Synthesis of oxazolidinylphosphine chalcogenides from aminoethyl vinyl ethers

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N-(2-Vinyloxyethyl)phosphinothioamides and -phosphinoselenoamides prepared by oxidative cross-coupling of 2-vinyloxyethylamine with secondary phosphine chalcogenides undergo thermal (75—100 °C) cyclization into the corresponding 3-(diorganylchalcogenophosphoryl)-2-methyl-1,3-oxazolidines in 80—90% yields.

Key words: vinyl ethers, amino alcohols, phosphinochalcogenoic acids, intramolecular cyclization, 1,3-oxazolidines, organophosphorus compounds.

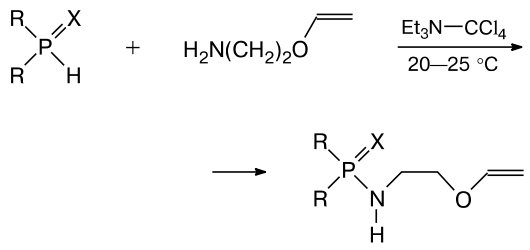
The chemistry of 1,3-oxazolidines and their derivatives has been extensively developed in the last decade.[1—16] They are used as precursors to drugs,[5—7,13,16] monomers and comonomers for the synthesis of special-purpose polymer materials,[17—20] and building blocks in organic synthesis.[4,8,13,21] For instance, the antitumor drug docetaxel is prepared from substituted 1,3-oxazolidine-5-carboxylates[3,16] and functionalized 1,3-oxazolidin-4-ones serve as selective cyclooxygenase-2 inhibitors.[10] 1,3-Oxazolidine structures are part of compounds exhibiting cardioprotectant,[5] antidiabetic,[22] antispasmodic,[23] antimicrobial,[6,9,24] herbicidal,[25] fungicidal,[11] and anthelmintic[12] activity. Oxazolidine-containing urethane formulations and the corresponding materials are highly resistant to hydrolysis.[8,17,19,20] 1,3-Oxazolidines are components of epoxy resins employed as impregnants for strengthening solid surfaces.[18]

A general method of constructing the 1,3-oxazolidine heterocycle involves cyclocondensation of β-amino alcohols with aldehydes or ketones (aminoacetalization).[1,6,9,13] The synthesis of 1,3-oxazolidines by isomerization (intramolecular cyclization) of β-aminoalkyl vinyl ethers (in the presence of Lewis acids) or that of organic acid amides containing the 2-vinyloxyethyl group has been reported. The latter isomerization is catalyzed by protic acids. In addition, the 1,3-oxazolidine ring can be constructed by thermal treatment (160—200 °C) of some functionalized *N*-(2-vinyloxyethyl)amides of carboxylic,[26] carbamic,[27] and dithiocarbamic[28,29] acids (in most cases, this process occurs during vacuum distillation of these amides).

The literature data on functionalized 1,3-oxazolidines containing organophosphorus substituents are scarce.[30—35] It has been reported that 1,3-oxazolidines with the fragment 2´-$C_6H_4PPh_2$ in position 2 are employed as P,N-ligands in the synthesis of efficient Pt- and Pd-based metal complex catalysts for asymmetric allylic alkylation[30,33—35] and the Diels—Alder reaction.[32,33] 1,3-Oxazolidines containing the organophosphorus substituent $P(=X)R_2$ directly bound to the N atom have not been documented prior to our investigations.[36]

In the present work, we propose a convenient route to earlier unknown 3-(diorganylchalcogenophosphoryl)-2-methyl-1,3-oxazolidines that involves thermal isomerization of *N*-(2-vinyloxyethyl)phosphinothioamides and -phosphinoselenoamides. The latter have recently become accessible through efficient oxidative cross-coupling of secondary phosphine chalcogenides with 2-vinyloxyethylamine[36] (Scheme 1).

Scheme 1



R = Ar, $ArCH_2CH_2$; X = O, S, Se

We found that heating of *N*-(2-vinyloxyethyl)amides **1a—e** (75—100 °C, 3.5—15 h, toluene, inert atmosphere) causes their easy and smooth cyclization into isomeric 3-(diorganylchalcogenophosphoryl)-2-methyl-1,3-oxazolidines **2a—e** in preparative 80—90% yields (Scheme 2,

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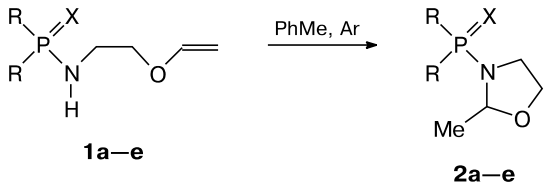
Table 1). The reaction proceeds as noncatalytic regioselective intramolecular hydroamination of the vinyloxy group.

Scheme 2



1a—e **2a—e**

R = Ph (**a**, **b**), $Ph(CH_2)_2$ (**c**, **d**), $PhCH(Me)CH_2$ (**e**)
X = S (**a**, **c**), Se (**b**, **d**, **e**)

Conditions: 75—100 °C, 3.5—15 h.

The course of the reactions was monitored using ^{31}P NMR spectroscopy: the integral intensities of the signals for the starting compounds **1a—e** (δ_P 58—66) decrease, with simultaneously increasing integral intensities of the signals for N-phosphorylated 2-methyl-1,3-oxazolidines **2a—e** (δ_P 64—72).

Because of the strong electron-withdrawing effect of the chalcogenophosphoryl group, *N*-(2-vinyloxyethyl)amides **1a—e** are stronger NH acids than amides of carboxylic and carbamic acids[26—29] and hence more easily undergo intramolecular electrophilic addition. It can be seen in Table 1 that the cyclization rate of selenophosphoryl-containing amides **1b**,**d** is higher than that of thiophosphoryl-containing amides **1a**,**c**, which can be attributed to better stabilization of the transition state by the bulky group $R_2P{=}Se$.

The structures of compounds **2a—e** were proved by ^1H, ^{13}C, ^{31}P, and ^{77}Se NMR spectroscopy and 2D NMR experiments (HMBC, $^{13}C—^1H$ HSQC, and $^{15}N—^1H$ HMBC). The $^{15}N—^1H$ HMBC spectra of these compounds show cross-peaks for the N atom and the protons of the methyl and OCH_2 groups (for compounds **2c—e**, an additional cross-peak reveals a correlation between the N atom and the PCH_2 protons).

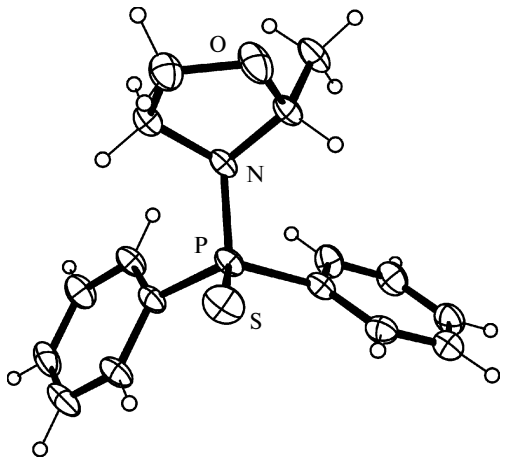


Fig. 1. One of two crystallographically independent molecules of compound **2a** according to X-ray diffraction data.

One of two crystallographically independent molecules of compound **2a** is depicted in Fig. 1 (X-ray diffraction data). The phenyl rings are planar; the five-membered ring adopts a conformation of a slightly distorted envelope. The molecular packing of compound **2a** in the crystal shows no shortened van der Waals contacts. Numerous intermolecular H...π-interactions are worth noting (the distances from the H atoms to the planes of the adjacent π-systems are 2.74—2.94 Å).

To sum up, we conducted and studied the intramolecular cyclization of *N*-(2-vinyloxyethyl)diorganylphosphinochalcogenoamides. The reactions proceed quantitatively at 75—100 °C to give 3-(diorganylchalcogenophosphoryl)-2-methyl-1,3-oxazolidines, which are promising ligands for the preparation of metal complex catalysts, potential drug precursors, and reactive building blocks for use in organic synthesis.

Experimental

IR spectra were recorded on a Bruker Vertex spectrometer (thin film and KBr pellets). ^1H, ^{13}C, ^{15}N, ^{31}P, and ^{77}Se NMR spectra were recorded on Bruker DPX-400 and Bruker AV-400

Table 1. Isomerization of *N*-(2-vinyloxyethyl)diorganylphosphinochalcogenoamides into the corresponding 2-methyl-1,3-oxazolidines

Entry	**1a—e**	R	X	*T*/°C	τ/h	Oxazolidine **2a—e**	Yield (%)
1	**1a**	Ph	S	75	15	**2a**	86
2	**1b**	Ph	Se	75	7	**2b**	90
3	**1c**	$Ph(CH_2)_2$	S	75	10	**2c***	80
4	**1d**	$Ph(CH_2)_2$	Se	75	3.5	**2d***	90
5	**1e**	$PhCH(Me)CH_2$	Se	100	12	**2e**	89

* The synthesis of compounds **2c** and **2d** has been briefly described in the study[36] dealing with oxidative cross-coupling of aminoalkyl vinyl ethers with secondary phosphine chalcogenides.

spectrometers (400.13, 100.62, 40.56, 161.98, and 76.31 MHz, respectively) in $CDCl_3$ with reference to $SiMe_4$ (^1H, ^{13}C), $MeNO_2$ (^{15}N), H_3PO_4 (^{31}P), and Me_2Se (^{77}Se). For signal assignments and determination of the structures of the compounds obtained, heteronuclear 2D NMR procedures were used ($^{13}C—^1H$ HSQC and HMBC). ^{15}N NMR spectra were recorded using 2D NMR experiments ($^{15}N—^1H$ HMBC). All steps of experimental work were carried out under an inert atmosphere (argon). Toluene was used as a solvent without further purification.

Single crystals of compound **2a** were obtained by crystallization from hexane. An X-ray diffraction experiment was performed with a single crystal (0.70×0.25×0.03 mm) on a Bruker KAPPA APEX II diffractometer equipped with a CCD area detector (Mo-Kα radiation, graphite monochromator, ω—φ scan mode, $2\theta \leq 50°$) at −73 °C. The crystals of compound **2a** are triclinic, a = 9.334(1) Å, b = 12.715(2) Å, c = 13.950(2) Å, $\alpha = 90.340(6)°$, $\beta = 93.266(6)°$, $\gamma = 108.219(5)°$, $V = 1569.6(4)$ Å^3, space group $P\bar{1}$, Z = 4, $C_{16}H_{18}NOPS$, d_{calc} = 1.284 g cm^{-3}, $\mu = 0.303$ mm^{-1}. The intensities of 5619 unique reflections were measured. Absorption corrections were applied with the SADABS program[37] (transmittance 0.53—0.97). The structure was solved by the direct methods with the SHELXS-97 program[38] and refined anisotropically by the full-matrix least-squares method with the SHELXL-97 program.[38] In each refinement cycle, the H atoms were located geometrically with respect to the coordinates of their parent carbon atoms. Final refinement of the structure was performed for all F^2 (wR_2 = = 0.2596, S = 1.10); the number of parameters refined is 363 (R = 0.0898 for 3947 $F > 4\sigma$). The rather high R factor is due to the poor quality of the crystals; the peak widths are 2.5—3.0° (unfortunately, better crystals were unavailable). The CIF file comprising comprehensive data on structure **2a** has been deposited with the Cambridge Structural Database (CCDC No. 906645).*

The starting compounds *N*-(2-vinyloxyethyl)phosphinochalcogenoamides **1a—e** were prepared as described earlier.[36]

3-(Diorganylchalcogenophosphoryl)-2-methyl-1,3-oxazolidines (2a—e) (general procedure). A solution of *N*-(2-vinyloxyethyl)phosphinochalcogenoamide **1a—e** (0.6 mmol) in toluene (2.3 mL) was heated in a sealed tube (75—100 °C, 3.5—15 h, argon) (see Table 1). The resulting reaction mixture was passed through a short column with Al_2O_3 (Brockmann activity II, column height 1.5 cm, toluene as an eluent). The solvent was removed under reduced pressure. The residue was evacuated to give compounds **2a—e**.

3-(Diphenylthiophosphoryl)-2-methyl-1,3-oxazolidine (2a). Yield 163 mg (86%), m.p. 76—77 °C (from hexane). Found (%): C, 63.29; H, 5.96; N, 4.61; P, 10.17; S, 10.54. $C_{16}H_{18}NOPS$. Calculated (%): C, 63.35; H, 5.98; N, 4.62; P, 10.21; S, 10.57. IR, ν/cm^{-1}: 1151, 1124, 1104, 1075 (O—C—N); 696 (P—N—C); 615 (P=S). ^1H NMR, δ: 1.07 (d, 3 H, Me, $^3J_{H,H}$ = 5.4 Hz); 3.18—3.27, 3.39—3.48 (both m, 1 H each, NCH_2); 3.90—3.92, 3.94—3.96 (both m, 1 H each, CH_2O); 5.12 (dq, 1 H, C<u>H</u>Me, $^3J_{P,H}$ = 10.9 Hz, $^3J_{H,H}$ = 5.4 Hz); 7.39—7.47 (m, 6 H, H_m, H_p); 7.99, 8.07 (both dd, 2 H each, H_o, $^3J_{P,H}$ = 13.3 Hz, $^3J_{H,H}$ = 7.2 Hz). ^{13}C NMR, δ: 22.1 (d, Me, $^3J_{P,C}$ = 5.5 Hz); 46.6 (NCH_2); 66.5 (d, CH_2O, $^3J_{P,C}$ = 2.6 Hz); 87.5 (d, <u>C</u>Me, $^2J_{P,C}$ = 2.2 Hz); 128.3—128.7 (m, C_m); 131.5—132.5 (m, C_o, C_p); 133.5 (d, C_{ipso}, $^1J_{P,C}$ = 105.7 Hz); 134.1 (d, C_{ipso}, $^1J_{P,C}$ = 103.2 Hz). ^{15}N NMR, δ: −311.9. ^{31}P NMR, δ: 66.4.

3-(Diphenylselenophosphoryl)-2-methyl-1,3-oxazolidine (2b). Yield 195 mg (90%), m.p. 71—72 °C (from hexane). Found (%): C, 54.82; H, 5.15; N, 4.01; P, 8.54; Se, 22.49. $C_{16}H_{18}NOPSe$. Calculated (%): C, 54.87; H, 5.18; N, 4.00; P, 8.84; Se, 22.54. IR, ν/cm^{-1}: 1179, 1128, 1099, 1072 (O—C—N); 695 (P—N—C); 584 (P=Se). ^1H NMR, δ: 1.05 (d, 3 H, Me, $^3J_{H,H}$ = 5.4 Hz); 3.13—3.23, 3.40—3.48 (both m, 1 H each, NCH_2); 3.92—4.02 (m, 2 H, CH_2O); 5.05 (dq, 1 H, C<u>H</u>Me, $^3J_{P,H}$ = 10.5 Hz, $^3J_{H,H}$ = 5.4 Hz); 7.43—7.51 (m, 6 H, H_m, H_p); 8.06, 8.13 (both dd, 2 H each, H_o, $^3J_{P,H}$ = 13.2 Hz, $^3J_{H,H}$ = 7.8 Hz). ^{13}C NMR, δ: 22.0 (d, Me, $^3J_{P,C}$ = 5.6 Hz); 47.3 (NCH_2); 66.3 (d, CH_2O, $^3J_{P,C}$ = 3.2 Hz); 88.2 (d, <u>C</u>Me, $^2J_{P,C}$ = 3.2 Hz); 128.3, 128.6 (both d, C_m, $^3J_{P,C}$ = 12.8 Hz); 132.0 (d, C_p, $^4J_{P,C}$ = 2.8 Hz); 132.4, 132.7 (both d, C_o, $^2J_{P,C}$ = 11.2 Hz); 133.0 (d, C_{ipso}, $^1J_{P,C}$ = 80.4 Hz). ^{15}N NMR, δ: −315.8. ^{31}P NMR, δ: 66.8 (s and d of the satellites, $^1J_{P,Se}$ = 763 Hz). ^{77}Se NMR, δ: −333.5 (d, $^1J_{P,Se}$ = 763 Hz).

3-[Bis(2-phenylethyl)thiophosphoryl]-2-methyl-1,3-oxazolidine (2c). Yield 174 mg (80%), viscous oil. Found (%): C, 66.79; H, 7.23; N, 3.87; P, 8.59; S, 8.88. $C_{20}H_{26}NOPS$. Calculated (%): C, 66.83; H, 7.29; N, 3.90; P, 8.62; S, 8.92. IR, ν/cm^{-1}: 1161, 1124, 1106, 1074 (O—C—N); 698 (P—N—C); 619 (P=S). ^1H NMR, δ: 1.32 (d, 3 H, Me, $^3J_{H,H}$ = 5.2 Hz); 2.06—2.25 (m, 4 H, PCH_2); 2.80—3.02 (m, 4 H, CH_2Ph); 3.08—3.16, 3.40—3.48 (both m, 1 H each, NCH_2); 3.76—3.82, 3.96—4.00 (both m, 1 H each, CH_2O); 5.42 (dq, 1 H, C<u>H</u>Me, $^3J_{P,H}$ = 10.0 Hz, $^3J_{H,H}$ = 5.2 Hz); 7.12—7.22 (m, 6 H, H_o, H_p); 7.24 (dd, 4 H, H_m, $^3J_{H,H}$ = 8.4 Hz, $^3J_{H,H}$ = 7.2 Hz). ^{13}C NMR, δ: 22.8 (d, Me, $^3J_{P,C}$ = 3.6 Hz); 28.3, 28.5 (both d, Ph<u>C</u>H_2, $^2J_{P,C}$ = 2.4 Hz and $^2J_{P,C}$ = 2.8 Hz, respectively); 34.2, 34.6 (both d, CH_2P, $^1J_{P,C}$ = = 61.1 Hz and $^1J_{P,C}$ = 64.3 Hz, respectively); 44.8 (NCH_2); 65.8 (d, CH_2O, $^3J_{P,C}$ = 4.0 Hz); 87.4 (d, <u>C</u>Me, $^2J_{P,C}$ = 3.2 Hz); 126.3 (C_p); 128.1 (d, C_o, $^4J_{P,C}$ = 6.0 Hz); 128.5 (d, C_m, $^5J_{P,C}$ = 0.8 Hz); 140.4 (d, C_{ipso}, $^3J_{P,C}$ = 15.2 Hz). ^{15}N NMR, δ: −317.9. ^{31}P NMR, δ: 72.1.

3-[Bis(2-phenylethyl)selenophosphoryl]-2-methyl-1,3-oxazolidine (2d). Yield 219 mg (90%), viscous oil. Found (%): C, 59.02; H, 6.47; N, 3.43; P, 7.56; Se, 19.38. $C_{20}H_{26}NOPSe$. Calculated (%): C, 59.11; H, 6.45; N, 3.45; P, 7.62; Se, 19.43. IR, ν/cm^{-1}: 1156, 1124, 1105, 1078 (O—C—N); 698 (P—N—C); 580 (P=Se). ^1H NMR, δ: 1.22 (d, 3 H, Me, $^3J_{H,H}$ = 5.1 Hz); 2.30—2.43 (m, 4 H, PCH_2); 2.91—3.05 (m, 4 H, CH_2Ph); 3.16—3.19, 3.51—3.53 (both m, 1 H each, NCH_2); 3.89—3.90, 4.04—4.05 (both m, 1 H each, CH_2O); 5.38 (dq, 1 H, C<u>H</u>Me, $^3J_{P,H}$ = 10.1 Hz, $^3J_{H,H}$ = 5.1 Hz); 7.19—7.26 (m, 6 H, H_o, H_p); 7.31 (dd, 4 H, H_m, $^3J_{H,H}$ = 8.6 Hz, $^3J_{H,H}$ = 7.1 Hz). ^{13}C NMR, δ: 22.8 (d, Me, $^3J_{P,C}$= 4.8 Hz); 29.2, 29.3 (both s, Ph<u>C</u>H_2); 35.0, 35.5 (both d, CH_2P, $^1J_{P,C}$ = 55.1 Hz and $^1J_{P,C}$ = 53.8 Hz, respectively); 45.6 (NCH_2); 65.9 (d, CH_2O, $^3J_{P,C}$ = 4.0 Hz); 88.1 (d, <u>C</u>Me, $^2J_{P,C}$ = 2.8 Hz); 126.4 (C_p); 128.2 (d, C_o, $^4J_{P,C}$ = 6.0 Hz); 128.7 (C_m); 140.4 (d, C_{ipso}, $^3J_{P,C}$ = 15.2 Hz). ^{15}N NMR, δ: −320.8. ^{31}P NMR, δ: 68.2 (s and d of the satellites, $^1J_{P,Se}$ = = 742 Hz). ^{77}Se NMR, δ: −334.7 (d, $^1J_{P,Se}$ = 742 Hz).

3-[Bis(2-phenylprop-1-yl)selenophosphoryl]-2-methyl-1,3-oxazolidine (2e). Yield 238 mg (89%), viscous oil. Found (%): C, 60.78; H, 6.95; N, 3.20; P, 7.06; Se, 18.09. $C_{22}H_{30}NOPSe$. Calculated (%): C, 60.83; H, 6.96; N, 3.22; P, 7.13; Se, 18.18. IR, ν/cm^{-1}: 1157, 1142, 1105, 1087 (O—C—N); 706 (P—N—C);

* The data can be made available free of charge from the website: www.ccdc.cam.ac.uk/data_request/cif.

532 (P=Se). ^{1}H NMR, δ: 1.07, 1.22, 1.24, 1.41, 1.43 (all d, 6 H, MeCHPh, $^3J_{H,H}$ = 6.9 Hz, $^3J_{H,H}$ = 7.2 Hz, $^3J_{H,H}$ = 7.1 Hz, $^3J_{H,H}$ = 7.4 Hz, $^3J_{H,H}$ = 7.8 Hz, respectively); 1.16, 1.17, 1.30, 1.33 (all d, 3 H, C(2)Me, $^3J_{H,H}$ = 5.2 Hz, $^3J_{H,H}$ = 5.3 Hz, $^3J_{H,H}$ = 6.4 Hz, $^3J_{H,H}$ = 5.6 Hz, respectively); 1.88—1.96, 1.99—2.08, 2.10—2.24, 2.27—2.54 (all m, 4 H, CH_2P); 2.96—2.99, 3.05—3.11, 3.24—3.32, 3.39—3.45, 3.51—3.61, 3.64—3.70, 3.74—3.87 (all m, 6 H, CHMe, NCH_2, CH_2O); 5.08—5.14, 5.21—5.27, 5.32—5.39 (all m, 2 H, C(2)H); 7.04—7.34 (m, 10 H, Ph). ^{13}C NMR, δ: 22.4 (d, MeCHPh, $^3J_{P,C}$ = 3.5 Hz); 22.7 (d, MeCHPh, $^3J_{P,C}$ = 4.8 Hz); 23.8—23.9 (four d, MeCHPh, $^3J_{P,C}$ = 11.9 Hz, $^3J_{P,C}$ = 11.2 Hz, $^3J_{P,C}$ = 11.0 Hz, $^3J_{P,C}$ = 12.3 Hz); 24.7—24.8 (four d, MeCHPh, $^3J_{P,C}$ = 10.2 Hz, $^3J_{P,C}$ = 9.2 Hz, $^3J_{P,C}$ = 11.4 Hz, $^3J_{P,C}$ = 11.2 Hz); 34.5, 34.8, 35.7, 36.0 (all d, C(2)Me, $^3J_{P,C}$ = 3.1 Hz, $^3J_{P,C}$ = 3.5 Hz, $^3J_{P,C}$ = 1.5 Hz, $^3J_{P,C}$ = = 4.2 Hz); 39.9, 40.4, 40.9, 42.1 (all d, CH_2P, $^1J_{P,C}$ = 55.7 Hz, $^1J_{P,C}$ = 57.7 Hz, $^1J_{P,C}$ = 51.6 Hz, $^1J_{P,C}$ = 51.3 Hz, respectively); 41.5, 41.6, 41.9, 42.4 (all s, PhCH); 44.7, 44.9, 45.0, 45.2 (all s, NCH_2); 65.4, 65.5, 65.6, 65.8 (all d, NCH_2, $^2J_{P,C}$ = 3.4 Hz, $^2J_{P,C}$ = 4.7 Hz, $^2J_{P,C}$ = 4.0 Hz, $^2J_{P,C}$ = 4.7 Hz, respectively); 87.7, 88.2, 88.3, 88.4 (all d, C(2), $^3J_{P,C}$ = 5.8 Hz, $^3J_{P,C}$ = 4.4 Hz, $^3J_{P,C}$ = 4.5 Hz, $^3J_{P,C}$ = 2.2 Hz); 126.2—128.9 (m, C_o, C_m, C_p); 145.4, 146.4 (both m, C_{ipso}). ^{15}N NMR, δ: −322.9, −320.9, −320.4, −318.2. ^{31}P NMR, δ: 64.3 (s and d of the satellites, $^1J_{P,Se}$ = 730 Hz); 66.4 (s and d of the satellites, $^1J_{P,Se}$ = 740 Hz); 67.3 (s and d of the satellites, $^1J_{P,Se}$ = 741 Hz); 70.2 (s and d of the satellites, $^1J_{P,Se}$ = 734 Hz). ^{77}Se NMR, δ: −333.4 (d, $^1J_{P,Se}$ = = 741 Hz); −329.72 (d, $^1J_{P,Se}$ = 740 Hz); −323.0 (d, $^1J_{P,Se}$ = = 730 Hz); −317.5 (d, $^1J_{P,Se}$ = 734 Hz).

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