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$\lambda^3\sigma^3$P and $\lambda^5\sigma^5$P Derivatives of (*E*)-1,2-Difluoro-2-(pentafluoro-λ^6-sulfanyl)ethylene and (*Z*)-1,2,3,3,3-Pentafluoropropylene

Holger Wessolowski, Andreas Gentzsch, and Gerd-Volker Röschenthaler*

Institut für Anorganische und Physikalische Chemie, Universtät Bremen, Leobener Strasse, 28334 Bremen, Germany

Gary L. Gard

Department of Chemistry, Portland State University, Portland, Oregon 97207-0751

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ABSTRACT

For the first time, the (E)-1,2-difluoro-2-(pentafluoro-λ^6-sulfanyl)ethenyl group has been bonded to $\lambda^3\sigma^3$ phosphorus using a Grignard reagent. Similar phosphorus derivatives containing the (Z)-1,2,3,3,3-pentafluoropropenyl moiety were also synthesized for comparison. In three cases, hexafluoroacetone was added to form 4,4,5,5-tetrakis(trifluoromethyl) 1,3,2$\lambda^5\sigma^5$-dioxaphospholanes. © 1997 John Wiley & Sons, Inc. Heteroatom Chem **8**:467–471, 1997

INTRODUCTION

The fluoroalkenes $R^FC(R)=CF_2$ (R^F = SF_5, CF_3; R = F, H) show similarities in their reactions with silylated phosphites and tri-*n*-butylphosphine [1–4]. Grignard reagents prepared from iodoperfluoroalkenes, $CF_2=CFI$ or (*Z*)-$CF_3CF=CFI$, have been used to obtain the respective phosphonites [5–8]. Compounds of the type (*Z*)-$CF_3CF=CFPR_2$ (R = OEt, NEt_2) were reacted with hexafluoroacetone to give a 1,3,2-$\lambda^5\sigma^5$-dioxaphospholane for R = OEt (1:2 molar ratio) and, surprisingly, a 1,2$\lambda^5\sigma^5$-oxaphospholene-(3) for R = NEt_2 (1:1 molar ratio) [9]. Since (*E*)-1,2-difluoro-1-iodo-2-(pentafluoro-λ^6-sulfanyl)-ethylene is now available [2,10], preparation of phosphonites (*E*)-$F_5SCF=CFPR_2$ is possible, and these compounds can be compared with their (*Z*)-$CF_3CF=CFPR_2$ analogues.

RESULTS AND DISCUSSION

The Grignard reagents "(*E*)-$F_5SCF=CFMgI$" and "(*Z*)-$CF_3CF=CFMgI$" have now been generated from magnesium and (*E*)-1,2-difluoro-1-iodo-2-(pentafluoro-λ^6-sulfanyl)ethylene (**1**) [2,10] or (*Z*)-1,2,3,3,3-pentafluoro-1-iodo-propylene **2** [11] and reacted with some selected phosphorus(III) chlorides $ClPR^1R^2$, **3a–e** [R^1 = R^2 = OEt (**a**) [12]; R^1 = OMe, R^2 = NMe_2 (**b**); R^1 = OEt, R^2 = NEt_2 (**c**); R^1R^2 = OCH_2CH_2NMe (**d**); R^1 = R^2 = *i*Pr (**e**)] to yield the $\lambda^3\sigma^3$P derivatives **4a–e** or **5a–e** (Scheme 1). Chlorobis(diethylamido) phosphite did not yield the expected phosphonoamidite (*E*)-$R^FCF=CFP(NEt_2)_2$ (R^F = F_5S) under the reaction conditions applied; a nonseparable mixture containing nonidentified species having been formed. The analogue with R^F =

*To whom correspondence should be addressed.
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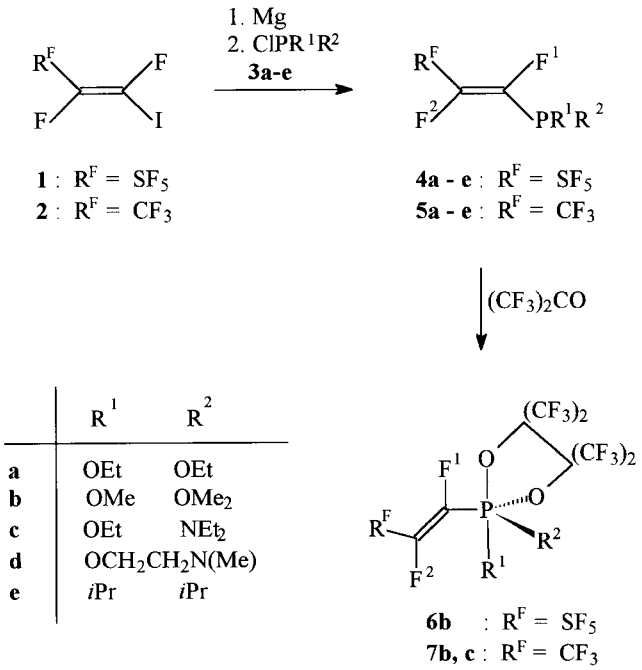

SCHEME 1

CF_3, however, had previously been prepared [8]. Hexafluoroacetone has now been added to **4b, 5b,** and **5c** in a 2:1 stoichiometry to furnish the 4,4,5,5-tetrakis(trifluoromethyl)-1,3,2$\lambda^5\sigma^5$-dioxaphospholanes **6b, 7b,** and **7c,** exclusively, as in the case of **5a** [9] (Scheme 1). In the case where the $R^FCF=CF$ group was involved in the ring formation, no 1:1 addition product was obtained [9]. The new compounds were colorless, moisture- and oxygen-sensitive liquids.

The ^1H, ^{19}F, and ^{31}P NMR data for compounds **4–7** support the proposed structures (Tables 1 and 2). The phosphorus resonances were found in the expected range [3]. Replacing the SF_5 group with CF_3 did not significantly alter the δ_P values for the same substituent R. The large coupling constants $^3J_{F^1F^2}$ (>136 Hz) are due to *trans* substitution for all compounds [1–3]. In the case of the amidophosphonite **4c,** the overlapping signals for F^1 and F^2 made it impossible to observe $^3J_{F^1F^2}$. The F^1 and F^2 resonances for the species containing the SF_5 group (**4** and **6**) appear at lower field than the respective signals for CF_3 analogues whose F^1 resonances for the $\lambda^3\sigma^3$P species (**5**) were clearly observed at higher field than for the F^2 signals; the opposite being found for the two phosphoranes (**7b** and **7c**). The difference $|\delta_F(F^1) - \delta_F(F^2)|$ is much smaller for the SF_5-substituted compounds. Coupling constants $^2J_{PF}$, $^3J_{F^1F^2}$, and $^3J_{F^2P}$ are of comparable magnitude for the two classes of phosphorus derivatives. Two broad signals were found for the four CF_3 groups bonded to the dioxaphospholane ring system in the $\lambda^5\sigma^5$P species **6b** and **7b,** due to a slowing down on the NMR time scale, at room temperature, of the first step of a two-step pseudorotation process [14].

EXPERIMENTAL

The appropriate precautions in handling moisture- and oxygen-sensitive compounds were observed throughout this work. Elemental analysis: Mikroanalytisches Laboratorium Beller, Göttingen. MS: MAT 8222 (EI, electron energy 70 eV). IR: BioRad Digilab FTS-7 spectrometer, liquids or solids as capillary film between NaCl disks. NMR: AC 80, operating at 80.13 MHz (^1H, internal standard TMS), 75.39 MHz (^{19}F, internal standard CCl_3F), and 32.44 MHz (^{31}P, external standard 85% H_3PO_4). Compounds **1** [2], **2** [11], and **3a–e** [15–19] were prepared according to literature procedures.

General Procedure for the Synthesis of Compounds 4–7 (see Table 3). In a typical experiment, the iodoolefins **1** or **2** (15 mmol) in 15 mL of diethyl ether were added at −40°C (**1**) or 20°C (**2**) to magnesium (3.6 g, 15 mmol) in 10 mL of diethyl ether. The mixture was reacted at −20 ÷ −10°C (**1**) or −10 ÷ 0°C (**2**) and stirred for 4 hours until the magnesium had completely disappeared. The phosphorus chloride (15 mmol) in 15 mL of diethyl ether was added at −80°C (**1**) or at −10°C (**2**), the reaction mixture then being allowed to warm to ambient temperature and stirred for 18 hours. All volatile materials were pumped off at 0.001 mm and collected. The fractional distillation furnished colorless products.

*(E)-[1,2-Difluoro-1-(pentafluoro-λ⁶-sulfanyl)]-ethenyl-diethylphosphonite (**4a**).* MS(120°C) *m/e* (%): 310 (M^+, 4), 271 ($M^+ - F - HF$, 27), 265 ($M^+ - OC_2H_5$, 6), 254 ($M^+ - 2\ C_2H_4$, 7), 127 (SF_5^+, 21), 121 [$P(OC_2H_5)_2^+$, 58], 105 [$PC_2H_5(OC_2H_5)^+$, 82], 89 (SF_3^+, 39), 77 [$P(H)OC_2H_5^+$, 52], 65 [$P(OH)_2^+$, 100], and other fragments. IR: $\tilde{\nu}$ (cm^{-1}): 1672 w (C=C), 879 vst (SF), 598 st (SF), and other bands. Anal. calcd for $C_6H_{10}F_7O_2PS$ (310.17): C, 23.23; H, 3.25; F, 42.88; P, 9.99. Found: C, 22.88; H, 3.16; F, 41.90; P, 9.61.

*(E)-[1,2-Difluoro-1-(pentafluoro-λ⁶-sulfanyl)]-ethenyl-dimethylamidomethyl-phosphonite (**4b**).* MS (20°C) *m/e* (%): 295 (M^+, 18), 264 ($M^+ - OCH_3$, 5), 251 [$M^+ - N(CH_3)_2$, 6], 127 (SF_5^+, 14), 106 [$P(OCH_3)N(CH_3)_2^+$, 100], 94 [$FPN(CH_3)_2^+$, 80], 89 (SF_3^+, 39), 81 ($C_2F_3^+$/$FPOCH_3^+$, 35), and other fragments. IR: $\tilde{\nu}$ (cm^{-1}): 1616 w (C=C), 877 vst (SF), 596

TABLE 1 ^{1}H, ^{19}F, and ^{31}P NMR Data for Compounds **4** and **6** (*J* values are given in Hz)

	δ_H^a				δ_F^a				δ_P^a
Compounds	$CH_3(OR)$ ($^3J_{PH}$)	$CH_3(NR_2)$ ($^3J_{PH}$)	$CH_2(OR)$ ($^3J_{PH}$, $^3J_{HH}$)	$CH_2(NR_2)$ ($^3J_{PH}$, $^3J_{HH}$)	F^1 ($^2J_{PF}$, $^4J_{FF^4}$)	F^2 ($^3J_{F^1F}$)	$SF^3{}_4F^4$ ($^3J_{F^2F}$)	$SF^3{}_4F^4$ ($^3J_{F^2F}$)	($^3J_{F^2P}$)
4a	1.3		4.1 (8.4, 7.0)		−142.9[b] (55.2, 3.8)	−146.5[c] (136.0)	57.3 (20.8)	72.5 (2.5)	134.8 (34.5)
4b	3.6 (14.0)	2.8 (9.5)			−147.6[f] (41.5, 3.7)	−148.1[g]	52.9[d] (18.8)	68.5[e] (2.7)	119.4 (35.3)
4c	1.3	1.1	3.9 (10.0, 7.0)	3.2 (9.8, 7.1)	−140.8–142.1[h]		57.1[d]	73.8[e]	118.7
4d		2.8 (13.4)	4.3[i]	3.1[i]	−151.1[c] (52.0, 3.7)	−147.9[c] (136.2)	53.0[d] (19.8)	68.0[e] (2.5)	113.8 (24.0)
4e[j]	1.3[k] (5.2)				−133.5[f] (62.5, 3.7)	−133.2[g]	57.6[d] (17.9)	73.2[e] (2.9)	7.2 (25.1)
6b[l]	3.5 (13.6)	2.7 (11.6)			−143.5[m] (83.5, 2.5)	−139.6[c] (136.6)	52.1[d] (22.2)	66.9[c] (4.0)	−44.7

[a]Highfield shifts from TMS, CCl_3F, and 85% H_3PO_4 were given negative signs
[b]ddd.
[c]ddpd.
[d]dm.
[e]Signal splits into 9 lines, Ref. [13].
[f]dd.
[g]dpd, $^3J_F^{1F}$ not obtained.
[h]Overlapping multiplets.
[i] m.
[j]$\delta_H = 2.3$
[k]$CH(CH_3)_2$.
[l]$\delta_F = -72.2$ (CF_3, 6 F), -73.9 (CF_3, 6 F).
[m]ddpdm. [$CH(CH_3)_2$, 2 H, m, $^3J_{HH} = 7.0$].

TABLE 2 ^{1}H, ^{19}F, and ^{31}P NMR Data for Compounds **5** and **7** (*J* values are given in Hz)

	δ_H^a				δ_F^a			δ_P^a
Compounds	$CH_3(OR)$ ($^3J_{PH}$)	$CH_3(NR_2)$ ($^3J_{PH}$)	$CH_2(OR)$ ($^3J_{PH}$, $^3J_{HH}$)	$CH_2(NR_2)$ ($^3J_{PH}$, $^3J_{HH}$)	F^1 ($^2J_{PF}$, $^4J_{FF^3}$)	F^2 ($^3J_{F^1F}$)	CF_3^3 ($^3J_{F^2F}$, $^4J_{PF}$)	($^3J_{F^2P}$)
5a[b]	1.2		3.7–4.2[c] (−, 7.1)		−171.5 (52.3, 10.1)	−158.1 (142.0)	−72.4 (21.2, 3.8)	134.2 (31.2)
5b	3.6 (13.9)	2.8 (9.0)			−172.0 (40.0, 10.3)	−155.7 (142.0)	−72.2 (21.5, 4.9)	117.5 (33.4)
5c	1.3	1.1	3.9 (10.1, 7.3)	3.2 (9.6, 7.1)	−170.9 (40.3, 10.3)	−154.1 (21.4, 5.2)	−72.4 (41.7)	112.3
5d		2.8 (13.5)	4.3 (7.8, 6.8)	3.1 (7.9, 6.8)	−175.3 (50.0, 10.5)	154.2 (140.3)	−72.3 (21.3, 1.8)	111.5 (20.5)
5e[d]	1.2 (4.7)				−160.9 (70.5, 10.3)	−146.3 (150.5)	−71.7 (21.3, 1.8)	−4.3 (7.4)
7b[e]	3.7 (13.3)	2.9 (11.3)			−150.9 (86.7, 21.6)	−163.3 (137.8)	−72.4 (9.5, 0.5)	−45.9 (11.5)
7c[f]	1.3	1.1	3.9–4.3 (−, 8.3)	3.3 (14.3, 7.1)	−148.0 (90.5, 21.7)	−160.4 (136.6)	−72.3 (9.2, 0.6)	−46.7 (12.5)

[a]Highfield shifts from TMS, CCl_3F, and 85% H_3PO_4 were given negative signs.
[b] Ref. [8].
[c]ABM_3X spin system, not resolved.
[d]$\delta_H = 2.2$ ($^2H_{PH} = 14.0$, $^3J_{HH} = 6.9$).
[e]$\delta_F = -71.5 \div -72.0$ (CF_3, 6 F), $-73.0 \div -73.9$ (CF_3, 6 F).
[f]$\delta_F = -71.2 \div -73.4$ (CF_3, 12 F).

TABLE 3 Experimental Details for the Preparation of Compounds **4** and **5** (for **5a,** see Ref. [8])

Compound	*Reactants [g (mmol)]*	*Yield [g (%)]*	*B.p. (°C/mm)*
4a	**1**: 4.0 (12.7); Mg: 0.3 **3a**: 2.0 g (12.7)	2.0 (50)	12/20
4b	**1**: 4.5 (14.1); Mg: 0.3 **3b**: 2.0 (13.8)	1.9 (45)	60/20
4c	**1**: 4.4 (13.9); Mg: 0.3 **3c**: 2.6 (13.9)	3.0 (65)	90/20
4d	**1**: 3.7 (11.8); Mg: 0.3 **3d**: 1.6 (11.8)	1.0 (28)	47/20
4e	**1**: 5.8 (18.3); Mg: 0.5 **3e**: 2.8 (18.0)[a]	3.6 (65)	74/20
5b	**2**: 12.9 (50); Mg: 1.2 **3b**: 7.1 (50)	8.4 (71)	38/12
5c	**2**: 19.4 (75); Mg: 1.8 **3c**: 13.8 (75)	15.6 (74)	67/12
5d	**2**: 6.5 (25); Mg: 0.6 **3d**: 4.6 (25)	2.9 (49)	55/12
5e	**2**: 6.5 (25); Mg: 0.6 **3e**: 3.8 (25)	2.1 (34)	59/12

[a]Addition of **3e** at −30°C.

vst (SF), and other bands. Anal. calcd for $C_5H_9F_7NOPS$ (295.16): C, 20.35; H, 3.07; F, 45.06; P, 10.49. Found: C, 20.33; H, 3.01; F, 44.80; P, 10.26.

*(E)-[1,2-Difluoro-1-(pentafluoro-λ^6-sulfanyl)]-ethenyl-diethylamidoethyl-phosphonite (***4c***).* MS (140°C) *m/e* (%): 337 (M^+, 29), 322 ($M^+ - CH_3$, 7), 292 ($M^+ - OC_2H_5$, 9), 237 [$M^+ - N(C_2H_5)_2 - C_2H_4$, 23], 210 ($M^+ - SF_5$, 2), 148 [$P(OC_2H_5)N(C_2H_5)_2^+$, 100], 120 [$P(OH)N(C_2H_5)_2^+$, 89], 89 ($SF_3^+$, 7), 72 [$N(C_2H_5)_2^+$, 18], and other fragments. IR: $\tilde{\nu}$ (cm^{-1}): 1635 vw (C=C), 873 vst (SF), 596 vst (SF), and other bands. Anal. calcd for $C_8H_{15}F_7NOPS$ (337.24): C, 28.49; H, 4.48; F, 39.43; P, 19.18. Found: C, 28.31; H, 4.42; F, 39.20; P, 19.07.

*2-[(E)-[1,2-Difluoro-1-(pentafluoro-λ^6-sulfanyl)]-ethenyl]-3-methyl-1,3,2$\lambda^3\sigma^3$-oxazaphospholan (***4d***).* MS (20°C) *m/e* (%): 293 (M^+, 54), 127 (SF_5^+, 14), 104 ($POC_2H_4NCH_3^+$, 100), 89 ($SF_3^+/POC_2H_4N^+$, 17), and other fragments. IR: $\tilde{\nu}$ (cm^{-1}): 1642 vw (C=C), 875 vst (SF), 595 vst (SF), and other bands. Anal. calcd for $C_5H_7F_7NOPS$ (293.14): C, 20.49; H, 2.41; F, 45.37; P, 10.57. Found: C, 20.11; H, 2.31; F, 44.60; P, 10.00.

*(E)-[1,2-Difluoro-I-(pentafluoro-λ^6-sulfanyl)]-ethenyl-diisopropylphosphine (***4e***).* MS (20°C) *m/e* (%): 306 (M^+, 20), 264 ($M^+ - C_3H_6$, 3), 189 ($SF_5CF=CF^+$, 3), 127 (SF_5^+, 21), 89 (SF_3^+, 39), 43 ($C_3H_7^+$, 100), and other fragments. IR: $\tilde{\nu}$ (cm^{-1}): 1604 vw (C=C), 878 vst (SF), 595 vst (SF), and other bands. Anal. calcd for $C_8H_{14}F_7PS$ (306.22): C, 31.38; H, 4.61; F, 43.43; P, 10.11. Found: C, 31.17; H, 4.60; F, 42.80; P, 9.97.

*(Z)-1,2,3,3,3-Pentafluoropropenyl-dimethylamidomethylphosphonite (***5b***).* MS (150 °C) *m/e* (%): 237 (M^+, 47), 206 ($M^+ - OCH_3$, 5), 193 [$M^+ - N(CH_3)_2$, 9], 106 [$P(OCH_3)N(CH_3)_2^+$, 100], 94 [$FPN(CH_3)_2^+$, 20], 93 ($C_3F_3^+$, 22), 81 ($C_2F_3^+/FPOCH_3^+$, 34), 76 [$HPN(CH_3)_2^+$, 33], 69 (CF_3^+, 16), 63 ($HPOCH_3^+$, 36), 60 ($PNCH_3^+$, 19), and other fragments. IR: $\tilde{\nu}$ (cm^{-1}): 1677 vw (C=C). Anal. calcd for $C_6H_9F_5NOP$ (237.11): C, 30.39; H, 3.38; F, 40.06; P, 13.06. Found: C, 30.21; H, 3.64; F, 40.10; P, 13.02.

*(Z)-1,2,3,3,3-Pentafluoropropenyl-diethylamidoethylphosphonite (***5c***).* MS (150°C) *m/e* (%): 279 (M^+, 33), 264 ($M^+ - CH_3$, 15), 234 ($M^+ - OC_2H_5$, 15), 220 ($M^+ - CH_3 - HNC_2H_5$, 15), 179 ($CF_3CF=CFPOH^+$, 32), 148 [$P(OC_2H_5)N(C_2H_5)_2^+$, 93], 120 [$P(OH)N(C_2H_5)_2^+$, 100], 72 [$N(C_2H_5)_2^+$, 22], 76 [$HPN(CH_3)_2^+$, 33], and other fragments. IR: $\tilde{\nu}$ (cm^{-1}): 1679 vw (C=C). Anal. calcd for $C_9H_{15}F_5NOP$ (279.19): C, 38.72; H, 5.42; F, 34.02; P, 11.09. Found: C, 38.69; H, 5.48; F, 34.00; P, 11.01.

*2-[(Z)-1,2,3,3,3-Pentafluoropropenyl]-3-methyl-1,3,2$\lambda^3\sigma^3$-oxaza-phospholan (***5d***).* MS (140°C) *m/e* (%): 235 (M^+, 9), 104 ($POC_2H_4NCH_3^+$, 100), 93 ($C_3F_3^+$, 6), 69 (CF_3^+, 5), 60 ($PNCH_3^+$, 19), and other fragments. IR: $\tilde{\nu}$ (cm^{-1}): 1668 vw (C=C) and other bands. Anal. calcd for $C_6H_7F_5NOP$ (235.10): C, 30.65; H, 3.00; F, 40.41; P, 13.18. Found: C, 30.34; H, 3.14; F, 40.00; P, 13.42.

*(Z)-1,2,3,3,3-Pentafluoropropenyl-diisopropylphosphine (***5e***).* MS (140°C) *m/e* (%): 248 (M^+, 6), 206 ($M^+ - C_3H_6$, 3), 43 ($C_3H_7^+$, 100), and other fragments. IR: $\tilde{\nu}$ (cm^{-1}): 1674 w (C=C) and other bands. Anal. calcd for $C_9H_{14}F_5P$ (248.18): C, 43.56; H, 5.69; F, 38.28; P, 12.48. Found: C, 42.53; H, 5.61; F, 35.60; P, 12.24.

*2-[(E)-[1,2-Difluoro-1-(pentafluoro-λ^6-sulfanyl)]-ethenyl]-2-dimethylamino-2-methoxy-4,4,5,5-tetrakis(trifluoromethyl)-1,3,2$\lambda^5\sigma^5$-dioxaphospholane (***6b***).* Compound **4b** (0.8 g, 2.6 mmol) in 5 mL diethylether and 1.6 g (6.6 mmol) hexafluoroacetone were reacted for 4 days. Fractional distillation at 43°C/0.001 mm yielded 1.1 g (67.6%) **6b.** MS (150°C) *m/e* (%): 627 (M^+, >0.05), 608 ($M^+ - F$, 10), 596 ($M^+ - OCH_3$, 46), 583 [$M^+ - N(CH_3)_2$, 100], 558 ($M^+ - CF_3$, 26), 438 ($M^+ - SF_5CF=CF$, 18), 127 (SF_5^+, 14), 106 [$P(OCH_3)N(CH_3)_2^+$, 15], 89 (SF_3^+, 4), 69 (CF_3^+, 19), and other fragments. IR: $\tilde{\nu}$ (cm^{-1}): 1653 vw (C=C), 879 vst (SF), 597 vst (SF), and other bands. Anal.

calcd for $C_{11}H_9F_{19}NO_3PS$ (627.20): C, 21.07; H, 1.45; P, 4.94. Found: C, 21.36; H, 1.83; P, 5.63.

*2-[(Z)-1,2,3,3,3-Pentafluoropropenyl]-2-dimethylamino-2-methoxy-4,4,5,5-tetrakis(trifluoromethyl)-1,3,2$\lambda^5\sigma^5$-dioxaphospholane (***7b***).* Compound **5b** (2.0 g, 8.5 mmol) and 2.8 g (17.0 mmol) hexafluoroacetone were reacted for 12 hours. Fractional distillation at 41°C/0.001 mm yielded 4.5 g (93.2%) **7b.** MS (150 °C) *m/e* (%): 550 ($M^+ - F$, 8), 538 ($M^+ - OCH_3$, 47), 525 [$M^+ - N(CH_3)_2$, 100], 500 ($M^+ - CF_3$, 28), 438 ($M^+ - CF_3CF{=}CF$, 10), 106 [$P(OCH_3)N(CH_3)_2^+$, 30], 81 ($C_2F_3^+$/$FPOCH_3^+$, 24), 69 (CF_3^+, 38), and other fragments. IR: $\tilde{\nu}$ (cm^{-1}): 1682 vw (C=C). Anal. calcd for $C_{12}H_9F_{17}NO_3P$ (569.16): C, 25.32; H, 1.59; F, 56.75; P, 5.44. Found: C, 25.44; H, 1.71; F, 56.20; P, 5.58.

*2-[(Z)-1,2,3,3,3-Pentafluoropropenyl]-2-diethylamino-2-ethoxy-4,4,5,5-tetrakis(trifluoromethyl)-1,3,2$\lambda^5\sigma^5$-dioxaphospholane (***7c***).* Compound **5c** (4.2 g, 15.0 mmol) and 5.0 g (30.0 mmol) hexafluoroacetone were reacted for 12 hours. Fractional distillation at 52°C/0.001 mm yielded 7.7 g (83.7%) **7c.** MS (150°C) *m/e* (%): 566 ($M^+ - OC_2H_5$, 33), 511 [$M^+ - N(C_2H_5)_2^- C_2H_4$, 100], 345 [$CF_3CF{=}CFP(OH){=}C(CF_3)_2^+$, 38], 148 [$CF_3CF{=}CF(OH)^+$, 20], 122 [$FPN(C_2H_5)_2^+$, 29], 97 ($OCCF_3^+$, 29), 69 ($CF_3^+$, 48), and other fragments. IR: $\tilde{\nu}$ (cm^{-1}): 1688 vw (C=C). Anal. calcd for $C_{15}H_{15}F_{17}NO_3P$ (611.24): C, 29.48; H, 2.47; F, 52.84; P, 5.07. Found: C, 29.79; H, 2.64; F, 51.90; P, 5.24.

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