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Methyl N-alkyl(phenyl)-P-(3-methylalka-1,2-dienyl)phosphonamidoates – synthesis and properties

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Abstract*: The synthesis of titled compounds has been described. Their reactivity towards sulfanyl- and selanylbromides has been investigated.*

Keywords*: Allenephosphonate, synthesis, cyclization, 2,5-dihydro-1,2-oxaphosphole.*

Introduction

It is well-known that 1,2- alkadienephosphonates react with different kind of reagents due to their unique structure. Their structural parameters allowed activation of different reaction centers in their molecules, i.e. two orthogonal double bonds and a phosphoryl group. This activation could be achieved by variation of the type and number of substituents at phosphorus and carbon atoms of the allenephosphonate systhem [1].

Continuing our investigations in the area of N-containing-1,2-alkadienephosphonates[2-8], we would like to report our results on the synthesis and reactions of the titled compounds.

Results and Discussion

The methyl-N-alkyl(phenyl)-P-(3-methylalka-1,2-dienylphosphonoamidoates **2a-f** were synthesized via a procedure described [2], i.e. via nucleophilic substitution of the two chlorine atoms at phosphorus in the 1,2-alkadienephosphonate dichlorides **1a,b** in the reaction with methanol and primary amines according to Scheme 1.

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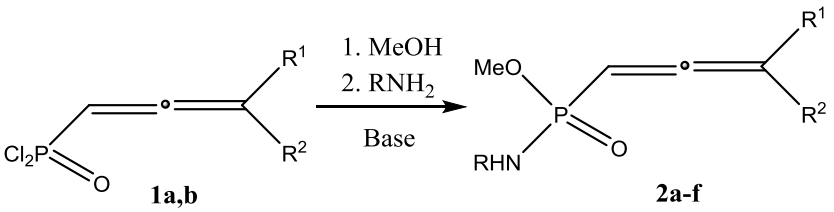
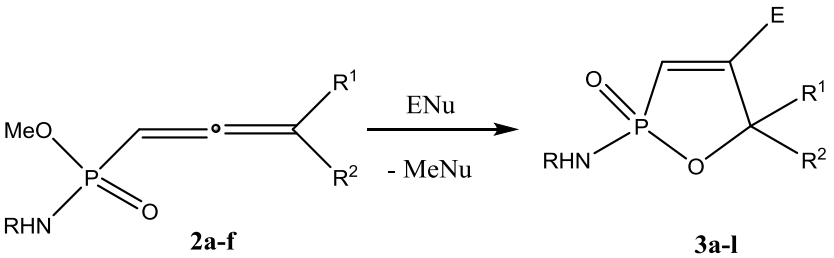


R= Pr, Bu, Ph
R^1= R^2= Me
R^1= Me, R^2= Et

Scheme 1. Synthesis of compounds **2a-f**

The obtained methyl-N-alkyl(phenyl)-P-(3-methylalka-1,2-dienylphosphonoamidoates **2a-f** were investigated in the reactions with sulphenyl- and selenenylbromides. In all cases, regardless of the type of the electrophile, 2,5-dihydro-1,2-oxaphosphole 2-oxide derivatives **3a-l** were obtained. The reaction follows the Scheme 2:



R= Pr, Bu, Ph
R^1= R^2= Me
R^1= Me, R^2= Et
ENu= PhSBr, PhSeBr

Scheme 2. Synthesis of compounds **3a-l**

All spectral data confirm the structure of obtained compounds **3a-l**. Thus, the characteristic band for allenic system in the IR spectra of **2a-f** at 1980-1990cm^{-1} disappears in the IR spectra of the compounds **3a-l** and a new characteristic band for endo-cyclc double bond appears at 1590-1580cm^{-1}. The signals for methoxy-group protons and for olefinic proton in the ^{1}H-NMR spectra of **2a-f** disappear in the same spectra of the compounds **3a-l** and two doublets for the protons, connected with the C3 atom of the oxaphosphole ring appears. The signal for ^{31}P in the ^{31}P NMR spectra of **2a-f** appears at 16.8-17.3ppm, while they shift up to 27.9-32.00ppm for compounds **3a-l**. The molecular formulae of **3a-l** were confirmed by elemental analysis data.

Experimental

Analytical Methods

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The ^{1}H NMR and ^{31}P NMR spectra were measured at normal probe temperature on a Brucker Advance DRX 250 MHz spectrometer, using TMS or H_3PO_4 as internal standards in $CDCl_3$ solution Chemical shift are given in ppm and are positively downfield from the standard. The IR spectra were recorded on a Shimadzu IRAffinity-1 spectrophotometer. Elemental analyses were carried out by the University of Shumen Microanalytical Service Laboratory.

Starting Materials

The dichlorides **1a,b** were prepared according to the procedure described[10].

Benzeneselanylbromide is commercially available. Benzenesulfanylbromide was synthesized according the procedure described [9].

The solvents were purified by standard methods. All reactions were carried out in oven-dried glassware under an argon atmosphere and exclusion of moisture. All compounds were checked for their purity on TLC plates.

Synthesis of compounds **2a-f**. *General procedure*:

To a solution of the appropriate dichloride **1a,b** (5mmol) in dry diethyl ether(100ml) at 0 - -5^{o}C and stirring, a mixture of methanol (5mmol) and pyridine (5mmol) dissolved at the same solvent (50ml) was added. Then a mixture of appropriate ammine (5mmol) and pyridine (5mmol) dissolved at the same solvent (50ml) was added too. After an additional stirring (1 hour) the reaction mixture was rest for additional time (12 hours/ 0^{o}C – to room temperature), the precipitate was filtered off, the solvent was removed under low pressure and the residue was distilled in vacuum.

Methyl-N-propyl-P-(3-methylbuta-1,2-dienyl)phosphonamidoate **2a**. Yellow-red liquid, b.p.(^{o}C/0.5mmHg) uncorrected 136-138; 0.83g(85%); IR(KBr): ν_{max}/cm^{-1} 1986, 1256, 980; ^{1}H NMR($CDCl_3$): δ/ppm 5.05-4.95(m, 1H), 3.39(d, $^{3}J_{HP}$ 11.2Hz, 3H), 2.67-2.54(m, 2H), 1.46(s, 3H), 1.51(s, 3H), 2.00(d, $^{2}J_{HP}$ 10.0Hz, 1H), 1.28-1.19(m, 2H), 0.91(t, 3H); ^{31}P NMR ($CDCl_3$) δ /ppm: ^{31}P 17.1; Anal. Calcd. For $C_9H_{18}NO_2P$ (Mr = 203.2): P 15.24, N 6.89%; Found: P 15.19, N 6.82%.

Methyl-N-butyl-P-(3-methylbuta-1,2-dienyl)phosphonamidoate **2b**. Yellow-red liquid, b.p.(^{o}C/0.5mmHg) uncorrected 136-138; 0.83g(85%); IR(KBr): ν_{max}/cm^{-1} 1988, 1260, 980; ^{1}H NMR($CDCl_3$): δ/ppm 5.05-4.97(m, 1H), 3.38(d, $^{3}J_{HP}$ 11.2Hz, 3H), 2.66-2.53(m, 2H), 1.46(s, 3H), 1.51(s, 3H), 2.00(d, $^{2}J_{HP}$ 10.0Hz, 1H), 1.27-1.18(m, 2H), 1.16-0.99(m, 2H), 0.63(t, 3H); ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 16.8; Anal. Calcd. For $C_{10}H_{20}NO_2P$ (Mr = 217.23): P 14.26, N 6.44%; Found: P 14.21, N 6.40%.

Methyl-P-3-methylbuta-1,2-dienyl-N-phenylphosphonamidoate **2c**. Colorless cryst., m.p.(^{o}C) uncorrected 143-146; 0.94g(80%); IR(KBr): ν_{max}/cm^{-1} 1987, 1264, 980; ^{1}H NMR($CDCl_3$): δ/ppm 7.29-7.17(m, 2H), 7.05(d, $^{2}J_{HH}$ 7.75Hz, 1H), 6.89(t, $^{2}J_{HH}$ 7.2Hz, 2H), 5.45-5.35(m, 1H), 4.03(d, $^{2}J_{HP}$ 11.2Hz, 1H), 3.74(d, $^{3}J_{HP}$ 11.5Hz, 3H), 1.46(s, 3H), 1.51(s, 3H); ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 17.0; Anal. Calcd. For $C_{12}H_{16}NO_2P$ (Mr = 237.22): P 13.06, N 5.90%; Found: P 13.00, N 5.88%.

Methyl-N-propyl-P-(3-methylpenta-1,2-dienyl)phosphonamidoate **2d**. Yellow-red liquid, b.p.(^{o}C/0.5mmHg) uncorrected 137-139; 1.03g(87%); IR(KBr): ν_{max}/cm^{-1} 1990, 1258, 980; ^{1}H NMR($CDCl_3$): δ/ppm 5.04-4.97(m, 1H), 3.38(d, $^{2}J_{HP}$ 11.2Hz, 1H), 2.67-2.54(m, 2H), 1.82-1.68(m, 3H),1.49(dd, $^{3}J_{HH}$ 10.0Hz, $^{4}J_{HH}$ 3.53Hz, 3H), 1.28-1.19(m, 2H), 0.77(dt, $^{2}J_{HH}$ 1.2Hz, 3H), 0.91(t, 3H);

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^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 16.8; Anal. Calcd. For $C_{10}H_{20}NO_2P$ (Mr = 217.23): P 14.26, N 6.44%; Found: P 14.21, N 6.40%.

Methyl-N-butyl-P-(3-methylpenta-1,2-dienyl)phosphonamidoate **2e**. Yellow-red liquid, b.p.(°C/0.5mmHg) uncorrected 427-145; 0.93g(81%); IR(KBr): ν_{max}/cm^{-1} 1989, 1260, 980; ^{1}H NMR($CDCl_3$): δ/ppm 5.04-4.97(m, 1H), 3.38(d, $^2J_{HP}$ 11.2Hz, 1H), 2.66-2.53(m, 2H), 1.82-1.68(m, 3H),1.49(dd, $^3J_{HH}$ 10.0Hz, $^4J_{HH}$ 3.5Hz, 3H), 1.27-1.18(m, 2H), 1.16-0.99(m, 2H), 0.77(dt, $^2J_{HH}$ 1.25Hz, 3H), 0.63(t, 3H); ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 17.3; Anal. Calcd. For $C_{10}H_{23}NO_2P$ (Mr = 231.26): P 13.39, N 6.05%; Found: P 13.34, N 6.00%.

Methyl-P-3-methylpenta-1,2-dienyl-N-phenylphosphonamidoate **2f**. Colorless cryst., m.p.(°C) uncorrected 145-147; 1.01g(81%); IR(KBr): ν_{max}/cm^{-1} 1992, 1259, 980; ^{1}H NMR($CDCl_3$): δ/ppm 7.29-7.17(m, 2H), 7.05(d, $^2J_{HH}$ 7.7Hz, 1H), 6.89(t, $^2J_{HH}$ 7.2Hz, 2H), 5.45-5.35(m, 1H), 4.03(d, $^2J_{HP}$ 11.2Hz, 1H), 3.74(d, $^3J_{HP}$ 11.5Hz, 3H), 1.98-1.75(m, 2H), 1.64(dd, $^3J_{HH}$ 8.5Hz, $^4J_{HH}$ 3.2Hz, 3H), 0.85(t, 3H); ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 16.9; Anal. Calcd. For $C_{13}H_{18}NO_2P$ (Mr = 251.24): P 12.33, N 5.57%; Found: P 12.29, N 5.52%.

Synthesis of compounds **3a-l**. *General procedu*re:

To a solution of the appropriate methyl-N-alkyl(phenyl)-P-(3-methylalka-1,2-dienyl)phosphonamidoates **2a-f** (5mmol) in dry methylene chloride (50ml) at -12 - -10°C and stirring, a solution of the appropriate electrophile (PhSBr or PhSeBr) (5mmol) dissolved at the same solvent (5ml) was added. After an additional stirring (1 hour) the reaction mixture was rest for additional time (12 hours/ -10°C – to room temperature), the solvent was removed under low pressure and the residue was purified by column chromatography.

(5,5-Dimethyl-2-oxo-4-phenylsulfanyl-2,5-dihydro-2λ^5-[1,2]oxaphosphol-2-yl)propylamine **3a**. Colorless cryst., m.p.(°C) uncorrected 113-115, 1.26g(85%), IR(KBr): ν_{max}/cm^{-1} 1592, 1259, 1000; ^{1}H NMR($CDCl_3$): δ/ppm 7.56-7.46(m, 2H), 7.29-7.23(m, 3H), 5.35(dd, $^2J_{HP}$ 27.7Hz, $^3J_{HH}$ 3.7Hz, 1H), 2.54(m, 2H), 1.46(s, 3H), 1.51(s, 3H), 2.00(d, $^2J_{HP}$ 10.0Hz, 1H), 1.28-1.19(m, 2H), 0.91(t, 3H) ; ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 29.0; Anal. Calcd. For $C_{14}H_{20}NO_2PS$ (Mr = 297.33): P 10.42, N 4.71, S 10.78%; Found: P 10.40, N 4.68, S 10.72%.

(5,5-Dimethyl-2-oxo-4-phenylsulfanyl-2,5-dihydro-2 λ^5-[1,2]oxaphosphol-2-yl)butylamine **3b**. Colorless cryst., m.p.(°C) uncorrected 115-118, 1.30g(84%), IR(KBr): □$_{max}$/cm^{-1} 1590, 1260, 1000; ^{1}H NMR($CDCl_3$): □/ppm 7.64-7.60(m, 5H), 5.35(dd, $^2J_{HP}$ 27.7Hz, $^3J_{HH}$ 3.7Hz, 1H), 2.66-2.53(m, 2H), 1.46(s, 3H), 1.51(s, 3H), 2.00(d, $^2J_{HP}$ 10.0Hz, 1H), 1.27-1.18(m, 2H),1.16-0.99(m, 2H), 0.63(t, 3H) ; ^{31}P NMR ($CDCl_3$) □/ppm: ^{31}P 28.9; Anal. Calcd. For $C_{15}H_{22}NO_2PS$ (Mr = 311.36): P 9.95, N 4.50, S 10.30%; Found: P 9.91, N 4.48, S 10.28%.

(5,5-Dimethyl-2-oxo-4-phenylsulfanyl-2,5-dihydro-2 λ^5-[1,2]oxaphosphol-2-yl)phenylamine **3c**. Colorless cryst., m.p.(°C) uncorrected 123-126, 1.35g(82%), IR(KBr): ν_{max}/cm^{-1} 1598, 1257, 1000; ^{1}H NMR($CDCl_3$): δ/ppm 7.63-7.59(m, 2H), 7.49-7.37(m, 3H), 7.26-7.18(m, 2H), 7.07-6.95(m, 3H), 5.44(dd, $^2J_{HP}$ 28.0Hz, $^3J_{HH}$ 5.00Hz, 1H), 1.46(s, 3H), 1.51(s, 3H); ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 30.0; Anal. Calcd. For $C_{17}H_{18}NO_2PS$ (Mr = 331.34): P 9.35, N 4.22, S 9.68%; Found: P 9.33, N 4.19, S 9.65%.

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(5-Ethyl-5-methyl-2-oxo-4-phenylsulfanyl-2,5-dihydro-2 λ^5-[1,2]oxaphosphol-2-yl)propylamine **3d**. Colorless cryst., m.p.(^{o}C) uncorrected 114-116, 1.30g(84%), IR(KBr): ν_{max}/cm^{-1} 1592, 1259, 1000; ^{1}H NMR($CDCl_3$): δ/ppm 7.56-7.46(m, 2H), 7.29-7.23(m, 3H), 5.35(dd, $^2J_{HP}$ 27.7Hz, $^3J_{HH}$ 3.75Hz, 1H), 2.67-2.54(m, 2H), 1.82-1.68(m, 3H), 1.49(dd, $^3J_{HH}$ 10.0Hz, $^4J_{HH}$ 3.5Hz, 3H), 1.28-1.19(m, 2H), 0.77(dt, $^2J_{HH}$ 1.2Hz, 3H), 0.91(t, 3H) ; ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 29.8; Anal. Calcd. For $C_{15}H_{22}NO_2PS$ (Mr = 311.36): P 9.95, N 4.50, S 10.30%; Found: P 9.91, N 4.48, S 10.28%.

(5-Ethyl-5-methyl-2-oxo-4-phenylsulfanyl-2,5-dihydro-2 λ^5-[1,2]oxaphosphol-2-yl)butylamine **3e**. Colorless cryst., m.p.(^{o}C) uncorrected 116-118, 1.33g(82%), IR(KBr): ν_{max}/cm^{-1} 1590, 1260, 1000; ^{1}H NMR($CDCl_3$): δ/ppm 7.64-7.60(m, 5H), 5.35(dd, $^2J_{HP}$ 27.7Hz, $^3J_{HH}$ 3.75Hz, 1H), 2.66-2.53(m, 2H), 1.82-1.67(m, 3H), 1.49(dd, $^3J_{HH}$ 10.0Hz, $^4J_{HH}$ 3.53Hz, 3H), 1.27-1.18(m, 2H), 1.16-0.99(m, 2H), 0.77(dt, $^2J_{HH}$ 1.2Hz, 3H), 0.63(t, 3H) ; ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 32.0; Anal. Calcd. For $C_{16}H_{24}NO_2PS$ (Mr = 325.38): P 9.52, N 4.30, S 9.85%; Found: P 9.50, N 4.28, S 9.81%.

(5-Ethyl-5-methyl-2-oxo-4-phenylsulfanyl-2,5-dihydro-2 λ^5-[1,2]oxaphosphol-2-yl)phenylamine **3f**. Colorless cryst., m.p.(^{o}C) uncorrected 125-127, 1.38g(80%), IR(KBr): ν_{max}/cm^{-1} 1589, 1257, 1000; ^{1}H NMR($CDCl_3$): δ/ppm 7.63-7.59(m, 2H), 7.49-7.37(m, 3H), 7.26-7.18(m, 2H), 7.07-6.95(m, 3K), 5.44(dd, $^2J_{HP}$ 28.0Hz, $^3J_{HH}$ 5.0Hz, 1H), 1.98-1.75(m, 2H), 1.64(dd, $^3J_{HH}$ 8.5Hz, $^4J_{HH}$ 3.2Hz, 3H), 0.85(t, 3H) ; ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 27.9; Anal. Calcd. For $C_{18}H_{20}NO_2PS$ (Mr = 345.37): P 8.97, N 4.05, S 9.22%; Found: P 8.94, N 4.00, S 9.22%.

(5,5-Dimethyl-2-oxo-4-phenylselanyl-2,5-dihydro-2 λ^5-[1,2]oxaphosphol-2-yl)propylamine **3g**. Colorless cryst., m.p.(^{o}C) uncorrected 114-116, 1.44g(84%), IR(KBr): ν_{max}/cm^{-1} 1592, 1259, 1000; ^{1}H NMR($CDCl_3$): δ/ppm 7.56-7.46(m, 2H), 7.29-7.23(m, 3H), 5.35(dd, $^2J_{HP}$ 27.7Hz, $^3J_{HH}$ 3.75Hz, 1H), 2.54(m, 2H), 1.46(s, 3H), 1.51(s, 3H), 2.00(d, $^2J_{HP}$ 10.0Hz, 1H), 1.28-1.19(m, 2H), 0.91(t, 3H) ; ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 29.7; Anal. Calcd. For $C_{14}H_{20}NO_2PSe$ (Mr = 344.23): P 9.92, N 4.48%; Found: P 9.90, N 4.43%.

(5,5-Dimethyl-2-oxo-4- phenylselanyl -2,5-dihydro-2 λ^5-[1,2]oxaphosphol-2-yl)butylamine **3h**. Colorless cryst., m.p.(^{o}C) uncorrected 119-120, 1.54(86%), IR(KBr): ν_{max}/cm^{-1} 1590, 1260, 1000; ^{1}H NMR($CDCl_3$): δ/ppm 7.64-7.60(m, 5H), 5.35(dd, $^2J_{HP}$ 27.7Hz, $^3J_{HH}$ 3.75Hz, 1H), 2.66-2.53(m, 2H), 1.46(s, 3H), 1.51(s, 3H), 2.00(d, $^2J_{HP}$ 10.0Hz, 1H), 1.27-1.18(m, 2H),1.16-0.99(m, 2H), 0.63(t, 3H) ; ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 30.05; Anal. Calcd. For $C_{15}H_{22}NO_2PSe$ (Mr = 358.26): P 9.49, N 4.29%; Found: P 9.45, N 4.23%.

(5,5-Dimethyl-2-oxo-4- phenylselanyl -2,5-dihydro-2 λ^5-[1,2]oxaphosphol-2-yl)phenylamine **3i**. Colorless cryst., m.p.(^{o}C) uncorrected 143-126, 1.55g(82%), IR(KBr): ν_{max}/cm^{-1} 1589, 1257, 1000; ^{1}H NMR($CDCl_3$): δ/ppm 7.63-7.59(m, 2H), 7.49-7.37(m, 3H), 7.26-7.18(m, 2H), 7.07-6.95(m, 3H), 5.44(dd, $^2J_{HP}$ 28.0Hz, $^3J_{HH}$ 5.0Hz, 1H), 1.46(s, 3H), 1.51(s, 3H); ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 28.8; Anal. Calcd. For $C_{17}H_{18}NO_2PSe$ (Mr = 378.24): P 8.94, N 4.04%; Found: P 8.90, N 4.00%.

(5-Ethyl-5-methyl-2-oxo-4- phenylselanyl -2,5-dihydro-2 λ^5-[1,2]oxaphosphol-2-yl)propylamine **3j**. Colorless cryst., m.p.(^{o}C) uncorrected 115-117, 1.48g(83%), IR(KBr): ν_{max}/cm^{-1} 1592, 1259, 1000; ^{1}H NMR($CDCl_3$): δ/ppm 7.56-7.46(m, 2H), 7.29-7.23(m, 3H), 5.35(dd, $^2J_{HP}$ 27.7Hz, $^3J_{HH}$ 3.75Hz, 1H), 2.67-2.54(m, 2H), 1.82-1.68(m, 3H), 1.49(dd, $^3J_{HH}$ 10.0Hz, $^4J_{HH}$ 3.53Hz, 3H), 1.28-1.19(m, 2H), 0.77(dt, $^2J_{HH}$ 1.2Hz, 3H), 0.91(t, 3H) ; ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 29.8; Anal. Calcd. For $C_{15}H_{22}NO_2PSe$ (Mr = 358.26): P 9.49, N 4.29%; Found: P 9.45, N 4.23%.

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(5-Ethyl-5-methyl-2-oxo-4- phenylselanyl -2,5-dihydro-2 λ^5-[1,2]oxaphosphol-2-yl)butylamine **3k**. Colorless cryst., m.p.(^oC) uncorrected 120-122, 1.52g(82%), IR(KBr): ν_{max}/cm^{-1} 1590, 1260, 1000; ^1H NMR($CDCl_3$): δ/ppm 7.64-7.60(m, 5H), 5.35(dd, $^2J_{HP}$ 27.7Hz, $^3J_{HH}$ 3.75Hz, 1H), 2.66-2.53(m, 2H), 1.82-1.67(m, 3H), 1.49(dd, $^3J_{HH}$ 10.0Hz, $^4J_{HH}$ 3.53Hz, 3H), 1.27-1.18(m, 2H), 1.16-0.99(m, 2H), 0.77(dt, $^2J_{HH}$ 1.2Hz, 3H), 0.63(t, 3H) ; ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 30.09; Anal. Calcd. For $C_{16}H_{24}NO_2PSe$ (Mr = 372.28): P 9.10, N 4.11%; Found: P 9.06, N 4.08%.

(5-Ethyl-5-methyl-2-oxo-4-phenylsulanyl-2,5-dihydro-2λ^5-[1,2]oxaphosphol-2-yl)phenylamine **3l**. Colorless cryst., m.p.(^oC) uncorrected 127-129, 1.58g(81%), IR(KBr): ν_{max}/cm^{-1} 1589, 1257, 1000; ^1H NMR($CDCl_3$): δ/ppm 7.63-7.59(m, 2H), 7.49-7.37(m, 3H), 7.26-7.18(m, 2H), 7.07-6.95(m, 3K), 5.44(dd, $^2J_{HP}$ 28.0Hz, $^3J_{HH}$ 5.0Hz, 1H), 1.98-1.75(m, 2H), 1.64(dd, $^3J_{HH}$ 8.5Hz, $^4J_{HH}$ 3.25Hz, 3H), 0.85(t, 3H) ; ^{31}P NMR ($CDCl_3$) δ/ppm: ^{31}P 31.7; Anal. Calcd. For $C_{18}H_{20}NO_2PSe$ (Mr = 392.27): P 8.59, N 3.88%; Found: P 8.55, N 3.85%.

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