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Te(II)-induced heterocyclization of 1,2-alkadienephosphonates

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Abstract: The reactivity of some 1,2-alkadienephosphonates towards phenyltelluryl halides was investigated. A plausible mechanism of the reaction is discussed.

Key words: 1,2-Alkadienephosphonates, electrophilic addition, phosphorus heterocycles

1. Introduction

The applications of organophosphorus compounds as pharmaceutical, agricultural, and chemical agents are well documented.[1,2] Among them, oxaphosphole derivatives, which have structures similar to those of phosphosugars, have received particular interest.[3,4] Consequently, many attempts for their synthesis have been made. One of the easiest and most fruitful methods for the synthesis of these derivatives is electrophile-induced heterocyclization of 1,2-alkadienephosphonates.[5]

Keeping in mind that the scope of applications of organotellurides has been known for years because of their ready transformation to other compounds via reactions with organometallic reagents,[6–10] here we wish to report the results of our study on the electrophilic addition of organotellurides to some 1,2-alkadienephosphonates.

2. Experimental

2.1. Analytical methods

The ^{1}H NMR and ^{31}P NMR spectra were measured at normal probe temperature on a Bruker Avance DRX 250 MHz spectrometer using tetramethylsilane (TMS) (^{1}H) and 85% H_3PO_4 (^{31}P) as internal references in CDCl3 solution.

Chemical shifts are given in parts per million (ppm) and are downfield from the internal standard. The infrared (IR) spectra were run on a Shimadzu IRAffinity-1 spectrophotometer. Elemental analyses were carried out by the University of Shumen Microanalytical Service Laboratory. Phenyltelluryl chloride was synthesized as described previously.[11–15]

Compounds **1**, **3**, **4**, **7**, and **9** were synthesized according to the literature.[16–18]

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

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2.2. Synthesis of 2-alkoxy-5-alkyl-5-alkyl-4-phenyltellanyl-5*H*-[1,2]-oxaphosphole 2-oxides and of 2-alkoxy-4-phenyltellanyl-1-oxa-2-phospha-[4,5]-dec-3-ene 2-oxide 2a–d

2.2.1. General procedure

To a solution of **1** (5 mmol) in methylene chloride (10 mL) was added a solution of phenyltelluryl chloride (1.24 g, 5.2 mmol) in 5 mL of methylene chloride under stirring and cooling (–10 to –12 °C). After 1 h of stirring at the same conditions, the reaction mixture stood overnight, and was concentrated and recrystallized in heptane/benzene (2:1).

2a, cryst. colorless needles; 1.59 g (87%), mp °C (147–149), IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1540, 1235, 960 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 6.46 (d, J_{HP} 26.0 Hz, 1H), 3.70 (d, J_{HP} 12.2 Hz, 3H), 1.59 (s, 3H), 1.55 (s, 3H). ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 33.09; *Anal.*, Calcd. for $C_{12}H_{15}O_3PTe$ (M_r = 365.81): P 8.47; Found P 8.43; **2b**, cryst. colorless needles; 1.38 g (73%), mp °C (150–152), IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1580, 1235, 1000 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 6.49 (d, J_{HP} 26.1 Hz, 1H), 4.17 (m, J_{HP} 10.0 Hz, 2H), 1.36 (t, J_{HH} 7.0 Hz, 3H) 1.52 (s, 3H), 1.57 (s, 3H). ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 32.0; *Anal.*, Calcd. for $C_{13}H_{17}O_3PTe$ (M_r = 379.836): P 8.15; Found P 8.11; **2c**, cryst. colorless needles; 1.46 g (77%), mp °C (149–150); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1545, 1235, 980 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 6.55, 6.59* (d, J_{HP} 26.0 Hz, 1H), 3.80, 3.82* (d, J_{HP} 11.6 Hz, 2H), 1.51, 154* (s, 3H), 1.89 (m, 2H), 0.92 (t, 3H). ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 33.12; *Anal.*, Calcd. for $C_{13}H_{17}O_3PTe$ (M_r = 379.836): P 8.15; Found P 8.10; (*Additional signals for diastereomers); **2d**, cryst. colorless needles; 1.44 g (71%), mp °C (155–157); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1540, 1235, 990 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 6.42 (d, J_{HP} 25.8 Hz, 1H), 3.80 (d, J_{HP} 11.2 Hz, 2H), 1.68 (m, 10H). ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 33.23; *Anal.*, Calcd. for $C_{15}H_{19}O_3PTe$ (M_r = 405.872): P 7.63; Found P 7.60.

2.3. Synthesis of (5-alkyl-5-alkyl-2-oxo-4-phenyltellanyl-2,5-dihydro-2λ^5-[1,2]-oxaphosphol-2-yl) dialkylamines 5a-c and of dialkyl-(2-oxo-4-phenyltellanyl-1-oxa-2λ^5phospha-spiro[4,5]-dec-3-ene 2-yl)amines 6a–c

2.3.1. General procedure

To a solution of **3** or **4** (5 mmol) in methylene chloride (10 mL) was added a solution of phenyltelluryl chloride (1.24 g, 5.2 mmol) in 5 mL of methylene chloride under stirring and cooling (–10 to –12 °C). After 1 h of stirring at the same conditions, the reaction mixture stood overnight, and was concentrated and recrystallized in heptane/benzene (2:1).

5a, cryst. colorless needles; 1.67 g (82%), mp °C (147–149); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1589, 1225, 1004 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 5.88 (d, J_{HP} 24.2 Hz, 1H), 1.40 (s, 3H), 1.58 (s, 3H), 1.00 (t, J_{HH} 7.0 Hz, 3H), 2.93 (m, J_{HP} 13.6 Hz, 2H). ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 28.3; *Anal.*, Calcd. for $C_{15}H_{22}O_2NPTe$ (M_r = 406.896): P 7.61, N 3.44; Found P 7.59, N 3.41; **5b**, cryst. colorless needles; 1.62 g (77%), mp °C (149–150); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1590, 1235, 980 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 6.55, 6.59* (d, J_{HP} 22.4 Hz, 1H), 1.51, 154* (s, 3H), 1.89 (m, 2H), 0.92 (t, 3H), 1.04 (t, J_{HH} 7.0 Hz, 3H), 3.00 (m, J_{HP} 12.1 Hz, 2H).

^{31}P NMR (250 MHz, $CDCl_3$) ppm: 27.9; *Anal.*, Calcd. for $C_{16}H_{24}O_2NPTe$ (M_r = 420.922): P 7.36, N 3.32; Found P 7.33, N 3.29 (*Additional signals for diastereomers); **5c**, cryst. colorless needles; 1.81 g (81%), mp °C (155–157); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1588, 1225, 1000 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 5.87 (d, J_{HP} 23.5 Hz, 1H), 1.68 (m, 10H), 0.98 (t, J_{HH} 7.0 Hz, 3H), 2.92 (m, J_{HP} 12.4 Hz, 2H). ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 32.3; *Anal.*, Calcd. for $C_{18}H_{26}O_2NPTe$ (M_r = 446.958): P 6.93, N 3.13; Found P 6.90, N 3.10.

6a, cryst. colorless needles; 1.89 g (87%), mp °C (147–149); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1580, 1230, 1000 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 6.08 (d, J_{HP} 24.2 Hz, 1H), 1.40 (s, 3H), 1.58 (s, 3H), 1.24 (ss, 6H), 2.93 (m, 1H). ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 28.3; *Anal.*, Calcd. for $C_{17}H_{26}O_2NPTe$ (M_r = 434.948): P 7.12, N 3.22; Found P 7.10, N 3.19; **6b**, cryst. colorless needles; 1.66 g (74%), mp °C (147–149); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1597, 1235, 900 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 6.55, 6.59* (d, J_{HP} 26.0 Hz, 1H), 1.51, 154* (s, 3H), 1.89 (m, 2H), 0.92 (t, 3H), 1.24 (ss, 6H), 2.93 (m, 1H). ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 28.3; *Anal.*, Calcd. for $C_{18}H_{28}O_2NPTe$ (M_r = 450.974): P 6.89, N 3.12; Found P 6.86, N 3.10; **6c**, cryst. colorless needles; 1.99 g (84%), mp °C (147–149); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1590, 1228, 1004 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 5.87 (d, J_{HP} 23.5 Hz, 1H), 1.68 (m, 10H), 1.24 (s, 6H), 2.93 (m, 1H); ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 28.3; *Anal.*, Calcd. for $C_{20}H_{30}O_2NPTe$ (M_r = 475.01): P 6.52, N 2.95; Found P 6.50, N 2.91.

2.4. Synthesis of (5-alkyl-5-alkyl-2-oxo-4-phenyltellanyl-2,5-dihydro-2λ^5-[1,2]-oxaphosphol-2-yl) alkylamines 8a,b and of alkyl-(2-oxo-4-phenyltellanyl-1-oxa-2λ^5phospha-spiro[4,5]-dec-3-ene 2-yl)amine 8c

2.4.1. General procedure

To a solution of **7** (5 mmol) in methylene chloride (10 mL) was added a solution of phenyltelluryl chloride (1.24 g, 5.2 mmol) in 5 mL of the same solvent under stirring and cooling (–10 to –12 °C). After 1 h of stirring at the same conditions, the reaction mixture stood overnight, and was concentrated and recrystallized in heptane/benzene (2:1).

8a, cryst. colorless needles; 1.61 g (82%), mp °C (147–149); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1580, 1245, 1004 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.56–7.46 (m, 2H); 7.29–7.23 (m, 3H); 5.35 (d, J_{HP} 27.75 Hz, 1H); 2.54 (m, 2H); 1.46 (s, 3H); 1.51 (s, 3H); 2.00 (d, J_{HP} 10.00 Hz, 1H); 1.28–1.19 (m, 2H); 0.91 (t, 3H); ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 29.0; *Anal.*, Calcd. for $C_{14}H_{20}O_2NPTe$ (M_r = 392.87): P 7.88, N 3.56; Found P 7.83, N 3.51; **8b**, cryst. colorless needles; 1.52 g (75%), mp °C (147–149); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1589, 1230, 960 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 6.55, 6.59* (d, J_{HP} 26.0 Hz, 1H), 1.51, 154* (s, 3H), 1.89 (m, 2H), 0.92 (t, 3H), 2.54 (m, 2H), 2.00 (d, J_{HP} 10.00 Hz, 1H); 1.28–1.19 (m, 2H); 0.91 (t, 3H). ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 28.3; *Anal.*, Calcd. for $C_{15}H_{22}O_2NPTe$ (M_r = 406.896): P 7.61, N 3.44; Found P 7.58, N 3.40 (*Additional signals for diastereomers); **8c**, cryst. colorless needles; 1.71 g (79%), mp °C (147–149); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1587, 1253, 1004 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 5.87 (d, J_{HP} 23.5 Hz, 1H), 1.68 (m, 10H), 2.54 (m, 2H), 2.00 (d, J_{HP} 10.00 Hz, 1H); 1.28–1.19 (m, 2H); 0.91 (t, 3H); ^{31}P NMR (250

MHz, $CDCl_3$) ppm: 28.3; *Anal.*, Calcd. for $C_{17}H_{24}O_2NPTe$ (M_r = 432.932): P 7.15, N 3.23; Found P 7.11, N 3.20.

2.5. Synthesis of 4-(5-alkyl-5-alkyl-2-oxo-4-phenyltellanyl-2,5-dihydro-2λ^5-[1,2]-oxaphosphol-2-yl) morpholines 10a,b and of 4-(2-oxo-4-phenyltellanyl-1-oxa-2λ^5phospha-spiro[4,5]-dec-3-ene 2-yl)morpholine 10c

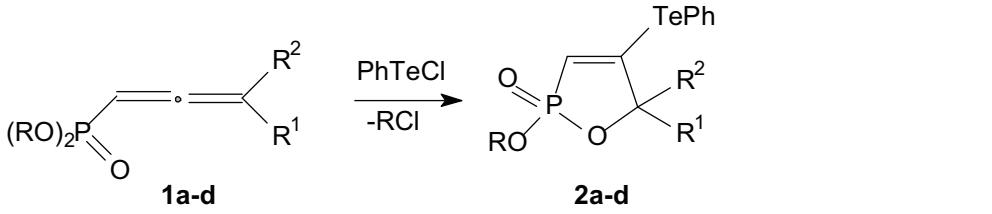
2.5.1. General procedure

To a solution of **9** (5 mmol) in methylene chloride (10 mL) was added a solution of phenyltelluryl chloride (1.24 g, 5.2 mmol) in 5 mL of methylene chloride under stirring and cooling (–10 to –12 °C). After 1 h of stirring at the same conditions, the reaction mixture stood overnight, and was concentrated and recrystallized in heptane/benzene (2:1).

10a, cryst. colorless needles; 1.30 g (62%), mp °C (147–149); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1589, 1225, 1004 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.56–7.46 (m, 2H); 7.29–7.23 (m, 3H); 6.34 (d, J_{HP} 23.0 Hz, 1H); 1.46 (s, 3H); 1.51 (s, 3H), 2.87, 3.76 (m, 8H); ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 33.42; *Anal.*, Calcd. for $C_{15}H_{20}O_3NPTe$ (M_r = 420.88): P 7.36, N 3.33; Found P 7.32, N 3.30; **10b**, cryst. colorless needles; 1.45 g (67%), mp °C (147–149); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1595, 1225, 1000 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 6.55, 6.59* (d, J_{HP} 26.0 Hz, 1H), 1.51, 154* (s, 3H), 1.89 (m, 2H), 0.92 (t, 3H), 2.87, 3.76 (m, 8H). ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 34.12; *Anal.*, Calcd. for $C_{16}H_{22}O_3NPTe$ (M_r = 434.906): P 7.12, N 3.22; Found P 7.09, N 3.18 (*Additional signals for diastereomers); **10c**, cryst. colorless needles; 1.40 g (61%), mp °C (147–149); IR (KBr) ν_{max}/cm^{-1} 2980, 2677, 1589, 1273, 998 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) ppm: 7.86–7.87 (m, 2H), 7.30–7.51 (m, 3H), 5.87 (d, J_{HP} 23.5 Hz, 1H), 1.68 (m, 10H), 2.87, 3.76 (m, 8H). ^{31}P NMR (250 MHz, $CDCl_3$) ppm: 33.22; *Anal.*, Calcd. for $C_{18}H_{24}O_3NPTe$ (M_r = 460.942): P 6.72, N 3.04; Found P 6.69, N 2.99.

3. Results and discussion

In our first report on this subject[19] we demonstrated that the reaction of dialkyl esters of 1,2-alkadienephosphonic acids with phenyltelluryl chloride leads to the formation of 4-phenyltelluro-2,5-dihydro-1,2-oxaphosphole 2-oxide derivatives (Figure 1).

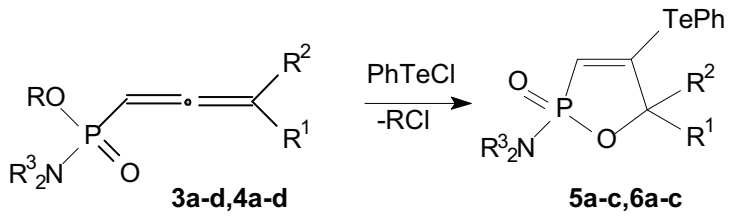


2a, R,R^1,R^2 = Me, 2b, R = Et, R^1 = R^2 = Me, 2c, R = R^1 = Me, R^2 = Et, 2d, R = Me, R^1+R^2 = cyclohexyl

Figure 1. Reaction of dialkyl esters of 1,2-alkadienephosphonic acids with phenyltelluryl chloride.

In 2007, Yuan and co-workers reported the same results using different synthetic conditions.[20]

Continuing our investigations on this reaction, we studied the reaction of N,N-dialkylamido-O-alkyl-1,2-alkadienephosphonates previously described by us,[17] with the same reagent, and established that in all cases with good yields the oxaphosphole derivatives **5a**–**c** and **6a**–**c** were obtained (Figure 2):



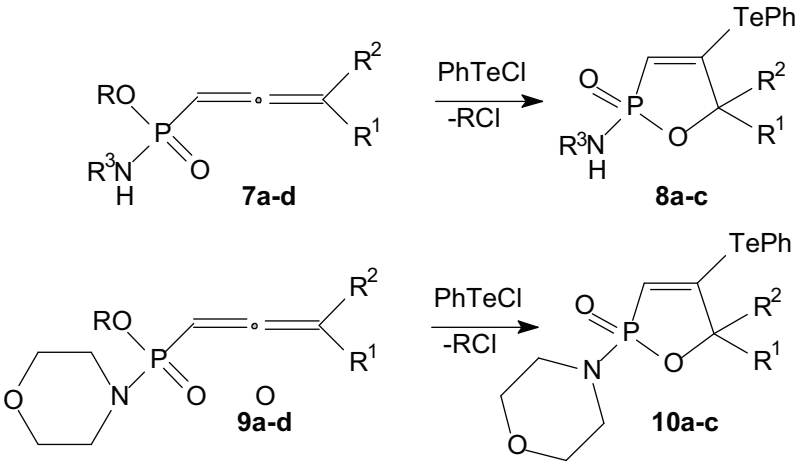
3a-d,4a-d 5a-c,6a-c

5a, $R^1 = R^2 =$ Me, $R^3 =$ Et; 5b, $R^1 =$ Me, $R^2 =$ Et, $R^3 =$ Et; 5c, $R^1+R^2 =$ cyclohexyl, $R^3 =$ Et
6a, $R^1 = R^2 =$ Me, $R^3 = {}^i$Pr; 6b, $R^1 =$ Me, $R^2 =$ Et, $R^3 = {}^i$Pr; 6c, $R^1+R^2 =$ cyclohexyl, $R^3 = {}^i$Pr

R = Me

Figure 2. Reaction of N,N-dialkylamido-O-alkyl-1,2-alkadienephosphonates with phenyltelluryl chloride.

The results reported above encourage us to investigate the reactivity of N-alkylamido-O-alkyl-1,2-alkadienephosphonates as well as the reactivity of N-morpholino-O-alkyl-1,2-alkadienephosphonates also previously reported by us.[18] We expected both substrates to react with phenyltelluryl chloride with formation of the corresponding 2,5-dihydro-1,2-oxaphosphole 2-oxide derivatives (Figure 3).



7a-d 8a-c 9a-d 10a-c

8a, $R^1 = R^2 =$ Me, $R^3 =$ Pr; 8b, $R^1 =$ Me, R^2=Rt, $R^3 =$ Pr; 8c, $R^1+R^2 =$ cyclohexyl, $R^3 =$ Pr
10a, $R^1 = R^2 =$ Me, 10b, $R^1 =$ Me, $R^2 =$ Et; 10c, $R^1+R^2 =$ cyclohexyl

R = Me

Figure 3. Reaction of N-alkylamido-O-alkyl-1,2-alkadienephosphonates and of N-morpholino-O-alkyl-1,2-alkadienephosphonates with phenyltelluryl chloride.

All the synthetic results obtained as well as our previous experience[5] give us reason to suggest the following plausible mechanism of the telluro-induced cyclization of 1,2-alkadienephosphonates (Figure 4):

The attack of the reagent affecting the C^2–C^3 double bond of the allenephosphonate system leads to the formation of "onium" intermediate **A**, which is in equilibrium with carbocation **B**. The latter can be transformed to quaziphosphonium intermediate **C**, which undergoes dealkylation (Michalis–Arbuzov reaction – second stage) to afford the final 2,5-dihydro-1,2-oxaphosphole 2-oxide derivatives **2**, **5**, **6**, **8**, and **10**.

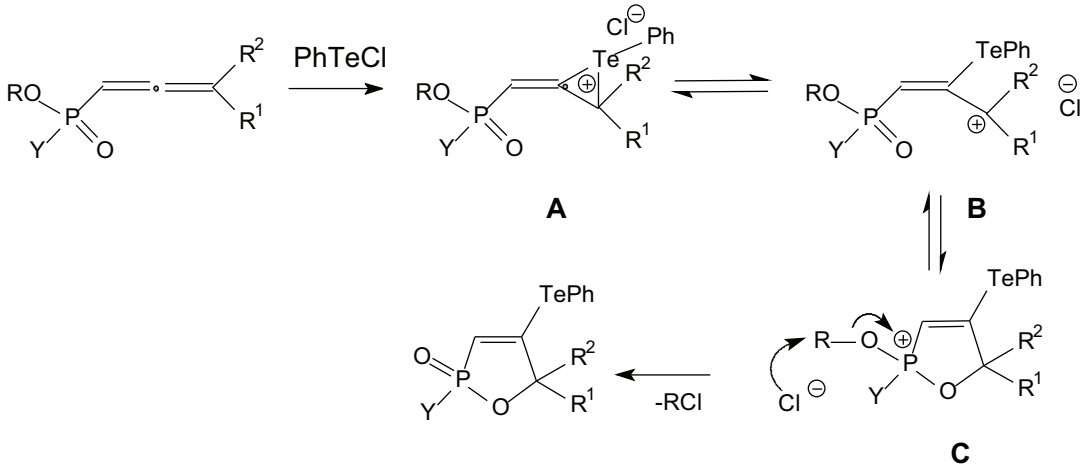


Figure 4. Plausible mechanism of the telluro-induced cyclization of 1,2-alkadienephosphonates.

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