Ferrocenylbis(ylene)phosphoranes [1]

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Abstract

The ferrocenylbis(methylene)phosphorane $Fc-P(=CTms_2)_2$ (1) ($Fc = -C_5H_4FeC_5H_5$) can be synthesized starting from ferrocenyldichlorophosphane $FcPCl_2$ or ferrocenyldilithiophosphane $FcPLi_2$. The X-ray structure of (1) shows some unusual structural features, which indicate considerable electronic interaction of the ferrocenyl group and the $\sigma^3\lambda^5$-phosphorane unit. As a consequence of this interaction, the rotational barrier of the methylene bonds is extraordinary low. Therefore, in contrast to other bis(methylene)phosphoranes, the *endo*- and *exo*-Tms groups are indistinguishable on the NMR time scale even at $-100°C$. The analogous ferrocenylbis(imino)phosphorane $Fc-P(=NTms)_2$ (9), can only be isolated in its dimeric form. The conditions, which are necessary for the formation of (9) from the corresponding azidophosphane $Fc-P(N_3)NTms_2$ under elimination of nitrogen lead to a self addition reaction of (9). The elimination of nitrogen is facilitated by polar solvents, and electrophilic reagents. © 1997 Elsevier Science S.A.

Keywords: Ferrocenylbismethylenephosphorane; X-ray crystallography

1. Introduction

The ferrocenyl substituent is known to stabilize a positive charge on an adjacent atom. While this is a well explored phenomenon in the case of carbenium ions [1], considerably less information is available concerning a third row element, acting as an electrophilic centre towards an adjacent ferrocenyl group [2]. Investigations of the electrochemical behaviour in correlation with Mossbauer spectroscopy showed, that also in neutral ferrocenyl phosphanes the phosphorus atom always acts as electron acceptor [3]. Further evidence for such interaction arises particularly from the unusual chemical behaviour of ferrocenyl phosphanes containing a low coordinated phosphorus centre [4], although no crystal structure of such a compound has been reported so far. The extent of a dative interaction should be even higher, when an electrophilic bis(ylene)phosphorane fragment —which formally derives from monomeric metaphosphorus acid—is attached to ferrocene. In this paper we report for the first time the crystal structure and spectroscopic properties of a ferrocenylbis(methylene)phosphorane, to give further insight into the nature of the ferrocenyl substituent's donation abilities.

2. Results and discussion

2.1. Ferrocenylbismethylenephosphorane

The title compound ferrocenyl-bis(bistrimethylsilylmethylene)phosphorane 1 was synthesized by addition of a threefold excess of bis(trimethylsilyl)methylenecarbenoid to ferrocenyldichlorophosphane at $-100°C$ (Scheme 1). The product was purified by subsequent recrystallization from toluene–pentane at $-30°C$, yielding dark red crystals, which were characterized by NMR and mass spectroscopy.

The ^{31}P chemical shift at 170.0 ppm is within the usual range for bis(methylene)phosphoranes. The ^{13}C NMR spectra show a doublet for the two methylene carbon atoms at 81.5 ppm ($^1J_{CP} = 54.0\,Hz$). The *endo*- and *exo*-trimethylsilyl groups are indistinguishable in

(1)
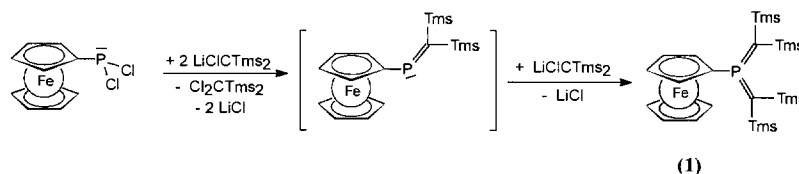
Scheme 1.

the ^{1}H-, ^{13}C- and ^{29}Si-NMR spectra. The constitution of **1** was further corroborated by an X-ray structure analysis (Fig. 1). Interestingly, in the solid state the molecule exhibits only C_1 symmetry. While the P=C bond lengths (1.657(3) Å, 1.667(3) Å; Table 1) are in common with values found in bis(methylene)phosphoranes [5], the P–C(ferrocenyl) bond distance (1.783(3) Å) is shorter than in ferrocenylphosphanes (1.810(3) Å) [6] but slightly longer than in the corresponding ferrocenylphosphonium salts (1.768(3) Å) [7]. In contrast to well established models describing the stabilization of an electrophilic centre by the ferrocenyl substituent [8], no direct interaction between the iron atom and the electrophilic phosphorus centre can be observed (P · · · Fe 3.699(3) Å). The phosphorus atom is even shifted slightly away from the iron atom to the opposite side of the cyclopentadienyl plane by 0.52 Å.

As common for bis(methylene)phosphoranes, the phosphorus-bonded carbon atoms surround the phosphorus centre in a propeller-like manner, which causes the helical chirality of the molecule [9]. The substituents at the phosphorus atom are twisted due to a second order Jahn–Teller-distortion, which allows the mixing of orbitals, which in the planar case would stand orthogonally to each other [10]. However, usually the difference between these twist angles of the identically substituted methylene units differs only slightly by 0–4° (Table 2). In **1** with respect to the C(11)PC(1)C(2) plane the C(1)Si(1)Si(2) and the C(2)Si(3)Si(4) planes are twisted by 38.2° and 48.5°, whereas the plane of the Cp ring is twisted by 37.6°. The resulting difference of the twist angles for the methylene groups is 10.3°. So far, only in the dimethylamino-substituted bis(methylene)phosphorane **2** has a twist difference of the same

magnitude been observed. Quantum chemical investigations proved that the twisting of the methylene groups is facilitated by π-donating substituents at the phosphorus atom, whereas the driving force for this phenomenon is the steric congestion of the substituents attached to the methylene carbon atoms [11]. Experimental evidence of the shallow potential of the twisted methylene groups arises from the fact that the *endo-* and *exo-*trimethylsilyl groups are indistinguishable on the NMR time scale even at -100 °C, which has never been observed for a bis(methylene)phosphorane so far.

The ferrocenyl unit shows a staggered conformation and the arrangement of the Cp-rings differs from linearity by 11.0°. Although both Cp-rings are planar and show almost the same Fe–Cp distance (1.660(2) Å and 1.662(2) Å), the bond lengths within the rings differ significantly. While in the substituted ring the values vary from 1.401(6) to 1.430(5) Å, in the unsubstituted one the values are in a range between 1.334(7) and 1.388(9) Å. The *ipso-*carbon atom is slightly pyramidalized ($\Sigma = 357.1(3)°$) and the distance to the adjacent ring carbon atoms are significantly widened (1.430(4) Å and 1.429(4) Å) by 0.03 Å with respect to the other C–C bond lengths within the same Cp-ring. Moreover, the Cp ring attached to the phosphorus atom shows a smaller twist angle with respect to the C(11)PC(1)C(2) plane, than both methylene units do, which allows better electronic interaction of the ferrocenyl group with the electrophilic phosphorus centre.

Therefore, it seems that in **1** the stabilization of the low-coordinated phosphorane centre rather occurs by resonance with the adjacent Cp-ring, than by direct interaction of the iron atom. This corresponds well with a formulation as described in Fig. 2 and stands in contrast to the stabilization of electrophilic carbon cen-
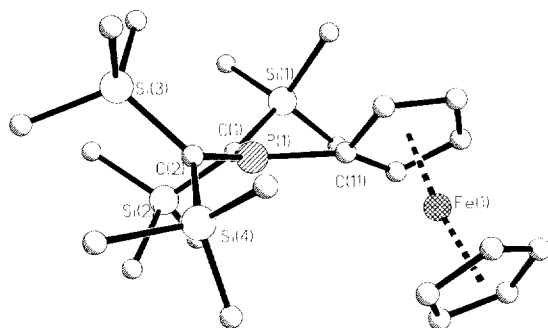
Fig. 1. Crystal structure of **1**.

Table 1
Selected interatomic distances (Å) and bond angles (deg)

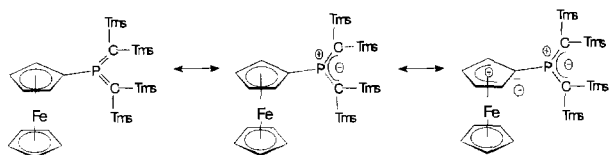| Distances			
P–C(11)	1.783(3)	C(1)–Si(1)	1.874(3)
P–C(1)	1.657(3)	C(1)–Si(2)	1.871(3)
P–C(2)	1.667(3)	C(2)–Si(3)	1.854(3)
		C(2)–Si(4)	1.852(3)
Angles			
C(1)–P–C(2)	127.5(2)	P(1)–C(1)–Si(1)	122.7(2)
C(2)–P–C(11)	118.7(2)	P(1)–C(1)–Si(2)	118.4(2)
C(1)–P–C(11)	113.2(2)	P(1)–C(2)–Si(3)	117.6(2)
Cp–Fe–Cp	168.96(2)	P(1)–C(2)–Si(4)	122.6(2)

Fig. 2. Illustration of the interaction of the ferrocenyl moiety with the phosphorane centre. The π-system of the Cp-ring is polarized by the electrophilic phosphorane fragment.

Table 2
Twist angles

R	twist angles	difference (°)	Ref.
-NMe₂ (2)	38° / 50°	12°	9
-Fc (1)	38.2° / 48.5°	10.3°	this work
-SPh	42°./.48°	4°	9
-Cl	37° / 41°	4°	9
-CHPh₂	40° / 41°	1	9
-CHMeEt	42° / 43°	1	9
-fluorenyl	40° / 41°	1	9
-CH₂R	43° / 43°	0	5

tres by an adjacent ferrocenyl unit. Moreover, the extent of stabilization seems to be comparable, but slightly smaller than in the corresponding amino-substituted compounds.

Bis(methylene)phosphorane **1** can also be obtained by utilizing precursors of the opposite polarity. Thus, reaction of dilithioferrocenylphosphanide [2] **3** with Cl₂CTms₂ at 0 °C, affords **1**. However the main product of this reaction is the diferrocenyldiphoshirane **4**. The formation of these products can be explained by assuming initial metal–halogen-exchange. The resulting phosphenoid and carbenoid, which are unstable under these conditions, immediately dimerize under elimination of LiCl. Further reaction with either the phosphenoid or the carbenoid leads to **1** or **4** respectively (Fig. 3). The predominant formation of the disphosphirane suggests an increased stability of the phosphenoid compared to the carbenoid under these conditions. Diphosphirane **4** was characterized by mass- and NMR-spectroscopy investigations.

2.2. Bis(imino)phosphorane

Considering the enhanced polarity of the PN compared with the PC bond, we were interested to synthe-

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[2] In contrast to the observations made by Cowley and coworkers [2], FcPLi₂ exhibits a ³¹P-chemical shift of −243 ppm (solvent DME). We found, that the value of −161.7 ppm claimed by these authors for FcPLi₂ has to be assigned to the monolithiated product, due to the ¹J_{PH}-coupling of 170.4 Hz (doublet).

size the corresponding ferrocenylbis(imino)phosphorane **9**. Starting from chlorophosphane **5** the reaction with trimethylsilylazide TmsN₃ furnishes azidophosphane **6**, which was isolated as a viscous oil in good yield (90%). While azidophosphane **6** remains unchanged in boiling toluene, heating or irradiation with UV light in THF solution leads to different products, which could not be characterized. In contrast, heating the same compound in DME solution, leads selectively to the diazadiphospetidines **10a,b**, which resemble the dimers of **9**. The trans isomer **10a** is formed in large excess and crystallizes on cooling to −30 °C. We suppose, that the initial step of the nitrogen elimination involves the formation of an unstable nitrilophosphorane **7**. Such species have been previously described as short living intermediates after nitrogen elimination from azidophosphanes [12]. Subsequently, **7** rearranges by migration of a trimethylsilyl
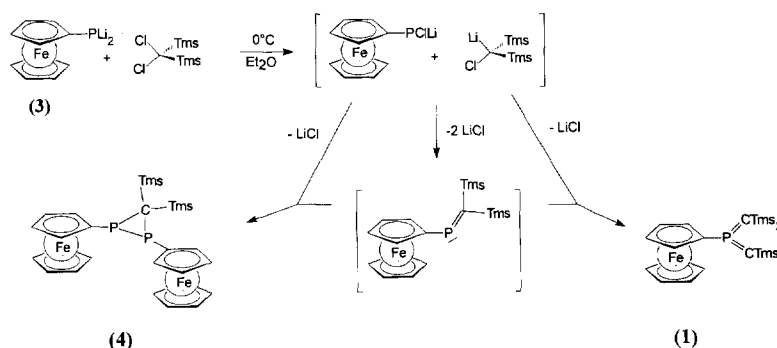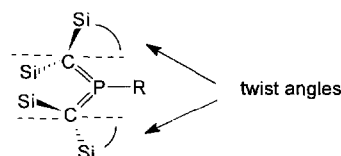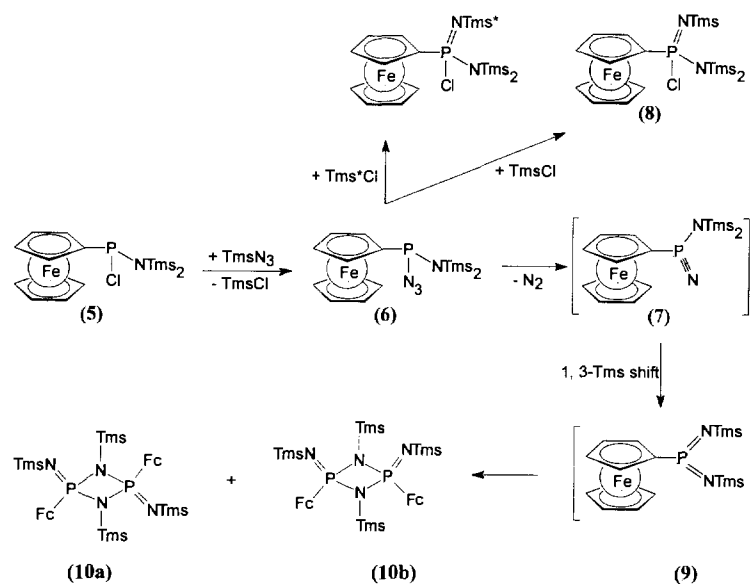

Fig. 3. Reaction of either the carbenoid or the phosphenoid with the intermediate methylenephosphane affords bis(methylene)phosphorane (**1**) or diphosphorane (**4**) respectively.

Fig. 4. Synthesis and reactions of azidophosphane (6).

group yielding **9**. The latter compound dimerizes to the diazadiphospetidine **10a,b**.

On the other hand, heating a toluene solution of **7** in the presence of electrophilic chlorosilanes leads to ferrocenylphosphorane **8** under elimination of molecular nitrogen at mild conditions (70 °C; Fig. 4). In this case, the occurrence of nitrilophosphorane **7** is unlikely, since the chlorosilanes obviously facilitate the elimination of the nitrogen, probably by initial addition of a silyl group to the azido function. Similar activation of azido groups by various electrophiles has already been reported for organic azides [13].

The unusual structural and spectroscopic properties of ferrocenyl-substituted bis(methylene)phosphorane **1** suggest, that the electronic situation in the ferrocenyl-substituted bis(imino)phosphorane **9** is comparable to amino-substituted bis(imino)phosphoranes $R_2N-P(=NTms)_2$. In the case of R = Tms, the Ni(0)-complexes of the latter are known to catalyse the ethene polymerization [14], as well as the α-ω-polymerization of α-olefins [15].

3. Experimental

3.1. General considerations

All steps were carried out under an atmosphere of dry argon using common Schlenk techniques. Solvents were dried and freshly distilled from Na–K alloy before use. NMR spectra were recorded on a Bruker AMX 300 spectrometer at 25 °C. ^{31}P NMR spectra were recorded using 85% H_3PO_4 as external standard, ^{1}H NMR spectra with tetramethylsilane as external standard.

3.2. Synthesis of 1

To a solution of Cl_2CTms_2 (0.48 g, 2.1 mmol) in 20 ml of a mixture of THF–Et_2O–pentane in the ratio 8:6:3 a solution of n-BuLi (1.44 ml, 2.3 mmol) was added slowly at −100 °C. After stirring for 0.5 h, the mixture was treated with a solution of FcPCl$_2$ (0.2 g, 0.7 mmol) in 11 ml ether at the same temperature. After stirring for a further 0.5 h the mixture was warmed to ambient temperature and filtered. The solvent was removed in vacuum and the dark red residue was recrystallized from toluene–pentane (yield: 0.2 g, 0.36 mmol, 52%). ^{31}P NMR (C_6D_6): δ 170.0. ^{1}H NMR (C_6D_6): δ 4.68 (2H, m), 4.26 (2H, m), 4.12 (5H, d, $^4J_{HP} = 0.2$ Hz), 0.53 (36H, s). ^{13}C NMR (C_6D_6): δ 83.6 (d, $^1J_{CP} = 52.8$ Hz, C(ipso)), 81.5 (d, $^1J_{CP} = 54.0$ Hz, = $CTms_2$), 76.6 (d, $^2J_{CP} = 13.7$ Hz, C_α), 72.2 (d, $^3J_{CP} = 9.7$ Hz, C_β), 71.1 (s, Cp'), 5.1 (d, $^3J_{CP} = 5.6$ Hz, Tms exo/endo). ^{29}Si NMR (C_6D_6): δ −11.49 (d, $^2J_{SiP} = 2.1$ Hz) Tms endo/exo. MS (EI, 25 eV, 200 °C): m/z 532.1677 (found), 532.1684 (calcd.), M^+, 41%; 495, $M^+ -$ Tms, 12%; 73, Tms^+, 100%.

3.3. Synthesis of 4

To a solution of 0.44 g (2 mmol) FcPH$_2$ in 20 ml of DME 2.2 mmol n-BuLi dissolved in hexane are added at −78 °C. The resulting dark red mixture is warmed to room temperature and stirred for 1 h. Thereafter it is treated with 0.46 g (2 mmol) Cl_2CTms_2, diluted in 4 ml DME at 0 °C. After further stirring for 2 h the solvent is evaporated in vacuum and the residue is extracted twice with 15 ml of pentane. The insoluble precipitate is

removed by filtration and the solvent and volatile organic by-products are removed in high vacuum (10^{-3} Torr). Diphosphirane **4** is obtained as a yellow viscous oil (0.77 g, 1.3 mmol, 65%). ^{31}P NMR (toluene): -154.8 (s). ^{1}H NMR (C_6D_6): 4.60 (4H, m), H_α; 4.47 (4H, m), H_β; 4.40 (10H, s), Cp'; 0.27, (18H, s), Tms. ^{13}C NMR (C_6D_6): 83.4 (dd, $^1J_{CP} = 61.4$ Hz, $^2J_{CP} = 22.5$ Hz) C_{ipso}, 77.5, ("t", $^{2/3}J_{CP} = 5.1$ Hz), C_α, 76.7 (s), C_β; 70.3 (s), Cp'; 3.41 (t, $^3J_{CP} = 4.5$ Hz), Tms; 1.0 (t, $^1J_{CP} = 49.7$ Hz), $>CTms_2$. MS (EI): 590.0529 (found) 590.0530 (calcd.) (^{56}Fe,^{56}Fe), 588.0581 (found) 588.0576 (calcd.) (^{54}Fe,^{56}Fe), M^+, 34%; 517 $M^+ -$ Tms, 13%; 401, 35%; 216, FcP$^+$, 37%; 147, Tms$_2^+$, 29%; 73, Tms$^+$, 100%.

3.4. Synthesis of 5

To a solution of FcPCl$_2$ (1.07 g, 3.73 mmol) in 30 ml of an Et$_2$O–pentane mixture (1:1), solid LiNTms$_2$ · Et$_2$O (0.99 g, 4.1 mmol) is added at -100°C. After stirring for 0.5 h the mixture was warmed to ambient temperature. The solvent was removed in vacuum and the residue extracted with pentane. The solution was filtered and the solvent evaporated, yielding **5** as a viscous oil (1.53 g, 3.72 mmol, 99.7%). ^{31}P NMR (C_6D_6): δ 138.2. ^{1}H NMR (C_6D_6): δ 4.62 (1H, m), 4.60 (1H, m), 4.39 (2H, m), 4.19 (5H, d, $^4J_{HP} = 3.9$ Hz), 0.2 (18H, s). ^{13}C NMR (C_6D_6): δ 84.4 (d, $^1J_{CP} = 40.8$ Hz, C(ipso)), 72.3 (d, $^2J_{CP} = 3.4$ Hz, C_α), 71.2 (d, $^2J_{CP} = 3.4$ Hz, $C_{\alpha'}$), 71.2 (d, $^3J_{CP} = 22.5$ Hz, C_β), 70.5 (s, $C_{\beta'}$), 69.9 (d, $^3J_{CP} = 2.7$ Hz, Cp'), 4.5/4.4 (s, Tms exo/endo). MS (EI, 25 eV, 200°C): m/z 411.0462 (found), 411.0458 (calcd.), 411, M$^+$, 18.3%; 376, M$^+ -$ Cl, 48.3%; 303, M$^+ -$ TmsCl, 65%; 73, Tms$^+$, 100%.

3.5. Synthesis of 6

A solution of **5** (0.42 g, 1.0 mmol) in 5 ml of THF is treated with TmsN$_3$ (0.13 g, 1.1 mmol) at room temperature. After stirring for 24 h, the solvent and volatile by-products are removed in vacuum. The product is obtained as a viscous oil (0.39 g, 0.9 mmol, 90%). ^{31}P NMR (C_6D_6); δ 116.8. ^{1}H NMR (C_6D_6): δ 4.53 (1H, m), 4.23 (1H, m), 4.17 (5H, s), 4.16 (2H, m), 0.33 (18H, d, $^4J_{HP} = 1.3$ Hz). ^{13}C NMR (C_6D_6): δ 81.5 (d, $^1J_{CP} = 12.9$ Hz, C(ipso)), 71.6 (d, $^2J_{CP} = 9.2$ Hz, C_α), 71.2 (d, $^2J_{CP} = 12.2$ Hz, $C_{\alpha'}$), 70.4 (s, C_β), 70.1 (d, $^3J_{CP} = 2.3$ Hz, Cp'), 4.7 (d, $^3J_{CP} = 8.4$ Hz; d, $^1J_{CSi} = 56.8$ Hz, Tms). Due to its decomposition tendency, no satisfying microanalysis or mass spectra of **6** could have been obtained.

3.6. Synthesis of (10a,b)

A solution of **6** (0.2 g, 0.5 mmol) in 2.0 ml of DME is heated to 110°C for 3 h. The solvent is evaporated in

vacuum and recrystallization of the residue from toluene–pentane affords **10** as pale yellow solid (0.1 g, 0.3 mmol, 60%). **10a,b**: ^{31}P NMR (C_6D_6): δ -24.2 and -19.0. **10a**: ^{1}H NMR (C_6D_6): δ 4.29 (4H, m), 4.26 (4H, m), 4.19 (10H, s), 0.38 (18H, s), 0.23 (18H, s). ^{13}C NMR (C_6D_6): δ 84.6 (d, $^1J_{CP} = 40.5$ Hz, C(ipso)), 71.4 (d, $^2J_{CP} = 15.6$ Hz, C_α), 70.9 (d, $^3J_{CP} = 17.4$ Hz, C_β), 70.4 (s, Cp'), 4.6 (t, $^3J_{CP} = 7.4$ Hz Tms), 2.4 (s, Tms). MS (EI, 25 eV, 200°C): m/z 780.1611 (found), 780.1623 (calcd), M$^+$, 28.2%; 709, M$^+ -$ Tms, 45%; 147, Tms$_2$H$^+$, 100%; 73, Tms, 12%.

3.7. Synthesis of 8

To a solution of **6** (0.84 g, 2.0 mmol) in 5 ml of toluene a large excess of TmsCl (0.5 ml) is added at room temperature. The mixture is heated to 100°C for 3 h. After evaporation of the solvent and excess TmsCl, **8** can be obtained as a viscous oil (0.95 g, 1.9 mmol, 95%). ^{31}P NMR (C_6D_6): δ 10.9. ^{1}H NMR (C_6D_6): δ 4.63 (1H, m), 4.52 (1H, m), 4.33 (5H, s), 4.15 (1H, m), 4.05 (1H, m), 0.59 (9H, s), 0.47 (18H, s). ^{13}C NMR (C_6D_6): δ 86.5 (d, $^1J_{CP} = 177.8$ Hz, C(ipso)), 73.5 (d, $^2J_{CP} = 16.0$ Hz, C_α), 72.6 (d, $^2J_{CP} = 19.0$ Hz, $C_{\alpha'}$), 71.7 (d, $^3J_{CP} = 14.9$ Hz, C_β), 71.0 (s, Cp'), 68.5 (d, $^3J_{CP} = 13.4$ Hz, $C_{\beta'}$), 5.7 (d, $^3J_{CP} = 2.6$ Hz, –NTms$_2$), 4.2 (d, $^3J_{CP} = 5.4$ Hz, =NTms). MS (EI, 25 eV, 200°C): 498.0968 (found), 498.0962 (calcd.), M$^+$, 6%; 411, M$^+ -$ NTms, 8%; 147, Tms$_2$H$^+$, 100%; 73, Tms$^+$, 19%.

3.8. X-ray crystallographic study of 1

[$C_{24}H_{45}$FePSi$_4$], $M_r = 532.78$, crystal dimensions $0.55 \times 0.35 \times 0.25$ mm^3, monoclinic, space group $P2_1/c$ (No. 14), $a = 8.004(4)$ Å, $b = 22.362(11)$ Å, $c = 17.281(10)$ Å, $\beta = 101.59(4)°$; $V = 3025(3)$ Å^3, $Z = 4$, $D_c = 1.170$ g cm^{-3}, $F(000) = 1144$, $T = 293(2)$ K, 283 parameters, 5562 data measured, 5374 used for calculations, no restraints, $R = 0.0418$, $wR2 = 0.105$ for all data, largest diff. peak and hole 0.277 and $-0.225\,e^-$ Å^{-3}. Diffraction data were collected on a Nicolet R3m with graphite-monochromatized Mo Kα radiation ($\lambda = 0.71073$ Å) in the range $\vartheta = 1.51$ to 25.05°. Intensity data were corrected every 147 reflections. The structure was solved by direct methods and refinement was done using the full-matrix least squares on F^2 using SHELXL-93 [16].

Acknowledgements

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