Supporting Information

Synthesis of a Stable 1,2-Bis(ferrocenyl)diphosphene

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General procedure

Unless otherwise stated, all experiments were performed under an atmosphere of dry argon. Prior to use, all solvents were dried and purified by standard methods and/or The Ultimate Solvent System (Glass Contour Company).[S1] Preparative column chromatography was performed on Wakogel C-200. All ^{1}H (300 MHz), ^{13}C (75 MHz) and ^{31}P (120 MHz) NMR spectra were recorded in C_6D_6 using a JEOL AL-300 spectrometer. Chemical shifts are referenced to residual C_6HD_5 (δ = 7.15 ppm for ^{1}H NMR) and C_6D_6 (δ = 128.0 ppm for ^{13}C NMR) as internal standards or 85% H_3PO_4 in water (δ = 0 ppm for ^{31}P) as an external standard. The multiplicity of the signals in the ^{13}C NMR spectra was determined by DEPT techniques. High-resolution mass spectrometry data were obtained from a JEOL JMS-700 spectrometer (FAB) or a Bruker microTOF (APPI-TOF). Electronic spectra were recorded on a JASCO V-570 UV/vis spectrometer. Raman spectra were recorded on a Raman spectrometer consisting of a Spex 1877 Triplemate and an EG&G PARC 1421 intensified photodiode array detector. An NEC GLG 108 He/Ne laser (633 nm) was used for Raman excitation. Melting points were determined on a BÜCHI Melting Point M-565. Elemental analyses were carried out in the Microanalytical Laboratory of the Institute for Chemical Research, Kyoto University. Calculations were conducted with the Gaussian 03 series[S2] of electronic structure programs. Geometries were optimized using density functional theory at the B3PW91 level, with basis sets of 6-31+G(2d) (P), 6-31G(d) (C, H) and DZVP (Fe)[S3]. Frequency calculations confirmed the optimized structures to have minimum energies. Computation time was generously provided from the Supercomputer Laboratory in the Institute for Chemical Research, Kyoto University. Ferrocenylphenylsulfoxide was prepared according to a literature procedure.[S4]

Experimental procedure

*Preparation of 1-(3,5-dimethylphenyl)-2-(phenylsulfoxide)ferrocene **2***

A solution of diisopropylamine (8.33 mL, 59.5 mmol) in THF (50 mL) was treated with *n*-BuLi (36.1 mL, 1.65 M in hexane, 59.5 mmol) before being added to a solution of ferrocenylphenylsulfoxide (12.3 g, 39.7 mmol) in THF (100 mL) at –78 °C. Stirring was continued at –78 °C for 30 min before the resulting solution was transferred to a suspension of $ZnCl_2$ (16.2 g, 119 mmol) in THF (100 mL) at –78 °C. Stirring was continued at –78 °C for 1 hour before the mixture was warmed to room temperature, where stirring was maintained for 1 hour. The resulting solution was added to a solution of 1-Iodo-3,5-dimethylbenzene (5.73 mL, 39.7 mmol) and $[Pd(PPh_3)_4]$ (2.30 g, 1.99 mmol) in THF (50 mL). The reaction mixture was heated to 60 °C for 3 hours, before the reaction was quenched with a saturated aqueous ammonium chloride solution. The reaction mixture was extracted with Et_2O and the combined organic phases were washed with water and dried over $MgSO_4$. After filtration, the filtrate was evaporated to dryness under reduced pressure and the obtained residue was purified by column chromatography on silica gel (hexane/ethyl acetate 1:1) to give **2** (14.4 g, 34.8 mmol, 88%). **2**: yellow solid; m.p. 138–139 °C; ^{1}H NMR (300 MHz, C_6D_6) δ = 2.22 (s, 6H), 3.96 (pt, $^3J_{HH}$ = 2.7 Hz, 1H), 4.05 (s, 5H), 4.14 (dd, $^3J_{HH}$ = 2.7 Hz, $^4J_{HH}$ = 1.5 Hz, 1H), 4.41 (dd, $^3J_{HH}$ = 2.7 Hz, $^4J_{HH}$ = 1.5 Hz, 1H), 6.71 (s, 1H), 6.85–6.96 (m, 3H), 7.63 (s, 2H), 7.67–7.70 (m, 2H); ^{13}C NMR (75 MHz, C_6D_6) δ = 21.4 (q), 69.0 (d), 71.5 (d), 71.5 (d), 71.8 (d), 89.7 (s), 93.9 (s), 125.6 (d), 128.2 (d), 128.3 (d), 129.1 (d), 130.0 (d), 136.3 (s), 137.3 (s), 145.3 (s); HRMS (FAB), *m/z*: 414.0743([M^+]), calcd. for $C_{24}H_{22}FeOS$: 414.0741; Anal. Calcd. for $C_{24}H_{22}FeOS$: C, 69.57; H, 5.35%. Found: C, 69.82; H, 5.37%.

*Preparation of 1-(3,5-dimethylphenyl)-2-(phenylsulfanyl)ferrocene **3***

A solution of trifluoroacetic anhydride (11.3 mL, 81.4 mmol) in acetone (50 mL) was added to a solution of compound **2** (14.4 g, 34.8 mmol) and NaI (12.2 g, 81.4 mmol) in acetone (100 mL) at 0 °C. After stirring for 30 min at 0 °C, the reaction mixture was extracted with Et_2O. The combined organic phases were washed with 10% sodium thiosulfate solution and dried over $MgSO_4$. After filtration, the filtrate was evaporated to dryness under reduced pressure and the obtained residue was purified by column chromatography on silica gel (hexane/ethyl acetate 1:1) to give **3** (12.6 g, 31.6 mmol, 91%). **3**: yellow solid; m.p. 120–121 °C; ^{1}H NMR (300 MHz, C_6D_6) δ = 2.15 (s, 6H), 4.05 (s, 5H), 4.10 (pt, $^3J_{HH}$ = 2.7 Hz 1H), 4.45 (dd, $^3J_{HH}$ = 2.7 Hz, $^4J_{HH}$ = 1.5 Hz, 1H), 4.54 (dd, $^3J_{HH}$ = 2.7 Hz, $^4J_{HH}$ = 1.5 Hz, 1H), 6.71 (s, 1H), 6.75–6.80 (m, 1H), 6.88–6.93 (m, 2H), 7.19–7.22 (m, 2H), 7.52 (s, 2H); ^{13}C NMR (75 MHz, C_6D_6) δ = 21.5 (q), 69.4 (d), 71.1 (d), 71.5 (d), 74.9 (d), 77.2 (s), 91.0 (s), 125.0 (d), 126.2 (d), 127.2 (d), 128.8 (d), 129.0 (d), 137.2 (s), 137.4 (s), 141.6 (s); HRMS (FAB), *m/z*: found 398.0793([M^+]), calcd. for $C_{24}H_{22}FeS$: 398.0792. Anal. Calcd. for $C_{24}H_{22}FeS$: C, 72.36; H, 5.57%. Found: C, 72.60; H, 5.57%.

*Preparation of 1-(3,5-dimethylphenyl)-2-(phenylsuloxide)ferrocene **4***

A solution of compound **3** (12.6 g, 31.6 mmol) in CH_2Cl_2 (100 mL) was cooled to 0 °C and a solution of *m*-CPBA (5.71 g, 33.1 mmol) in CH_2Cl_2 (150 mL) was added drop-wise. The reaction mixture was stirred at 0 °C for 3 hours before being extracted with CH_2Cl_2. The combined organic phases were consecutively washed with aqueous NaOH solution and water. After drying over $MgSO_4$, filtration and solvent evaporation under reduced pressure, the resulting residue was purified by column chromatography on silica gel (hexane/ethyl acetate 1:1) to give **4** (11.8 g, 28.5 mmol, 90%). **4**: yellow solid; m.p. 138–139 °C; ^{1}H NMR (300 MHz, C_6D_6) δ = 2.17 (s, 6H), 4.00 (pt, $^3J_{HH}$ = 2.7 Hz, , 1H), 4.29 (s, 5H), 4.35 (dd, $^3J_{HH}$ = 2.7 Hz, $^4J_{HH}$ = 1.5 Hz, 1H), 4.78 (dd, $^3J_{HH}$ =

2.7 Hz, $^4J_{HH}$ = 1.5 Hz, 1H), 6.75 (s, 1H), 6.80–6.89 (m, 3H), 7.43 (s, 2H), 7.56–7.59 (m, 2H); ^{13}C NMR (75 MHz, C_6D_6) δ = 21.4 (q), 65.7 (d), 69.1 (d), 70.0 (d), 71.8 (d), 88.9 (s), 95.6 (s), 124.3 (d), 127.5 (d), 128.9 (d), 129.3 (d), 130.0 (d), 137.0 (s), 138.0 (s), 148.7 (s); HRMS (FAB), *m/z*: found 414.0738([M$^+$]), calcd. for $C_{24}H_{22}FeOS$: 414.0741. Anal. Calcd. for $C_{24}H_{22}FeS$: C, 69.57; H, 5.35%. Found: C, 69.55; H, 5.34%.

Preparation of 1,3-bis(3,5-dimethylphenyl)ferrocenylphenylsulfoxide **5**

Compound **4** (11.8 g, 28.5 mmol) was dissolved in THF (10 mL) and cooled to –78 °C. To this solution a freshly prepared solution of diisopropylamine (6.24 mL, 44.6 mmol) and *n*-BuLi (27.0 mL, 1.65 M, 44.6 mmol) in THF (50 mL), was added. After stirring at –78 °C for 30 min, the resulting solution was added to a suspension of Zinc chloride (12.1 g, 89.1 mmol) in THF (100 mL) at –78 °C. The reaction mixture was stirred at –78 °C for 1 hour and warmed to room temperature, where stirring was continued for 1 hour. The resulting solution was added to the solution of 1-Iodo-3,5-dimethylbenzene (4.29 mL, 29.7 mmol) and [$Pd(PPh_3)_4$] (1.72 g, 1.49 mmol) in THF (50 mL). The resulting solution was heated to 60 °C for 3 hours, before the reaction was quenched by addition of saturated aqueous ammonium chloride. The reaction mixture was extracted with Et_2O and the combined organic phases were washed with water and dried over $MgSO_4$. After filtration and evaporation of all volatiles under reduced pressure, the obtained residue was purified by column chromatography on silica gel (hexane/ethyl acetate 1:1) to give **5** (9.72 g, 23.5 mmol, 82%). **5**: yellow solid; m.p. 121–122 °C; ^{1}H NMR (300 MHz, C_6D_6) δ = 2.17 (s, 6H), 2.21 (s, 6H), 4.40 (s, 5H), 4.52 (d, $^3J_{HH}$ = 2.7 Hz, 1H), 4.56 (d, $^3J_{HH}$ = 2.7 Hz, 1H), 6.62 (s, 1H), 6.63–6.73 (m, 3H), 6.80 (s, 1H), 7.32–7.34 (m, 2H), 7.54 (s, 2H), 7.59 (s, 2H); ^{13}C NMR (75 MHz, C_6D_6) δ = 21.3 (q), 21.4 (q), 69.4 (d), 69.8 (d), 73.3 (d), 90.0 (s), 94.3 (s), 94.5 (s), 127.7 (d), 128.3 (d), 128.6 (d), 128.8 (d), 129.03 (d), 129.05 (d), 129.4 (d), 136.5 (s), 136.5 (s), 136.7 (s), 137.7 (s), 145.7 (s); HRMS (FAB), *m/z*: found 518.1363 ([M+]), calcd. for $C_{24}H_{22}FeS$: 518.1367. Anal. Calcd. for $C_{32}H_{30}FeOS$: C, 74.13; H, 5.83%. Found: C, 74.99; H, 5.76%.

Preparation of 1,3-bis(3,5-dimethylphenyl)-2-iodoferrocene **6**

A solution of compound **5** (9.72 g, 23.5 mmol) in Et_2O (100 mL) was treated with *t*–BuLi in pentane (34.4 mL, 1.58 M, 54.3 mmol) at –78 °C. After 5 min, iodine (15.6 g, 0.124 mol) was added to the reaction mixture, which was allowed to warm to room temperature. After 3 hours, the reaction mixture was extracted with Et_2O. The combined organic phases were washed with 10% sodium thiosulfate solution and dried over $MgSO_4$. After filtration and evaporation of all volatiles under reduced pressure, the obtained residue was purified by column chromatography on silica gel (hexane/ethyl acetate 1:1). The obtained solid was precipitated from Et_2O to afford iodide **6** (8.56 g, 16.5 mmol, 70%). **6**: brown solid; m.p. 149–150 °C; ^{1}H NMR (300 MHz, C_6D_6) δ = 2.26 (s, 12H), 4.03 (s, 5H), 4.50 (s, 2H), 6.82 (s, 2H), 7.53 (s, 4H); ^{13}C NMR (75 MHz, CDCl$_3$) δ = 21.6 (q), 48.6 (s), 68.7 (d), 73.9 (d), 90.8 (s), 128.6 (d), 129.0 (d), 137.2 (s), 137.5 (s); HRMS (FAB), *m/z*: found 520.0354 ([M+]), calcd. for $C_{26}H_{25}FeI$: 520.0351; Anal. Calcd. for $C_{28}H_{31}FeI$: C, 60.03; H, 4.84%. Found: C, 59.95; H, 4.83%.

Preparation of 1,3-bis(3,5-dimethylphenyl)ferrocene-2-yl-dichorophosphine 7

Compound **6** (1.32 g, 2.54 mmol) was dissolved in Et$_2$O (10 mL) and cooled to –78 °C before *n*–BuLi (1.85 mL, 1.65 M, 3.04 mmol) was added drop-wise to the solution. The reaction mixture was stirred at room temperature for 30 min and then cooled to –78 °C. Neat PCl$_3$ (0.665 mL, 7.62 mmol) was added to the reaction mixture and stirring was continued for 1 hour. All volatiles were removed under reduced pressure, the resulting residue was extracted with hexane (50 mL) and all insoluble inorganic salts were removed by filtration through a pad of Celite. After evaporation of the filtrate to dryness, the ensuing residue was taken up in Et$_2$O (ca. 5 mL), filtered over Celite before all volatiles were removed to afford ferrocenyldicholophospine **7** (921 mg, 1.84 mmol, 72%). **7**: brown solid; m.p. 138–139 °C; ^{1}H NMR (300 MHz, C$_6$D$_6$) δ = 2.26 (s, 12H), 4.05 (s, 5H), 4.53 (s, 2H), 6.80 (s, 2H), 7.51 (s, 4H); ^{31}P NMR (120 MHz, C$_6$D$_6$) δ = 155 (s); HRMS (FAB), *m/z*: found 494.0420 ([M$^+$]), calcd. for $C_{26}H_{25}Cl_2FeP$: 494.0421; Anal. Calcd. for $C_{26}H_{25}Cl_2FeP$: C, 63.06; H, 5.09%. Found: C, 63.06; H, 5.14%.

*Preparation of 1,3-bis(3,5-dimethylphenyl)ferrocene-2-yl-phosphine **8***

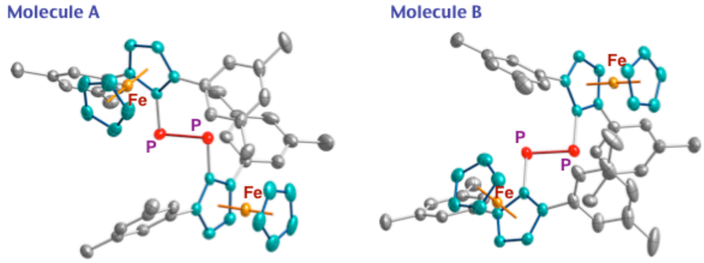
A solution of **7** (412 mg, 0.832 mmol) in THF (10 mL) was treated with a solution of LiAlH$_4$ (316 mg, 8.32 mmol) in THF (10 mL) and stirred for 5 h. The reaction mixture was quenched with MeOH and was all volatiles were removed under reduced pressure. The ensuing residue was extracted with hexane (30 mL) and filtered through a pad of Celite. After removal of all volatiles from the filtrate, the ensuing residue was taken up in Et$_2$O (ca. 5 mL) and filtered over Celite before all volatiles were removed to afford ferrocenylphosphine **8** (328 mg, 0.769 mmol, 92%). **8**: orange crystals; m.p. 136–137 °C; ^{1}H NMR (300 MHz, C$_6$D$_6$) δ = 2.25 (s, 12H), 4.00 (s, 5H), 4.07 (d, 2H, $^1J_{PH}$ = 198 Hz), 4.56 (s, 2H), 6.81 (s, 2H), 7.43 (s, 4H); ^{13}C{^{1}H} NMR (75 MHz, C$_6$D$_6$) δ = 21.4 ($\underline{C}H_3$), 30.2 (brs, β-Cp), 71.7 (d, J_{PC} = 1.5 Hz), 72.7 (Cp), 94.7 (brs, α-Cp), 128.3 (brs, $\underline{C}$), 128.8 ($\underline{C}H$), 137.4 ($\underline{C}$), 138.7 (brs, $\underline{C}$); ^{31}P NMR (120 MHz, C$_6$D$_6$) δ = –148 (t, $^1J_{PH}$ = 198 Hz); HRMS (FAB), *m/z*: found 426.1192 ([M+]), calcd. for $C_{26}H_{27}FeP$:426.1200; Anal. Calcd. for $C_{26}H_{27}FeP$: C, 73.25; H, 6.38%. Found: C, 73.49; H, 6.45%.

*Preparation of 1,2-bis[1,3-bis(3,5-dimethylphenyl)ferrocen-2-yl]diphosphene **1***

A solution of phosphine **8** (94.7 mg, 0.222 mmol) in THF (2.0 mL) was cooled to –78 °C before *n*–BuLi (0.135 mL, 1.65 M, 0.222 mmol) was added drop-wise. Stirring was continued for 5 min at –78 °C before the reaction mixture was transferred to a solution of compound **7** (110 mg, 0.222 mmol) in THF (1.0 mL). After 5 min, a solution of DBU (0.139 mL, 1.60 M in THF, 0.222 mmol) was added. The temperature was kept at –78 °C for 1 hour, before the reaction mixture was allowed to warm to room temperature within 1 hour. A characteristic color change from red to purple was observed. All volatiles were removed under reduced pressure before the obtained residue was extracted with hexane (20 mL) and filtered through Celite. After removal of the solvent, the residue was reprecipitated from Et_2O/hexane to afford 1,2-bis(ferrocenyl)diphosphene **1** (76.1 mg, 89.7 µmol, 40%). **1**: red-purple crystals, 204–205 °C (decomp). ^1H NMR (300 MHz, C_6D_6) δ = 2.20 (s, 12H), 4.05 (s, 5H), 4.65 (s, 2H), 6.74 (s, 2H), 7.39 (s, 4H); ^{13}C NMR (75 MHz, C_6D_6) δ = 21.5 (q), 67.8 (s), 72.5 (d), 72.7 (d), 93.3 (s), 128.7 (d), 130.0 (d), 136.9 (s), 138.6 (s); ^{13}P NMR (120 MHz, C_6D_6) δ = 482 (s). FT-Raman (solid state): 621 cm^{-1} ($\nu_{P=P}$). UV/vis: λ_{max} = 351 (ε 9,000), 448 (1,400), 556 (3,000) nm (THF); λ_{max} = 349 (6,100), 446 (990), 546 (2,000) nm (hexane). HRMS (APPI TOF), *m/z*: found 849.2115 ($[M+H]^+$), calcd. for $C_{52}H_{51}Fe_2P_2$: 849.2162.

X-Ray crystallographic analysis of [1·(C$_6$H$_{14}$)$_{0.25}$].

Intensity data for **[1·(C_6H_{14})$_{0.25}$]** were collected on a RIGAKU Saturn70 CCD(system) with VariMax Mo Optic using MoKα radiation (λ = 0.71070 Å). Single crystals suitable for X-ray diffraction analysis were obtained by slow recrystallization of **1** from hexane at –40 °C. A single crystal was mounted on a glass fiber. Structures were solved by direct methods (SHELXS-97)[S5] and refined by full-matrix least-squares procedures on F^2 for all reflections (SHELXL-97)[S5]. All hydrogen atoms were placed using AFIX instructions, while all other atoms were refined anisotropically. Two crystallographically independent molecules were found per unit cell. A cif file containing all crystallographic information for **[1·(C_6H_{14})$_{0.25}$]** has been deposited at the Cambridge Crystallographic Data Centre (CCDC-879016). These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. **[1·(C_6H_{14})$_{0.25}$]**: Formula $C_{52}H_{50}Fe_2P_2$, $(C_6H_{14})_{0.25}$, M_W = 870.10; T = 103(2) K; triclinic; space group P–1 (#2); a = 14.0951(1) Å, b = 17.4920(2) Å, c = 19.8743(2) Å; Z = 4; V = 4513.86(8) Å^3; μ = 0.749 mm^{-1}; D_{calcd} = 1.280 g/cm^3; $2\theta_{max}$ = 51.0°; 38846 measured reflections, 16401 independent reflections, [R_{int} = 0.0289], 1053 refined parameters; GOF = 1.031; R_1 ($I>2\sigma(I)$) = 0.0386; wR_2 (all data) = 0.1054; largest diff. peak and hole 1.653 (around Fe atom) and −0.425 e.Å^{-3}.



	P–P/Å	P–C/Å	C–P–P/°	∠(Cp)and(CPPC)
molecule A	2.0312(8)	1.826(2), 1.815(2)	99.81(7), 101.69(7)	44.4°, 41.8°
molecule B	2.0379(8)	1.816(2), 1.824(2)	100.77(7), 101.04(7)	45.4°, 41.7°
Mes*P=PMes*[a)]	2.034(2)	1.861(2)	102.81(1)	—
FcP=PTbt[b)]	2.0285(15)	1.805(4), 1.848(3)	101.64(16), 103.15(12)	5.1°

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Figure S1. Molecular structure of **1** (ORTEP at 50% probability level) and selected structural parameters for **1** and related diphosphenes.

Cyclic voltammograms of 1.

Electrochemical experiments were carried out on an ALS 600A or 1140A potentiostat/galvanostat using Pt wire electrodes. Electrochemical samples were recorded with scan rates of 10–500 mVs^{-1} at –78 $^{\circ}$C or room temperature, respectively. Samples solutions (CH_2Cl_2 or THF) were 2.0 mM in analyte and 0.1 M in *n*-Bu_4NBF_4 as the supporting electrolyte.

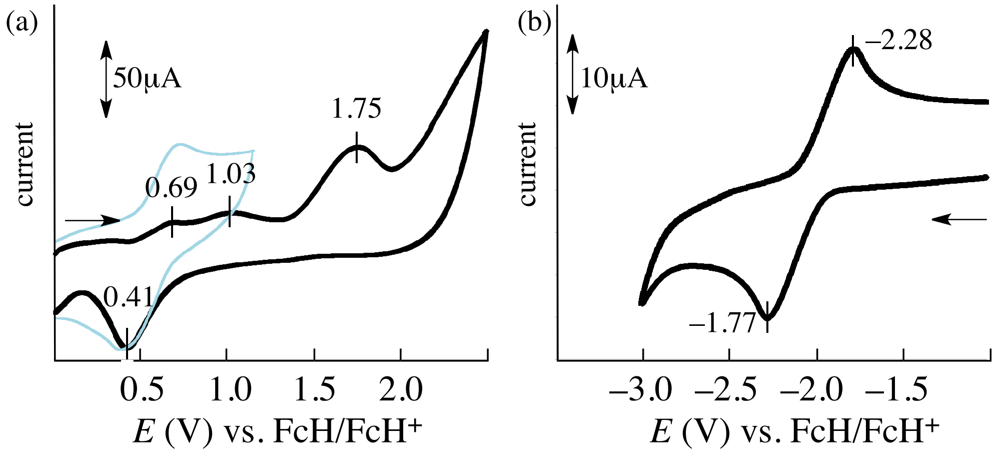

Figure S2. Cyclic voltammograms of **1** (2.0 mM in analyte, 0.1 M in *n*-Bu_4NBF_4): (a) Oxidation region, –78 °C, in CH_2Cl_2 (blue line: reversed after the first oxidation step) (b) Reduction region, r. t., THF.

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