The Ambiguous Behaviour of Diphosphines Towards The Quasilinear Iron(I) Complex $[Fe(N(SiMe_3)_2)_2]^2$ – Between Inertness, P–C Bond Cleavage and C–C Double Bond Isomerisation

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General considerations

All manipulations were carried out in a glovebox or using Schlenk-type techniques under a dry argon atmosphere. Used solvents were dried by continuous distillation over sodium metal for several days, degassed via three freeze-pump-thaw cycles and stored over molecular sieves 4 Å. THF-d₈ was degassed via three freeze-pump-thaw cycles and stored over molecular sieves 4 Å. The ¹H NMR spectra were recorded on a Bruker AV III 500 or Bruker AV II 300 NMR spectrometers. Chemical shifts are reported in ppm relative to the residual proton signals of the solvent (for ¹H) or relative to the signal of the solvent itself (¹³C). $w_{1/2}$ is the line width of a signal at half its maximum intensity. Integrals of the broad signals of the silylamide units were obtained directly or by peak fitting (in case of overlapping signals) using the MestreNova software package. IR measurements were conducted on a Bruker Alpha ATR-IR spectrometer. The UV/VIS measurement were recorded on an Analytik Jena Specord S600 diode array UV/Vis spectrometer using the WinASPECT software. Elemental analysis was performed by the "in-house" service of the Chemistry Department of the Philipps University Marburg, Germany using a CHN(S) analyzer vario MICRO Cube (Elementar). Solution magnetic susceptibilities were determined by the Evans method.^[i] All substrates were obtained commercially (Sigma-Aldrich, Acros, Strem, Alfa Aesar) and - if not noted otherwise - used as received. Dimethoxy ethane, N-methyl-2-pyrrolidone, NEt₃, TMEDA and 4-tert-butylpyridine were degassed, transferred into the glovebox and stored over molecular sieves. 18-crown-6 was sublimed prior use to remove traces of water. $[Fe{N(SiMe_3)_2}_2]^{ii}$ and $K{18c6}[Fe{N(SiMe_3)_2}_2], 1$,ⁱⁱⁱ were prepared according to literature procedures.

1. Synthesis of K{18-crown-6}[Fe(N(SiMe₃)₂)₃] (2)

A solution of 38 mg (0.1 mmol, 1 equiv.) Fe(N(SiMe₃)₂)₂ and 27 mg (0.1 mmol, 1 equiv.) 18-crown-6 in 2 ml Et₂O was treated with 20 mg (0.1 mmol 1 equiv.) KN(SiMe₃)₂. After 1 minute the colourless solution was filtered, layered with 3 ml pentane and stored at -35°C. After 16 h the supernatant solution was removed via a Pasteur pipette. The remaining colourless crystalline solid was washed with 2x2 ml pentane. Drying of the solid in vacuo yielded 74 mg (0.088 mmol, 88%) **K{18-crown-6}[Fe(N(SiMe₃)₂)₃]**, **2**. ¹H NMR (THF-d₈, 298 K, ppm, 500.1 MHz): δ = 3.38 (24 H, $w_{1/2}$ = 7.3 Hz, O-CH₂), - 2.36 (54H, $w_{1/2}$ = 530 Hz, SiMe₃). IR (ATR, cm⁻¹): v = 2944 (m), 2892 (m), 1471 (vw), 1454 (vw), 1351 (m), 1233 (s), 1105 (vs), 977 (vs), 960 (s), 866 (m), 824 (vs), 780 (m), 750 (m), 709 (m), 661 (s), 609 (m), 529 (w). Elemental analysis calcd. (%) for C₃₀H₇₈FeKN₃O₆Si₆ (840.42 g/mol): C 42.87, H 9.36, N 5.00; found: C 42.47, H 9.02, N 4.88. μ_{eff} = 5.15 μ_B (Evans, THF-d₈ + 1% Si(CH₃)₄), $\mu_{s.o.}$ = 4.90 μ_B).



Figure S 1. ¹H NMR spectrum of K{18-crown-6}[Fe(N(SiMe₃)₃)] (2) in THF-d₈ (500.1 MHz).

2. Synthesis of K{18-crown-6}[Fe(Ph)(N(SiMe₃)₂)(ppbz*] (3)

A solution of 136 mg (0.2 mmol, 1 equiv.) K{18-crown-6}[Fe(N(SiMe₃)₂)₂] (1) in 5 ml Et₂O was treated with 45 mg dppbz (0.1 mmol, 0.5 equiv.). After stirring over night the blood red suspension was filtered, the dark red crystalline residue washed with 3 ml Et₂O and shortly dried *in vacuo* yielding 19 mg (0.017 mmol) of K{18-crown-6}[Fe(Ph)(N(SiMe₃)₂)(ppbz]*2Et₂O (3*2Et₂O). The combined filtrates were slightly reduced in volume *in vacuo* and kept at room temperature for several days to give a second crop of blood red crystals of 3*2Et₂O (45 mg, 0.041 mmol, combined yield 58%). ¹H NMR (THF-d₈, 298 K, ppm, 500.1 MHz): δ = 85.8, 27.6, 18.2, 15.6, 13.3, 7.1 ($w_{1/2}$ = 762 Hz, SiMe₃), 3.14 ($w_{1/2}$ = 120 Hz, O-*CH*₂), -11.34, -13.95, -22.65, -23.0, -53.0.). IR (ATR, cm⁻¹): v = 3050 (vw), 2943 (w), 2891 (m), 1562 (w), 1469 (vw), 1433 (w), 1416 (w), 1350 (m), 1282 (m), 1235 (m), 1102 (vs), 1023 (m), 995 (s), 960 (s), 871 (m), 820 (s), 778 (s), 744 (m), 719 (s), 695 (s), 659 (m), 615 (m), 528 (m), 514 (m), 460 (m), 437 (w). Elemental analysis calcd. (%) for C₅₆H₈₄FeKNO₈P₂Si₂ (1110.45 g/mol): C 60.36, H 7.78, N 1.26; found: C 60.77, H 7.04, N 1.59. μ_{eff} = 5.29 μ_B (Evans, THF-d₈ + 1% Si(CH₃)₄), $\mu_{s.o.}$ = 4.90 μ_B).



Figure S 2. ¹H NMR spectrum of K{18-crown-6}[Fe(Ph)(N(SiMe₃)₂)(ppbz^{*}] (3) in THF-d₈ (500.1 MHz). (*) denotes Et_2O .



Figure S 3. *In-situ* ¹H NMR spectrum of the reaction of complex **1** with 1 equivalent dppbz (formally 0.5 equivalent excess). (*) signals belonging to complex **3**; (#) signal belonging to complex **2**. Integral ratio of the signals attributed to the respective SiMe₃ groups is 2.8 (complex **2**, signal at -2.36 ppm) : 1 (complex **3**, broad signal around 7 ppm). Deviation from the expected 3:1 integral ratio can be attributed to differences in paramagnetically induced line broadening and very short relaxation times.



Figure S 4. UV/Vis spectrum of K{18-crown-6}[Fe(Ph)(N(SiMe₃)₂)(ppbz*] (3) in THF.

3. Synthesis of K{18-crown-6}[Fe(N(SiMe₃)₂)₂(trans-dppee)] (4)

Method A: A solution of 34 mg (0.05 mmol, 1 equiv.) K{18-crown-6}[Fe(N(SiMe₃)₂)₂] (1) in 3 ml THF was treated with 20 mg (0.05 mmol, 1 equiv.) *cis*-dppee. After stirring for 3 days, the brown red solution was stripped of all volatiles *in vacuo*. The residue was extracted with a mixture of 2 ml Et₂O and 0.5 ml THF, the filtrate layered with 3 ml pentane and stored at -40°C. After 2 days the supernatant solution was removed using a Pasteur pipette. The remaining brown crystals were rinsed with 2x2 ml pentane and dried *in vacuo* to yield 33 mg (0.031 mmol, 61%) of K{18-crown-6}[Fe(N(SiMe₃)₂)(trans-dppee)], 4.

Method B: A solution of 34 mg (0.05 mmol, 1 equiv.) K{18-crown-6}[Fe(N(SiMe₃)₂)₂] (1) in 3 ml THF was treated with 20 mg (0.05 mmol, 1 equiv.) *trans*-dppee. After stirring for 30 minutes, the brownish red solution was stripped of all volatiles *in vacuo*. The residue was extracted with a mixture of 2 ml Et₂O and 0.5 ml THF, the filtrate layered with 3 ml pentane and stored at -40°C. After 2 days the supernatant solution was removed using a Pasteur pipette. The remaining brown crystals were rinsed with 2x2 ml pentane and dried *in vacuo* to yield 51 mg (0.047 mmol, 94% yield) of K{18-crown-6}[Fe(N(SiMe₃)₂)(*trans*-dppee)], 4.

¹H NMR (THF-d₈, 298 K, ppm, 500.1 MHz): $\delta = 14.71 (w_{1/2} = 303 \text{ Hz}), 10.23 (w_{1/2} = 57 \text{ Hz}), 9.54 (w_{1/2} = 51 \text{ Hz}), 7.33 (w_{1/2} = 52 \text{ Hz}), 6.58 (w_{1/2} = 52 \text{ Hz}), 2.44 (w_{1/2} = 65 \text{ Hz}, \text{O-CH}_2), -4.53 (w_{1/2} = 237 \text{ Hz}, \text{SiMe}_3), -18.27 (w_{1/2} = 52 \text{ Hz}). IR (ATR, cm⁻¹): v = 2939 (w), 2892 (w), 1583 (w), 1471 (w), 1451 (w), 1431 (w), 1351 (m), 1283 (w), 1244 (m), 1232 (m), 1105 (vs), 1053 (w), 989 (s), 959 (s), 885 (s), 864 (s), 819 (vs), 776 (m), 748 (m), 731 (s), 695 (s), 657 (s), 609 (s), 509 (m), 464 (s), 446 (m), 428 (m). Elemental analysis calcd. (%) for C₅₀H₈₂FeKNO₈P₂Si₄ (1076.44 g/mol): C 55.79, H 7.68, N 2.60; found: C 55.60, H 7.78, N 2.66. <math>\mu_{eff} = 4.38 \ \mu_B$ (Evans, THF-d₈ + 1% Si(CH₃)₄), $\mu_{s.o.} = 3.87 \ \mu_B$).



Figure S 5. ¹H NMR spectrum of K{18-crown-6}[Fe(N(SiMe₃)₂)₂(trans-dppee)] (4) in THF-d₈ (500.1 MHz).



Figure S 6. UV/Vis spectrum of K{18-crown-6}[Fe(N(SiMe₃)₂)₂(trans-dppee)] (4) in THF.

4. Synthesis of K{18-crown-6}[Fe(N(SiMe₃)₂)₂)(cis-dppee)] (5)

A solution of 34 mg (0.05 mmol, 1 equiv.) K{18-crown-6}[Fe(N(SiMe₃)₂)₂] in 3 ml THF was treated with 20 mg (0.05 mmol, 1 equiv.) *cis*-dppee. After stirring for 1 minute, the brown red solution was stripped of all volatiles *in vacuo*. The residue was extracted with a mixture of 2 ml Et₂O and 0.5 ml THF and the filtrate layered with 3 ml pentane and stored at -40°C. After 2 days, the supernatant solution was removed using a Pasteur pipette. The remaining brown crystals were rinsed with 2x2 ml pentane and dried *in vacuo* to yield 33.6 mg (0.031 mmol, 62%) of K{18-crown-6}[Fe(N(SiMe₃)₂)(*cis*-dppee)], 5.

¹H NMR (THF-d₈, 298 K, ppm, 500.1 MHz): δ = 15.25 (4H, $w_{1/2}$ = 110 Hz), 12.40 (2H, $w_{1/2}$ = 80 Hz), 7.25 (8H, $w_{1/2}$ = 75 Hz), 3.81 (2H, $w_{1/2}$ = 140 Hz), 3.09 (24 H, $w_{1/2}$ = 110 Hz, O-CH₂), 2.11 (2H, $w_{1/2}$ = 100 Hz), -2.66 (36 H, $w_{1/2}$ = 500 Hz, SiMe₃). IR (ATR, cm⁻¹): v = 2943 (w), 2891 (w), 1583 (w), 1472 (w), 1452 (w), 1431 (m), 1351 (w), 1283 (m), 1234 (m), 1104 (vs), 1026 (w), 960 (vs), 884 (m), 860 (m), 823 (vs),

779 (m), 735 (s), 691 (s), 657 (s), 609 (m), 533 (s), 509 (m), 480 (m). Elemental analysis calcd. (%) for $C_{50}H_{82}FeKNO_8P_2Si_4$ (1076.44 g/mol): C 55.79, H 7.68, N 2.60; found: C 55.37, H 7.62, N 2.58. μ_{eff} = 4.88 μ_B (Evans, THF-d₈ + 1% Si(CH₃)₄), $\mu_{s.o.}$ = 3.87 μ_B).



Figure S 7. ¹H NMR spectrum of K{18-crown-6}[Fe(N(SiMe₃)₂)₂(*cis*-dppee)] (**5**) in THF-d₈ (500.1 MHz).



Figure S 8. ¹H NMR spectrum of K{18-crown-6}[Fe(N(SiMe₃)₂)₂(*cis*-dppee)] (**5**) in THF-d₈ after 16 h (500.1 MHz).



Figure S 9. UV/Vis spectrum of K{18-crown-6}[Fe(N(SiMe₃)₂)₂(cis-dppee)] (5) in THF.



Figure S 10. Overlay of UV/Vis spectra of K{18-crown-6}[Fe(N(SiMe₃)₂)₂(*trans*-dppee)] (4) and K{18-crown-6}[Fe(N(SiMe₃)₂)₂(*cis*-dppee)] (5) in THF.

5. Catalytic *cis->trans* isomerization of *cis*-dppee mediated by 1

3.4 mg (0.005 mmol, 1 equiv.) K{18c6}[Fe(N(SiMe₃)₂)₂], **1**, and 20 mg (0.05 mmol, 10 equiv.) *cis*-dppee were dissolved in 0.7 mmol THF-d₈ and transferred into a Young NMR tube. The transformation was monitored via ³¹P NMR spectroscopy.



Figure S 11. Immediately taken ${}^{31}P{}^{1}H$ NMR spectrum of the reaction mixture of 5 µmol K{18-crown-6}[Fe(N(SiMe_3)_2)_2] (1) and 0.5 mmol *cis*-dppee in THF-d₈ (121.54 MHz).



Figure S 12. ³¹P{¹H} NMR spectrum of the reaction mixture of 5 μ mol K{18-crown-6}[Fe(N(SiMe₃)₂)₂] (**1**) and 0.5 mmol *cis*-dppee in THF-d₈ after 72h (121.54 MHz).



Figure S 13³¹P{¹H} NMR spectrum of the reaction mixture of 5 μ mol K{18-crown-6}[Fe(N(SiMe_3)_2)_2] (1) and 0.5 mmol *cis*-dppee in THF-d₈ after 144h (121.54 MHz).

6. X-Ray diffraction analysis and molecular structures

Data for **2** (CCDC 1858799), **3** (CCDC 1858802), **4** (CCDC 1964496), and **5** (CCDC 1964494) were collected at 100 K on a Bruker Quest D8 diffractometer using a graphite-monochromated Mo-K α radiation and equipped with an *Oxford Instrument Cooler Device*. The structures have been solved using OLEX SHELXT V2014/1^[iv] and refined by means of least-squares procedures on F^2 with the aid of the program SHELXL-2016/6^[v] included in the softwares package WinGX version 1.63^[vi] or using CRYSTALS.^[vii]

The Atomic Scattering Factors were taken from *International Tables for X-Ray Crystallography*.^[viii] All non-hydrogen atoms were refined anisotropically. All hydrogens atoms were refined by using a riding model. Absorption corrections were introduced by using MULTISCAN.^[ix] Drawings of molecules are performed with the program DIAMOND with 50% probability displacement ellipsoids for non-H atoms. Depiction of H atoms is omitted for clarity.

Table S1. Crystal data and structure refinement for **2**. The high number of restraints is necessary in order to properly model a disorder of the potassium cation and the coordinated solvent (Et_2O).

Identification code	K18c6_Fehmds3				
Empirical formula	$C_{38}H_{98}FeKN_3O_8Si_6$				
Formula weight ∕ g mol⁻¹	988.68				
Temperature / K	110.0				
Crystal system	monoclinic				
Space group	P2 ₁ /n				
<i>a</i> / Å	15.4864(6)				
<i>b</i> / Å	17.4686(7)				
c / Å	21.7327(9)				
α/°	90				
β/°	92.3350(10)				
k/°	90				
V / Å ³	5874.4(4)				
Ζ	4				
$\rho_{calc}/g \text{ cm}^{-3}$	1.118				
μ/mm^{-1}	0.491				
F(000)	2160.0				
Crystal size / mm ³	0.37 × 0.3 × 0.19				
Radiation	ΜοΚα (λ = 0.71073)				
20 range for data collection / °	4.418 to 55.142				
Index ranges	$-20 \le h \le 20, -22 \le k \le 22, -28 \le l \le 28$				
Reflections collected	160701				
Independent reflections	13562 [R _{int} = 0.0512, R _{sigma} = 0.0251]				
Data/restraints/parameters	13562/164/592				
Goodness-of-fit on F ²	1.027				
Final <i>R</i> indexes [<i>I</i> ≥2σ (<i>I</i>)]	R ₁ = 0.0367, wR ₂ = 0.0839				
Final <i>R</i> indexes [all data]	$R_1 = 0.0539$, w $R_2 = 0.0913$				
Largest diff. peak/hole / e $Å^{-3}$	0.56/-0.49				
	/				
	N2				

Figure S 14. Section of the crystal structure of 2. The K{18-crown-6} cation and H atoms are omitted.

Table S2. Crystal data and structure refinement for 3.

Identification code	K18c6_Feppbz_hmds
Empirical formula	$C_{56}H_{82.48}FeKNO_8P_2Si_2$
Formula weight∕g mol⁻¹	1110.77
Temperature/K	100.0
Crystal system	triclinic
Space group	P-1
a / Å	13.1704(6)
b / Å	15.3361(7)
<i>c</i> / Å	15.4398(7)
α/°	97.045(2)
6 / °	103.248(2)
γ/°	95.214(2)
V / Å ³	2989.7(2)
Ζ	2
$\rho_{calc}/g\ cm^{-3}$	1.234
μ / mm ⁻¹	0.465
F(000)	1185.0
Crystal size / mm ³	$0.446 \times 0.186 \times 0.126$
Radiation	ΜοΚα (λ = 0.71073)
2Θ range for data collection / °	4.432 to 53.614
Index ranges	$-16 \le h \le 16, -18 \le k \le 19, -19 \le l \le 19$
Reflections collected	93235
Independent reflections	12781 [$R_{int} = 0.0451$, $R_{sigma} = 0.0265$]
Data/restraints/parameters	12781/0/674
Goodness-of-fit on F ²	1.024
Final <i>R</i> indexes [/≥2σ (/)]	$R_1 = 0.0336$, w $R_2 = 0.0712$
Final R indexes [all data]	$R_1 = 0.0453$, w $R_2 = 0.0754$
Largest diff. peak/hole / e Å ⁻³	0.56/-0.43





Table S3. Crystal data and structure refinement for **4**. The high number of restraints stems from the modeling of the disorder of one of the phenyl groups.

Identification code	K18c6_Fehmds2_trans_dppe
Empirical formula	$C_{58}H_{98}FeKN_2O_8P_2Si_4$
Formula weight / g mol⁻¹	1220.63
Temperature/K	100.0
Crystal system	monoclinic
Space group	P21/c
a / Å	12.5633(5)
b / Å	35.5283(14)
<i>c</i> / Å	15.3551(7)
α/°	90
6 / °	99.326(2)
v/°	90
<i>V</i> / Å ³	6763.2(5)
Z	4
ρ_{calc} / g cm ⁻³	1.199
μ / mm ⁻¹	0.451
F(000)	2620.0
Crystal size / mm ³	0.333 × 0.287 × 0.12
Radiation	ΜοΚα (λ = 0.71073)
20 range for data collection / $^{\circ}$	4.366 to 53.954
Index ranges	-16 ≤ h ≤ 16, -45 ≤ k ≤ 45, -19 ≤ l ≤ 19
Reflections collected	158557
Independent reflections	14674 [R _{int} = 0.0413, R _{sigma} = 0.0222]
Data/restraints/parameters	14674/231/751
Goodness-of-fit on F ²	1.064
Final <i>R</i> indexes [/≥2σ (/)]	R ₁ = 0.0414, wR ₂ = 0.0973
Final R indexes [all data]	R ₁ = 0.0511, wR ₂ = 0.1018
Largest diff. peak/hole / e Å $^{-3}$	1.10/-0.55



Figure S 16. Section of the crystal structure of **4**. The disorder found for the phenyl rings, the K{18crown-6} counter ion and all H atoms except the ones at C1 and C2 are omitted.

Table S4. Crystal data and structure refinement for **5**. The very high number of restraints is necessary to model the disorder of the K{18c6} cation and of the coordinated dppee ligand. The disorder of the complex 5 is shown in figure S 18.

Identification code	K18c6_Fehmds2_cis_dppe
Empirical formula	$C_{50}H_{82}FeKN_2O_6P_2Si_4$
Formula weight ∕ g mol ⁻¹	1076.42
Temperature/K	100.0
Crystal system	triclinic
Space group	P-1
<i>a</i> / Å	11.660(2)
<i>b</i> /Å	13.061(2)
c / Å	20.875(4)
α / °	73.368(5)
6 / °	74.116(6)
γ/°	75.985(5)
<i>V</i> / Å ³	2883.1(9)
Z	2
ρ_{calc} / g cm ⁻³	1.240
μ / mm ⁻¹	0.518
F(000)	1150.0
Crystal size / mm ³	0.226 × 0.135 × 0.117
Radiation	ΜοΚα (λ = 0.71073)
2Θ range for data collection / $^\circ$	4.308 to 49.998
Index ranges	$-13 \leq h \leq 13, -15 \leq k \leq 15, -24 \leq l \leq 24$
Reflections collected	61411
Independent reflections	10137 [R_{int} = 0.1130, R_{sigma} = 0.0826]
Data/restraints/parameters	10137/2529/919
Goodness-of-fit on F ²	1.120
Final <i>R</i> indexes [/≥2σ (/)]	R ₁ = 0.1185, wR ₂ = 0.2694
Final R indexes [all data]	R ₁ = 0.1565, wR ₂ = 0.2835
Largest diff. peak/hole / e Å ⁻³	1.48/-0.92



Figure S 17. Section of the crystal structure of **5**. K{18-crown-6} counter ion, disordered dppee ligand and all H atoms except the ones at C1 and C2 are omitted.



Figure S 18. Depiction of the disorder found for of **5**. All H atoms are omitted. The disordered *cis*-dppee ligand with 50% occupation with similar bond metrics is rotated by approx. 180° around the iron-ethylene axis. The crown ether of the K{18c6} cation is also disordered and rotated by 23° around the potassium ion.

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