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Synthesis of tricyclic (λ^5)-phosphoranes *N*-demethylation/*N*-alkylation reactions during the oxidative addition perfluorinated α-diketones to P-bis(2-chloroethyl)amino-substituted λ^3P-compounds

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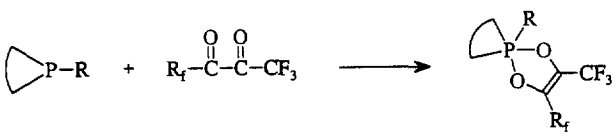
Abstract

In the reaction of various 2,3-dihydro-1,3,2-benzodiazaphosphorin-4(1*H*)-ones with perfluorinated α-diketones, $CF_3C(:O)C(:O)R_f$, an oxidative addition reaction with concomitant *N*-alkylation of the $\underline{CH}_3(N)$ atom, and formation of tricyclic phosphoranes was found to take place. In one case no *N*-alkylation reaction was observed and a perfluoropinacolyl spirophosphorane was formed instead. The course of the reaction mainly depends on the steric demand of R_f and the *N*-3 substituents of the benzodiazaphosphorinones. © 1997 Elsevier Science S.A.

Keywords: Benzodiazaphosphorinone derivatives; Phosphoranes; NMR spectroscopy

1. Introduction

The oxidative addition of hexafluoroacetone (HFA) $(CF_3)_2C(:O)$, *o*-quinones and perfluorinated α-diketones $R_fC(:O)C(:O)R_f$ to phosphorus(III) compounds is a well-known method of synthesizing dioxaphosphoranes, involving λ^5P [1,2]. Thus, in the reaction of perfluorinated α-diketones with diaza- and triazaphosphorinone derivatives, the formation of spirophosphoranes, involving the 1,3,2-dioxaphospholene ring system, was observed (Eq. (1)) [3,4]:



$\triangleright$P = diaza- and triazaphosphorinone ring systems

R = CH_2Cl, $N(CH_2CH_2Cl)_2$, $NH(CH_2CH_2Cl)$

R_f = perfluoroalkyl (1)

When such reactions were studied on some new benzodiaza-λ^3-phosphorinones, involving P-[bis(2-chloroethyl)-amino] substitutents [5–7], an unusual course of reaction was observed: the oxidative addition of HFA or tetrachloro-*o*-benzoquinone (TOB) to a series of such P(III) compounds was accompanied by an unusual and unexpected attack of one of the two 2-chloroethylamino substituents at the NCH_3 group of the benzodiaza-λ^3-phosphorinones. A ring-closure reaction occurred with loss of chloromethane, and formation of a tricyclic (λ^5)phosphorane structure took place. A possible mechanism of formation of these compounds has been proposed [4,6,7].

In the present work, the reactivity of two 2-[bis(2-chloroethyl)amino]-3-(halobenzyl)-1-methyl-2,3-dihydro-1,3,2-benzodiazaphosphorin-4(1*H*)-ones (**1**, **2**) and of 2-[bis(2-chloroethyl)-amino]-1,3-dimethyl-2,3-dihydro-1,3,2-benzodiazaphosphorin-4(1*H*)-one (**9**) towards two perfluoroalkyl-substituted diketones has been investigated. Structures were assigned from NMR data, supported by high-resolution mass spectra.

2. Results and discussion

The reactions of 2-[bis(2-chloroethyl)amino]-3-(4-fluorobenzyl)-1-methyl-2,3-dihydro-1,3,2-benzodiazaphosphorin-4(1*H*)-one (**1**) and of 2-[bis(2-chloroethyl)-

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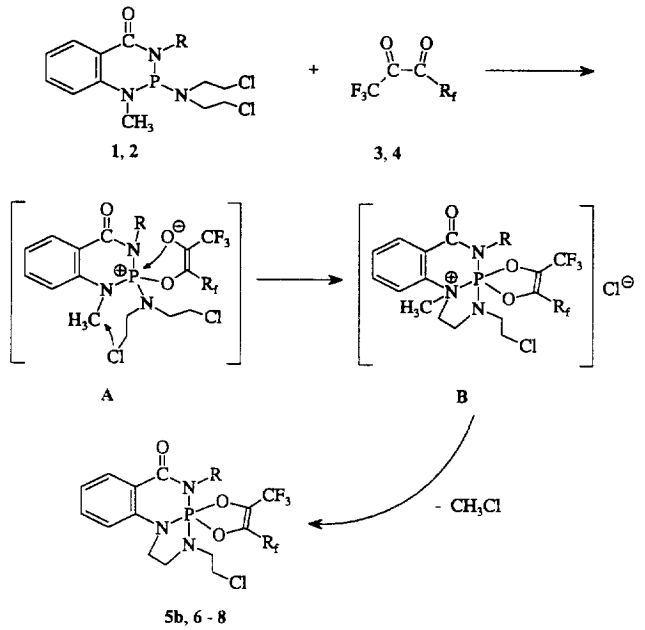
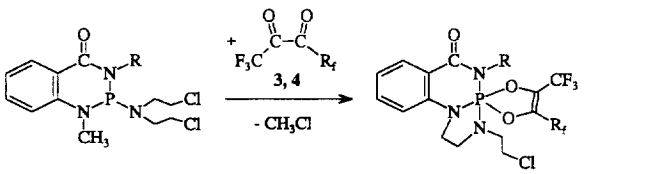


Scheme 1.

amino]-3-(2-chlorobenzyl)-1-methyl-2,3-dihydro-1,3,2-benzodiazaphosphorin-4(1*H*)-one (**2**) with perfluoromethylethyl-diketone (**3**) and perfluoromethylpropyl diketone (**4**) led to the tricyclic (λ^5) phosphoranes **5–8** in good yield (Eq. (2)).

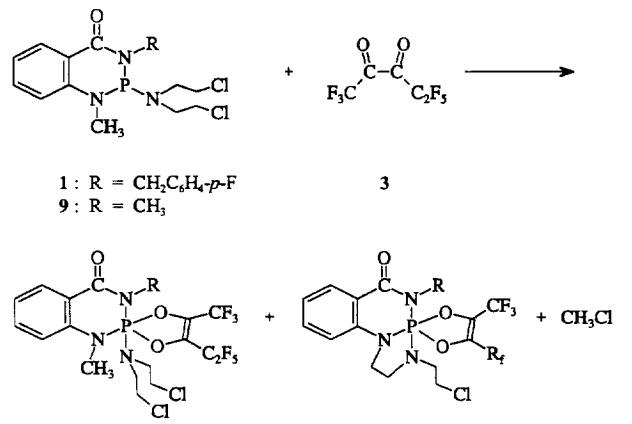


1: R = $CH_2C_6H_4$-*p*-F
2: R = $CH_2C_6H_4$-*o*-Cl

5b: R = $CH_2C_6H_4$-*p*-F; R_f = C_2F_5
6: R = $CH_2C_6H_4$-*p*-F; R_f = C_3F_7
7: R = $CH_2C_6H_4$-*o*-Cl; R_f = C_2F_5
8: R = $CH_2C_6H_4$-*o*-Cl; R_f = C_3F_7

(2)

A possible mechanism of formation of **5–8** is presented in Scheme 1. None of the proposed intermediates in this reaction, occurring under mild conditions, were actually observed, but the route as described in Scheme 1 would appear plausible. It is suggested that, in the first step of the reaction, addition of the diketone **3** or **4** to the benzodiazaphosphorinone **1** or **2**, respectively, gives rise to a zwitterionic intermediate **A**. Its rearrangement leads to an intermediate **B**, in a similar fashion which has been described in the literature for HFA [6–10]. In the ^{31}P-NMR spectrum of the reaction mixture, $\delta(^{31}P)$ values in the region $\delta = 8–10$ ppm were observed within 30 min after the reactants were combined. This indicates the presence of species involving pentacoordinate (λ^5) phosphorus such as the ammonium salt **B**. Elimination of chloromethane was established by ^{1}H-NMR spectroscopy after a further three-day period of stirring the reaction mixture at room temperature. It is suggested that the



1: R = $CH_2C_6H_4$-*p*-F
9: R = CH_3

5a: R = $CH_2C_6H_4$-*p*-F (8 %) **5b**: R = $CH_2C_6H_4$-*p*-F (92 %)
10a: R = CH_3 (100 %) **10b**: R = CH_3 (0 %)

Scheme 2.

ring-closure reaction is a consequence of the low stability of the intermediates **A** and **B** proposed in Scheme 1.

This low stability is related to steric effects of the methyl and benzyl substituents in the intermediate **B**, which breaks down to form the tricyclic phosphoranes **5b** and **6–8**. The identity of **5b** and **6–8** was established by ^{1}H-, ^{13}C-, ^{31}P- and ^{19}F-NMR spectroscopy and by high-resolution mass spectrometry. $\delta(^{31}P)$ values for **5–8** typical of phosphoranes (with λ^5P) were observed [11]. It turned out that the course of the reaction also depends on the steric demand of the *N*-3 substituent of the benzodiazaphosphorinone (Scheme 2).

In the case of **9** (R = CH_3), no *N*-alkylation reaction took place and the bicyclic spirophosphorane **10a** was formed exclusively. In the case of **1** with a higher steric demand of R (*p*-fluorobenzyl), a mixture of the bicyclic (**5a**) and the tricyclic phosphorane (**5b**) was obtained in an approximate ratio of 8:92 (integration of the ^{31}P-NMR signals). The composition of the product mixture could not be influenced either by excess **3** or by changing the reaction conditions. The *N*-alkylation is, apparently, favoured by *N*-3 substituents with greater steric demand (*p*-fluorobenzyl (**1**) in contrast to methyl (**9**)). Separation of **5a** and **5b** was impossible but high-resolution mass spectrometry confirmed the presence of both products.

3. Experimental details

Solvents were dried using standard procedures [12] and were stored over molecular sieves. All melting points are uncorrected. 'In vacuo' corresponds to a pressure of 0.1 mm Hg unless stated otherwise. ^{1}H-, ^{13}C- and ^{31}P-NMR spectra were recorded on a Bruker AC-200 instrument (^{1}H at 200.1 MHz, ^{13}C at 50.3 MHz, ^{31}P at 81.0 MHz) using TMS and 85% H_3PO_4 as external references. ^{19}F-NMR spectra were recorded on the same instrument using $CFCl_3$ as external reference ($CDCl_3$ as a solvent). Mass spectra were recorded on a Finnigan MAT 8430 instrument. IR spectra were

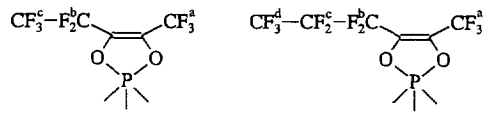


Fig. 1. Labelling scheme for NMR spectra (δ(F), J(FF), J(CF) and J(PF)).

recorded on a Nicolet 320 FT-IR spectrometer. Elemental analyses were conducted by Mikroanalytisches Laboratorium Beller, Göttingen. Designations are as follows. NMR: s, singlet; d, doublet; t, triplet; q quartet; sept, septet; m, multiplet; br, broad. IR: s, strong; vs, very strong.

The following compounds were obtained according to the methods described in the literature: **1** [13], **2** [13], **3** [14], **4** [15], and **9** [5].

δ(^{19}F) values and J(FF), J(CF) and J(PF) coupling constants were assigned in accordance with the labelling scheme depicted in Fig. 1.

3.1. Reaction of *1* with *3*: synthesis of *5a* and *5b*

A solution of 0.32 g (1.3 mmol) of perfluoroethylmethyl diketone (**3**) in 5 ml of dichloromethane was added dropwise with stirring at room temperature to a solution of 0.51 g (1.2 mmol) of **1** in 20 ml of dichloromethane. Stirring was continued for 3 days at 20°C. Subsequently, the solvent was pumped off in vacuo and the residue dissolved in 5 ml of diethyl ether. Within 3 days at −35°C a light yellow solid was precipitated, which was filtered through a sintered glass disc and washed with two 1 ml portions of diethyl ether. The product was then dried in vacuo. Yield, 0.52 g (70%).

5a (8%, mixture with **5b**: 92%): ^{1}H-NMR ($CDCl_3$, 200.1 MHz) δ: 3.10–3.56 (m, CH_2CH_2Cl, NCH_2CH_2N); 3.30 [d, ^3J(PH) = 13.3 Hz, NCH_3, **5a**]; 4.69 [dd, ^3J(FH) = 6.6 Hz, ^2J(HH) = 14.8 Hz, $FC_6H_4CH_2N$]; 5.33 [dd, ^3J(FH) = 6.3, ^2J(HH) = 12.4 Hz, $FC_6H_4CH_2N$]; 6.80–8.15 (m, C_6H_4). ^{13}C-NMR ($CDCl_3$, 50.3 MHz) δ: 30.25 [d, NCH_3, ^2J(PC) = 42.4 Hz, **5a**]; 37.56 [d, J(PC) = 8.3 Hz], 41.30 [d, J(PC) = 2.3 Hz],49.78 [d, J(PC) = 8.8 Hz], 51.66 (s), 52.19 (s), ($PNCH_2C_6H_4F$, $PNCH_2CH_2Cl$, $PNCH_2CH_2N$); 113.12–137.89 [12 s, C_6H_4]; 108.29 [tq, ^1J(CF^c) = 256.7 Hz, ^2J(CF^b) = 35.6 Hz, CF_3^c]; 114.72 [m, CF_2^b]; 119.49 [dq, ^1J(CF^a) = 264.0 Hz, ^3J(PC) = 12.6 Hz, CF_3^a]; 127.70 [dt, ^2J(CF^b) = 32.9 Hz, ^2J(PC) = 5.1 Hz, CCF_2^b]; 134.22 [dq, ^2J(CF^a) = 37.9 Hz, ^2J(PC) = 5.2 Hz, CCF_3^a]; 166.80 [d, ^2J(PC) = 3.3 Hz, C(:O)NP]. ^{19}F-NMR ($CDCl_3$, 188.3 MHz) δ: −64.72 [t, ^3J(F^{b}F^c) = 15.3 Hz, F^c]; −84.59 (s, F^a); −117.50 [s, $CH_2C_6H_4F$]; −117.24 [dq, ^3J(F^{b}F^c) = 16.8 Hz, ^4J(PF^b) = 3.4 Hz, F^b]. ^{31}P-NMR ($CDCl_3$, 81.0 MHz) δ: −36.35 (s, **5b**); −31.53 (s, **5a**). EI-MS m/z(%): 671 (2) [M (**5a**)]$^+$; 621 (5) [M (**5b**)]$^+$; 602 (1) [**5a** $-CF_3$]$^+$; 531 (10) [**5a** $-N(CH_2CH_2Cl)_2$]$^+$; 512 (<1) [**5b** $-CH_2C_6H_4F$]$^+$; 502 (<1) [**5b** $-C_2F_5$]$^+$; 109 (100) [$CH_2C_6H_4F$]$^+$. High-resolution mass spectrometry (res.: 10000, 10%): **5b**: theoret. 621.0631, exp. 621.063 ± 2 ppm, **5a**: theoret. 671.0554, exp. 671.055 ± 2 ppm. −**5b**: $C_{23}H_{18}ClF_9N_3O_3P$ (621.83), **5a**: $C_{24}H_{21}Cl_2F_9N_3O_3P$ (672.31). For an explanation of F^a, F^b, F^c, and F^d, see Fig. 1.

3.2. Reaction of *1* with *4*: synthesis of *6*

The preparation of **6** was conducted in a fashion similar to that described for compounds **5a** and **5b** from 0.64 g (1.5 mmol) of **1** and 0.47 g (1.6 mmol) of perfluoromethylpropyl diketone **4**. Yield, 0.63 g (63%); m.p. 85°C.

^{1}H-NMR ($CDCl_3$, 200.1 MHz) δ: 3.15–3.51 (m, CH_2CH_2Cl, NCH_2CH_2N); 4.69 [dd, ^3J(FH) = 6.7, ^2J(HH) = 15.3 Hz, $FC_6H_4CH_2N$]; 5.34 [dd, ^3J(FH) = 6.6, ^2J(HH) = 12.4 Hz, $FC_6H_4CH_2N$]; 6.87–8.14 (m, C_6H_4). ^{13}C-NMR ($CDCl_3$, 50.3 MHz) δ: 37.63 [d, J(PC) = 8.8 Hz]; 41.35 [d, J(PC) = 1.9 Hz]; 49.62 [d, J(PC) = 12.1 Hz]; 51.64 (s), 52.39 (s), ($PNCH_2C_6H_4F$, $PNCH_2CH_2Cl$, $PNCH_2CH_2N$); 114.67–137.42 [12 s, C_6H_4]; 109.68 [tq, ^1J(CF^d) = 253.32 Hz, ^2J(CF^c) = 39.1 Hz, CF_3^d]; 117.08 [dtt, ^1J(CF^b) = 262.1, ^2J(CF^c) = 31.3, ^3J(PC) = 14.8 Hz, CF_2^b]; 122.41 [dq, ^1J(CF^a) = 259.1, ^3J(PC) = 19.18 Hz, CF_3^a]; 124.91 [m, CF_2^c]; 126.08 [dt, ^2J(CF^b) = 33.7, ^2J(PC) = 3.92 Hz, CCF_2^b]; 131.71 [dq, ^2J(CF^a) = 38.61, ^2J(PC) = 4.21 Hz, CCF_3^a]; 166.77 [d, ^2J(PC) = 3.1 Hz, C(:O)NP]. ^{19}F-NMR ($CDCl_3$, 188.3 MHz) δ: −64.95 [t, br, ^5J(F^{a}F^b) = 2.8 Hz, F^a]; −80.90 [t, ^3J(F^{c}F^d) = 9.6 Hz, F^d]; −114.47 (m, F^c), −117.66 (s, $CH_2C_6H_4F$); −127.06 (m, br, F^b). ^{31}P-NMR ($CDCl_3$, 81.0 MHz) δ: −36.34. EI-MS m/z (%): 671 (5) [M]$^+$; 622 (6) [M–CH_2Cl]$^+$; 321 (16) [POC(CF_3)C(C_3F_7)]$^+$; 188 (8) [$CF_3CF_2CF_3$]$^+$; 169 (14) [C_3F_7]$^+$; 109 (100) [$CH_2C_6H_4F$]$^+$; 69 (58) [CF_3]$^+$. High-resolution mass spectrometry (res.: 10000, 10% Taldef.): theoret. 671.0598, exp. 671.056 ± 2 ppm. –$C_{24}H_{18}ClF_{11}N_3O_3P$ (671.83).

3.3. Reaction of *2* with *3*: synthesis of *7*

The preparation of **7** was conducted in a fashion similar to that described for compounds **5a** and **5b** from 0.54 g (1.2 mmol) of **2** and 0.34 g (1.4 mmol) of perfluoroethylmethyl diketone **3**. Yield, 0.64 g (84%); m.p. 142°C.

^{1}H-NMR ($CDCl_3$, 200.1 MHz) δ: 3.26–3.54 (m, CH_2CH_2Cl, NCH_2CH_2N); 4.80 [dd, ^3J(FH) = 8.9, ^2J(HH) = 16.5 Hz, $ClC_6H_4CH_2N$]; 5.29 [dd, ^3J(FH) = 9.7, ^2J(HH) = 16.6 Hz, ClC6H4CH_2N]; 7.03–8.25 (m, C_6H_4). ^{13}C-NMR ($CDCl_3$, 50.3 MHz) δ: 30.36 [d, J(PC) = 4.2 Hz], 42.12 [d, J(PC) = 4.1 Hz], 42.44 [d, J(PC) = 14.1 Hz], 46.76 (s), 49.66 (s), ($PNCH_2C_6H_4Cl$, $PNCH_2CH_2Cl$, $PNCH_2CH_2N$); 113.05–142.53 [12 s, C_6H_4]; 107.08 [tq, ^1J(CF^c) = 259.8, ^2J(CF^b) = 35.7 Hz, CF_3^c]; 114.02 [m, CF_2^b]; 118.63 [dq, ^1J(CF^a) = 265.2, ^3J(PC) = 11.26 Hz, CF_3^a]; 127.43 [dt, ^2J(CF^b) = 34.2, ^2J(PC) = 4.7 Hz, CCF_2^b]; 133.66 [dq, ^2J(CF^a) = 39.8, ^2J(PC) = 5.5 Hz, CCF_3^a]; 163.60 [d, ^2J(PC) = 3.74 Hz, C(:O)NP]. ^{19}F-NMR ($CDCl_3$, 188.3 MHz) δ: −65.51 [t, ^3J(F^{b}F^c) = 21.1 Hz, F^c]; −81.90 (s, F^a); −121.92 [dq, ^3J(F^{b}F^c) = 15.3, ^4J(PF^b) = 5.5 Hz, F^b]. ^{31}P-NMR ($CDCl_3$, 81.0 MHz) δ:

−35.98. EI-MS *m/z* (%): 637 (42) $[M]^+$; 602 (100) $[M-Cl]^+$; 461 (1) $[M-CH_2C_6H_4Cl-CH_2Cl]^+$; 243 (14) $[C_6H_4C(:O)NCH_2C_6H_4Cl]^+$; 132 (17) $[C_6H_4NC(:O)N]^+$; 125 (73) $[CH_2C_6H_4Cl]^+$; 69 (11) $[CF_3]^+$. High-resolution mass spectrometry (res.: 10000, 10% Tal-def.): theoret. 637.0335, exp. 637.034±2 ppm. $-C_{23}H_{18}Cl_2F_8N_3O_3P$ (638.28).

3.4. Reaction of 2 with 4: synthesis of 8

The preparation of **8** was conducted in a fashion similar to that described for compounds **5a** and **5b** from 0.54 g (1.2 mmol) of **2** and 0.38 g (1.3 mmol) of perfluoromethylpropyl diketone **4**. Yield 0.35 g (43%); m.p. 89°C.

^{1}H-NMR ($CDCl_3$, 200.1 MHz) δ: 3.18–3.59 (m, CH_2CH_2Cl, NCH_2CH_2N); 4.78 [dd, ^3J(FH) = 6.7, ^2J(HH) = 16.5Hz, $ClC_6H_4CH_2N$]; 5.33 [dd, ^3J(FH) = 6.8, ^2J(HH) = 12.8 Hz, $ClC_6H_4CH_2N$]; 6.83–8.15 (m, C_6H_4). ^{13}C-NMR ($CDCl_3$, 50.3 MHz) δ: 36.62 [d, J(PC) = 2.2 Hz]; 40.33 [d, J(PC) = 2.3 Hz]; 50.09 [d, J(PC) = 12.4 Hz], 51.69 (s), 52.40 (s), ($PNCH_2C_6H_4Cl$, $PNCH_2CH_2Cl$, $PNCH_2CH_2N$); 114.96–142.82 [12 s, C_6H_4]; 106.67 [tq, $^1J(CF^d)$ = 259.13, $^2J(CF^c)$ = 38.3 Hz, CF^d_3]; 113.69 [dtt, $^1J(CF^b)$ = 258.0, $^2J(CF^c)$ = 31.9, ^3J(PC) = 14.9 Hz, CF^b_2]; 119.81 [dq, $^1J(CF^a)$ = 263.7, ^3J(PC) = 18.23 Hz, CF^a_3]; 123.94 [m, CF^c_2]; 127.17 [dt, $^2J(CF^b)$ = 33.6, ^2J(PC) = 4.02 Hz, CCF^b_2]; 131.00 [dq, $^2J(CF^a)$ = 40.01, ^2J(PC) = 3.31 Hz, CCF^a_3]; 165.44 [d, ^2J(PC) = 10.1 Hz, $C(:O)NP$]. ^{19}F-NMR ($CDCl_3$, 188.3 MHz) δ: −65.26 [t, br, $^5J(F^aF^b)$ = 3.1 Hz, F^a]; −80.97 [t, $^3J(F^cF^d)$ = 9.5 Hz, F^d]; −114.68 (m, F^c); −127.26 (m, br, F^b). ^{31}P-NMR ($CDCl_3$, 81.0 MHz) δ: −36.57 (s). EI-MS *m/z* (%): 687 (10) $[M]^+$; 652 (22) $[M-Cl]^+$; 169 (25) $[C_3F_7]^+$; 125 (100) [CH2C6H4Cl]$^+$; 69 (16) $[CF_3]^+$. High-resolution mass spectrometry (res.: 10000, 10% Taldef.): theoret. 687.0303, exp. 687.030±2 ppm. $-C_{24}H_{18}Cl_2F_{10}N_3O_3P$ (688.28).

3.5. Reaction of 9 with 3: synthesis of 10a

The preparation of **10a** was conducted in a fashion similar to that described for compounds **5a** and **5b** from 0.54 g (1.6 mmol) of **9** and 0.40 g (1.6 mmol) of perfluoroethylmethyl diketone **3**. Yield, 0.55 g (59%); m.p. 132°C.

^{1}H-NMR ($CDCl_3$, 200.1 MHz) δ: 3.21–3.62 [m, CH_2CH_2Cl]; 3.28, 3.33 [2 d, ^3J(PH) = 12.7, 13.6 Hz, NCH_3]; 7.03–8.25 (m, C_6H_4). ^{13}C-NMR ($CDCl_3$, 50.3 MHz) δ: 27.77, 27.84 [2 d, ^2J(PC) = 41.4, 42.0 Hz, NCH_3]; 42.26 [s, $PNCH_2CH_2Cl$]; 49.78 [d, ^2J(PC) = 5.0 Hz, $PNCH_2CH_2Cl$]; 112.90–142.20 [6 s, C_6H_4]; 105.29 [tq, $^1J(CF^c)$ = 262.6, $^2J(CF^b)$ = 28.6 Hz, CF^c_3]; 115.79 [m, CF^b_2]; 118.69 [dq, $^1J(CF^a)$ = 266.1, ^3J(PC) = 13.7 Hz, CF^a_3]; 127.83 [dt, $^2J(CF^b)$ = 34.8, ^2J(PC) = 4.2 Hz, CCF^b_2]; 136.21 [dq, $^2J(CF^a)$ = 39.0, ^2J(PC) = 5.3 Hz, CCF^a_3]; 163.93 [d, ^2J(PC) = 3.8 Hz, $C(:O)NP$]. ^{19}F-NMR ($CDCl_3$, 188.3 MHz) δ: −68.90 [t, $^3J(F^bF^c)$ = 21.3 Hz, F^c]; −81.94 (s, F^a); −119.90 [dq, $^3J(F^bF^c)$ = 15.2, $^4J(PF^b)$ = 5.2 Hz, F^b]. ^{31}P-NMR ($CDCl_3$, 81.0 MHz) δ: −32.72 (s). EI-MS *m/z* (%): 577 (<1) $[M]^+$; 243 (100) $[M-CF_3C(:O)C(:O)C_2F_5]^+$; 69 (15) $[CF_3]^+$. $C_{18}H_{18}Cl_2F_8N_3O_3P$ (578.22) Ber.: C, 37.39; H, 3.14; P, 5.36. Gef.: C, 37.93; H, 4.20; P, 5.42.

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References

[1] F. Ramirez, C.P. Smith, J.F. Pilot, A.S. Gulati, J. Org. Chem. 33 (1968) 3787.
[2] F. Ramirez, Synthesis (1974) 90.
[3] I. Neda, M. Farkens, A.K. Fischer, P.G. Jones, R. Schmutzler, Z. Naturforsch. 50b (1995) 1785.
[4] A. Kadyrov, I. Neda, T. Kaukorat, R. Sonnenburg, A. Fischer, P.G. Jones, R. Schmutzler, Chem. Ber. 129 (1996) 725.
[5] I. Neda, A. Fischer, P.G. Jones, R. Schmutzler, Phosphorus, Sulfur, Silicon 78 (1993) 271.
[6] I. Neda, C. Melnicky, A. Vollbrecht, R. Schmutzler, Synthesis (1996) 473.
[7] I. Neda, C. Melnicky, A. Vollbrecht, A. Fischer, P.G. Jones, R. Schmutzler, Z. Anorg. Allg. Chem. 622 (1996) 1047.
[8] R.E. Duff, R.K. Oram, S. Tripett, J. Chem. Soc., Chem. Comm. (1971) 1011.
[9] E. Evangelidou-Tsolis, F. Ramirez, Phosphorus, 4 (1974) 121.
[10] R.K. Oram, S. Tripett, J. Chem. Soc., Perkin Trans I (1973) 1300.
[11] V. Mark, C.H. Dungan, M.M. Crutchfield, J.R. Van Wazer, in M. Grayson, E.J. Griffith (Eds.), Topics in Phosphorus Chemistry, Vol. 5, Wiley Interscience, New York, London, Sydney, Toronto, 1967, p. 227.
[12] D.D. Perrin, W.L.F. Armarego, Purification of Laboratory Chemicals, 4th edn., Pergamon, Oxford, New York, Beijing, Frankfurt, Sao Paulo, Sydney, Tokyo, Toronto, 1988.
[13] A.A. Kadyrov, I. Neda, T. Kaukorat, A. Fischer, P.G. Jones, R. Schmutzler, J. Fluorine Chem. 72 (1995) 29.
[14] A.Ya. Zapevalov, T.I. Filyakova, I.P. Kolenko, Izv. Akad. Nauk SSSR, Ser. Khim. (1979) 2812.
[15] M.A. Kurikin, L.S. German, Yu.N. Studnev, A.V. Fokin, Izv. Akad. Nauk SSSR, Ser. Khim. (1980) 1679.