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One-Pot Vinylation of Secondary Phosphine Chalcogenides with Vinyl Sulfoxides

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ONE-POT VINYLATION OF SECONDARY PHOSPHINE CHALCOGENIDES WITH VINYL SULFOXIDES

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A facile, one-pot vinylation of secondary phosphine chalcogenides with alkyl(or aryl) vinyl sulfoxides has been elaborated. The vinylation comprises the nucleophilic addition of secondary phosphine chalcogenides to the vinyl sulfoxides (~50 mol% KOH, dioxane, 25–40°C, 1 h) followed by the elimination of sulfenic acids from the adducts (additional equivalent of KOH, 60–70°C, 1.5–2.0 h), the yields of target tertiary vinyl phosphine chalcogenides reaching 92%.

Keywords Secondary phosphine chalcogenides; tertiary vinyl phosphine chalcones; vinyl sulfoxides; vinylation

INTRODUCTION

Vinyl phosphine chalcogenides are highly reactive building blocks that readily add various nucleophiles (amines,¹ phosphines,² thiols,^{1c} carbanions³) and other⁴ reagents to afford functionalized phosphine chalcogenides. The latter are widely applied as ligands for the design of advanced catalysts,⁵ flame retardants,⁶ extractants of rare earth, transuranic and noble elements,⁷ and coordinating solvents for the synthesis of conductive nanomaterials.⁸

Conventional syntheses of vinyl phosphine chalcogenides⁹ employ toxic and hazardous phosphorus halides and organometallics under inert atmosphere, and are laborious and do not meet modern ecological requirements. In the example of *tert*-butylphenylphosphine oxide and 4-methylphenyl vinyl sulfoxide, the synthesis of *tert*-butylphenylvinylphosphine oxide was briefly noted.¹⁰ However, experimental details as well as proofs of the structure of the vinylphosphine oxide formed are not given in this communication.¹⁰ The synthesis¹¹ of two tertiary bis(arylalkyl)vinylphosphine oxides in low (19–34%) yields by the elimination of sulfenic acids from the adducts of secondary phosphine oxides to alkyl vinyl sulfoxides has also been reported. Consequently, this version

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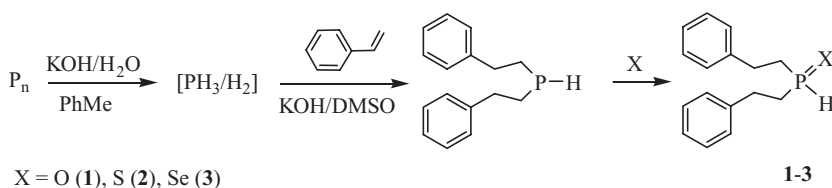
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of reaction could not be considered as synthetically useful. Meanwhile, in principal, such an approach, when further properly elaborated up to a high-yield version and extended to phosphine sulfides and phosphine selenides as well as to aryl vinyl sulfoxides, could become general efficient synthesis of vinylphosphine chalcogenides.

To reach this goal, in this work we have thoroughly studied the nucleophilic addition of secondary phosphine chalcogenides to organyl vinyl sulfoxides followed by the elimination of sulfenic acids from the intermediary adducts without their isolation. Eventually, our efforts led to elaboration of the one-pot synthesis of tertiary vinyl phosphine chalcogenides, mostly in high preparative yield.

RESULTS AND DISCUSSION

As representative secondary phosphine chalcogenides, bis[2-(phenyl)ethyl]phosphine oxide (**1**), bis[2-(phenyl)ethyl]phosphine sulfide (**2**), and bis[2-(phenyl)ethyl]phosphine selenide (**3**), directly prepared from red phosphorus and styrene¹² have been chosen (Scheme 1).



Scheme 1

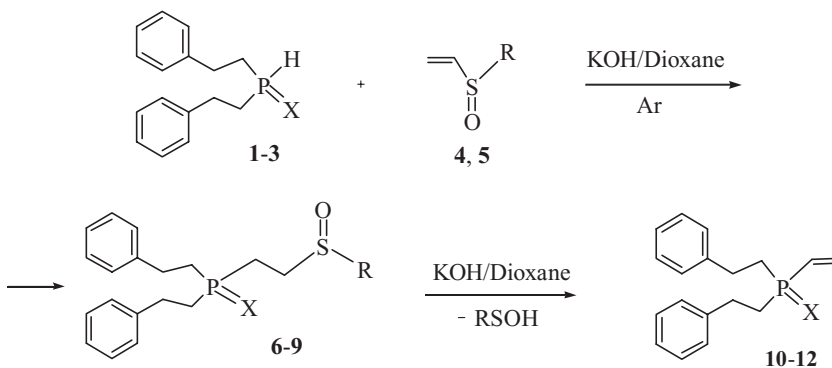
It was found that phosphine chalcogenides **1–3** added to ethyl vinyl sulfoxide (**4**) or phenyl vinyl sulfoxide (**5**) under mild conditions (~ 0.5 equivalent of KOH, dioxane, 25–40°C, 1 h) to give regioselectively adducts **6–9** (Stage 1). The latter, without isolation, have been further heated (60–70°C, 1.5–2.0 h) in the presence of an additional amount (~ 1 equivalent) of KOH (Stage 2) to afford the target vinylphosphine chalcogenides **10–12** in 87%, 92%, and 30% yields, respectively (Scheme 2).

The decreased yield of vinyl phosphine selenide **12** is due to selenium extrusion from starting **3**, intermediary **8**, and final **12** products. Elemental selenium thus precipitated reacts with secondary phosphine selenide **3** and KOH to furnish the potassium salt of phosphinic acid **13** (identified by ^{31}P NMR technique using external standard¹³), as shown in Scheme 3.

The vinylation of secondary phosphine chalcogenides **1–3** involves their addition to organyl vinyl sulfoxides **4**, **5**, and elimination of organyl sulphenic acids from the adducts **6–9**. The sulfenic acids in the presence of KOH are transformed to salts **14** and **15** (Scheme 4).

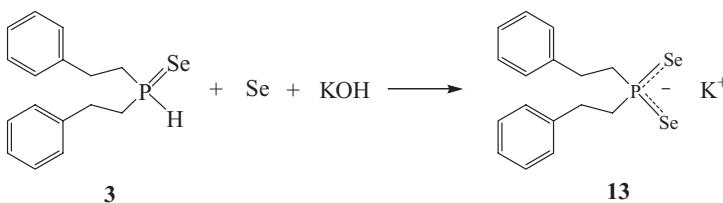
Salt **14** was isolated in the mixture with KOH. The data of potentiometric acidimetric titration have shown that the content of salt **14** in the mixture reaches 92%.

The excess KOH (~ 1.5 equivalent) in this vinylation is crucial to tie up the released sulfenic acids, which, otherwise, could add back to the active double bond of vinylphosphine chalcogenides **10–12** to restore the adducts **6–9**. Indeed, with ~ 0.5 equivalent of KOH, both the conversion of the adducts **6–9** and the yields of vinylated phosphine chalcogenides **10–12** drop significantly (^{31}P NMR monitoring).

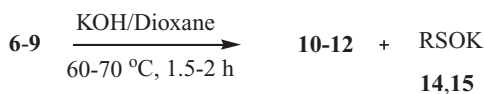


R = Et, X = O (**1**, **4**, **6**, **10**); R = Et, X = S (**2**, **4**, **7**, **11**); R = Et, X = Se (**3**, **4**, **8**, **12**); R = Ph, X = S (**2**, **5**, **9**, **11**)

Scheme 2



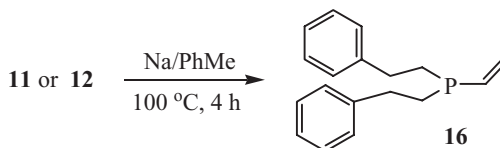
Scheme 3



R = Et (**14**); Ph (**15**)

Scheme 4

The previously unknown vinylphosphine chalcogenides **11** and **12** which are now available prove to be convenient precursors of the novel vinylphosphine **16**. Thus, the reduction of vinyl sulfide **11** and vinyl selenide **12** with sodium metal in toluene (100°C, 4 h) proceeds smoothly and almost quantitatively (Scheme 5).



Scheme 5

Surprisingly, vinylphosphine oxide **10**, under the same conditions, was found to be practically inert (the conversion was near to zero). In this case, the conventional protocol¹⁴ for the phosphine oxide reduction with SiHCl_3 also failed; a complex mixture of

various products containing neither initial vinylphosphine oxide **10**, nor the expected vinyl phosphine **16**, was formed.

In conclusion, in the examples of ethyl vinyl and phenyl vinyl sulfoxides, it has been shown that organyl vinyl sulfoxides are convenient and effective vinyl synthons for the secondary phosphine oxides, phosphine sulfides, and phosphine selenides and allow for preparation of hitherto unknown or scarcely available tertiary vinylphosphine chalcogenides. The latter are recognized to be reactive building blocks for organic synthesis and convenient models for physicochemical studies, e.g., for conformational analysis and stereochemical dependences of ^{31}P – ^1H spin–spin coupling constants of di(2-phenethyl)vinyl phosphine and related phosphine chalcogenide.¹⁵

EXPERIMENTAL

IR spectra were measured with a Bruker IFS 25 instrument in microlayers or KBr (cm^{-1}). ^1H , ^{13}C , and ^{31}P NMR spectra were recorded on a Bruker DPX-400 spectrometer (400.13, 100.61, and 161.98 MHz, respectively) in CDCl_3 solutions and referenced to internal HMDS (^1H), CHCl_3 (^{13}C), and external 85% H_3PO_4 (^{31}P). Two-dimensional homo- and heteronuclear NMR correlation experiments (NOESY, HSQC) were used to assign the signals in ^1H and ^{13}C NMR spectra. The reaction was monitored by ^{31}P NMR (decrease of signals in the region of 3–33 ppm for phosphine chalcogenides **1–3** and increase of signals at 33–43 ppm for vinyl phosphine chalcogenides **10–12**). Potentiometric titration in water was performed on an EV-74 instrument at 20°C using glass chlorosilver electrodes filled with aqueous solution of LiCl and AgCl salts, with 0.1 N aqueous solution of HCl being a titrant. The concentration of the salt studied ranged 5–10 mmol. The concentration basicity constant of the salt studied was determined by potentiometry in water⁶ using imidazole as a standard.

Synthesis of Tertiary Phosphine Oxide **10**

A mixture of phosphine oxide **1** (1.4 mmol), sulfoxide **4** (1.4 mmol), and powdered $\text{KOH}\cdot 0.5\text{H}_2\text{O}$ (0.7 mmol) in dioxane (10 mL) was stirred at ambient temperature for 1 h and analyzed by ^{31}P NMR. In the spectrum, a signal of initial phosphine oxide **1** at 28.07 ppm disappeared, and a signal at 47.89 ppm attributable to the adduct **6** was observed (^{31}P NMR using external standard¹⁰). To the reaction mixture, $\text{KOH}\cdot 0.5\text{H}_2\text{O}$ (1.4 mmol) was also added, and the suspension was stirred at 60°C for 2 h. In the ^{31}P NMR spectrum, a signal at 36.62 ppm assigned to vinyl phosphine oxide **10** was detected. The reaction mixture was cooled, the residue was filtered off, dioxane was removed, and the product obtained was dried under lowered pressure to give 0.35 g (87%) of vinyl phosphine oxide **10** (purified by recrystallization from acetone).

Bis(2-phenethyl)vinylphosphine Oxide (**10**)

Colorless crystals, 0.35 g (87%) yield, mp 92°C (acetone). ^1H NMR, δ (ppm), J (Hz): 1.99–2.11 (m, 4H, CH_2P), 2.86–2.94 (m, 4H, CH_2Ph), {6.14 [1H, H(3)], 6.25 [1H, H(1)], 6.38 [1H, H(2)]}, $^2J_{\text{H}(1)\text{H}(2)} = 1.4$, $^3J_{\text{H}(1)\text{H}(3)} = 12.6$, $^3J_{\text{H}(2)\text{H}(3)} = 18.5$, $^2J_{\text{PH}(3)} = 28.0$, $^3J_{\text{PH}(1)} = 37.9$, $^3J_{\text{PH}(2)} = 20.8$, 7.18–7.28 (m, 10H, Ph). ^{13}C NMR, δ (ppm), J (Hz): 27.60 (d, $^2J_{\text{PC}} = 3.0$, CH_2Ph), 31.60 (d, $^1J_{\text{PC}} = 67.0$, $\text{PCH}_2\text{CH}_2\text{Ph}$), 126.50 (C-*p*), 128.10 (C-*o*), 128.70 (C-*m*), 130.70 (d, $^1J_{\text{PC}} = 86.8$, $=\text{CH}_2$), 135.20 ($=\text{CH}$), 141.0 (d, $^3J_{\text{PC}} = 14.1$,

C-*i*). ^{31}P NMR, δ (ppm): 36.0. IR (KBr), ν , cm^{-1} : 1603 ($\text{C}=\text{C}_{\text{vinyl}}$); 1167 ($\text{P}=\text{O}$). Anal. Calcd. for $\text{C}_{18}\text{H}_{21}\text{OP}$: C, 75.35; H, 7.24; P, 9.93. Found: C, 76.04; H, 7.44; P, 10.89.

Synthesis of Tertiary Phosphine Sulfide 11

A mixture of phosphine sulfide **2** (2.2 mmol), sulfoxide **4** (2.2 mmol), and powdered $\text{KOH}\cdot 0.5\text{H}_2\text{O}$ (1.1 mmol) in dioxane (10 mL) was stirred at 25°C for 1 h. In the spectrum, a signal of initial phosphine sulfide **2** at 22.24 ppm disappeared, and a signal at 48.92 ppm assigned to the adduct **7** was observed (^{31}P NMR using external standard^{12a}). To the reaction mixture, $\text{KOH}\cdot 0.5\text{H}_2\text{O}$ (0.12 g, 2.2 mmol) was added, and the resulting mixture was then heated at 70°C for 1.5 h. In the ^{31}P NMR spectrum, a signal at 42.21 ppm assigned to vinyl phosphine oxide **11** was detected. Next, the precipitate was filtered off, the solvent was removed, the residue was dried in vacuum, and the product obtained was reprecipitated from diethyl ether to hexane to afford 0.60 g (yield 91%) of vinyl phosphine sulfide **11**.

The residue was subsequently washed with dioxane and ether, dried in vacuum to give 0.17 g of the product.

The product obtained was analyzed by potentiometric acidimetric titration in aqueous solution. The titration curve shows two jumps, the first one corresponding to the neutralization of excess KOH, the second one relating to the neutralization of potassium salt of ethanesulfenic acid **14**. The basicity of potassium ethanesulfenate (**14**) (the value of the acid dissociation constant $\text{pK}_a = 6.18$) is significantly weaker than that of potassium hydroxide totally ionized in water. According to the potentiometric titration data, the product contains 92% of this salt.

Vinyl phosphine sulfide **11** was synthesized in 92% yield from secondary phosphine sulfide **2** and phenyl vinyl sulfoxide **5** under the same conditions.

Bis(2-phenethyl)vinylphosphine Sulfide (11)

Viscous, paraffin-like, light-yellowish product, 0.60 g (91%). ^1H NMR, δ (ppm), J (Hz): 2.13–2.28 (m, 4H, CH_2P), 2.79–3.06 (m, 4H, CH_2Ph), {6.22 [1H, H(3)], 6.25 [1H, H(1)], 6.47 [1H, H(2)]}, $^2J_{\text{H}(1)\text{H}(2)} = 1.6$, $^3J_{\text{H}(1)\text{H}(3)} = 11.5$, $^3J_{\text{H}(2)\text{H}(3)} = 17.6$, $^2J_{\text{PH}(3)} = 26.8$, $^3J_{\text{PH}(1)} = 44.0$, $^3J_{\text{PH}(2)} = 24.4$, 7.20–7.33 (m, 10H, Ph). ^{13}C NMR, δ (ppm), J (Hz): 28.30 (d, $^2J_{\text{PC}} = 2.1$, CH_2Ph), 34.20 (d, $^1J_{\text{PC}} = 52.8$, CH_2P), 126.50 (C-*p*), 128.30 (C-*o*), 128.70 (C-*m*), 130.10 (d, $^1J_{\text{PC}} = 69.9$, $\text{CH}_2=$), 135.20 (d, $^2J_{\text{PC}} = 1.1$, $=\text{CH}$), 140.8 (d, $^3J_{\text{PC}} = 15.1$, C-*i*). ^{31}P NMR, δ (ppm): 42.52. IR, ν , cm^{-1} : 1602 ($\text{C}=\text{C}_{\text{vinyl}}$); 543 ($\text{P}=\text{S}$). Anal. Calcd. for $\text{C}_{18}\text{H}_{21}\text{PS}$: C, 71.82; H, 6.99; P, 10.91; S, 10.51. Found: C, 71.97; H, 7.05; P, 10.31; S 10.67.

Synthesis of Tertiary Phosphine Selenide 12

A mixture of 0.64 g phosphine selenides **3** (0.21 g, 2.0 mmol), sulfoxide **4** (2.0 mmol), and ground KOH (0.06 g, 1.0 mmol) in dioxane (10 mL) was stirred at 40°C for 1 h. In the spectrum, a signal of initial phosphine selenide **3** at 2.99 ppm disappeared, and the signals at 36.49 ppm assigned tentatively to phosphine selenide **8**, 33.36 ppm related to vinyl phosphine oxide **12**, and 24.70 ppm corresponding to potassium salt of phosphinic acid were observed (^{31}P NMR using external standard¹³). To the reaction mixture, KOH (0.11 g, 2.0 mmol) was added, and the mixture was heated at 70°C for 1.5 h. The ^{31}P NMR spectrum showed the signals at 33.36 ppm assigned to vinyl phosphine oxide **12** and

24.70 ppm assigned to potassium salt of phosphinic acid. The reaction mixture was cooled, diethyl ether (5 mL) was added, the suspension obtained was filtered off, the solvent was removed, the product was dissolved in chloroform and reprecipitated in hexane, and the latter was removed to give 0.21 g (yield 30%) of phosphine selenide **12**.

The residue was consequently washed with chloroform, dioxane, and water and dried in air to give 0.01 g of elemental selenium.

Bis(2-phenethyl)vinylphosphine Selenide (**12**)

Light-yellowish oil. ^1H NMR, δ (ppm), J (Hz): 2.22–2.31 (m, 4H, CH_2P), 2.71–3.06 (m, 4H, CH_2Ph , {6.21 [1H, H(3)], 6.25 [1H, H(1)], 6.47 [1H, H(2)]}, $^2J_{\text{H(1)H(2)}} 2.2$, $^3J_{\text{H(1)H(3)}} 11.6$, $^3J_{\text{H(2)H(3)}} 17.6$, $^3J_{\text{PH(3)}} 24.6$, $^3J_{\text{PH(1)}} 45.4$, $^3J_{\text{PH(2)}} 24.6$ }, 7.17–7.26 (m, 10H, Ph). ^{13}C NMR, δ (ppm), J (Hz): 29.10 (d, $^2J_{\text{PC}} = 2.2$, CH_2Ph), 33.91 (d, $^1J_{\text{PC}} = 45.4$, CH_2P), 126.60 (C-*p*), 128.30 (C-*o*), 128.70 (C-*m*), 128.80 (d, $^1J_{\text{PC}} = 75.4$, $=\text{CH}_2$), 137.60 (d, $^2J_{\text{PC}} = 2.2$, $=\text{CH}$), 140.6 (d, $^3J_{\text{PC}} = 15.3$, C-*i*). ^{31}P NMR, δ (ppm): 33.26. ^{77}Se NMR (76.31 MHz, CDCl_3): $\delta = -433.0$ (d, $^1J_{\text{PSe}} = 716.1$ Hz). IR, ν , cm^{-1} : 1602 ($\text{C}=\text{C}_{\text{vinyl}}$); 448 ($\text{P}=\text{Se}$). Anal. Calcd. for $\text{C}_{18}\text{H}_{21}\text{PSe}$: C, 61.82; H, 6.13; P, 9.01; Se, 22.51. Found: C, 62.25; H, 6.09; P, 8.92; S 22.74.

Synthesis of Tertiary Phosphine **16**

The suspension of phosphine sulfide **11** (1.5 mmol) or phosphine selenide (1.5 mmol) **12** and sodium (4.5 mmol) in toluene (5 mL) was vigorously stirred at 100°C for 4 h. In the ^{31}P NMR spectrum, the signals at 41.21 ppm assigned to phosphine sulfide **11** or 33.09 ppm assigned to phosphine selenide **12** disappeared, and a signal at -26.37 ppm corresponding to phosphine **16** appeared. The reaction mixture was cooled, filtered off, and toluene was removed to afford phosphine **16**.

Bis(2-phenethyl)vinylphosphine (**16**)

Light-yellowish oil, 0.4 g (yield 90%). ^1H NMR, δ (ppm), J (Hz): 1.67–1.71 (m, 4H, CH_2P), 2.68–2.74 (m, 4H, CH_2Ph), {6.14 [1H, H(1)], 6.18 [1H, H(2)], 6.58 [1H, H(1), H(3)], $^2J_{\text{H(1)H(2)}} = 2.2$, $^3J_{\text{H(1)H(3)}} = 11.7$, $^3J_{\text{H(2)H(3)}} = 18.4$, $^3J_{\text{PH(3)}} = 5.8$, $^3J_{\text{PH(1)}} = 30.4$, $^3J_{\text{PH(2)}} = 13.17$ }, 7.11–7.25 (m, 10H, Ph). ^{13}C NMR, δ (ppm), J (Hz): 32.56 (d, $^2J_{\text{PC}} = 14.0$, CH_2Ph), 29.73 (d, $^1J_{\text{PC}} 13.2$, CH_2P), 126.02 (C-*p*), 127.80 (C-*o*), 128.40 (C-*m*), 140.0 (d, $^1J_{\text{PC}} = 19.6$, $\text{CH}_2=$), 128.10 (d, $^2J_{\text{PC}} = 19.6$, $\text{CH}=\text{}$), 143.11 (d, $^3J_{\text{PC}} 10.0$ C-*i*). ^{31}P NMR, δ (ppm): -26.80 . IR, ν , cm^{-1} : 1603 ($\text{C}=\text{C}_{\text{vinyl}}$). Anal. Calcd. for $\text{C}_{18}\text{H}_{21}\text{P}$: C, 80.82; H, 7.99; P, 11.21. Found: C, 80.57; H, 7.89; P, 11.54.

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