



## A novel simple synthesis of bis(diorganoselenophosphoryl)selenides (R<sub>2</sub>PSe)<sub>2</sub>Se from secondary phosphines and elemental selenium

Alexander V. Artem'ev, Nina K. Gusarova, Svetlana F. Malysheva, Igor A. Ushakov, Boris A. Trofimov \*

A. E. Favorsky Irkutsk Institute of Chemistry, Siberian Branch of the Russian Academy of Sciences, 1 Favorsky Str., 664033 Irkutsk, Russian Federation

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### ABSTRACT

Secondary phosphines R<sub>2</sub>PH [R = Ph(CH<sub>2</sub>)<sub>2</sub>, 4-*t*-BuC<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>)<sub>2</sub>, 4-MeOC<sub>6</sub>H<sub>4</sub>(CH<sub>2</sub>)<sub>2</sub>, 2-Naphth(CH<sub>2</sub>)<sub>2</sub>] react with two equivalents of elemental selenium under mild conditions (85 °C, 3 h, toluene) to afford bis(diorganoselenophosphoryl)selenides (R<sub>2</sub>PSe)<sub>2</sub>Se in high yields (75–92%).

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Bis(diorganoselenophosphoryl)selenides (R<sub>2</sub>PSe)<sub>2</sub>Se are used as single-source precursors in metal organic chemical vapor deposition processes for the design of semiconducting thin films<sup>1</sup> and nanoparticles,<sup>2</sup> possessing magneto-optical,<sup>3</sup> electrical and other properties.<sup>4</sup> These compounds are promising extractants of heavy, rare and transuranium elements,<sup>5</sup> potent biologically active compounds,<sup>6</sup> RAFT-agents,<sup>7</sup> and intermediates for the generation of diselenophosphinate anions [R<sub>2</sub>PSe<sub>2</sub>]<sup>-</sup> as reactants used extensively for the design of diverse nanomaterials.<sup>2,8</sup>

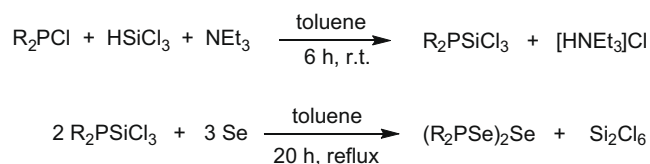
Reported methods for the preparation of bis(diorganoselenophosphoryl)selenides are low yielding and time-consuming. Furthermore, these methods utilize toxic phosphorus halides and hence do not conform to modern environmental requirements. Thus, bis(diphenylselenophosphoryl)selenide was synthesized by the reaction of Ph<sub>2</sub>PCl with Na<sub>x</sub>Se<sub>y</sub>, generated from sodium metal and elemental selenium in liquid ammonia solution at -78 °C.<sup>9</sup> The reaction afforded a mixture of products (along with unreacted sodium). In addition, the results of this method were not reproducible.<sup>10</sup> Recently, the synthesis of two representative selenides, (R<sub>2</sub>PSe)<sub>2</sub>Se (R = *i*-Pr, Ph) were reported.<sup>11</sup> These compounds were prepared in moderate yield (41–43%) via multistep reactions of toxic and difficult to obtain monochlorophosphines (R<sub>2</sub>PCl) with highly flammable and harmful trichlorosilane, triethylamine and selenium in toluene<sup>11</sup> (Scheme 1).

In addition, this method is laborious requiring an argon atmosphere, Schlenk equipment and especially pure anhydrous sol-

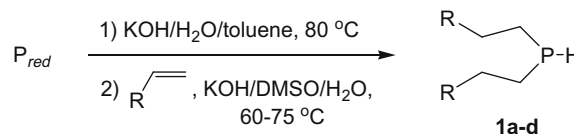
vents. Along with the target compounds the reaction also gave several byproducts.

Herein, we report a simple and convenient approach to the synthesis of bis(diorganoselenophosphoryl)selenides via the reaction of readily available secondary phosphines **1a–d** with elemental selenium. The initial phosphines were easily prepared from red phosphorus and styrenes<sup>12</sup> or 2-vinylnaphthalene<sup>13</sup> in one step (Scheme 2).

We found that secondary phosphines **1a–c** reacted easily with two equivalents of elemental selenium under mild conditions



Scheme 1.

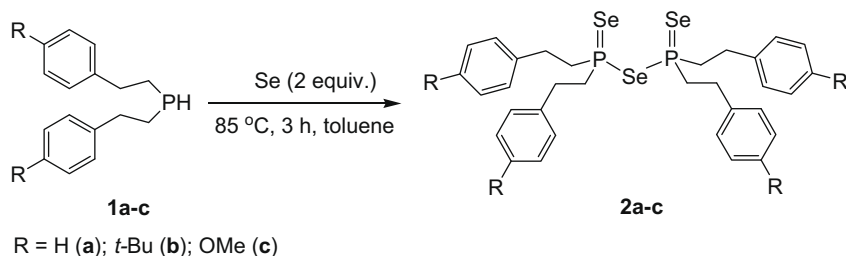


R = Ph (a), 4-*t*-BuC<sub>6</sub>H<sub>4</sub> (b), 4-MeOC<sub>6</sub>H<sub>4</sub> (c), 2-Naphth (d)

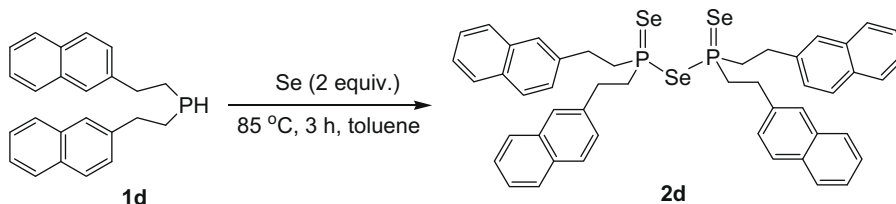
Scheme 2.

\* Corresponding author. Tel.: +7 395242 14 11; fax: +7 395241 93 46.

E-mail address: [boris\\_trofimov@irioch.irk.ru](mailto:boris_trofimov@irioch.irk.ru) (B.A. Trofimov).



Scheme 3.



Scheme 4.

(85 °C, toluene, 3 h) to give bis[di(2-arylethyl)selenophosphoryl]selenides **2a–c** in 83–92% isolated yields<sup>14</sup> (Scheme 3). The use of more than two equivalents of selenium did not affect the reaction.

Secondary bis[2-(2-naphthylethyl)]phosphine **1d** with bulky naphthyl groups was also utilized, thus demonstrating the generality of the process. Using the same conditions (85 °C, 3 h, PhMe) the reaction of phosphine **1d** with two equivalents of elemental selenium afforded selenide **2d** in 75% yield<sup>14</sup> (Scheme 4).

It is noteworthy that diselenophosphinoic acids  $\text{R}_2\text{P(Se)SeH}$  and bis(diorganoselenophosphoryl)diselenides  $(\text{R}_2\text{PSe})_2\text{Se}_2$ , as the expected products of the oxidation of secondary phosphines by two or threefold amounts of elemental selenium, were not formed under the present conditions. For example, the physico-chemical and spectral characteristics of selenide **2a** were different from those of the diselenide,  $[(\text{PhCH}_2\text{CH}_2)_2\text{PSe}]_2\text{Se}_2$ , which was synthesized independently from potassium bis(2-phenylethyl)diselenophosphinate and iodine.<sup>15</sup>

The mechanism for the formation of selenides **2** can be rationalized as follows (Scheme 5). In the first stage (1), the secondary phosphine **1** reacts with elemental selenium to furnish secondary phosphine selenide **A**. The latter, with a second equivalent of elemental selenium, gives diselenophosphinoic acid **B** (stage 2). Inter-

molecular condensation of two molecules of diselenophosphinoic acid **B** (stage 3) leads to bis(diorganoselenophosphoryl)selenide **2** and hydrogen selenide (identified by qualitative reaction with cadmium acetate).

The structures of compounds **2** were confirmed by multinuclear  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{31}\text{P}$  and  $^{77}\text{Se}$  NMR techniques. For example, the  $^{77}\text{Se}$  NMR spectra of bis[di(2-arylethyl)selenophosphoryl]selenides **2** showed a doublet of doublets integration of that of the bridging selenium with a chemical shift at 138–140 ppm ( $^1J_{\text{P}=\text{Se}} = 745\text{--}749\text{ Hz}$ ), assignable to the  $\text{P}=\text{Se}$  bond.<sup>12b,16</sup> The resonance signals of the bridging selenium atom with a chemical shift at 284–285 ppm ( $^1J_{\text{P}=\text{Se}} = 377\text{--}379\text{ Hz}$ ) appear as triplets due to the splitting of two magneto-equivalent phosphorus atoms. In the  $^{31}\text{P}$  NMR spectra of compounds **2**, the signals of the magneto-equivalent phosphorus atoms were observed as a singlet (ca. 56 ppm) showing a set of satellite signals from the  $^{77}\text{Se}$  nuclei, as is typical for such systems.<sup>17</sup> Analysis of the satellite signals allowed determination of the values of the  $^2J_{\text{P,P}}$  constants (17–10 Hz), transmitted through the bridging selenium atom.<sup>17</sup>

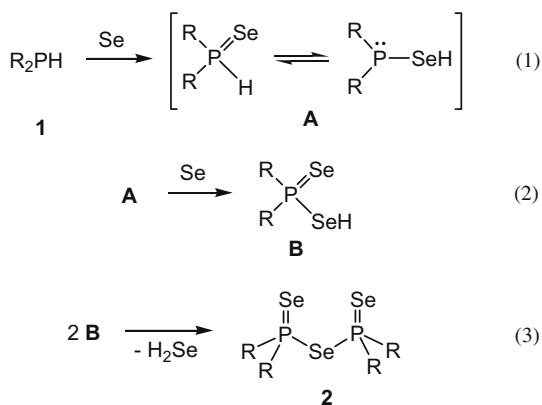
In summary, a novel and simple method for the synthesis of bis(diorganoselenophosphoryl)selenides via the unexpected reaction of secondary phosphines with elemental selenium is reported. The selenides obtained can be applied as ligands for the design of metal complexes, as potential single-source precursors for conductive, magneto-optical and other nanomaterials, as promising extractants of rare elements, as potent biologically active compounds, and as reactive building blocks for organic and elemento-organic synthesis.

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Scheme 5.

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14. **General procedure for the preparation of bis(diorganoselenophosphoryl)selenides 2a–d from secondary phosphines and elemental selenium.** To a solution of secondary phosphine **1a–d** (1.0 mmol) in toluene (8 mL), amorphous grey selenium (0.158 g, 2.0 mmol) was added at 85 °C under argon. The suspension was stirred for 3 h at 85 °C. The solvents were removed under reduced pressure and the residue was washed with cold hexane (3 × 5 mL), dried in vacuo (1 Torr, rt) to give compounds **2a–d**.
- Bis[di(2-phenethyl)selenophosphoryl]selenide 2a:** yellow powder, yield 0.331 g (92%), mp 112–114 °C (hexane). IR (KBr,  $\nu/\text{cm}^{-1}$ ): 3059, 3023, 2917, 2890, 2855, 1949, 1878, 1805, 1640, 1599, 1492, 1447, 1389, 1329, 1269, 1211, 1133, 1019, 1006, 938, 901, 825, 743, 699, 571, 501, 454.  $^1\text{H}$  NMR (400.13 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 2.61–2.69 (m, 4H,  $\text{CH}_2\text{P}$ ), 3.05–3.14 (m, 12H,  $\text{CH}_2\text{P}$ ,  $\text{CH}_2\text{Ph}$ ), 7.19–7.30 (m, 20H, Ph).  $^{13}\text{C}$  NMR (100.62 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 29.53 ( $\text{CH}_2\text{Ph}$ ), 37.88 (d,  $^1J_{\text{PC}} = 32.0$  Hz,  $\text{CH}_2\text{P}$ ), 126.32 (C-p, Ph), 128.04 (C-o, Ph), 128.40 (C-m, Ph), 139.09 (d,  $^3J_{\text{PC}} = 18.0$  Hz, C-i, Ph).  $^{31}\text{P}$  NMR (161.98 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 56.04 (+ satellites:  $^1J_{\text{P-Se}} = 378.5$  Hz,  $^1J_{\text{P-Se}} = 749.2$  Hz,  $^2J_{\text{PP}} = 19.1$  Hz).  $^{77}\text{Se}$  NMR (76.31 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : -140 (d,  $^1J_{\text{P-Se}} = 749.1$  Hz, P=Se), 284 (t,  $^1J_{\text{P-Se}} = 378.4$  Hz, P-Se-P). Calcd for  $\text{C}_{32}\text{H}_{36}\text{P}_2\text{Se}_3$ : C, 53.42; H, 5.04; P, 8.61; Se, 32.92. Found: C, 53.48; H, 5.10; P, 8.50; Se, 32.81.
- Bis[di(2-(4-tert-butylphenethyl))selenophosphoryl]selenide 2b:** yellow powder, yield 0.392 g (83%), mp 195–197 °C (hexane). IR (KBr,  $\nu/\text{cm}^{-1}$ ): 3093, 3055, 3022, 2959, 2902, 2864, 1902, 1791, 1685, 1634, 1607, 1517, 1475, 1462, 1443, 1413, 1391, 1363, 1268, 1201, 1135, 1108, 1019, 942, 927, 852, 837, 813, 769, 740, 728, 669, 563, 519, 485, 429.  $^1\text{H}$  NMR (400.13 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 1.27 (s, 36H, Me), 2.55–2.64 (m, 4H,  $\text{CH}_2\text{P}$ ), 2.96–3.09 (m, 12H,  $\text{CH}_2\text{P}$ ,  $\text{CH}_2\text{C}_6\text{H}_4$ ), 7.11–7.29 (m, 16H,  $\text{C}_6\text{H}_4$ ).  $^{13}\text{C}$  NMR (100.62 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 29.02 ( $\text{CH}_2\text{C}_6\text{H}_4$ ), 30.96 (Me), 34.04 (CMe), 37.89 (d,  $^1J_{\text{PC}} = 31.9$  Hz,  $\text{CH}_2\text{P}$ ), 125.27 (C-2 in  $\text{C}_6\text{H}_4$ ), 127.75 (C-3 in  $\text{C}_6\text{H}_4$ ), 136.02 (d,  $^3J_{\text{PC}} = 16.5$  Hz, C-1 in  $\text{C}_6\text{H}_4$ ), 149.24 (C-4 in  $\text{C}_6\text{H}_4$ ).  $^{31}\text{P}$  NMR (161.98 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 56.13 (+ satellites:  $^1J_{\text{P-Se}} = 377.1$  Hz,  $^1J_{\text{P-Se}} = 744.8$  Hz,  $^2J_{\text{PP}} = 17.6$  Hz).  $^{77}\text{Se}$  NMR (76.31 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : -139 (d,  $^1J_{\text{P-Se}} = 744.5$  Hz, P=Se), 284 (t,  $^1J_{\text{P-Se}} = 377.6$  Hz, P-Se-P). Calcd for  $\text{C}_{48}\text{H}_{68}\text{P}_2\text{Se}_3$ : C, 61.08; H, 7.26; P, 6.56; Se, 25.10. Found: C, 61.17; H, 7.34; P, 6.58; Se, 24.97.
- Bis[di(4-methoxyphenethyl)selenophosphoryl]selenide 2c:** yellow powder, yield 0.374 g (89%), mp 150–152 °C (hexane). IR (KBr,  $\nu/\text{cm}^{-1}$ ): 3126, 3099, 3061, 3027, 3004, 2955, 2929, 2910, 2858, 2834, 2595, 2545, 1610, 1582, 1512, 1463, 1440, 1420, 1397, 1318, 1246, 1176, 1130, 1032, 945, 817, 789, 736, 722, 708, 543, 528, 481, 467, 437, 400.  $^1\text{H}$  NMR (400.13 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 2.42–2.66 (m, 4H,  $\text{CH}_2\text{P}$ ), 2.85–3.11 (m, 12H,  $\text{CH}_2\text{P}$ ,  $\text{CH}_2\text{Ph}$ ), 3.76 (s, 12H, MeO), 6.8–7.11 (m, 16H,  $\text{C}_6\text{H}_4$ ).  $^{13}\text{C}$  NMR (100.62 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 28.61, 29.07 ( $\text{CH}_2\text{Ph}$ ), 38.31, 38.46, 38.65 and 38.97 ( $\text{CH}_2\text{P}$ ), 55.29 (MeO), 114.19 (C-2 in  $\text{C}_6\text{H}_4$ ), 129.45 (C-3 in  $\text{C}_6\text{H}_4$ ), 131.47 (d,  $^3J_{\text{PC}} = 16.9$  Hz, C-1 in  $\text{C}_6\text{H}_4$ ), 158.42 (C-4 in  $\text{C}_6\text{H}_4$ ).  $^{31}\text{P}$  NMR (161.98 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 55.94 (+ satellites:  $^1J_{\text{P-Se}} = 378.8$  Hz,  $^1J_{\text{P-Se}} = 746.0$  Hz,  $^2J_{\text{PP}} = 19.6$  Hz).  $^{77}\text{Se}$  NMR (76.31 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : -140 (d,  $^1J_{\text{P-Se}} = 746.0$  Hz, P=Se), 284 (t,  $^1J_{\text{P-Se}} = 378.5$  Hz, P-Se-P). Calcd for  $\text{C}_{36}\text{H}_{44}\text{O}_4\text{P}_2\text{Se}_3$ : C, 51.50; H, 5.28; P, 7.38; Se, 28.21. Found: C, 51.44; H, 5.30; P, 7.26; Se, 28.11.
- Bis[di(2-naphthylethyl)selenophosphoryl]selenide 2d:** yellow powder, yield 0.345 g (75%), mp 110–114 °C (hexane). IR (KBr,  $\nu/\text{cm}^{-1}$ ): 3050, 3020, 2924, 2855, 2330, 1957, 1800, 1634, 1597, 1506, 1445, 1367, 1277, 1125, 1018, 996, 950, 895, 858, 817, 796, 746, 477.  $^1\text{H}$  NMR (400.13 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 2.71–2.88 (m, 4H,  $\text{CH}_2\text{P}$ ), 3.12–3.38 (m, 12H,  $\text{CH}_2\text{P}$ ,  $\text{CH}_2\text{Naphth}$ ), 7.25–7.81 (m, 28H, Naphth).  $^{13}\text{C}$  NMR (100.62 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 30.20 ( $\text{CH}_2\text{Ph}$ ), 38.1 (d,  $^1J_{\text{PC}} = 32.0$  Hz,  $\text{CH}_2\text{P}$ ), 125.77, 126.34, 126.88, 127.64, 127.76, 128.59, 132.33, 133.60 (Naphth), 136.83–137.0 (d,  $^3J_{\text{PC}} = 16.9$  Hz, C-1 in Naphth).  $^{31}\text{P}$  NMR (161.98 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 55.55 (+ satellites:  $^1J_{\text{P-Se}} = 378.7$  Hz,  $^1J_{\text{P-Se}} = 749.1$  Hz,  $^2J_{\text{PP}} = 20.1$  Hz).  $^{77}\text{Se}$  NMR (76.31 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : -138 (d,  $^1J_{\text{P-Se}} = 749.4$  Hz, P=Se), 285 (t,  $^1J_{\text{P-Se}} = 378.3$  Hz, P-Se-P). Calcd for  $\text{C}_{48}\text{H}_{44}\text{P}_2\text{Se}_3$ : C, 62.69; H, 4.82; P, 6.74; Se, 25.76. Found: C, 62.50; H, 4.91; P, 6.61; Se, 25.81.
15. **Procedure for the preparation of bis[di(2-phenethyl)selenophosphoryl]diselenide.** A solution of iodine (0.127 g, 0.5 mmol) in EtOH (10 mL) was added to a solution of potassium bis(2-phenylethyl)diselenophosphinate (0.438 g, 1.0 mmol) in  $\text{H}_2\text{O}$  (10 mL) at rt under argon. The reaction mixture was stirred for 10 min, diluted with  $\text{H}_2\text{O}$  (30 mL) and extracted with toluene (2 × 25 mL). The toluene extract was dried over  $\text{K}_2\text{CO}_3$  and concentrated in vacuo to 10 mL. The remaining solution was diluted with hexane (15 mL) and kept overnight at 5–10 °C. The solvents were removed from the precipitate by decanting. The residue was washed with cold hexane (1 × 5 mL), dried in vacuo (1 Torr, rt) to give 0.323 g (81%) of bis[di(2-phenethyl)selenophosphoryl]diselenide as orange-red crystals, mp 117–118 °C (hexane–toluene). IR (KBr,  $\nu/\text{cm}^{-1}$ ): 3060, 3020, 2919, 2888, 2854, 1951, 1878, 1806, 1644, 1590, 1491, 1444, 1387, 1330, 1270, 1210, 1131, 1020, 1005, 939, 906, 825, 745, 670, 570, 507, 458.  $^1\text{H}$  NMR (400.13 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 2.78–2.91 (m, 8H,  $\text{CH}_2\text{P}$ ), 3.23–3.33 (m, 8H,  $\text{CH}_2\text{Ph}$ ), 7.37–7.48 (m, 20H, Ph).  $^{13}\text{C}$  NMR (100.62 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 29.74 ( $\text{CH}_2\text{Ph}$ ), 38.04 (d,  $^1J_{\text{PC}} = 32.0$  Hz,  $\text{CH}_2\text{P}$ ), 126.61 (C-p, Ph), 128.29 (C-o, Ph), 128.64 (C-m, Ph), 139.33 (d,  $^3J_{\text{PC}} = 17.3$  Hz, C-i, Ph).  $^{31}\text{P}$  NMR (161.98 MHz,  $\text{CDCl}_3$ , ppm),  $\delta$ : 55.64 (+ satellites:  $^1J_{\text{P-Se}} = 379$  Hz,  $^1J_{\text{P-Se}} = 747$  Hz). Calcd for  $\text{C}_{32}\text{H}_{36}\text{P}_2\text{Se}_4$ : C, 48.24; H, 4.54; P, 7.76; Se, 39.56. Found: C, 48.28; H, 4.50; P, 7.61; Se, 39.51.
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