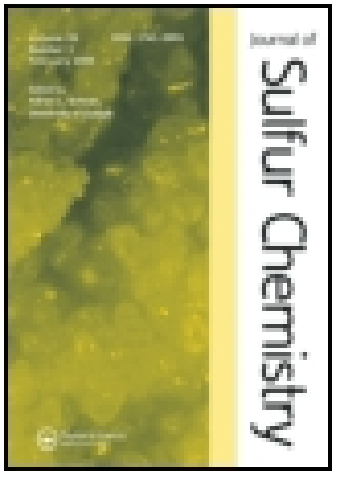

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Ludmila A. Oparina[a], Oksana V. Vysotskaya[a], Nikita A. Kolyvanov[a], Alexander V. Artem'ev[a], Nina K. Gusarova[a] & Boris A. Trofimov[a]
[a] A. E. Favorsky Irkutsk Institute of Chemistry, Siberian Branch of the Russian Academy of Sciences, Irkutsk, Russian Federation
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A. E. Favorsky Irkutsk Institute of Chemistry, Siberian Branch of the Russian Academy of Sciences, Irkutsk, Russian Federation

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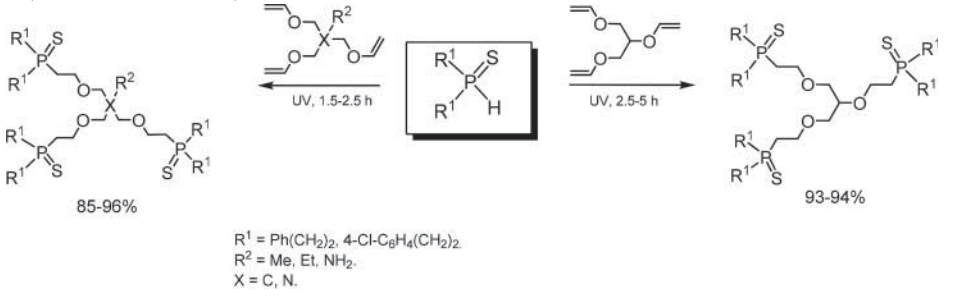
The tertiary polyfunctional triphosphine sulfides with amino and (or) ether groups have been synthesized in excellent yields by the exhaustive regioselective (in anti-Markovnikov manner) addition of secondary phosphines sulfides to trivinyl ethers of aminotriols and triols under free-radical conditions (UV-irradiation, 1.5–5 h).



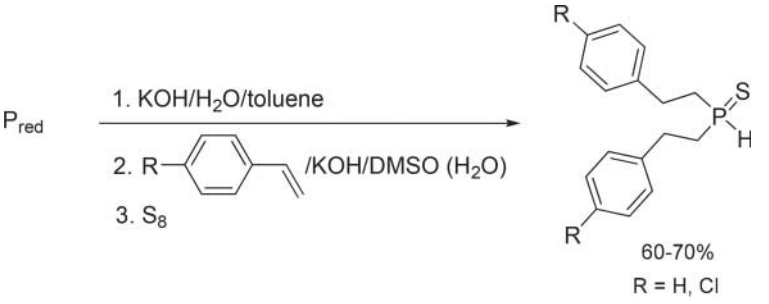
Keywords: secondary phosphine sulfides; trivinyl ethers; addition; tertiary triphosphine sulfides

1. Introduction

The tertiary phosphine sulfides are important organophosphorus compounds, which find diverse application. The are widely used as extractants of noble metals and radionuclides,[1–4] ligands for catalytically active metal complexes,[5–9] capping agents for stabilization of metal chalcogenide nanoparticles [10–12] as well as reagents for organic synthesis.[13] Furthermore, tertiary phosphine sulfides can serve as modifiers of rubbers and resins,[14] chemical sensitizers in photographic materials,[15,16] additives to lubricating oils and electrolytes.[17]

Less available and hence less understood are the triphosphine sulfides, though they are intriguing ligands for synthesis of useful metal complexes,[18] some of which show promising results in methanol carbonylation reaction.[19,20] Therefore, synthesis and investigation of new triphosphine sulfides represent challenging task in organophosphorus chemistry.

*Corresponding author. Email: boris_trofimov@irioch.irk.ru



Scheme 1. Elemental phosphorus-based synthesis of secondary phosphine sulfides.

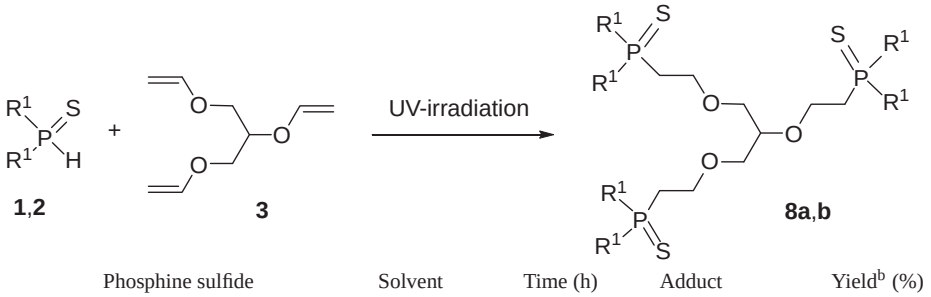
As a promising approach to the synthesis of novel functional 'tripod-like' triphosphine sulfides might be a reaction of radical addition of secondary phosphine sulfides to trivinyl ethers of triols, which earlier has been successfully realized on the example of secondary phosphines.[21] Starting trivinyl ethers of triols are readily assessable via the direct vinylation of the triols with acetylene in superbasic catalytic systems like KOH/DMSO.[22–25] Secondary phosphine sulfides are also easily prepared from red phosphorus, styrenes and elemental sulfur according to Scheme 1.[26,27]

2. Results and discussion

This paper is devoted to the analysis of the experimental results obtained for the reaction of secondary phosphine sulfides **1**, **2** with trivinyl ethers of glycerol **3**, triols **4**, **5** and aminotriols **6**, **7**. The phosphorylation has been carried out via exhaustive free-radical addition protocol: the molar ratio of secondary phosphine sulfides **1**, **2**:trivinyl ethers **3–7** = 3:1, UV-irradiation, inert atmosphere, and organic solvent (acetonitrile or 1,4-dioxane).

Under these conditions, phosphine sulfides **1**, **2** react with trivinyl ether **3** for 2.5–5 h to afford in 93–94% yield triphosphine sulfides **8a**,**b** separated by three-dimensional alkane triol spacers (Table 1). Notably, the reaction proceeds chemo- and regioselectively: no corresponding

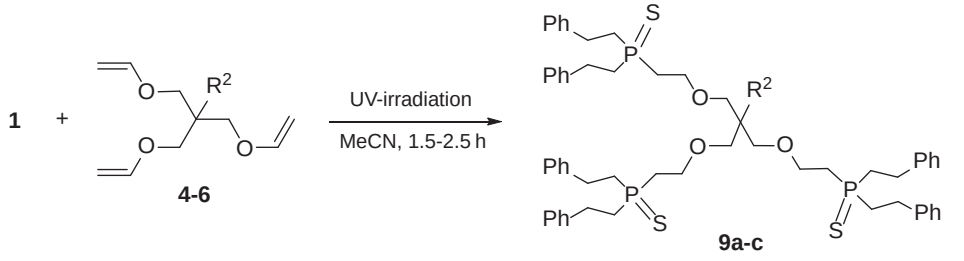
Table 1. Exhaustive free-radical addition of secondary phosphine sulfides to trivinyl ether of glycerol **3**[a].

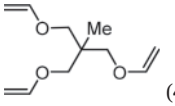
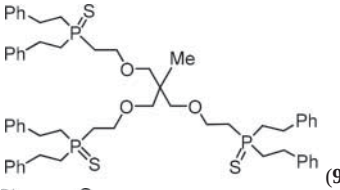
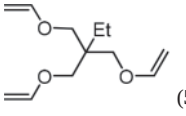
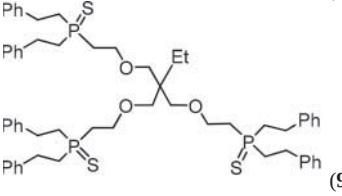
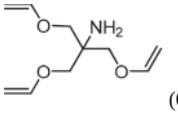
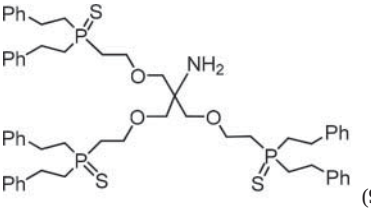


Entry	Phosphine sulfide	Solvent	Time (h)	Adduct	Yield[b] (%)
1	$Ph(CH_2)_2P(S)H$ (**1**)	Acetonitrile	2.5	**8a**	93
2	4-Cl-$C_6H_4(CH_2)_2P(S)H$ (**2**)	1,4-dioxane	5	**8b**	94

[a]Standard reaction conditions: molar ratio **1**,**2**/**3** = 3:1, argon. UV-irradiation (200 W Hg arc lamp).
[b]Isolated yield.

Table 2. Exhaustive free-radical addition of secondary phosphine sulfide 1 to trivinyl ether of triols **4–6**[a].



Entry	Trivinyl ether	Adduct	Time (h)	Yield[b] (%)
1	 (**4**)	 (**9a**)	2.5	90
2	 (**5**)	 (**9b**)	2.5	85
3	 (**6**)	 (**9c**)	1.5	95

[a]Standard reaction conditions: molar ratio **1**/**4**–**6** = 3:1, argon. UV-irradiation (200 W Hg arc lamp).
[b]Isolated yield.

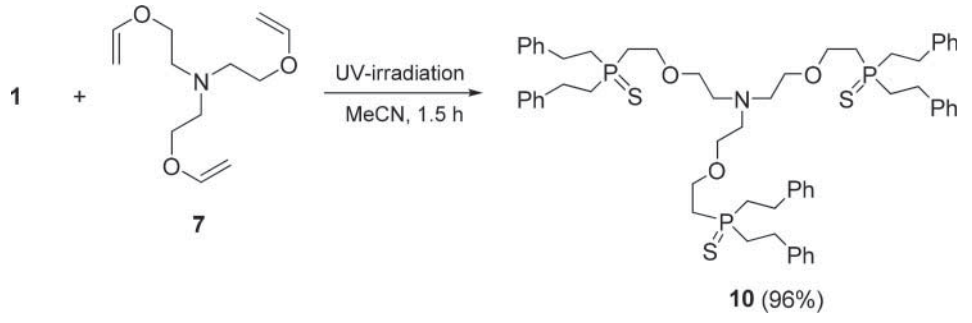
mono-, di- and Markovnikov adducts as well as cyclization and telomerization products have been observed (^{1}H and ^{31}P NMR).

Using phosphine sulfide **1** as an example, we have shown that under UV-irradiation during 1.5–2.5 h, phosphorylation of trivinyl ether **4–6** is also realized chemo- and regioselectively to give functional 'tripod-like' triphosphine sulfides **9a–9c** in 85–95% yield (Table 2).

Besides, the general character of the reaction studied is supported by the fact that trivinyl ether of aminotriol **7** adds three molecules of phosphine sulfide **1** (UV-irradiation, 1.5 h, acetonitrile) to form in 96% yield triphosphine sulfide **10** with amino and ether groups (Scheme 2).

3. Conclusion

In summary, the atom-economic chemo- and regioselective synthesis of highly branched polyfunctional triphosphine sulfides with amino and (or) ether groups has been developed by exhaustive free-radical addition of secondary phosphine sulfides to trivinyl ethers of aminotriols



Scheme 2. Exhaustive free-radical addition of secondary phosphine sulfides **1** to *N,N,N*-tris[2-(vinyloxy) ethyl]amine **7**.

or triols thus providing a facile short-cut to a new family of prospective tripodal ligands for the design of multi-purpose metal complexes.

4. Experimental

4.1. *General*

All reactions were carried out under an argon atmosphere. All solvents were dried and/or purified according to standard procedures. Secondary phosphine sulfides **1**, **2** were synthesized from red phosphorus and styrenes.[26,27] Trivinyl ethers **3–7** were prepared according to a published method.[22–25] The ^{1}H, ^{13}C and ^{31}P NMR spectra were recorded on a Bruker DPX 400 and Bruker AV-400 spectrometers (400.13, 100.62 and 161.98 MHz, respectively) at ambient temperature for $CDCl_3$ solutions. Chemical shifts were reported in δ (ppm) relative to $CDCl_3$ (^{1}H, ^{13}C) as the internal standard or H_3PO_4 (^{31}P) as the external standard. IR-FT spectra were taken on a Bruker Vertex 70 spectrometer. The C, H, S microanalyses were performed on a Flash EA 1112 CHNS-O/MAS analyzer, while the P content was determined by the combustion method. The content of chlorine in **8b** was determined by mercurimetric titration. Melting points (uncorrected) were recorded on a 'Stuart melting point apparatus'.

4.2. *General procedure for the synthesis of triphosphine sulfides 8–10*

A solution of phosphine sulfide **1**, **2** (0.9 mmol) and trivinyl ether **3–7** (0.3 mmol) in solvent (0.5 ml) was irradiated (200 W Hg arc lamp) in a quartz ampoule (the reaction time is given in Tables 1 and 2). The reaction was monitored by ^{31}P NMR spectroscopy following the disappearance of the peaks of the starting phosphine sulfides **1**, **2** (the 20–21 ppm region) and the appearance of new peaks in the 48–49 ppm region corresponding to triphosphine sulfides **8–10**. The reaction mixture was dissolved in diethyl ether (3 mL), and passed through a layer of basic Al_2O_3 (activity level II, 0.5 cm), the latter was additionally washed with 3 mL of n-hexane/diethyl ether mixture (1:1). The solvents were removed under reduced pressure to give the triphosphine sulfides **8–10**.

4.2.1. *2-{2,3-Bis[2-(diphenethylphosphorothioyl)ethoxy]propoxy}ethyl(diphenethyl)-phosphine sulfide (**8a**)*

Colorless oil; yield: 0.277 g (93%). ^{1}H NMR (400.13 MHz, $CDCl_3$): $\delta = 1.89–2.04$ (m, 6 H, PCH_2CH_2O), 2.08–2.17 (m, 12 H, PCH_2), 2.82–2.92 (m, 12 H, $PhCH_2$), 3.40–3.51 (m, 5 H,

CH_2O, CHO), 3.68–3.88 (m, 6 H, PCH_2CH_2O), 7.16–7.28 (m, 30 H, Ph). ^{13}C NMR (100.62 MHz, $CDCl_3$): $\delta = 28.52$ ($PhCH_2$), 31.27 and 33.54 (2 d, $^1J_{PC} = 50.2$ and 49.1 Hz, PCH_2), 63.93 (d, $^2J_{PC} = 2.4$ Hz, CH_2O), 65.32 (d, $^2J_{PC} = 2.3$ Hz, CH_2O), 70.65 (CH_2O), 77.82 (CHO), 126.45 (C_p in Ph), 128.18 (C_o in Ph), 128.64 (C_m in Ph), 140.67 (d, $^3J_{PC} = 14.2$ Hz, C_i in Ph). ^{31}P NMR (161.98 MHz, $CDCl_3$): $\delta = 49.00$ (br s). IR (film, ν, cm^{-1}): 753 (P – C), 600 (P = S). Anal. Calcd for $C_{57}H_{71}O_3P_3S_3$: C, 68.92; H, 7.20; P, 9.35; S, 9.68. Found: C, 68.78%; H, 7.15%; P, 9.30%; S, 9.73%.

4.2.2. *2-(2,3-Bis{2-[bis(4-chlorophenethyl)phosphorothioyl]ethoxy}propoxy)ethyl-[bis(4-chlorophenethyl)]phosphine sulfide (**8b**)*

Colorless oil; yield: 0.338 g (94%). ^1H NMR (400.13 MHz, $CDCl_3$): $\delta = 1.72$–2.08 (m, 18 H, PCH_2), 2.76–2.83 (m, 12 H, CH_2Ar), 3.35–3.42 (m, 5 H, CH_2O, CHO), 3.73–3.82 (m, 6 H, PCH_2CH_2O), 7.00–7.16 (m, 24 H in Ar). ^{13}C NMR (100.62 MHz, $CDCl_3$): $\delta = 27.72$ (CH_2Ar), 30.79 and 33.30 (2 d, $^1J_{PC} = 50.0$ and 48.3 Hz, PCH_2), 65.06 (d, $^2J_{PC} = 2.4$ Hz, CH_2O), 66.81 (d, $^2J_{PC} = 2.3$ Hz, CH_2O), 70.99 (CH_2O), 78.97 (CHO), 128.56 (C-2,6 in Ar), 129.43 (C-3,5 in Ar), 132.01 (C-4 in Ar), 138.87 (d, $^3J_{PC} = 14.2$ Hz, C-1 in Ar). ^{31}P NMR (161.98 MHz, $CDCl_3$): $\delta = 48.09$ (br s). IR (film, ν, cm^{-1}): 775 (P – C), 653 (P = S). Anal. Calcd for $C_{57}H_{65}Cl_6O_3P_3S_3$: C, 57.05; H, 5.46; Cl, 17.73; P, 7.74; S, 8.02. Found: C, 57.42%; H, 5.12%; Cl, 17.53%; P, 7.82%; S, 8.13%.

4.2.3. *2-(3-[2-(Diphenethylphosphorothioyl)ethoxy]-2-[2-(diphenethylphosphorothioyl)-ethoxy]methyl-2-methylpropoxy)ethyl(diphenethyl)phosphine sulfide (**9a**)*

Colorless oil; yield: 0.276 g (90%). ^1H NMR (400.13 MHz, $CDCl_3$): $\delta = 0.82$ (s, 3 H, Me), 2.03–2.09 (m, 6 H, PCH_2CH_2O), 2.17–2.24 (m, 12 H, PCH_2), 2.96 (dt, $^3J_{PH} = 9.8$, $^3J_{HH} = 7.2$ Hz, 12 H, $PhCH_2$), 3.16 (s, 6 H, CH_2O), 3.62 (dt, $^3J_{PH} = 18.4$, $^3J_{HH} = 5.9$ Hz, 6 H, PCH_2CH_2O), 7.20–7.29 (m, 30 H, Ph). ^{13}C NMR (100.62 MHz, $CDCl_3$): $\delta = 17.85$ (Me), 28.66 ($PhCH_2$), 31.69 and 33.83 (2 d, $^1J_{PC} = 49.9$ and 48.3 Hz, PCH_2), 40.58 (C), 65.50 (d, $^2J_{PC} = 2.8$ Hz, CH_2O), 73.89 (CCH_2O), 126.59 (C_p in Ph), 128.31 (C_o in Ph), 128.78 (C_m in Ph), 140.76 (d, $^3J_{PC} = 14.4$ Hz, C_i in Ph). ^{31}P NMR (161.98 MHz, $CDCl_3$): $\delta = 48.40$. IR (film, ν, cm^{-1}): 758 (P – C), 599 (P = S). Anal. Calcd for $C_{59}H_{75}O_3P_3S_3$: C, 69.38; H, 7.40; P, 9.10; S, 9.42. Found: C, 69.40%; H, 7.72%; P, 9.29%; S, 9.58%.

4.2.4. *2-(2,2-Bis[2-(diphenethylphosphorothioyl)ethoxy]methylbutoxy)ethyl-(diphenethyl)phosphine sulfide (**9b**)*

Colorless oil; yield: 0.264 g (85%). ^1H NMR (400.13 MHz, $CDCl_3$): $\delta = 0.71$ (t, $^3J = 7.4$ Hz, 3 H, Me), 1.22 (q, $^3J_{HH} = 7.4$ Hz, 2 H, CH_2Me), 2.02–2.07 (m, 6 H, PCH_2CH_2O), 2.15–2.22 (m, 12 H, PCH_2), 2.93 (dt, $^3J_{PH} = 9.5$, $^3J_{HH} = 7.2$ Hz, 12 H, $PhCH_2$), 3.14 (s, 6 H, CH_2O), 3.62 (dt, $^3J_{PH} = 18.4$, $^3J_{HH} = 6.0$ Hz, 6 H, PCH_2CH_2O), 7.18–7.31 (m, 30 H, Ph). ^{13}C NMR (100.62 MHz, $CDCl_3$): $\delta = 7.59$ (Me), 23.01 (CH_2Me), 28.64 ($PhCH_2$), 31.40 and 33.54 (2 d, $^1J_{PC} = 50.3$ and 48.3 Hz, PCH_2), 42.93 (C), 65.44 (d, $^2J_{PC} = 3.6$ Hz, CH_2O), 71.49 (CCH_2O), 126.57 (C_p in Ph), 128.28 (C_o in Ph), 128.75 (C_m in Ph), 140.875 (d, $^3J_{PC} = 14.4$ Hz, C_i in Ph). ^{31}P NMR (161.98 MHz, $CDCl_3$): $\delta = 48.47$. IR (film, ν, cm^{-1}): 753 (P – C), 600 (P = S). Anal. Calcd for $C_{60}H_{77}O_3P_3S_3$: C, 69.60; H, 7.50; P, 8.97; S, 9.29. Found: C, 69.40%; H, 7.72%; P, 8.59%; S, 9.14%.

4.2.5. *2-[2-(Diphenethylphosphorothioyl)ethoxy]-1,1-bis[2-(diphenethylphosphorothioyl)ethoxy]methylethylamine (**9c**)*

White powder; yield: 0.291 g (95%); m.p. 90°C. ^{1}H NMR (400.13 MHz, $CDCl_3$): $\delta = 1.39$ (br s, 2 H, NH_2), 1.96–2.02 (m, 6 H, PCH_2CH_2O), 2.08–2.15 (m, 12 H, PCH_2), 2.85–2.91 (m, 12 H, $PhCH_2$), 3.16 (s, 6 H, OCH_2), 3.61 (dt, $^3J_{PH} = 17.5$, $^3J_{HH} = 6.2$ Hz, 6 H, PCH_2CH_2O), 7.13–7.26 (m, 30 H, Ph). ^{13}C NMR (100.62 MHz, $CDCl_3$): $\delta = 28.20$ ($PhCH_2$), 31.10 and 33.50 (2 d, $^1J_{PC} = 50.3$ and 48.3 Hz, PCH_2), 55.43 (CNH_2), 65.24 (d, $^2J_{PC} = 2.4$ Hz, CH_2O), 72.89 (OCH_2C), 126.20 (C_p in Ph), 127.86 (C_o in Ph), 128.37 (C_m in Ph), 140.25 (d, $^3J_{PC} = 14.4$ Hz, C_i in Ph). ^{31}P NMR (161.98 MHz, $CDCl_3$): $\delta = 48.25$. IR (KBr, ν, cm^{-1}): 3375, 3288 (N – H), 752 (P – C), 599 (P = S). Anal. Calcd for $C_{58}H_{74}NO_3P_3S_3$: C, 68.14; H, 7.30; N, 1.37; P, 9.09; S, 9.41. Found: C, 68.40%; H, 7.62%; N, 1.44%; P, 9.56%; S, 9.08%.

4.2.6. *N,N,N-tris2-[2-(diphenethylphosphorothioyl)ethoxy]ethylamine (**10**)*

Colorless oil; yield: 0.303 g (96%). ^{1}H NMR (400.13 MHz, $CDCl_3$): $\delta = 2.01$–2.06 (m, 6 H, PCH_2CH_2O), 2.10–2.16 (m, 12 H, PCH_2), 2.59 (t, $^3J = 5.8$ Hz, 6 H, NCH_2), 2.86 (dt, $^3J_{PH} = 8.9$, $^3J_{HH} = 8.3$ Hz, 12 H, $PhCH_2$), 3.35 (t, $^3J_{HH} = 5.8$ Hz, 6 H, OCH_2), 3.68 (dt, $^3J_{PH} = 17.6$, $^3J_{HH} = 6.1$ Hz, 6 H, PCH_2CH_2O), 7.13–7.25 (m, 30 H, Ph). ^{13}C NMR (100.62 MHz, $CDCl_3$): $\delta = 28.25$ ($PhCH_2$), 31.40 and 33.26 (2 d, $^1J_{PC} = 49.5$ and 48.3 Hz, PCH_2), 54.17 (NCH_2), 64.61 (d, $^2J_{PC} = 2.8$ Hz, CH_2O), 69.30 (d, $^2J_{PC} = 2.4$ Hz, CH_2O), 126.15 (C_p in Ph), 127.88 (C_o in Ph), 128.33 (C_m in Ph), 140.39 (d, $^3J_{PC} = 14.4$ Hz, C_i in Ph). ^{31}P NMR (161.98 MHz, $CDCl_3$): $\delta = 48.47$. IR (film, ν, cm^{-1}): 752 (P – C), 599 (P = S) cm^{-1}. Anal. Calcd for $C_{60}H_{78}NO_3P_3S_3$: C, 68.61; H, 7.48; N, 1.33; P, 8.85; S, 9.16. Found: C, 68.40%; H, 7.72%; N, 1.62%; P, 8.39%; S, 9.61%.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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