229813	2.114720	-0.176486
C6	0.019690	1.400357	-0.007788
Pd7	0.010594	-0.603315	0.039799
Se8	-2.356564	-0.388843	-0.477612
C9	-2.480437	1.404373	0.367892
C10	-3.213404	-1.487510	0.901189
C11	2.519699	1.363114	-0.383272
Se12	2.378785	-0.384781	0.549892
C13	3.229316	-1.561653	-0.766705
H14	2.173201	4.060911	-0.300422
H15	0.037603	5.318748	-0.104628

H16	-2.109372	4.091668	0.152368
H17	-3.353270	1.937021	-0.027075
H18	-2.639895	1.213975	1.437325
H19	-2.860271	-1.178864	1.888943
H20	-2.882940	-2.512282	0.704567
H21	-4.301830	-1.400895	0.819917
H22	3.397623	1.906345	-0.014930
H23	2.676435	1.119338	-1.442170
H24	4.318285	-1.486436	-0.681591
H25	2.882474	-2.570426	-0.521401
H26	2.887642	-1.298593	-1.771512
C127	-0.000680	-3.016133	0.095999

E(RB+HF-PW91) = -774.987869 Hartree

C1	0.236049	0.059786	0.076879
C2	0.169422	0.121102	1.473004
C3	1.342766	0.049816	2.241003
C4	2.601851	-0.129748	1.622167
C5	2.648976	-0.206033	0.210874
C6	1.475830	-0.090764	-0.553077
C7	1.270139	0.212264	3.737009
Se8	2.771954	-0.808006	4.540634
C9	3.195171	0.369806	6.047726
Pd10	4.305093	-0.228818	2.739472
Se11	5.420239	0.308139	0.645186
C12	6.769799	-1.093719	0.401134
C13	3.965316	-0.446885	-0.479935
Se14	6.441316	-0.138016	4.111234
C15	6.372544	-1.691084	5.245272
H16	-0.798359	0.243143	1.963023
H17	-0.674460	0.132211	-0.517941
H18	1.526235	-0.133069	-1.642751
H19	1.430028	1.257056	4.033959
H20	0.322435	-0.135228	4.165032
H21	4.187483	-1.519075	-0.561373
H22	4.014832	-0.002408	-1.480884
H23	7.511189	-0.937493	1.190889
H24	6.308054	-2.076738	0.528455
H25	7.225126	-0.993737	-0.589958
H26	4.139850	0.004170	6.460615

H27	3.325981	1.395325	5.692888
H28	2.396149	0.300183	6.793847
C29	5.714881	-2.882387	4.877993
C30	5.705789	-3.989875	5.734593
C31	6.363334	-3.940696	6.971034
C32	7.030968	-2.764421	7.339545
C33	7.032756	-1.650990	6.491014
H34	5.218121	-2.942387	3.909420
H35	5.186241	-4.898887	5.427627
H36	6.359244	-4.805342	7.634883
H37	7.550656	-2.708458	8.297245
H38	7.550023	-0.739967	6.794160

TS 5a

E(RB+HF-PW91) = -905.932598 Hartree

C1	0.142174	0.140744	-0.017906
C2	0.092926	0.016674	1.382028
C3	1.289538	-0.139574	2.104442
C4	2.516315	-0.183280	1.429755
C5	2.564311	-0.061511	0.035135
C6	1.372680	0.103807	-0.684186
Se7	-1.571583	0.121597	2.345117
Pd8	-3.363448	-1.186189	1.040348
Se9	-2.107420	-2.864824	-0.264732
C10	-3.386578	-4.285716	0.246838
C11	-4.751490	-3.677996	0.090247
C12	-4.928345	-2.304517	0.365628
C13	-6.219795	-1.746016	0.240357
C14	-7.309655	-2.549408	-0.131170
C15	-7.119086	-3.905196	-0.416158
C16	-5.841388	-4.464493	-0.314910
C17	-6.418056	-0.277872	0.492454
Se18	-5.149254	0.246847	1.923145
C19	-4.715245	2.062385	1.319509
C20	-0.495446	-3.590489	0.583295
C21	-1.449712	-2.004330	3.619255
C22	-1.073736	-1.634588	4.944141
C23	-0.749596	-1.287092	6.067565
C124	-1.704047	-4.489098	3.801727
H25	-5.693186	-5.522480	-0.536305
H26	-7.964789	-4.524925	-0.713504
H27	-8.307763	-2.114546	-0.205795
H28	-3.148923	-4.533187	1.292104
H29	-3.226744	-5.164922	-0.387719
H30	-6.146701	0.328648	-0.381098

H31	-7.437628	-0.022817	0.802644
H32	-3.862346	2.398513	1.917273
H33	-4.439637	2.048907	0.261546
H34	-5.578402	2.713337	1.494194
H35	0.233915	-2.778088	0.654793
H36	-0.745452	-4.007971	1.569471
H37	-0.109666	-4.368830	-0.084705
H38	-0.780706	0.268671	-0.583785
H39	1.397771	0.202122	-1.769911
H40	3.520685	-0.094534	-0.486514
H41	3.436449	-0.312967	2.000039
H42	1.263421	-0.227761	3.190787
H43	-0.688718	-2.339421	2.932106
H44	-2.490504	-2.181441	3.390941
H45	-0.470949	-1.015663	7.066691

TS 5b

E(RB+HF-PW91) = -905.930516 Hartree

C1	0.144858	-0.049470	0.125216
C2	0.057719	-0.024906	1.526156
C3	1.235181	0.003551	2.295273
C4	2.485877	0.036674	1.665677
C5	2.573571	0.028444	0.266611
C6	1.399800	-0.021230	-0.497257
Se7	-1.653829	-0.107086	2.423644
C8	-3.069166	-0.561476	0.761659
C9	-3.220255	-1.799253	0.479001
C10	-3.874550	-2.753511	-0.346644
Pd11	-2.279255	2.177671	3.360309
Se12	-4.651866	1.574856	3.484356
C13	-4.888803	2.513598	5.213152
C14	-4.092280	3.789009	5.147879
C15	-2.908436	3.836236	4.375502
C16	-2.152999	5.030213	4.381253
C17	-2.561508	6.132500	5.149696
C18	-3.746456	6.077372	5.890130
C19	-4.515364	4.909569	5.880614
C20	-0.919830	5.139101	3.528342
Se21	-0.128286	3.332817	3.293727
C22	0.358481	3.519075	1.402358
C23	-4.820266	-0.281613	4.094923
C124	-5.179389	-2.088363	-1.664161
H25	-1.960009	7.043212	5.159373
H26	-4.068424	6.940158	6.472906
H27	-5.440281	4.865932	6.458309

H28	-1.163174	5.483596	2.514637
H29	-0.159655	5.806226	3.951257
H30	-4.501420	1.832286	5.981452
H31	-5.957623	2.679632	5.391008
H32	-4.607015	-0.915475	3.227497
H33	-4.103846	-0.484797	4.895403
H34	-5.849381	-0.445110	4.432276
H35	0.770837	2.562392	1.072681
H36	-0.527874	3.770702	0.814253
H37	1.120037	4.302245	1.321801
H38	1.174928	-0.010515	3.384167
H39	3.392146	0.058426	2.271816
H40	3.547409	0.048005	-0.222738
H41	1.456625	-0.042897	-1.586034
H42	-0.764228	-0.107293	-0.471760
H43	-3.411760	0.428790	0.473008
H44	-4.492492	-3.459893	0.213839
H45	-3.180523	-3.295135	-0.995159

TS 8

E(RB+HF-PW91) = -494.893318 Hartree

C1	0.203443	-0.939993	-0.035023
C2	0.353472	-1.013190	1.356199
C3	1.182925	-0.089819	2.008513
C4	1.873804	0.887087	1.273108
C5	1.715877	0.949387	-0.116275
C6	0.878036	0.039172	-0.773365
Se7	1.464439	-0.184552	3.925123
Sn8	0.073801	2.004620	4.652453
C9	1.342828	3.648819	4.116642
C10	-0.402408	-1.360751	4.657548
C11	-0.176859	-2.756935	4.432942
C12	-1.757014	1.892416	3.561944
C13	-0.153168	1.708923	6.756934
C114	-2.771501	-1.160652	5.795477
H15	2.541435	1.587522	1.775190
H16	2.253637	1.709144	-0.683422
H17	0.756996	0.089403	-1.855199
H18	-0.443995	-1.657638	-0.538581
H19	-0.163227	-1.792569	1.913110
H20	-0.436111	2.658974	7.228674
H21	-0.958040	0.979629	6.920484
H22	0.773146	1.355543	7.224821
H23	0.911259	4.582268	4.501306
H24	2.343122	3.528594	4.548619

H25	1.431191	3.736461	3.027631
H26	-2.243904	2.876427	3.570812
H27	-1.583577	1.599566	2.520235
H28	-2.412433	1.161440	4.055217
H29	-0.337289	-1.016848	5.683888
H30	-1.137832	-0.866841	4.032932
C31	0.056975	-3.933702	4.216990
H32	0.228240	-4.980155	4.054774

E(RB+HF-PW91) = -130.983149 Hartree

C1	0.008719	0.000000	-0.003537
C12	-0.097655	0.000000	1.803843
C3	1.386423	0.000000	-0.478866
H4	-0.521479	-0.892637	-0.348848
H5	-0.521479	0.892637	-0.348848
C6	2.523340	0.000000	-0.910304
H7	3.529522	0.000000	-1.282440

PhSeSnMe₃ (2)

E(RB+HF-PW91) = -363.970389 Hartree

C1	-2.397483	0.127380	-1.213316
C2	-1.792998	0.499207	-0.000514
C3	-2.397671	0.129443	1.212825
C4	-3.588378	-0.609021	1.210684
C5	-4.186836	-0.982681	0.000557
C6	-3.588190	-0.611079	-1.210110
Se7	-0.183534	1.586933	-0.001328
Sn8	1.513738	-0.366523	0.000258
C9	1.214628	-1.555254	-1.763771
C10	3.440969	0.584673	-0.000452
C11	1.214460	-1.552459	1.766134
H12	-1.938194	0.424688	2.155760
H13	-4.049039	-0.889006	2.158581
H14	-5.114161	-1.555892	0.000975
H15	-4.048696	-0.892683	-2.157602
H16	-1.937851	0.421021	-2.156676
H17	4.237159	-0.170967	0.000252
H18	3.560854	1.213022	-0.890993
H19	3.560760	1.214581	0.888999
H20	1.892034	-2.416414	1.763883
H21	1.408208	-0.956526	2.665712
H22	0.182268	-1.919889	1.804343
H23	1.892616	-2.418884	-1.760365

H24	0.182605	-1.923231	-1.801235
H25	1.407919	-0.960573	-2.664275

4a

E(RB+HF-PW91) = -356.834060 Hartree

C1	-0.151829	0.000000	-0.374939
C2	-0.036740	0.000000	1.076556
C3	0.043648	0.000000	2.291469
Se4	-2.081076	0.000001	-0.900254
H5	0.317322	-0.893337	-0.801485
H6	0.317322	0.893336	-0.801485
H7	0.123449	0.000000	3.360860
C8	-1.795855	0.000000	-2.812737
C9	-1.706703	-1.214332	-3.513456
C10	-1.518829	-1.212273	-4.901816
C11	-1.423216	0.000000	-5.597336
C12	-1.518829	1.212273	-4.901817
C13	-1.706703	1.214333	-3.513457
H14	-1.789458	-2.156728	-2.972219
H15	-1.450897	-2.158429	-5.439225
H16	-1.279321	0.000000	-6.678053
H17	-1.450898	2.158429	-5.439226
H18	-1.789459	2.156728	-2.972219

7

E(RB+HF-PW91) = -356.844472 Hartree

C1	0.271240	0.127840	-0.150402
C2	-0.013630	0.432846	1.186694
C3	0.944358	0.210403	2.183210
C4	2.207869	-0.299296	1.840892
C5	2.501290	-0.591936	0.499194
C6	1.528654	-0.390053	-0.487304
Se7	3.511744	-0.512388	3.243551
C8	4.378763	-2.099022	2.588401
C9	5.679411	-2.280094	2.680160
C10	6.980568	-2.456870	2.787985
H11	3.486579	-0.968209	0.224705
H12	1.762170	-0.626134	-1.525915
H13	-0.480263	0.291052	-0.922749
H14	-0.990842	0.830712	1.461369
H15	0.702614	0.422196	3.225359

H16	3.721064	-2.884366	2.211627
H17	7.422598	-2.858304	3.701636
H18	7.652083	-2.208981	1.963963

Me₃SnCl

E(RB+HF-PW91) = -138.143034 Hartree

Sn1	-0.000753	0.000000	-0.151437
Cl2	-0.001786	0.000000	2.228540
C3	2.062113	0.000000	-0.716111
C4	-1.030952	-1.786120	-0.719290
C5	-1.030952	1.786120	-0.719290
H6	-1.088375	1.869088	-1.812270
H7	-2.049389	1.779632	-0.314093
H8	-0.505969	2.665147	-0.327941
H9	2.164447	0.000000	-1.808963
H10	2.562676	0.889683	-0.316920
H11	2.562676	-0.889683	-0.316920
H12	-1.088375	-1.869088	-1.812270
H13	-0.505970	-2.665147	-0.327941
H14	-2.049389	-1.779632	-0.314093

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