Reactions of Phosphaalkenes with Hexafluoroacetone☆

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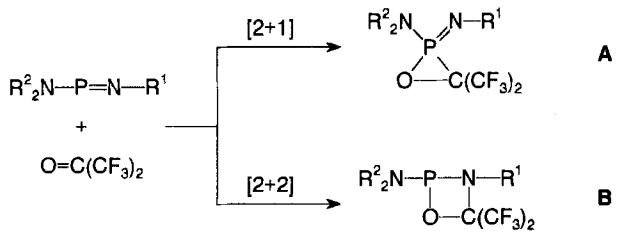
The dimethylamino-substituted phosphaalkene **1** reacts with hexafluoroacetone (HFA) with addition at the P=C bond to form the 1,5,2-dioxaphosphorinane **2**. The structure of **2** was confirmed by an X-ray crystal structure analysis. The six-membered ring displays an envelope conformation with the phosphorus atom out of the plane, but the phosphorus is disordered over two sites. Reaction of *P*-trimethylsilyl-substituted phosphaalkenes with HFA proceeds with retention of the P=C double bond and insertion of HFA into the P–Si bond. Two isomeric products are obtained and are characterized by ^{1}H-, ^{13}C-, ^{19}F-, and ^{31}P-NMR spectroscopy, IR spectroscopy, mass spectrometry, and elemental analysis.

The reactivity of λ^4-phosphorus double bond systems >P=E–R (E = N, P; R = hydrocarbon group) towards HFA has been intensively studied[1]. Compounds of phosphorus in low coordination number, e.g. iminophosphanes, are converted by HFA either to λ^4-oxaphosphiranes **A**[2] ([2 + 1] cycloaddition) or to 1,3,2-oxazaphosphetanes **B**[3] ([2 + 2] cycloaddition), depending on the substituents R^1 and R^2 (Scheme 1).

Scheme 1



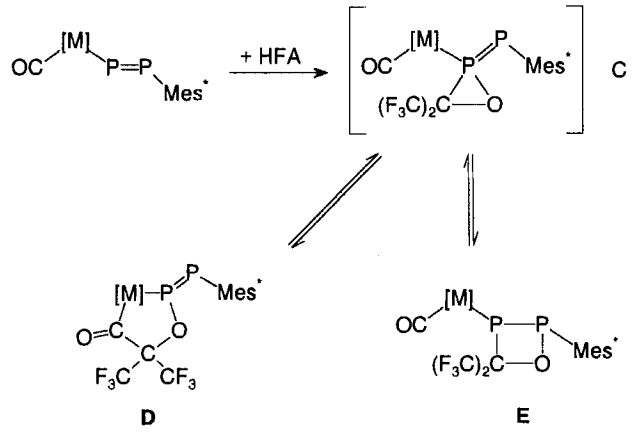
A : $R^1 = Me_3Si$ or ^tBu, $R^2 = Me_3Si$

B : $R^1 = {}^tBu$, $R^2 = {}^iPr$

Transition metal-substituted diphosphenes[4] (η^5-C_5R_5)$(CO)_2$M–P=P–Mes* (C_5R_5 = C_5Me_5, C_5Me_4Et, $C_5(1,2,4\text{-}iPr)_3H_2$, $C_5(1,3\text{-}t\text{Bu})_2H_3$, $C_5[1,3\text{-}(Me_3Si)_2]H_3$; M = Fe, Ru) react with HFA to afford either five-membered metallaheterocycles (η^5-C_5R_5)(CO)M–P(=P–Mes*)OC-$(CF_3)_2$C(O) **D** or metallated 1,2,3-oxadiphosphetanes (η^5-C_5R_5)$(CO)_2$M–P–P(Mes*)OC$(CF_3)_2$ **E**, which result from an intramolecular rearrangement of **D**.

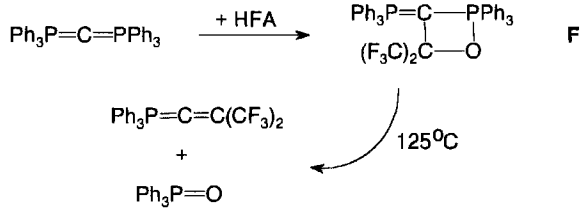
The course of the reaction and the nature of the products are largely influenced by the basicity of the cyclopentadienyl metal fragment. It was suggested that the proposed mechanism for the formation of **D** and **E** involves a transient [2 + 1] cycloadduct **C** which, however, could not be detected[5] (Scheme 2).

Scheme 2



The present knowledge of reactions of HFA with P=C double bonds is restricted to λ^4P=C systems; thus the 1,2λ^5-oxaphosphetane **F** has been isolated[6] and structurally characterized[7] as the stable intermediate of a Wittig reaction between HFA and hexaphenyl-1λ^4,3λ^4-diphosphaallene (Scheme 3).

Scheme 3



At 125°C a Wittig reaction occurs, and 1,1,1-triphenyl-3,3-bis(trifluoromethyl)-1λ^4-phosphaallene is formed to-

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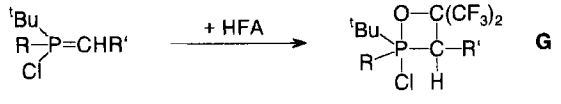
gether with triphenylphosphane oxide. The same type of [2 + 2] cycloaddition has been observed for a series of *P* chlorophosphane ylides, which add HFA to form stable 1,2λ⁵-oxaphosphetanes **G** in quantitative yield[8] (Scheme 4).

Scheme 4

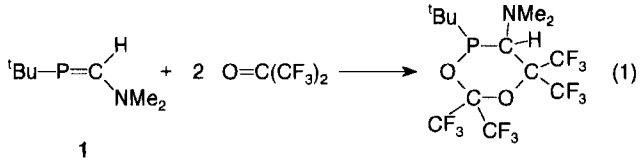


R = tBu, R' = H;
R = Et_2N, R' = H;
R = Et_2N, R' = Me.

1,2λ⁵-Oxaphosphetanes have also been obtained by Röschenthaler among a mixture of products in the reaction of HFA with $Me_2POCH(CF_3)_2$ and $MeP[OCH(CF_3)_2]_2$, respectively[9].

Results and Discussion

The reaction of the *C*-aminosubstituted phosphaalkene *tert*-butyl[(dimethylamino)methylene]phosphane (**1**) with HFA gave the addition product **2** in good yield (eq. 1). With other phosphaalkenes, for example *tert*-butyl[2,2-dimethyl-1-(trimethylsiloxy)propylidene]phosphane[10], no reaction was observed. The reactivity of **1** can be explained by the delocalisation of the π-electron charge density of the double bond because of its conjugation with the lone electron pair of the neighbouring NMe_2 group[11,12]. Reaction of **1** with HFA in a 1:1 ratio also gave **2** in poor yield. With excess HFA no further reaction of **2** took place.



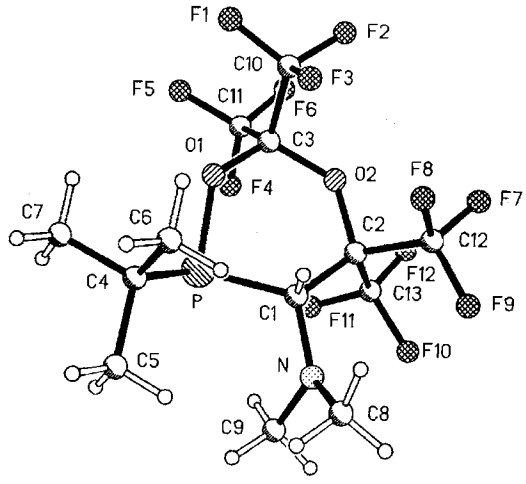
(1)

1 **2**

The structure of **2** has been proved by means of ^1H-, ^{13}C-, ^{19}F-, and ^{31}P-NMR spectroscopy, IR spectroscopy, mass spectrometry, and by a single-crystal X-ray structure determination (Figure 1). A similar connectivity has been observed in the five-membered cyclic 1,3,4λ⁵-dioxaphospholanes[13–21].

The diastereotopic NCH_3 groups show two singulets in the ^1H- and two doublets in the ^{13}C-NMR spectrum, respectively. The ^{31}P-NMR spectrum of **2** displays a septuplet at δ = 137.84 with *J*(PF) = 27.5 Hz. The ^{19}F-NMR spectrum of **2** consists of four signals indicating the magnetic inequivalence of the CF_3 groups. The $PCC(CF_3)_2$ groups exhibit two doublets of multiplets with a *J*(PF) value of 24.7 Hz. The complicated multiplicity is probably due to coupling to neighbouring magnetically active atoms (P, F). Further coupling constants could not be determined; ^{13}C satellites are not observed. The resonance of the $POC(CF_3)_2$ groups appears as two virtual septuplets, apparently caused by overlapping of two quadruplets. The "coupling" of 8.9 Hz could not be assigned unambiguously.

Figure 1. Structure of **2** in the crystal; radii are arbitrary[a]



[a] Selected bond lengths and angles: P–O(1) 169.8(2), P–C(1) 185.5(3), P–C(4) 185.7(3), O(1)–C(3) 137.2(3), O(2)–C(3) 140.4(4), O(2)–C(2) 143.1(4), N–C(1) 143.0(4), N–C(8) 144.0(4), N–C(9) 144.7(4), C(1)–C(2) 158.3(4) pm; O(1)–P–C(1) 93.42(12), O(1)–P–C(4) 94.41(12), C(1)–P–C(4) 104.30(13), C(3)–O(1)–P 122.5(2), C(3)–O(2)–C(2) 128.0(2), N–C(1)–P 115.6(2), C(2)–C(1)–P 111.6(2), O(2)–C(2)–C(1) 115.9(2), O(1)–C(3)–O(2) 117.3(2)°.

X-Ray Investigation

Compound **2** consists of a phosphorus-containing heterocycle with one *tert*-butyl group, four trifluoromethyl groups, and one dimethylamino group as substituents.

The phosphorus atom is disordered, with an alternative position on the other side of the six-membered ring (occupation 10%). For this reason, the bond lengths and angles should be interpreted with caution. The minor disordered component is not discussed.

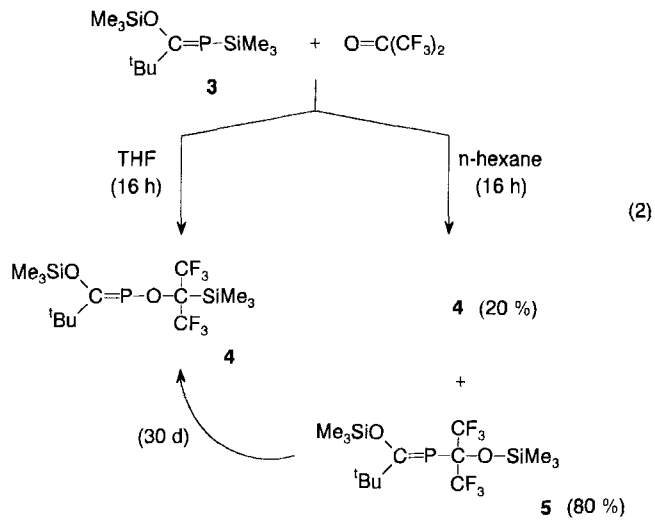
The phosphorus atom displays a distorted trigonal-pyramidal coordination geometry. Its distance to the plane of the atoms C1, O1, and C4 is 90 pm with bond angles between 104.30(13) (C1–P–C4) and 93.42(12)° (O1–P–C1). Despite the steric demands of the *tert*-butyl group, the observed bond lengths P–C4 [185.9(8) pm] and P–C1 [185.5(3) pm] lie in the expected range. The normal length of a P–C single bond is 185 pm[22].

The six-membered ring exhibits an envelope conformation, in which the phosphorus atom lies 89 pm out of the plane of the other ring members; the mean deviation from the plane is 1.4 pm.

The four CF_3 groups adopt an eclipsed conformation, whereby the pseudo-torsion angles lie between 7.3 (F1–C10···C11–F5) and 16.9° (F3–C10···C11–F4). The nitrogen atom lies only 19 pm out of the plane of C1, C8, and C9 with an associated angle sum of 354.7°.

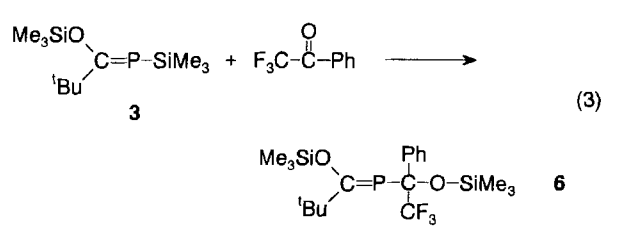
The reaction of HFA with a silylphosphane has been reported, and two alternative insertion products (because of the migration of the trimethylsilyl group) were found[23]. The reactivity of HFA towards *P*-trimethylsilyl-substituted

phosphaalkenes has so far not been studied. After 16 hours the reaction of **3** (*E* isomer)[24] with HFA in *n*-hexane resulted in the formation of two isomers, **4** and **5**, in an approximate ratio of 1:4 (by integration of the ^{31}P-NMR signals) (eq. 2). Both products exhibit a septuplet, with $\delta(^{31}P)$ 124.78 (**4**) and 150.52 (**5**), which are typical shift values for λ^2P=C double bond systems[25]. The identification of the products is mainly based on the *J*(PF) values in the ^{31}P- and ^{19}F-NMR spectra. The smaller PF coupling constant (7.2 Hz) is assigned to **4**, the larger (25.2 Hz) to **5**. This remarkable difference suggests that **4** and **5** are formed as *E* isomers; thus *Z* isomers can be excluded. Separation of the isomers by distillation was impossible, but microanalysis confirmed the composition of the mixture.



Stirring for 30 days in *n*-hexane leads to isomerisation of **5**; pure **4** is obtained, which is thus thermodynamically more stable than **5**. Compound **4** is exclusively formed within 16 hours when THF is used as a solvent.

Under the same reaction conditions as in eq. (2), the reaction of trifluoroacetophenone with **3** results in only one product, **6**, corresponding to **5** [eq. (3)]. Stirring for 40 hours caused no change in the ^{31}P NMR spectrum.



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Experimental

All experiments were carried out with exclusion of air and moisture, solvents were purified and dried according to the usual methods[26]. "In vacuo" refers to a pressure of 0.05 Torr at 25°C, unless otherwise stated. – NMR: Bruker AC 200 (^{1}H: 200.1 MHz, ^{13}C: 50.3 MHz, ^{19}F: 188.3 MHz, ^{31}P: 81.0 MHz); reference substances were $SiMe_4$ (TMS) ext. (^{1}H, ^{13}C), 85% H_3PO_4 ext. (^{31}P), and $CFCl_3$ ext. (^{19}F); high-field shifts are given negative, low-field shifts positive signs. – MS: Finnigan MAT 8430. – Melting points: sealed capillaries, Büchi 510 instrument. – IR: Nicolet 320 FT-IR spectrometer. – Elemental analyses: Mikroanalytisches Laboratorium Beller, Göttingen, and Analytisches Laboratorium des Instituts für Anorganische und Analytische Chemie der Technischen Universität, Braunschweig.

(*E*)-*tert*-Butyl[(dimethylamino)methylene]phosphane (**1**)[27] and (*E*)-[2,2-dimethyl-1-(trimethylsiloxy)propylidene](trimethylsilyl)phosphane (**3**)[24] were prepared as described in the literature. All other reagents were obtained commercially.

2-tert-Butyl-3-(dimethylamino)-4,4,6,6-tetrakis(trifluoromethyl)-1,5,2-dioxaphosphorinane (**2**): Into a heavy-walled glass tube, equipped with a Teflon® stopcock, was placed a solution of 2.15 g (14.80 mmol) of *tert*-butyl[(dimethylamino)methylene]phosphane (**1**) in 20 ml of *n*-hexane. The solution was cooled to −196°C, and 5.02 g (30.24 mmol) of hexafluoroacetone was condensed into the tube. The reaction mixture was allowed to warm up to room temp., and within 1 h the solution turned yellow. After stirring for 16 h at room temp. the solvent and all volatile components were removed in vacuo, and the yellow residue was recrystallized from diethyl ether. The product was obtained as colourless crystals. Yield: 5.2 g (74%), m.p. 32°C. – ^{1}H NMR ($CDCl_3$): δ = 1.11 [d, *J* = 12.6 Hz, 9H, $C(CH_3)_3$], 2.50 (s, 3H, NCH_3), 2.51 (s, 3H, NCH_3), 9.01 (d, *J* = 12.1 Hz, 1H, PCH). – ^{13}C NMR ($CDCl_3$): δ = 24.17 [d, *J* = 15.6 Hz, $C(CH_3)_3$], 38.79 (d, *J* = 3.3 Hz, NCH_3), 39.16 (d, *J* = 3.3 Hz, NCH_3), 59.69 [d, *J* = 35.8 Hz, $C(CH_3)_3$], 82.68 [septd, *J* = 14.0, 28.7 Hz, $C(CF_3)_2$], 93.88 (septd, *J* = 34.9, 2.9 Hz, $HCNMe_2$), (septd = septuplet of doublets), 119.90, 121.98 [qd, *J* = 292.9, 288.7 Hz, *J* = 28.9, 20.42 Hz, $C(CF_3)_2$]. – ^{19}F NMR ($CDCl_3$): δ = −69.45, −79.42 [m, $PCC(CF_3)_2$], −74.89, −81.26 [m, $POC(CF_3)_2$]. – $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ = 137.84 (sept, *J* = 27.5 Hz). – EI MS (70 eV), *m/z* (%): 477 (2) $[M]^+$, 420 (100) $[M - C_4H_9]^+$. – IR (*n*-hexane): $\tilde{\nu}$ = 966 cm^{-1} (s), 945 (s), 734 (m). – $C_{13}H_{16}F_{12}NO_2P$ (477.2): calcd. C 32.72, H 3.38, F 47.78; found C 32.81, H 3.64, F 46.8.

[Bis(trifluoromethyl)(trimethylsilyl)methoxy][2,2-dimethyl-1-(trimethylsiloxy)propylidene]phosphane (**4**) *and [Bis(trifluoromethyl)(trimethylsiloxy)methyl][2,2-dimethyl-1-(trimethylsiloxy)propylidene]phosphane* (**5**): A solution of 4.94 g (18.82 mmol) of [2,2-dimethyl-1-(trimethylsiloxy)propylidene](trimethylsilyl)phosphane (**3**) in 30 ml of either THF or *n*-hexane was placed into a heavy-walled glass tube, equipped with a Teflon® stopcock. The solution was cooled to −196°C, and 3.13 g (18.85 mmol) of hexafluoroacetone was condensed into the tube. The reaction mixture was allowed to warm to room temp., and within 1 h the solution turned yellow. After stirring for 16 h at room temp. the solvent and all volatile components were removed in vacuo, and the yellow residue was fractionally distilled in vacuo. The products were obtained as yellow viscous liquids.

4: Yield: 4.98 g (62%), b.p. 74°C (0.2 Torr). – ^{1}H NMR ($CDCl_3$): δ = 0.22 [s, 9H, $Si(CH_3)_3$], 0.33 [s, 9H, $Si(CH_3)_3$], 1.23 [d, *J* = 2.9 Hz, 9H, $C(CH_3)_3$]. – ^{13}C NMR ($CDCl_3$): δ = 2.22 [d, *J* = 3.7 Hz, $OCSi(CH_3)_3$], 2.55 [s, $PCOSi(CH_3)_3$], 29.34 [d, *J* = 17.0 Hz, $C(CH_3)_3$], 44.23 [d, *J* = 31.3 Hz, $C(CH_3)_3$], 82.37 [dsept, *J* = 52.4, 31.3 Hz, $C(CF_3)_2$], 123.49 (qm, *J* = 285.7 Hz, CF_3), 218.04 (d, *J* = 87.5 Hz, C=P). – ^{19}F NMR ($CDCl_3$): δ = −71.00 (d, *J* = 7.2 Hz, CF_3). – $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ = 124.78 (sept, *J* = 7.7 Hz). –

EI MS (70 eV), *m/z* (%): 428 (0.2) $[M]^+$, 263 (0.7) $[M - OC(CF_3)_2]^+$, 239 (0.5) $[(CF_3)_2COSiMe_3]^+$, 147 (100) $[C_2F_5CO]^+$ – IR (*n*-hexane): $\tilde{\nu}$ = 946 cm^{-1} (s), 982 (s). – $C_{14}H_{27}F_6O_2PSi_2$ (428.5): calcd. C 39.24, H 6.35, P 7.23; found C 39.54, H 6.57, P 6.89.

5: Mixture with **4**, b.p. 74°C (0.2 Torr). – ^1H NMR ($CDCl_3$): δ = 0.24 [s, 9H, $Si(CH_3)_3$], 0.32 [s, 9H, $Si(CH_3)_3$], 1.23 [d, *J* = 2.9 Hz, 9H, $C(CH_3)_3$]. – ^{13}C NMR ($CDCl_3$): δ = 1.94 [d, *J* = 4.2 Hz, $C(CF_3)_2OSi(CH_3)_3$], 2.35 [s, $P{=}COSi(CH_3)_3$], 30.00 [d, *J* = 12.5 Hz, $C(CH_3)_3$], 45.25 [d, *J* = 23.7 Hz, $C(CH_3)_3$], 87.89 [dsept, *J* = 71.6, 33.7 Hz, $C(CF_3)_2$], 123.49 (qm, *J* = 285.7 Hz, CF_3), 216.71 (d, *J* = 82.3 Hz, C=P). – ^{19}F NMR ($CDCl_3$): δ = −69.57 (d, *J* = 24.5 Hz, CF_3). – $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ = 150.52 (sept, *J* = 25.2 Hz). – IR (*n*-hexane): $\tilde{\nu}$ = 995 cm^{-1} (s), 1020 (s). – $C_{14}H_{27}F_6O_2PSi_2$ (428.5): calcd. C 39.24, H 6.35, P. 7.23; found C 39.45, H 6.68, P 6.43 (mixture with **4**).

[2,2-Dimethyl-1-(trimethylsiloxy)propylidene][phenyl-(trifluoromethyl)(trimethylsiloxy)methyl]phosphane (**6**): A solution of 3.5 g (20.10 mmol) of trifluoroacetophenone in 10 ml of *n*-hexane was added to a solution of 5.0 g (19.05 mmol) of **3** in 10 ml of *n*-hexane. After stirring for 12 d at room temp. the solvent and all volatile components were removed in vacuo, and the yellow residue was fractionally distilled in vacuo. The product was obtained as a pale yellow viscous liquid. Yield: 5.8 g (70%), b.p. 72°C (0.4 Torr). – ^1H NMR ($CDCl_3$): δ = 0.22 [s, 9H, $Si(CH_3)_3$], 0.26 [s, 9H, $Si(CH_3)_3$], 1.22 [d, *J* = 2.5 Hz, 9H, $C(CH_3)_3$], 7.23–7.29 (m, 5H, C_6H_5). – ^{13}C NMR ($CDCl_3$): δ = 0.81 [d, *J* = 3.0 Hz, $C(CF_3)(Ph)OSi(CH_3)_3$], 2.12 [s, $P{=}COSi(CH_3)_3$], 29.07 [d, *J* = 16.0 Hz, $C(CH_3)_3$], 47.31 [d, *J* = 25.9 Hz, $C(CH_3)_3$], 82.85 [dq, *J* = 57.7, 26.2 Hz, $C(CF_3)_2$], 125.84 (qd, *J* = 27.6, 287.3 Hz, CF_3), 215.80 (dq, *J* = 2.2, 74.0 Hz, C=P). – ^{19}F NMR ($CDCl_3$): δ = −74.64 (d, *J* = 28.2 Hz, CF_3). – $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ = 153.49 (q, *J* = 28.4 Hz). – EI MS (70 eV), *m/z* (%): 279 (4) $[PC(Ph)(CF_3)OSiMe_3]^+$, 247 (5) $[PhC(CF_3)OSiMe_3]^+$, 77 (56) $[C_6H_5]^+$, 58 (100) $[C_4H_{10}]^+$. – IR (*n*-hexane): $\tilde{\nu}$ = 992 cm^{-1} (s), 1009 (s). – $C_{19}H_{32}F_3O_2PSi_2$ (436.6): calcd. C 52.27, H 7.39, P 7.09; found C 51.94, H 7.27, P 6.85.

X-Ray Structure Determination of Compound **2**: *Crystal data:* $C_{13}H_{16}F_{12}NO_2P$, *M* = 477.24, monoclinic, $P2_1/c$, *a* = 887.76(14), *b* = 1759.6(3), *c* = 1266.2(2) pm, β = 108.097(11)°, *V* = 1.8801(5) nm^3, *Z* = 4, D_x = 1.686 Mg m^{-3}, λ(Mo-K_α) = 71.073 pm, μ = 0.27 mm^{-1}, *F*(000) = 960, *T* = 143 K. – *Data collection and reduction:* A colourless prism of 0.9 × 0.6 × 0.4 mm was used to collect 3485 intensities on a Stoe Stadi-4 diffractometer fitted with a Siemens LT-2 low-temperature device ($2\Theta_{max}$ 50°, 3304 unique, R_{int} 0.019). The cell constants were refined from ±ω angles of 60 reflections in the 2Θ range 20–23°. – *Structure solution and refinement:* The structure was solved by direct methods and refined anisotropically on F^2 (program SHELXL-93, G. M. Sheldrick, University of Göttingen). H atoms were included by using a riding model or with rigid methyl groups. The phosphorus atom was disordered (see Discussion). The final $wR(F^2)$ for 269 parameters was 0.142, with conventional *R*(*F*) 0.050. The weighting scheme was $w^{-1} = [\sigma^2(F^2) + (aP)^2 + bP]$, where $3P = (2F_c^2 + F_o^2)$, and *a* and *b* are constants set by the program. Max. Δ/σ = 0.001; max. $\Delta\rho$ = 509 e nm^{-3}; *S* = 1.05. – Full details of the structure determination have been deposited at the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-76344 Eggenstein-Leopoldshafen, Germany, from where this material may be obtained on quoting the full literature citation and the reference number CSD-401 537.

☆ Dedicated to Professor *H. M. R. Hoffmann* on the occasion of his 60th birthday.

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