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Radical Addition of Secondary Phosphine Chalcogenides to Allylamine: Atom-Economic Synthesis of Aminopropylphosphine Chalcogenides

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Abstract—Secondary phosphine sulfides and phosphine selenides react with allylamine under the conditions of radical initiation (UV or AIBN) to form the anti-Markovnikov adducts in up to 93 % yield.

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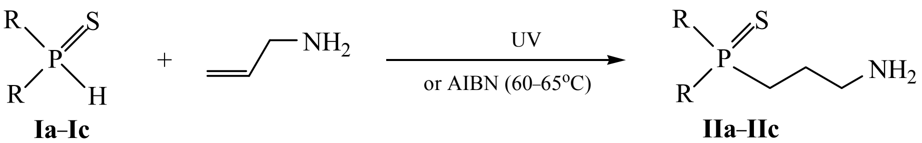
Organic phosphines and phosphine chalcogenides containing the $PCH_2CH_2CH_2NR_2$ fragment are efficient *P,N*-ligands for the design of metal complexes [1–11] and highly reactive building blocks for organic and organoelemental synthesis [1, 4, 5, 12–14]. Their complexes with metals are used, for example, as catalysts for hydrogenation of alkenes [4], ketones [6, 11] (including enantioselective hydrogenation [6]), imines [11], as well as in the Heck reaction [1]. Metal complexes of diphenylaminopropylphosphines of general formula $[M(R_1R_2N(CH_2)_3PPh_2)_2]^{2+}$ [M = Pt(II), Pd(II); R_1, R_2 = H, Me, Bn, cyclohexyl] were proposed as potential anticancer drugs [2].

A convenient approach to the synthesis of the title aminophosphines is the reaction of radical addition to the allylamine derivatives of phosphine [15–18] and the primary [10, 12, 13, 19–22] and secondary phosphines [3, 23]. At the same time, the data on the reactions of allylamines with secondary phosphine chalcogenides are scarce. The addition of dimethylphosphine oxide [24] and diphenylphosphine oxide [25] to the double bond of allylamines in the presence of azobisisobutyronitrile (AIBN) [24] or peroxides [24], or under UV radiation was briefly reported [25].

In the present work the reaction of radical addition of secondary phosphine sulfides and phosphine selenides to allylamine has been first studied with the aim to elaborate a general and convenient atom-economic method for the synthesis of functional tertiary phosphine chalcogenides containing amino group.

We have found that secondary phosphine sulfides **Ia–Ic** react with allylamine (the ratio of the reagents 1:2.5–3.5) in dioxane or benzene under UV irradiation (200-W Hg arc Lamp) or in the presence of AIBN (3 wt %, 60–65°C) in 1–3 h to form aminopropylphosphine sulfides **IIa–IIc** in 48–93% preparative yield (Scheme 1, table).

Scheme 1.



As can be seen from table, the reaction should be carried out under UV radiation in dioxane. In the presence of AIBN the addition proceeds slower and is less selective (cf. runs 1 and 3). The same effect is observed at the replacement of dioxane by benzene (cf. runs 1 and 2).

Conditions of the reaction of secondary phosphine sulfides with allylamine

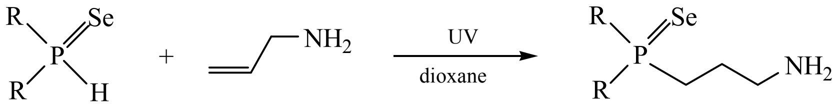
Run	Phosphine sulfide	R	Initiator	Solvent	Time, h	Adduct	Yield [a], %
1	**Ia**	Ph	UV	Dioxane	1	**IIa**	70 (48)
2	**Ia**	Ph	UV	Benzene	1	**IIa**	60 (35)
3	**Ia**	Ph	AIBN	Dioxane	2	**IIa**	64 (42)
4	**Ib**	$PhCH_2CH_2$	UV	Dioxane	2	**IIb**	96 (93)
5	**Ic**	$PhCH(Me)CH_2$	UV	Dioxane	3	**IIc**	92 (66)

[a] Yield calculated from ^{31}P NMR spectra of the crude product. Preparative yield calculated with respect to the phosphine sulfide $R_2P(S)H$ taken in the reaction is given in parentheses.

A rather low yield of phosphine sulfide **IIa** in the reaction of diphenylphosphine sulfide **Ia** with allylamine (runs 1–3) is due to the formation of side products as indicated by the presence of signals at 60–61 ppm (10–12%) and 55–56 ppm (20–26%) in the ^{31}P NMR spectra of the reaction mixtures.

Secondary phosphine selenides **IIIa, IIIb** react with allylamine under UV radiation (dioxane, 1–2.5 h) to give adducts **IVa, IVb** in ~71–76% chemoselectivity according to the ^{31}P NMR spectra (Scheme 2).

Scheme 2.

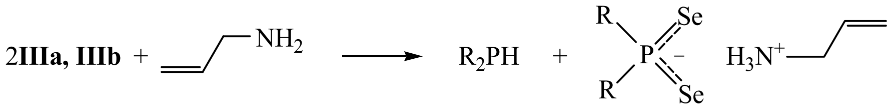


IIIa, IIIb **IVa, IVb**

R = $PhCH_2CH_2$ (**a**), $PhCH(Me)CH_2$ (**b**).

The corresponding secondary phosphines **Va, Vb** and diselenophosphinates **VIa, VIb** were also identified in the reaction mixture. In spite of the fact that their total contents does not exceed 12–15% (from ^{31}P NMR), their presence hampers the isolation of the target aminophosphine selenides **IVa, IVb** and reduces the preparative yield to 51–58%. Side products **Va, Vb, VIa, VIb** apparently are formed as shown in Scheme 3, which was described earlier by the example of the reaction of secondary phosphine selenides with amines [26].

Scheme 3.

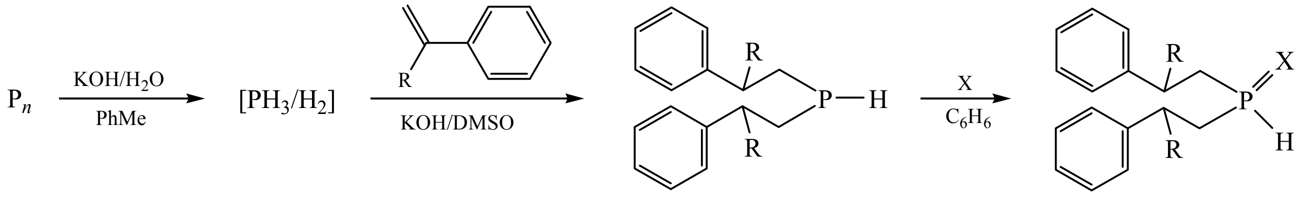


Va, Vb **VIa, VIb**

R = $PhCH_2CH_2$ (**a**), $PhCH(Me)CH_2$ (**b**).

Note that secondary phosphine oxides under these conditions (UV radiation or in the presence of AIBN) practically do not react with allylamine. This is in contrast to the results of [24, 25] cited in the introduction but is consistent with the commonly accepted notions on relatively low reactivity of secondary phosphine oxides in the reactions of radical addition to the double bond [27–29].

Scheme 4.



Ib, Ic, IIIa, IIIb

R = H, X = S (**Ib**); R = Me, X = S (**Ic**); R = H, X = Se (**IIIa**); R = Me, X = Se (**IIIb**).

It is worth mentioning that the starting secondary sulfides **Ib, Ic** and selenides **IIIa, IIIb** are now available because they are easily prepared from elemental phosphorus, styrenes, and elemental chalcogens [30, 31] (Scheme 4).

Therefore, the reaction of free-radical addition of secondary phosphine sulfides and phosphine selenides to allylamine was performed for the first time and on its basis an atom-economic method of synthesis of aminopropylphosphine chalcogenides, promising N,P=X (X = S, Se) ligands for the design of metal complex catalysts of new generation and building blocks for organoelement synthesis, was elaborated.

EXPERIMENTAL

^{1}H, ^{13}C, ^{31}P, and ^{77}Se NMR spectra were taken on a Bruker DPX 400 (400.13, 101.61, 161.98 and 76.31 MHz, respectively) in CDCl$_3$, internal references HMDS (^{1}H, ^{13}C), Me$_2$Se (^{77}Se), external reference 85% H$_3$PO$_4$ (^{31}P). The assignment of the signals in the ^{1}H, ^{13}C NMR spectra was based on the 2D homo- and heteronuclear NMR techniques: COSY, HSQC. Compounds **IIc, IVb, Vb**, and **VIb** consist of three diastereomers (one pair of enantiomers and two meso-forms) due to the presence of two asymmetric carbon atoms and the pseudoasymmetric phosphorus atom.

3-(Diphenylphosphorothioyl)propylamine (IIa) (see table, run 1). The solution of diphenylphosphine sulfide **Ia** (0.054 g, 0.25 mmol) and allylamine (0.050 g, 0.88 mmol) in 1 mL of dioxane was placed into a quartz ampule, flushed with argon, and irradiated with UV light for 1 h. The reaction mixture was passed through a layer of Al$_2$O$_3$ (2 cm) (eluent dioxane). Dioxane was removed under a reduced pressure, the residue was dried in a vacuum. 0.033 g (48%) of 3-(diphenylphosphorothioyl)propylamine **IIa** was obtained as light-yellow oil. ^{1}H NMR, δ_H, ppm: 1.30 br.s (2H, NH$_2$), 1.72 m (2H, $CH_2C\underline{H}_2CH_2$), 2.49 m (2H, CH$_2$P), 2.74 t (2H, CH$_2$N, $^3J_{HH}$ 6.7 Hz), 7.43 m (6H, *m*-Ph, *p*-Ph), 7.81 m (4H, *o*-Ph, $^3J_{HP}$ 13.0 Hz). ^{13}C NMR, δ_C, ppm: 26.21 d ($CH_2\underline{C}H_2CH_2$, $^2J_{CP}$ 2.6 Hz), 30.02 d (CH$_2$P, $^1J_{CP}$ 57.5 Hz), 42.58 d (CH$_2$N, $^3J_{CP}$ 17.3 Hz), 128.53 d (C$_m$, $^3J_{CP}$ 12.2 Hz), 131.01 d (C$_o$, $^2J_{CP}$ 10.3 Hz), 131.38 d (C$_p$, $^4J_{CP}$ 3.0 Hz), 132.80 d (C$_i$, $^1J_{CP}$ 80.0 Hz). ^{31}P NMR, δ_P, ppm: 43.8. Found, %: C 65.65; H 6.50; N 5.07; P 10.95; S 11.39. $C_{15}H_{18}NPS$. Calculated, %: C 65.43; H 6.59; N 5.09; P 11.25; S 11.65.

3-(Diphenethylphosphorothioyl)propylamine (IIb) (see table, run 4). The solution of bis(2-phenethyl)phosphine sulfide **Ib** (0.089 g, 0.32 mmol) and allylamine (0.064 g, 1.12 mmol) in 1 mL of dioxane was placed into a quartz ampule, flushed with argon, and irradiated with UV light for 2 h. The reaction mixture was passed through a layer of Al$_2$O$_3$ (2 cm) (eluent dioxane). Dioxane was removed under a reduced pressure, the residue was dried in a vacuum. 0.099 g (93%) of 3-(diphenethylphosphorothioyl)propylamine **IIb** was obtained as light-yellow oil. ^{1}H NMR, δ_H, ppm: 1.72 m (2H, $CH_2C\underline{H}_2CH_2$), 1.85 m (2H, $CH_2CH_2C\underline{H}_2P$), 2.12 m (4H, $PhCH_2C\underline{H}_2P$), 2.73 t (2H, CH$_2$N, $^3J_{HH}$ 6.7 Hz), 2.92 m (4H, CH$_2$Ph), 7.18 m (4H, *o*-Ph), 7.20 m (2H, *p*-Ph), 7.28 m (4H, *m*-Ph). ^{13}C NMR, δ_C, ppm: 26.20 d ($CH_2\underline{C}H_2CH_2$, $^2J_{CP}$ 3.4 Hz), 28.59 d (CH$_2$Ph, $^2J_{CP}$ 2.7 Hz), 28.72 d ($CH_2CH_2\underline{C}H_2P$, $^1J_{CP}$ 50.5 Hz), 32.76 d ($PhCH_2\underline{C}H_2P$, $^1J_{CP}$ 48.6 Hz), 42.58 d (CH$_2$N, $^3J_{CP}$ 16.1 Hz), 126.58 (C$_p$), 128.29 (C$_o$), 128.73 (C$_m$), 140.64 d (C$_i$, $^3J_{CP}$ 13.8 Hz). ^{31}P NMR, δ_P, ppm: 49.6. Found, %: C 68.58; H 7.86; N 3.99; P 9.18; S 9.48. $C_{19}H_{26}NPS$. Calculated, %: C 68.85; H 7.91; N 4.23; P 9.34; S 9.67.

3-[Bis(2-phenylpropyl)phosphorothioyl]propylamine (IIc) (see table, run 5). The solution of bis(2-phenylpropyl)phosphine sulfide **Ic** (0.094 g, 0.31 mmol) and allylamine (0.044 g, 0.77 mmol) in 1 mL of dioxane was placed into a quartz ampule, flushed with argon, and irradiated with UV light for 3 h. The reaction mixture was passed through a layer of Al$_2$O$_3$

(2 cm) (eluent dioxane). Dioxane was removed under a reduced pressure, the residue was dried in a vacuum. 0.063 g (66%) of 3-[bis(2-phenylpropyl)phosphorothioyl)propylamine **IIc** was obtained as light-yellow oil. ^{1}H NMR, δ_H, ppm: 1.12 d, 1.12 d, 1.30 d, 1.33 d (6H, Me, $^3J_{HH}$ 7.2 Hz), 1.22 m (2H, $CH_2C\underline{H}_2CH_2$), 1.77 m (2H, $CH_2C\underline{H}_2P$), 1.71–2.08 m, 1.82–2.08 m, 1.93–2.08 m (4H, $C^*HC\underline{H}_2P$), 2.31 m, 2.33 m, 2.40 m (2H, CH_2N), 3.02 m, 3.25 m, 3.39 m, 3.41 m (2H, C^*H), 7.15–7.26 m (10H, Ph). ^{13}C NMR, δ_C, ppm: 24.75 d, 24.85 d, 24.88 d, 24.97 d (Me, $^3J_{CP}$ 5.6, 2.2, 4.2 and 2.2 Hz, respectively), 26.12 d, 26.66 d, 27.03 d ($\underline{C}H_2CH_2P$, $^2J_{CP}$ 3.6, 4.0 and 4.2 Hz, respectively), 28.63 d, 29.80 d, 29.94 d ($CH_2\underline{C}H_2P$, $^1\underline{J}_{CP}$ 48.9, 50.9 and 50.2 Hz, respectively), 34.29 d, 34.58 d, 34.62 d, 35.05 d (C^*H, $^2J_{CP}$ 2.8, 2.8, 3.0 and 3.3 Hz, respectively), 38.01 d, 39.03 d, 39.81 d, 39.94 d ($C^*H\underline{C}H_2P$, $^1J_{CP}$ 47.9, 47.2, 48.9 and 49.2 Hz, respectively), 42.42 d, 42.55 d, 42.60 d (CH_2N, $^3J_{CP}$ 17.5, 17.1 and 17.1 Hz, respectively), 126.61, 126.72, 126.75, 126.76 (C_p), 127.09, 127.23, 127.25 (C_o), 128.54, 128.59, 128.67, 128.69 (C_m), 145.71 d, 145.95 d, 146.01 d, 146.10 d (C_i, $^3J_{CP}$ 4.3, 4.6, 4.7 and 4.2 Hz, respectively). ^{31}P NMR, δ_P, ppm: 49.0, 49.1, 49.6. Found, %: C 70.32; H 8.40; N 3.68; P 8.34; S 9.19. $C_{21}H_{30}NPS$. Calculated, %: C 70.16; H 8.41; N 3.90; P 8.62; S 8.92.

3-(Diphenylethylphosphoroselenoyl)propylamine (IVa). The solution of bis(2-phenylethyl)phosphine selenide **IIIa** (0.103 g, 0.32 mmol) and allylamine (0.028 g, 0.49 mmol) in 1 mL of dioxane was placed into a quartz ampule, flushed with argon, and irradiated with UV light for 1 h. In the ^{31}P NMR spectrum of the reaction mixture the signals were observed at –69.05 ppm ($^1J_{PH}$ 199 Hz), 24.85 ppm (satellites: $^1J_{PSe}$ 556 Hz), 37.50 ppm (satellites: $^1J_{PSe}$ 696.3 Hz), from the secondary phosphine **Va**, diselenophosphinate **VIa** (identified with the authentic sample [32]) and adduct **IVa**, as well as unidentified signals at 65.52 and 66.03 ppm (the ratio of signals 2:2:25:1:3, respectively). Dioxane was removed under a reduced pressure. The oily residue was washed with hexane (3×0.5 mL), the residue was extracted with isopropanol, isopropanol was removed from the extract under a reduced pressure to give 0.07 g (58%) of 3-(diphenethylphosphoroselenoyl)propylamine **IVa** as light-yellow oil. ^{1}H NMR, δ_H, ppm: 1.85 m (2H, $CH_2C\underline{H}_2CH_2$), 1.99 m (2H, $CH_2CH_2C\underline{H}_2P$), 2.19 m (4H, $PhCH_2C\underline{H}_2P$), 2.88 m (6H, CH_2N, CH_2Ph), 4.71 br.s (2H, NH_2), 7.16 m (6H, *o*-Ph, *p*-Ph), 7.24 m (4H, *m*-Ph). ^{13}C NMR, δ_C, ppm: 25.10 d ($CH_2\underline{C}H_2CH_2$, $^2J_{CP}$ 3.1 Hz), 28.04 d ($CH_2CH_2\underline{C}H_2P$, $^1J_{CP}$ 44.3 Hz), 29.28 d (CH_2Ph, $^2J_{CP}$ 2.7 Hz), 32.39 d ($PhCH_2\underline{C}H_2P$, $^1J_{CP}$ 41.6 Hz), 41.27 d (CH_2N, $^3J_{CP}$ 16.1 Hz), 126.56 (C_p), 128.27, 128.67 (C_o, C_m), 140.21 d (C_i, $^3J_{CP}$ 13.8 Hz). ^{31}P NMR, δ_P, ppm: 37.5 (+ satellites: $^1J_{PSe}$ 696.3 Hz). ^{77}Se NMR, δ_{Se}, ppm: –384.2 d ($^1J_{SeP}$ 696.3 Hz). Found, %: C 60.59; H 7.15; N 3.52; P 7.90; Se 20.91. $C_{19}H_{26}NPSe$. Calculated, %: C 60.32; H 6.93; N 3.70; P 8.19; Se 20.87.

3-[Bis(2-phenylpropyl)phosphoroselenoyl]propylamine (IVb). The solution of bis(2-phenylpropyl) phosphine selenide **IIIb** (0.100 g, 0.29 mmol) and allylamine (0.025 g, 0.44 mmol) in 1 mL of dioxane was placed into a quartz ampule, flushed with argon, and irradiated with UV light for 2.5 h. In the ^{31}P NMR spectrum of the reaction mixture the signals are observed at –82.61, –81.70, –80.79 ppm ($^1J_{PH}$ ~200 Hz), 23.70, 24.69, 26.10 ppm (satellites: $^1J_{PSe}$ ~580 Hz) and 37.96, 38.54 38.78 ppm (satellites: $^1J_{PSe}$ ~690 Hz) from the secondary phosphine **Vb**, diselenophosphinate **VIb**, and adduct **IVb**, and unidentified signals at 63–67 ppm (the content of the target product **IVb** ~71%). Dioxane was removed under a reduced pressure, the residue was reprecipitated from ether into cold hexane. The formed oil was separated and dried in a vacuum to give 0.06 g (51%) of 3-[bis(2-phenylpropyl)phosphoroselenoyl]propylamine **IVb** as light-yellow oil. ^{1}H NMR, δ_H, ppm: 1.12 d, 1.12 d, 1.30 d, 1.33 d (6H, Me, $^3J_{HH}$ 7.0 Hz), 1.23 m (2H, $CH_2C\underline{H}_2CH_2$), 1.77 m (2H, $CH_2C\underline{H}_2P$), 1.70–2.08 m, 1.83–2.07 m, 1.92–2.08 m (4H, $C^*HC\underline{H}_2P$), 2.24 m, 2.34 m, 2.39 m (2H, CH_2N), 3.01 m, 3.26 m, 3.39 m, 3.40 m (2H, C^*H), 7.10–7.27 m (10H, Ph). ^{13}C NMR, δ_C, ppm: 24.71 d, 24.82 d, 24.84 d, 24.94 d (Me, $^3J_{CP}$ 5.0, 4.6, 3.8 and 4.6 Hz, respectively), 24.11 d, 24.17 d, 24.23 d ($\underline{C}H_2CH_2P$, $^2J_{CP}$ 4.2, 4.2 and 3.8 Hz, respectively), 27.57 d, 28.56 d, 28.89 d ($CH_2\underline{C}H_2P$, $^1J_{CP}$ 41.8, 44.1 and 43.7 Hz, respectively), 35.13 d, 35.51 d, 35.57 d, 35.85 d (C^*H, $^2J_{CP}$ 2.3, 2.7, 2.7 and 3.5 Hz, respectively), 37.50 d, 38.54 d, 39.45 d ($C^*H\underline{C}H_2P$, $^1J_{CP}$ 41.4, 40.3, and 42.1 Hz, respectively), 40.11 d, 40.20 d, 40.40 d (CH_2N, $^3J_{CP}$ 19.2, 17.6 and 17.3 Hz respectively), 126.64, 126.72, 126.76 (C_p), 127.14, 127.21, 127.31 (C_o), 128.64, 128.69, 128.75 (C_m), 145.32 d, 145.62 d, 145.67 d, 145.78 d (C_i, $^3J_{CP}$ 3.8, 4.2, 5.0 and 4.2 Hz, respectively). ^{31}P NMR, δ_P, ppm: 38.2 (+ satellites: $^1J_{PSe}$ 691.8 Hz), 38.7 (+ satellites: $^1J_{PSe}$ 683.8 Hz), 38.8 (+ satellites: $^1J_{PSe}$ 696.3 Hz). ^{77}Se NMR, δ_{Se}, ppm: –375.6 d ($^1J_{SeP}$ 691.8 Hz), –365.4 d ($^1J_{SeP}$ 683.8 Hz), –348.9 d ($^1J_{SeP}$ 696.3 Hz). Found, %: C 61.81; H 7.26; N 3.21; P 7.74;

Se 19.22. $C_{21}H_{30}NPSe$. Calculated, %: C 62.06; H 7.44; N 3.45; P 7.62; Se 19.43.

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