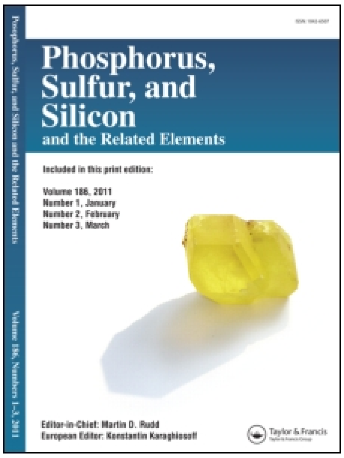


Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

[2,2-DIFLUORO-1-(PENTAFLUORO-λ⁶- SULFANYL)]ETHYLENE AND 1,1,3,3,3- PENTAFLUOROPROPENE: REACTIONS WITH DIETHYL(TRIMETHYLSILYL) AND TRIS(TRIMETHYLSILYL)PHOSPHITE

Holger Wessolowski[a]; Gerd-Volker Roschenthaler[a]; Rolf Winter[b]; Gary L. Gard[a]
[a] Institut für Anorganische and Physikalische Chemie, Bremen, F.R.G. [b] Department of Chemistry, Portland State University Portland, Oregon, U.S.A.

To cite this Article Wessolowski, Holger , Roschenthaler, Gerd-Volker , Winter, Rolf and Gard, Gary L.(1991) '[2,2-DIFLUORO-1-(PENTAFLUORO-λ⁶- SULFANYL)]ETHYLENE AND 1,1,3,3,3- PENTAFLUOROPROPENE: REACTIONS WITH DIETHYL(TRIMETHYLSILYL) AND TRIS(TRIMETHYLSILYL)PHOSPHITE', Phosphorus, Sulfur, and Silicon and the Related Elements, 60: 3, 201 — 208

To link to this Article: DOI: 10.1080/10426509108036782
URL: http://dx.doi.org/10.1080/10426509108036782

Phosphorus, Sulfur, and Silicon, 1991, Vol. 60, pp. 201–208
Reprints available directly from the publisher
Photocopying permitted by license only

[2,2-DIFLUORO-1-(PENTAFLUORO-λ^6-SULFANYL)]ETHYLENE AND 1,1,3,3,3-PENTAFLUOROPROPENE: REACTIONS WITH DIETHYL(TRIMETHYLSILYL) AND TRIS(TRIMETHYLSILYL)PHOSPHITE

HOLGER WESSOLOWSKI,[a] GERD-VOLKER RÖSCHENTHALER,*[a]
ROLF WINTER[b] and GARY L. GARD*†[b]
[a]*Institut für Anorganische und Physikalische Chemie, Leobener Straße, 2800 Bremen 33, F.R.G.;* [b]*Department of Chemistry, Portland State University Portland, Oregon 97207, U.S.A.*

(Received November 12, 1990; in final form November 21, 1990)

The 2-H olefins, $R^1CH{=}CF_2$ (**1**: CF_3 **2**: SF_5) react with the silylphosphites $(R^2O)_2POSiMe_3$ (**3a**: R^2 = Et; **3b**: R^2 = $SiMe_3$) to yield the (E/Z) phosphonates $R^1CH{=}CFP(O)(OR^2)_2$, **4a/b** and **5a/b** and fluorotrimethylsilane. In the case of R^2 = $SiMe_3$ hydrolyses furnished the acids $R^1CH{=}CFP(O)(OH)_2$, **4c** and **5c**. The preparation of (E)-$SF_5CF{=}CFP(O)(OH)_2$, **7** is reported, too.

Key words: 1,1,3,3,3-pentafluoropropene; 2,2-difluoro-1-(pentafluoro-λ^6-sulfanyl)ethylene; diethyltrimethylsilylphosphite; tris(trimethylsilyl)phosphite; phosphonates; phosphonic acids.

INTRODUCTION

Fluoroalkenylphosphonates[1] are precursors for potential pharmaceuticals[2] and fuel cell electrolytes.[3] One straightforward preparative route has been shown to be an ARBUZOV type reaction of hexafluoropropene, $CF_3CF{=}CF_2$, and silylated phosphites,[4] e.g., $(EtO)_2POSiMe_3$ or $(Me_3SiO)_3P$. Products were fluorotrimethylsilane and $CF_3CF{=}CFP(O)(OEt)_2$ (Z/E = 97:3) or $CF_3CF{=}CFP(O)(OSiMe_3)_2$ (Z/E = 99:1), which yielded $CF_3CF{=}CFP(O)(OH)_2$ upon treating with methanol. Similarly (E)−$F_5SCF{=}CFP(O)(OEt)_2$ and (E)−$F_5SCF{=}CFP(O)(OSiMe_3)_2$ could be obtained from $SF_5CF{=}CF_2$.[5] In this work, we report the influence of a hydrogen atom in 2-position for the reaction of the olefins $CF_3CH{=}CF_2$[6] and $F_5SCH{=}CF_2$[7] with $(EtO)_2POSiMe_3$ and $(Me_3SiO)_3P$. Also, new SF_5 containing phosphonic acids are reported for the first time.

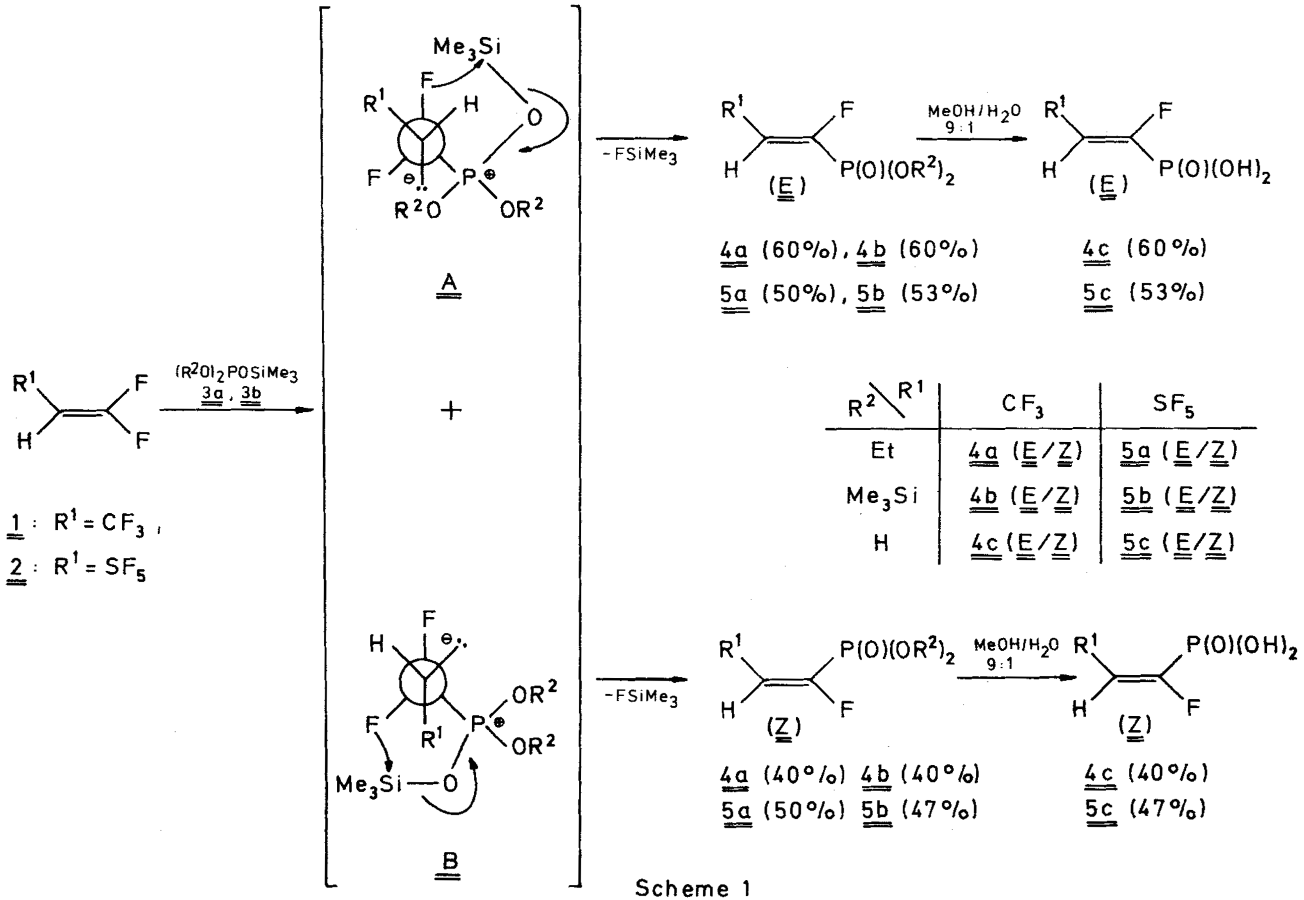
RESULTS AND DISCUSSION

1,1,3,3,3-Pentafluoropropene, $CF_3CH{=}CF_2$ (**1**) and 2,2-difluoro-1-(pentafluoro-λ^6-sulfanyl)ethylene, $F_5SCH{=}CF_2$ (**2**) reacted with diethyl(trimethylsilyl)phos-

---

* Correspondence to: Prof. G.-V. Röschenthaler, Institut für Anorganische und Physikalische Chemie, Leobener Straße, 2800 Bremen 33, F.R.G.

† Senior Fulbright Fellow 1989/90 at the University of Bremen.



R² \ R¹	CF₃	SF₅
Et	4a (E/Z)	5a (E/Z)
Me₃Si	4b (E/Z)	5b (E/Z)
H	4c (E/Z)	5c (E/Z)

Scheme 1

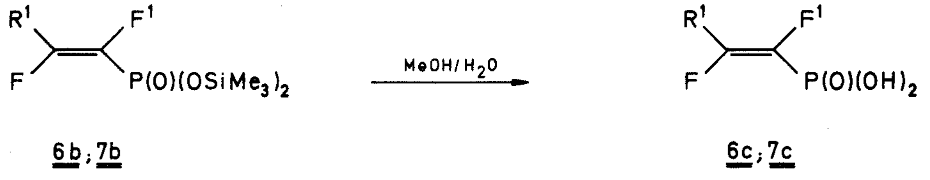
phite (**3a**) and tris(trimethylsilyl)phosphite (**3b**) to yield the phosphonates **4a/b**, **5a/b** and fluorotrimethylsilane (Scheme 1). The new compounds were colorless, moisture-sensitive liquids. Unexpectedly the 2-H olefins gave E/Z product ratios of 60:40 ($R^1 = CF_3$), 50:50 ($R^1 = SF_5$, $R^2 = Et$) and 53:47 ($R^1 = SF_5$, $R^2 = SiMe_3$). For the systems $R^1CF{=}CF_2/(R^2O)_2POSiMe_3$ ($R^1 = CF_3$, SF_5; $R^2 = Et$, $SiMe_3$) one observes a dramatic change in the ratio of the E/Z isomers; in this work the proposed intermediates **A** and **B** (Scheme 1) are of similar energy thereby alternating the expected E/Z ratios as found for the CF_3 derivatives.[8] Attempts to separate the (E)- and (Z)-isomers by distillation failed.

Hydrolysis of the (E/Z) phosphonates **4b** and **5b** in methanol led to the corresponding phosphonic acids **4c** and **5c** with no change in configuration; both acids are extremely hygroscopic solids (Scheme 1). Using the same method, (Z)$-CF_3CF{=}CFP(O)(OSiMe_3)_2$, **6b**, and (E)$-F_5SCF{=}CFP(O)(OSiMe_3)_2$, **7b**, could be converted into the corresponding phosphonic acids, **6c**[8] and **7c**, Scheme 2. In this manner, the first SF_5 containing phosphonic acids have been prepared.

The mass spectra (70 eV) of the new compounds (E/Z)-**4a–c** and (E/Z)-**5a,b** contained no molecular ions. The peaks of highest M/Z value were $[MH]^+$ for **4a**, **4c**, **5a**, and **5c** (see Reference 9); $[M - CH_3]^+$ for **4b** and **5b**. The fragment of highest abundance for **4a**, **4c**, **5a** was $P(OH)_2^+$; for **4b**, **5b** $Me_3SiOSiMe_2^+$; and for **5c** F_2PO^+. Additional characteristic ions found were $[M - CF_3]^+$, **4a**, **4b**; $[M-SF_5]^+$, **5a**, **5b**, **5c**; and SF_5^+, **5a–c**.

The C—C bond vibration for **4a–c** isomers was found in the 1656 to 1685 cm^{-1} region; for **5a–c** the 1630–1660 cm^{-1} region. The C—F and P—O stretching modes were found to overlap thereby preventing a definite assignment. Broad P—OH absorption bands were located at 2836 and 2275 cm^{-1} (**4a**); at 2792, 2294 cm^{-1} (**5c**) and at 2815 cm^{-1} (**7c**). These absorption bands are attributed to intermolecular hydrogen bonding.[10]

Structures (configurations) of the isomeric mixtures were determined from the corresponding coupling constants $^3J_{F1H}$, $^3J_{PH}$ and $^4J_{F1F2}$ (see Tables I and II). The values for *trans* $^3J_{F3H}$ [(E)-isomers] and for *trans* $^3J_{PH}$ [(Z)-isomers] should be larger than the corresponding *cis* couplings[11,12] (see Table III); the opposite applies for



6b; 7b

6c; 7c

	R^1
6	CF_3
7	$SF^2_4F^3$

Scheme 2

TABLE I

^1H, ^{19}F and ^{31}P NMR data of (E)/(Z)-$CF_3{}^2CH-CF^1P(O)(OR^2)_2$, **4a–c** (J given in Hz)

		δ_{Ha}	δ_{Fa}		δ_{Pa}
		=CH ($^3J_{PH}$, $^3J_{F1H}$, $^3J_{F2H}$)	$CF_3{}^2$ ($^4J_{F1F2}$, $^4J_{PF2}$)	F^1 ($^3J_{PF1}$)	
R^2 = Et **4a**^{b,c}	(E)	5.90 (8.6, 34.3, 7.5)	−63.5 (17.2, 0.6)	−115.2 (91.7)	0.1
	(Z)	6.00 (25.5, 23.9, 8.1)	−59.9 (11.3, 3.1)	−107.4 (100.9)	−2.1
R^2 = $SiMe_3$ **4b**^{c,d}	(E)	5.90 (8.5, 34.6, 7.6)	−63.6 (17.1, 0.6)	−115.1 (95.5)	−19.1
	(Z)	5.90 (25.5, 24.2, 8.2)	−59.5 (11.4, 3.2)	−114.9 (106.9)	−21.6
R^2 = H **4c**^{c,e}	(E)	6.20 (7.9, 35.6, 7.6)	−58.7 (17.5, −)	−111.9 (102.4)	3.2
	(Z)	6.40 (24.6, 24.5, 8.3)	−55.0 (11.3, −)	−103.0 (106.7)	1.8

[a] Upfield from TMS, CCl_3F and 85% H_3PO_4 given as negative.
[b] ^1H: δ = 1.30 (CH_3, $^3J_{HH}$ = 7.0), 4.20 (CH_2, $^3J_{PH}$ = 8.4).
[c] Signals for (E) and (Z) isomers are overlapping.
[d] ^1H: δ = 0.20 ($SiMe_3$).
[e] ^1H: δ = 8.5 (OH).

TABLE II

^1H, ^{19}F and ^{31}P NMR data of (E)/(Z)-$F^3SF_4{}^2CH-CF^1P(O)(OR^2)_2$, **5a–c** (J given in Hz)

		δ_{Ha}	δ_{Fa}			δ_{Pa}
		=CH ($^3J_{PH}$, $^3J_{F1H}$, $^3J_{F2H}$)	F^3 ($^4J_{F1F3}$)	F^2	F^1 ($^2J_{PF1}$, $^4J_{F2F1}$)	($^4J_{PF2}$)
R^1 = Et **5a**^{b,c}	(E)	6.90 (8.3, 30.6, 7.9)	+74.7^d (3.9)	+64.9^e	−113.7 (91.2, 21.9)	−0.1 (2.6)
	(Z)	6.90 (23.5, 25.6, 8.3)	+74.7 (2.7)	+68.5	−112.9 (97.6, 14.6)	−3.1 (8.1)
R^2 = $SiMe_3$ **5b**^{c,f}	(E)	6.50 (8.1, 30.7, 8.1)	+75.5^d (3.5)	+64.8^e	−112.4 (94.3, 21.6)	−19.4 (2.4)
	(Z)	6.70 (23.6, 25.6, 8.3)	+75.5 (2.6)	+68.8	−112.2 (103.8, 15.4)	−22.7 (2.7)
R^1 = H **5e**^{e,g}	(E)	6.90 (7.9, 31.6, 8.0)	+80.2^d (4.0)	+69.6^e	−108.3 (99.0, 22.4)	−0.6
	(Z)	6.90 (22.8, 25.8, 2.5)	+80.2 (2.2)	+73.4	−108.7 (102.5, 16.1)	−2.8

[a] Upfield from TMS, CCl_3F and 85% H_3PO_4 reported as negative.
[b] ^1H: δ = 1.20 (CH_3, $^3J_{HH}$ = 7.0), 4.10 (CH_2, $^3J_{PH}$ = 7.8).
[c] Signals for (E) and (Z) isomers are overlapping.
[d] Two overlapping multiplet systems centered at line 6 (see Reference 13).
[e] Multiplet.
[f] ^1H: δ = 0.30 ($SiMe_3$).
[g] ^1H: δ = 7.9 (OH).

TABLE III

Comparison of some selected shift values and coupling constants for the compounds $R^1CH{=}CF^1P(O)(OR^2)_2$ (J given in Hz)

R¹ \ R²		Et		SiMe₃		H	
		(E)	(Z)	(E)	(Z)	(E)	(Z)
CF₃	δ_P	0.1	−2.1	−19.1	−21.6	3.2	1.8
SF₅		0.1	−3.1	−19.4	−22.7	−0.6	−2.8
CF₃	δ_{F1}	−115.2	−107.4	−115.1	−114.9	−111.9	−103.0
SF₅		−113.7	−112.9	−112.4	−112.2	−108.3	−108.7
CF₃	$^3J_{PH}$	8.6	25.5	8.5	25.5	7.9	24.6
SF₅		8.3	23.5	8.1	23.6	7.9	22.8
CF₃	$^3J_{F1H}$	34.3	23.9	34.6	24.2	35.6	24.5
SF₅		30.6	25.6	30.7	25.6	31.6	25.8
CF₃	$^3J_{PF1}$	91.7	100.9	95.5	106.9	102.4	106.7
SF₅		91.2	97.6	94.3	103.8	99.0	102.5
CF₃	$^4J_{F1F2}$	17.2	11.3	17.1	11.4	17.5	11.3
SF₅		21.9	14.6	21.6	15.4	22.4	16.1

$^4J_{F1F2}$ in which *cis* $^4J_{F1F2}$ > *trans* $^4J_{F1F2}$ and is probably due to an additional through-space contribution.[11,12]

The δ_P values of the new phosphonates were in the region typical for SF_5-substituted compounds but at slightly higher field.[5]

The trimethylsilyl group in **4b** and **5b** were responsible for an additional high field contribution[5]; the signals for the (Z) isomers were also found at higher field compared with their corresponding (E) analogues.

EXPERIMENTAL

The appropriate precautions in handling moisture and oxygen-sensitive compounds were observed throughout this work. The reactions were carried out in 100 ml heavy-wall glass tubes, fitted with a TEFLON® stopcock. At −196°C compounds **1** or **2** were condensed to **3a** and **3b** by using a vacuum line. The phosphonic acids were obtained by adding a 9:1 mixture of MeOH/H_2O to **4b** and **5b**. After completion of the reactions the tubes were opened in vacuo at −196°C and fluorotrimethylsilane, excess alkene or other volatiles were removed by pumping at ambient temperature (see Table IV).

MS: MAT 8222 spectrometer (electron energy 70 eV), −IR: Nicolet 5 DX FT spectrometer, spectra were recorded either as liquid films or as solids between sodium chloride plates, −NMR: AC 80 Bruker spectrometer, 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); d = doublet, p = pentet, dec = decet. Compounds **2**, **3a**, **3b** and **7b** were synthesized by literature procedures.[7,14,5]

(E)/(Z)-1,3,3,3-Tetrafluoroethenyl-diethylphosphonate (**4a**). MS: m/z (%): 251 (M^+ + H, 1), 249 (M^+ − H, 1), 223 (M^+ + H − C_2H_4, 17) 195 (M^+ + H − $2C_2H_4$, 85), 181 (M^+ − CF_3, 27), 175 (M^+ − C_2H_4 − F, 57), 93 ($(P(OH)(OC_2H_5))^+$, 53), 65 ($P(OH)_2^+$, 100), 29 ($C_2H_5^+$, 49) and other fragments.

IR: $\tilde{\nu}$ = 3046 cm^{-1} w (CH), 2991 w (CH), 2943 w (CH), 2919 w (CH), 1669 m (C=C), 1270 s (CF, P=O), 1024 vs (P—O—C) and other absorptions.

$C_7H_{11}F_4O_3P$	Calcd.	C 33.61	H 4.43	F 30.4	P 12.38
(250.13)	Found	C 33.72	H 4.47	F 29.8	P 12.17

(E)/(Z)-1,3,3,3-Tetrafluoroethenyl-bis(trimethylsilyl)phosphonate (**4b**). MS: *m/z* (%): 323 (M^+ − CH_3, 7), 299 (M^+ − F − HF, 2), 269 (M^+ − CF_3, 4), 229 (M^+ − $OSi(CH_3)_3$ -HF, 7), 147 ($((CH_3)_5Si_2O^+$, 100), 89 ($OSi(CH_3)_3^+$, 16), 73 ($Si(CH_3)_3^+$, 41) and other fragments.

IR: $\tilde{\nu}$ = 3060 cm^{-1} w (CH), 2967 m (CH), 2907 w (CH), 1673 m (C=C), 1262 vs (CF, P=O) and other absorptions.

$C_9H_{19}F_4O_3PSi_2$	Calcd.	C 31.95	H 5.66	F 22.4	P 9.15
(338.39)	Found	C31.75	H 5.79	F 21.7	P 9.19

TABLE IV

Reaction conditions for the synthesis of compounds (E)/(Z)-$R^1CH-CFP(O)(OR^2)_2$, **4a–c**, **5a–c** (R^1 = CF_3, SF_5; R^2 = Et, $SiMe_3$, H) and **7c**

Reactants g (mmol)	Reaction time Temperature (°C)	Products E/Z Ratio	b.p. (°C)/Torr *m.p. (°C)*	Yield g/%
1: 7.11 (54.8) **3a**: 11.10 (52.8)	5h/60, 18h/25	**4a** (60:40)	88/114	9.53 (72)
1: 1.50 (11.4) **3b**: 3.39 (11.4)	14h/25	**4b** (60:40)	99/11	3.15 (82)
4b: 1.38 (4.1) MeOH/H_2O:2.00	24/25	**4c** (60:40)	[1]	0.79 (100)
2: 2.35 (12.4) **3a**: 1.60 (7.6)	15d/−30	**5a** (50:50)	37/0.001	1.39 (59)
2: 2.13 (11.2) **3b**: 3.35 (11.2)	2d/25	**5b** (53:47)	38/0.001	2.32 (52)
5b: 0.60 (1.5) MeOH/H_2O:2.00	2d/25	**5c** (53:47)	*60*	0.47 (100)
7b: 1.56 (3.8) MeOH/H_2O:2.00	2h/25	**7c**[2]	*101*	1.00 (98)

[1] Viscous liquid, no b.p. obtained. [2] 100% (E) isomer.

(E)/(Z)-1,3,3,3-Tetrafluoropropenylphosphonic acid (**4c**). MS: m/z (%): 195 (M^+ + H, 9), 175 (M^+ − F, 4), 155 (M^+ − F − HF, 6), 135 (M^+ − F − 2HF, 2), 81 ($OP(OH)_2^+$, 28), 69 (CF_3^+, 12), 65 ($P(OH)_2^+$, 100), 47 (PO^+, 19) and other fragments.

IR: $\tilde{\nu}$ = 3073 cm^{-1} w (CH), 2836 vs (P—OH), 2275 vs (P—OH), 1674 m (C=C), 1280 vs (CF, P=O) and other absorptions.

(E)/(Z)-[1-Fluoro-2-(pentafluoro-λ⁶-sulfanyl)ethenyl]diethylphosphonate (**5a**). MS: m/z (%): 309 (M^+ + H, 2), 293 (M^+ − CH_3, 3), 281 (M^+ + H − C_2H_4, 24), 253 (M^+ + H − $2C_2H_4$, 83), 233 (M^+ − $2C_2H_4$ − F, 52), 213 (M^+ + H − $2C_2H_4$ − 2HF, 49), 181 (M^+ − SF_5, 4), 153 ($F_5SCH{=}CH^+$, 66), 127 (SF_5^+, 37), 65 ($P(OH)_2^+$, 100), 29 ($C_2H_5^+$, 49) und andere Fragmente.

IR: $\tilde{\nu}$ = 3030 cm^{-1} s (CH), 2970 vs (CH), 2870 vs (CH), 1655 vs (C=C), 1270 vs (CF, P=O), 1020 vs (P—O—C), 860 vs (SF) and other absorptions.

$C_6H_{11}F_6O_3PS$	Calcd.	C 23.38	H 3.60	F 36.99	P 10.05
(308.18)	Found	C 23.44	H 3.56	F 37.11	P 10.15

(E)/(Z)-[1-Fluoro-2-(pentafluoro-λ⁶-sulfanyl)ethenyl]bis(trimethylsilyl)phosphonate (**5b**). MS: m/z (%): 381 (M^+ − CH_3, 14), 269 (M^+ − SF_5, 3), 229 ($FP(O)[OSi(CH_3)_3][OSi(CH_3)_2]^+$, 14), 211 ($HP(O)[OSi(CH_3)_3][OSi(CH_3)_2]^+$, 15), 147 ($((CH_3)_5Si_2O^+$, 100), 127 ($SF_5^+$, 2), 73 ($Si(CH_3)_3^+$, 60) and other fragments.

IR: $\tilde{\nu}$ = 3020 cm^{-1} m (CH), 2960 s (CH), 1660 s (C=C), 1260 vs (CF, P=O), 940 vs (P—O—Si), 860 vs (SF) and other absorptions.

$C_8H_{19}F_6O_3PSSi_2$	Calcd.	C 24.24	H 4.83	F 28.7	P 7.81
(396.44)	Found	C 25.32	H 5.10	F 28.7	P 7.64

(E)/(Z)-[1-Fluoro-2-(pentafluoro-λ⁶-sulfanyl)ethenyl]phosphonic acid (**5c**). MS: m/z (%): 253 (M^+ + H, 8), 252 (M^+, 1), 251 (M^+ − H, 7), 233 (M^+ − F, 3), 213 (M^+ − F − HF, 11), 127 (SF_5^+, 7), 125 (M^+ − SF_5, 7), 101 ($FP(OH)_3^+$, 39), (F_2PO^+, 100), 81 ($P(O)(OH)_2^+$, 18), 65 ($P(OH)_2^+$, 39), 85 (F_2PO^+, 100), 81 ($P(O)(OH)_2^+$, 18), 65 ($P(OH)_2^+$, 51) and other fragments.

IR: $\tilde{\nu}$ = 3108 cm^{-1} w (CH), 3088 w (CH), 2792 m (P—OH), 2294 m (P—OH), 1649 m (C=C), 1284 s (CF, P=O), 899 vs (CF), 855 vs (SF), 605 s (SF) and other absorptions.

*(E)-[1,2-Difluoro-2-(pentafluoro-λ⁶-sulfanyl)ethenyl]*phosphonic acid (**7c**). MS: m/z (%): 271 (M^+ + H, 27), 270 (M^+, 2), 151 ($C_2F_5S^+$, 44), 143 (M^+ − SF_5, 76), 127 (SF_5^+, 20), 123 (M^+ − SF_5 − HF, 21), 89 (SF_3^+, 70), 85 (F_2PO^+, 85), 65 ($P(OH)_2^+$, 100), 47 (PO^+, 36) and other fragments.

IR: $\tilde{\nu}$ = 2815 vs (broad, P—OH), 1195 s (CF), 868 vs (SF), 664 m (SF) and other absorptions.

NMR: ^1H: δ = 8.3 (OH); ^{19}F: δ = +70.9 (F^3, multiplet centered at line 6 (see Reference 13), $^3J_{F3F}$ = 3.9, $^4J_{F3F1}$ = 2.5 Hz), +57.1 (F^2, multiplet), −133.6 ($F_5SC(F)$ = ddd, $^3J_{PF}$ = 9.7, $^3J_{F2F1}$ = 22.4 Hz); ^{31}P: δ = 0.8.

$C_2H_2F_7O_3PS$	Calcd.	C 8.89	H 0.75	F 49.24	P 11.47
(270.06)	Found	C 8.96	H 0.78	F 48.90	P 11.39

ACKNOWLEDGEMENTS

The authors are grateful to the Fonds der Chemischen Industrie. We thank Mrs. I. Erxleben for carrying out mass spectroscopic measurements. G. L. G. and R. W. express their appreciation to the U.S. Department of Energy (DE-FG 21-88 MC 25142) for support of this work. G. L. G. wishes to thank the Fulbright Commission for a Senior Fulbright Scholarship.

REFERENCES

1. A. A. Kadyrov and E. M. Rokhlin, *Usp. Khim.*, **57**, 1488 (1988).
2. K. Issleib, *Nachr. Chem. Tech. Lab.*, **35**, 1037 (1987).
3. T. Mahmood and J. M. Shreeve, *Inorg. Chem.*, **25**, 3128 (1986).

4. U. von Allwörden and G.-V. Röschenthaler, *Chem.-Ztg.*, **112,** 71 (1988).
5. H. Wessolowski, G.-V. Röschenthaler, R. Winter and G. L. Gard, *Z. Naturforsch.*, in press.
6. A. L. Henne and T. P. Waalkes, *J. Am. Chem. Soc.*, **68,** 496 (1946).
7. R. A. De Marco and W. B. Fox, *J. Fluorine Chem.*, **12,** 137 (1978).
8. U. von Allwörden and G.-V. Röschenthaler, *Chem.-Ztg.*, **112,** 69 (1988).
9. T. R. Spalding in: *Mass Spectrometry of Inorganic and Organometallic Compounds* (Litzow-Spalding, Ed.), pp. 408, Elsevier, Amsterdam-London-New York, 1973.
10. L. C. Thomas in: *Interpretation of the Infrared Spectra of Organophosphorus Compounds*, pp. 40, Heyden, London-New York-Rheine, 1974.
11. J. W. Emsley, L. Phillips and V. Wray, *Progr. NMR Spectrosc.*, **10,** 109 (1976).
12. J. W. Emsley, J. Feeney and L. H. Sutcliffe, *High Resolution Nuclear Magnetic Spectroscopy*, Vol. 2, pp. 906 Pergamon Press (1966).
13. N. Boden, J. Feeney and L. H. Sutcliffe, *Spectrochim. Acta*, **21,** 627 (1965).
14. L. Wozniak and J. Chojnowski, *Tetrahedron*, **45,** 2465 (1989).