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Table S1: Spectroscopic and analytical data for compounds 2a-b, 3a-b and 4a-b

Cmpd	IR (ν(CO), cm^{-1})[a]	$^{31}P\{^{1}H\}$ δ[b]	^{1}H (δ), J(Hz)[b]	$^{13}C\{^{1}H\}$ (δ), J(Hz)[b]	Analytical data
2a	2043 m, 1997 s, 1976 m, 1945 m, 1933 w	194.2 (s, μ-PPh_2), 17.0 (s, $P(OMe)_3$)	7.87 - 7.10 (m, C_6H_5, 10H), 3.24 (d, $^{3}J_{PH}$ = 11.4 Hz, 9H, $P(OCH_3)_3$), 2.22 (d, $^{4}J_{PH}$ = 1.4 Hz, 3H, $C{\equiv}CCH_3$)	222.7 (dd, $^{2}J_{PC}$ = 14.3 Hz, $^{2}J_{PC}$ = 10.1 Hz, $CH_3C{=}C$), 216.4 (dd, $^{2}J_{PC}$ = 9.0 Hz, $^{2}J_{PC}$ = 6.0 Hz, CO), 215.6 (dd, $^{2}J_{PC}$ = 22.0 Hz, $^{2}J_{PC}$ = 4.5 Hz, CO), 212.1 (t, $^{2}J_{PC}$ = 14.0 Hz, CO), 127-143 (m, C_6H_5), 101.5 (dd, $^{1}J_{PC}$ = 51.0 Hz, $^{2}J_{PC}$ = 7.8 Hz, $C{=}C\{P(OMe)_3\}$), 53.7 (d, $^{2}J_{PC}$ = 5.4 Hz, $P(OCH_3)_3$), 37.8 (d, $^{3}J_{PC}$ = 12.6 Hz, $C{=}CCH_3$)	Calcd: C, 45.90; H 3.53. Found: C, 46.08; H, 3.29.
2b	2040 m, 1996 s, 1975 s, 1944 m, 1930 w.	193.5 (d, $^{3}J_{PP}$ = 2.6 Hz, μ-PPh_2), 13.1 (d, $^{3}J_{PP}$ = 2.6 Hz, $P(OEt)_3$).	7.83 - 7.81 (m, C_6H_5, 2H), 7.61-7.57 (m, C_6H_5, 2H), 7.19-7.09 (m, C_6H_5, 6H), 3.48 (qAB, $^{2}J_{HH}$ = 10.0 Hz, $^{3}J_{HH}$ = 7.0 Hz, 3H, $P(OCH_2CH_3)_3$), 3.40 (qAB, $^{2}J_{HH}$ = 10.0 Hz, $^{3}J_{HH}$ = 7.0 Hz, 3H, $P(OCH_2CH_3)_3$), 2.19 (d, $^{4}J_{PH}$ = 4.8 Hz, 3H, $C{\equiv}CCH_3$), 1.14 (dt, $^{3}J_{HH}$ = 7.0 Hz, $^{4}J_{PH}$ = 0.9 Hz, 9H, $P(OCH_2CH_3)_3$)	218.3 (dd, $^{2}J_{PC}$ = 16.0 Hz, $^{2}J_{PC}$ = 11.7 Hz, $CH_3C{=}C$), 216.4 (dd, $^{2}J_{PC}$ = 9.0 Hz, $^{2}J_{PC}$ = 7.0 Hz, CO), 215.6 (dd, $^{2}J_{PC}$ = 26.0 Hz, $^{2}J_{PC}$ = 5.5 Hz, CO), 212.2 (t, $^{2}J_{PC}$ = 14.0 Hz, CO), 127-144 (m, C_6H_5), 103.4 (dd, $^{1}J_{PC}$ = 53.3 Hz, $^{2}J_{PC}$ = 7.8 Hz, $C{=}C\{P(OEt)_3\}$), 63.9 (d, $^{2}J_{PC}$ = 5.7 Hz, $P(OCH_2CH_3)_3$), 37.7 (d, $^{3}J_{PC}$ = 12.4 Hz, $C{=}CCH_3$), 15.7 (d, $^{3}J_{PC}$ = 6.7 Hz, $P(OCH_2CH_3)_3$)	Calcd: C, 48.39; H 4.21. Found: C, 48.33; H, 4.04.
3a	2069 m, 2037 s, 2005 m 1990 m, 1983 s	171.4 (d, $^{3}J_{PP}$ = 28.3 Hz, μ-PPh_2), 24.1 (d, $^{3}J_{PP}$ = 28.3 Hz, $PO(OMe)_2$)	7.51-7.19 (m, C_6H_5, 10H), 3.40 (d, $^{3}J_{PH}$ = 10.8 Hz, 6H, $O{=}P(OCH_3)_2$), 3.21 (s, 3H, $CH_3C{=}CH$), 2.14 (dd, $^{2}J_{PH}$ = 7.4 Hz, $^{3}J_{PH}$ = 9.7 Hz, 1H, $CH_3C{=}CH$)	212.0 (br, s, CO), 195.4 (dd, $^{2}J_{PC}$ = 19.1 Hz, $^{2}J_{PC}$ = 10.7 Hz, $CH_3C{=}CH$), 127-138 (m, C_6H_5), 68.8 (dd, $^{1}J_{PC}$ = 157.8 Hz, $^{2}J_{PC}$ = 14.4 Hz, $CH_3C{=}CH$), 52.2 (d, $^{2}J_{PC}$ = 6.2 Hz, $PO(OCH_3)_2$), 51.7 (d, $^{2}J_{PC}$ = 6.8 Hz, $PO(OCH_3)_2$), 41.7 (dd, $^{3}J_{PC}$ = 10.7 Hz, $^{3}J_{PC}$ = 3.1 Hz, $CH_3C{=}CH$)	Calcd: C, 45.82; H 3.68. Found: C, 45.39; H, 3.16.

Table S1 continued:

Cmpd.	IR (ν(CO), cm^{-1})[a]	$^{31}P\{^1H\}$ δ[b]	^1H (δ), J(Hz)[b]	$^{13}C\{^1H\}$ (δ), J(Hz)[b]	Analytical data
3b	2069 m, 2036 s, 2006 m, 1988 m, 1982 s	171.1 (d, $^3J_{PP}$ = 28.9 Hz, μ-PPh_2), 20.6 (d, $^3J_{PP}$ = 28.9 Hz, $P(OEt)_3$)	7.52-7.19 (m, C_6H_5, 10H), 3.83-3.48 (m, 4H, OCH_2CH_3), 3.15 (s, 3H, CH_3C≡C), 2.18 (dd, $^2J_{PH}$ = 9.8 Hz, $^3J_{PH}$ = 7.0 Hz, C*H*≡C), 1.01 (t, $^3J_{HH}$ = 7.0 Hz, 6H, OCH_2CH_3)	212.0 (br, s, CO), 195.2 (dd, $^2J_{PC}$ = 18.6 Hz, $^2J_{PC}$ = 10.3 Hz, CH_3C=CH), 128-138 (m, C_6H_5), 70.0 (dd, $^1J_{PC}$ = 157.2 Hz, $^2J_{PC}$ = 14.6 Hz, CH_3C=*C*H), 61.6 (d, $^2J_{PC}$ = 6.2 Hz, $PO(OCH_2CH_3)_2$), 60.9 (d, $^2J_{PC}$ = 6.2 Hz, $PO(OCH_2CH_3)_2$), 41.1 (dd, $^3J_{PC}$ = 10.3 Hz, $^3J_{PC}$ = 3.1 Hz, CH_3C=CH), 16.3 (d, $^3J_{PC}$ = 6.2 Hz, $PO(OCH_2CH_3)_2$), 16.0 (d, $^3J_{PC}$ = 6.2 Hz, $PO(OCH_2CH_3)_2$)	Calcd: C, 46.76; H 3.77. Found: C, 47.08; H, 3.39.
4a	2042 w, 2002 s, 1975 m 1963 m, 1942 w	177.3 (d, $^2J_{PP}$ = 141.4 Hz, μ-PPh_2), 148.8 (d, $^2J_{PP}$ = 141.4 Hz, $P(OMe)_3$)	7.0 - 7.6 (m, C_6H_5, 10H), 3.54 (d, $^3J_{PH}$ = 11.4 Hz, 9H, $P(OCH_3)_3$), 1.53 (t, $^4J_{PH}$ = 3.5 Hz, $CCCH_3$)	214.9 (t, $^2J_{PC}$ = 14.6 Hz, CO), 213.7 (dd, $^2J_{PC}$ = 31.2 Hz, $^2J_{PC}$ = 11.0 Hz, CO), 127-140 (m, C_6H_5), 103.6 (s, *C*≡CCH_3), 89.6 (d, $^2J_{PC}$ = 8.7 Hz, C≡*C*CH_3), 52.2 (d, $^2J_{PC}$ = 3.2 Hz, $P(OCH_3)_3$), 9.8 (s, C≡*C*CH_3)	Calcd: C, 46.01; H 3.69. Found: C, 46.42; H, 4.11.
4b	2042 w, 2000 s, 1977 w 1963 m, 1940 w	171.9 (d, $^2J_{PP}$ = 113.3 Hz, μ-PPh_2), 146.5 (d, $^2J_{PP}$ = 113.3 Hz, $P(OEt)_3$)	7.0 - 7.6 (m, C_6H_5, 10H), 4.10 (multiplet, 6H, $P(OCH_2CH_3)_3$), 1.51 (t, $^4J_{PH}$ = 3.4 Hz, $CCCH_3$), 1.22 (t, $^4J_{PH}$ = 11.6 Hz, $P(OCH_2CH_3)_3$)	215.2 (t, $^2J_{PC}$ = 15.0 Hz, CO), 214.0 (dd, $^2J_{PC}$ = 27.9 Hz, $^2J_{PC}$ = 7.7 Hz, CO), 127-140 (m, C_6H_5), 104.2 (s, *C*≡CCH_3), 88.9 (d, $^2J_{PC}$ = 4.7 Hz, C≡*C*CH_3), 61.1 (d, $^2J_{PC}$ = 4.1 Hz, $P(OCH_2CH_3)_3$), 16.0 (d, $^3J_{PC}$ = 6.6 Hz, $P(OCH_2CH_3)_3$), 9.8 (s, C≡*C*CH_3)	Calcd: C, 48.60; H 4.11. Found: C, 48.92; H, 4.44.

[a] In n-hexane. [b] In $CDCl_3$ (293 K)

Table S2. Crystal data, structure solution and refinement for 2a.

Identification code	sd24
Chemical formula	$C_{24}H_{22}Fe_2O_9P_2$
Formula weight	628.06
Temperature	160(2) K
Radiation and wavelength	MoKα, 0.71073 Å
Crystal system, space group	monoclinic, $P2_1/c$
Unit cell dimensions	a = 10.0143(8) Å α = 90° b = 16.3022(13) Å β = 100.709(2)° c = 16.5641(13) Å γ = 90°
Volume	2657.1(4) Å^3
Z	4
Density (calculated)	1.570 g/cm^3
Absorption coefficient μ	1.262 mm^{-1}
F(000)	1280
Reflections for cell refinement	13079 (θ range 1.77 to 28.36°)
Crystal colour	yellow
Crystal size	0.34 × 0.19 × 0.14 mm
Data collection method	Siemens SMART CCD diffractometer, ω rotation with narrow frames
θ range for data collection	1.77 to 28.38°
Index ranges	$-12 \le h \le 12$, $-20 \le k \le 20$, $-22 \le l \le 12$
Intensity decay	0%
Reflections collected	18990
Independent reflections	6079 (R_{int} = 0.0321)
Reflections with I>2σ(I)	5145
Absorption correction	semi-empirical from ψ-scans
Max. and min. transmission	0.862 and 0.762
Structure solution	direct methods
Refinement method	full-matrix least-squares on F^2
Weighting parameters a, b	0.0201, 3.5255
Data / restraints / parameters	6079 / 153 / 364
Final R indices [I>2σ(I)]	R1 = 0.0384, wR2 = 0.0753
R indices (all data)	R1 = 0.0507, wR2 = 0.0795
Goodness-of-fit on F^2	1.069
Extinction coefficient	0.00021(13)
Largest and mean shift/esd	0.001 and 0.000
Largest diff. peak and hole	1.132 and -0.960 eÅ^{-3}

Table S3. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for 2a. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	Y	z	U(eq)
Fe(1)	4962.7(4)	8274.3(2)	1833.0(2)	18.03(10)
Fe(2)	3120.0(4)	7117.7(2)	1345.4(2)	18.28(10)
C(1)	4424(3)	8442.8(18)	3598.9(17)	27.1(6)
C(2)	4046(3)	8016.6(16)	2780.2(16)	19.7(5)
C(3)	3077(3)	7455.9(16)	2529.4(16)	19.4(5)
P(1)	1847.3(8)	7178.2(5)	3079.6(5)	28.21(18)
O(1)	1130(3)	6399(2)	2702(2)	27.7(9)
C(4)	-99(3)	6087(2)	2969(2)	41.9(8)
O(2)	594(3)	7818.2(16)	2957.6(17)	31.3(7)
O(3)	2251(3)	7130.1(17)	4009.0(15)	29.9(7)
O(1A)	755(11)	6635(7)	2583(6)	30(3)
O(2A)	1458(10)	7802(4)	3711(5)	34(2)
O(3A)	2464(9)	6426(5)	3686(5)	37(2)
C(5)	823(4)	8614(2)	3317(3)	55.0(11)
C(6)	3414(4)	6577(2)	4385(2)	44.2(8)
C(7)	3727(3)	9076.3(17)	1659.6(18)	27.1(6)
O(7)	2909(2)	9580.2(13)	1569.6(16)	44.9(6)
C(8)	6427(3)	8719.5(18)	2446.8(19)	28.0(6)
O(8)	7377(2)	9005.4(16)	2833.8(15)	48.2(6)
C(9)	5514(3)	8438.0(17)	868.3(19)	26.0(6)
O(9)	5842(3)	8582.7(16)	258.3(14)	43.7(6)
C(10)	1677(3)	7776.3(17)	1084.5(18)	26.2(6)
O(10)	743(2)	8200.8(14)	953.0(15)	39.5(6)
C(11)	2417(3)	6113.6(18)	1364.3(18)	26.4(6)
O(11)	1999(2)	5457.7(13)	1340.7(15)	41.2(6)
C(12)	3460(3)	7022.2(17)	327.7(18)	25.2(6)
O(12)	3680(2)	6947.3(14)	-322.5(13)	37.3(5)
P(2)	5294.6(7)	6943.2(4)	1871.1(4)	16.91(15)
C(13)	5776(3)	6373.0(16)	2832.3(16)	20.3(5)
C(14)	5214(3)	5606.6(18)	2925.9(19)	31.7(7)
C(15)	5641(4)	5146(2)	3627(2)	42.2(9)
C(16)	6646(4)	5444(2)	4245(2)	40.2(8)
C(17)	7197(3)	6205.9(19)	4166.0(19)	33.8(7)
C(18)	6767(3)	6672.3(17)	3464.7(17)	24.2(6)
C(19)	6441(3)	6456.2(16)	1271.0(16)	19.3(5)
C(20)	7655(3)	6828.3(18)	1176.6(18)	26.5(6)
C(21)	8558(3)	6428(2)	767.6(19)	32.5(7)
C(22)	8259(3)	5653.4(19)	447.3(19)	32.2(7)
C(23)	7052(3)	5283.1(19)	528(2)	35.3(7)
C(24)	6151(3)	5678.5(18)	937.4(19)	29.3(7)

Table S4. Bond lengths (Å) and angles (°) for 2a.

Fe(1)-C(8)	1.776(3)	Fe(1)-C(7)	1.786(3)
Fe(1)-C(9)	1.805(3)	Fe(1)-C(2)	2.004(3)
Fe(1)-P(2)	2.1943(8)	Fe(1)-Fe(2)	2.6569(5)
Fe(2)-C(11)	1.784(3)	Fe(2)-C(12)	1.788(3)
Fe(2)-C(10)	1.788(3)	Fe(2)-C(3)	2.045(3)
Fe(2)-P(2)	2.2083(8)	C(1)-C(2)	1.508(4)
C(2)-C(3)	1.342(4)	C(3)-P(1)	1.723(3)
P(1)-O(3)	1.519(3)	P(1)-O(1A)	1.524(8)
P(1)-O(1)	1.534(3)	P(1)-O(2A)	1.559(6)
P(1)-O(2)	1.616(3)	P(1)-O(3A)	1.632(7)
O(1)-C(4)	1.473(4)	C(4)-O(1A)	1.463(8)
O(2)-C(5)	1.429(4)	O(3)-C(6)	1.513(4)
O(2A)-C(5)	1.558(7)	O(3A)-C(6)	1.376(7)
C(7)-O(7)	1.150(4)	C(8)-O(8)	1.144(4)
C(9)-O(9)	1.144(4)	C(10)-O(10)	1.151(3)
C(11)-O(11)	1.146(3)	C(12)-O(12)	1.145(3)
P(2)-C(13)	1.829(3)	P(2)-C(19)	1.833(3)
C(13)-C(14)	1.390(4)	C(13)-C(18)	1.391(4)
C(14)-C(15)	1.382(4)	C(15)-C(16)	1.384(5)
C(16)-C(17)	1.376(5)	C(17)-C(18)	1.388(4)
C(19)-C(24)	1.392(4)	C(19)-C(20)	1.393(4)
C(20)-C(21)	1.389(4)	C(21)-C(22)	1.381(4)
C(22)-C(23)	1.379(5)	C(23)-C(24)	1.384(4)
C(8)-Fe(1)-C(7)	105.44(13)	C(8)-Fe(1)-C(9)	95.21(13)
C(7)-Fe(1)-C(9)	93.78(13)	C(8)-Fe(1)-C(2)	94.99(12)
C(7)-Fe(1)-C(2)	82.81(12)	C(9)-Fe(1)-C(2)	169.78(12)
C(8)-Fe(1)-P(2)	106.44(10)	C(7)-Fe(1)-P(2)	145.60(9)
C(9)-Fe(1)-P(2)	95.86(9)	C(2)-Fe(1)-P(2)	81.90(8)
C(8)-Fe(1)-Fe(2)	156.44(10)	C(7)-Fe(1)-Fe(2)	92.83(9)
C(9)-Fe(1)-Fe(2)	98.35(9)	C(2)-Fe(1)-Fe(2)	72.29(8)
P(2)-Fe(1)-Fe(2)	53.12(2)	C(11)-Fe(2)-C(12)	94.68(13)
C(11)-Fe(2)-C(10)	104.56(13)	C(12)-Fe(2)-C(10)	96.40(13)
C(11)-Fe(2)-C(3)	98.73(12)	C(12)-Fe(2)-C(3)	165.78(12)
C(10)-Fe(2)-C(3)	84.82(12)	C(11)-Fe(2)-P(2)	103.79(10)
C(12)-Fe(2)-P(2)	90.79(9)	C(10)-Fe(2)-P(2)	150.03(9)
C(3)-Fe(2)-P(2)	81.47(8)	C(11)-Fe(2)-Fe(1)	154.12(10)
C(12)-Fe(2)-Fe(1)	96.03(9)	C(10)-Fe(2)-Fe(1)	97.57(9)
C(3)-Fe(2)-Fe(1)	69.79(8)	P(2)-Fe(2)-Fe(1)	52.64(2)
C(3)-C(2)-C(1)	129.7(3)	C(3)-C(2)-Fe(1)	108.10(19)
C(1)-C(2)-Fe(1)	122.2(2)	C(2)-C(3)-P(1)	124.2(2)
C(2)-C(3)-Fe(2)	109.72(19)	P(1)-C(3)-Fe(2)	125.51(15)
O(3)-P(1)-O(1)	112.71(18)	O(1A)-P(1)-O(2A)	119.8(6)
O(3)-P(1)-O(2)	102.37(14)	O(1)-P(1)-O(2)	100.93(16)
O(1A)-P(1)-O(3A)	92.7(5)	O(2A)-P(1)-O(3A)	101.0(4)
O(3)-P(1)-C(3)	117.90(14)	O(1A)-P(1)-C(3)	112.3(4)
O(1)-P(1)-C(3)	109.21(17)	O(2A)-P(1)-C(3)	118.4(4)
O(2)-P(1)-C(3)	112.27(13)	O(3A)-P(1)-C(3)	107.3(4)
C(4)-O(1)-P(1)	121.2(3)	C(5)-O(2)-P(1)	118.3(2)
C(6)-O(3)-P(1)	119.1(2)	C(4)-O(1A)-P(1)	122.5(7)
C(5)-O(2A)-P(1)	114.1(5)	C(6)-O(3A)-P(1)	120.4(6)
O(7)-C(7)-Fe(1)	177.5(3)	O(8)-C(8)-Fe(1)	179.1(3)
O(9)-C(9)-Fe(1)	176.5(3)	O(10)-C(10)-Fe(2)	176.9(3)
O(11)-C(11)-Fe(2)	176.2(3)	O(12)-C(12)-Fe(2)	178.9(3)
C(13)-P(2)-C(19)	99.30(12)	C(13)-P(2)-Fe(1)	122.79(9)
C(19)-P(2)-Fe(1)	121.30(9)	C(13)-P(2)-Fe(2)	119.31(9)
C(19)-P(2)-Fe(2)	120.86(9)	Fe(1)-P(2)-Fe(2)	74.24(2)
C(14)-C(13)-C(18)	118.5(3)	C(14)-C(13)-P(2)	120.5(2)

C(18)-C(13)-P(2)	120.9(2)	C(15)-C(14)-C(13)	120.9(3)
C(14)-C(15)-C(16)	120.0(3)	C(17)-C(16)-C(15)	119.7(3)
C(16)-C(17)-C(18)	120.4(3)	C(17)-C(18)-C(13)	120.4(3)
C(24)-C(19)-C(20)	118.3(3)	C(24)-C(19)-P(2)	120.3(2)
C(20)-C(19)-P(2)	121.3(2)	C(21)-C(20)-C(19)	120.7(3)
C(22)-C(21)-C(20)	120.2(3)	C(23)-C(22)-C(21)	119.6(3)
C(22)-C(23)-C(24)	120.4(3)	C(23)-C(24)-C(19)	120.8(3)

Table S5. Anisotropic displacement parameters ($Å^2 \times 10^3$) for 2a.

The anisotropic displacement factor exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12})$.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
Fe(1)	18.8(2)	16.95(18)	18.6(2)	-0.89(15)	4.25(15)	-0.42(15)
Fe(2)	17.15(19)	19.71(19)	17.8(2)	-2.51(15)	2.79(14)	-0.08(15)
C(1)	29.3(16)	30.3(15)	22.6(15)	-8.3(12)	7.6(12)	-3.3(12)
C(2)	19.2(13)	21.5(13)	18.9(13)	-1.4(10)	4.8(10)	5.1(10)
C(3)	19.8(13)	22.3(13)	17.1(13)	0.9(10)	6.1(10)	3.5(11)
P(1)	28.2(4)	31.4(4)	28.9(4)	-6.3(3)	15.1(3)	-6.3(3)
O(1)	20.2(17)	34.9(17)	28.9(17)	0.1(13)	7.0(13)	-5.6(14)
C(4)	27.1(17)	53(2)	47(2)	8.0(17)	10.4(15)	-14.0(15)
O(2)	23.5(14)	33.6(14)	38.9(17)	0.1(12)	10.9(12)	4.2(11)
O(3)	28.4(14)	42.5(16)	20.5(13)	2.2(11)	8.9(11)	-0.1(12)
O(1A)	10(5)	57(8)	27(6)	-1(4)	10(4)	-6(5)
O(2A)	32(5)	45(5)	31(5)	-6(3)	26(4)	2(4)
O(3A)	38(5)	38(5)	39(5)	-2(4)	20(3)	-4(4)
C(5)	41(2)	38.7(18)	89(3)	-12.5(19)	22(2)	6.6(16)
C(6)	53(2)	45(2)	38.5(19)	14.3(16)	18.1(15)	5.1(16)
C(7)	30.2(16)	21.9(14)	29.0(16)	-1.9(12)	4.7(12)	-4.9(12)
O(7)	36.6(14)	27.8(12)	68.2(18)	1.7(11)	4.0(12)	11.7(10)
C(8)	27.4(16)	28.0(15)	29.9(16)	-2.4(13)	9.0(13)	-2.0(12)
O(8)	32.9(13)	61.3(16)	46.5(15)	-11.9(13)	-2.4(11)	-17.0(12)
C(9)	23.4(15)	26.0(14)	28.5(16)	0.5(12)	4.8(12)	1.1(11)
O(9)	49.4(15)	56.2(15)	30.1(13)	9.2(11)	19.2(11)	-0.8(12)
C(10)	26.4(15)	26.9(14)	23.8(15)	-6.6(12)	1.0(12)	-3.0(12)
O(10)	28.3(12)	40.7(13)	45.0(14)	-7.2(11)	-4.9(10)	12.9(10)
C(11)	22.8(14)	29.4(15)	28.0(16)	-6.1(12)	7.1(12)	0.3(12)
O(11)	47.8(15)	26.6(12)	51.5(15)	-10.3(10)	15.4(12)	-10.6(10)
C(12)	26.1(15)	23.4(14)	24.6(15)	-1.4(12)	0.7(12)	2.9(12)
O(12)	49.4(14)	42.4(13)	20.6(11)	-3.4(10)	8.0(10)	8.5(11)
P(2)	17.4(3)	17.2(3)	16.6(3)	-0.6(3)	4.3(3)	0.6(2)
C(13)	19.8(13)	22.2(13)	20.1(14)	0.5(11)	7.0(11)	3.6(11)
C(14)	37.7(18)	26.8(15)	28.3(16)	3.2(13)	-0.1(13)	-4.3(13)
C(15)	58(2)	28.4(16)	37.8(19)	10.6(14)	2.2(17)	-9.3(16)
C(16)	54(2)	36.1(18)	28.0(17)	14.4(14)	0.6(15)	4.0(16)
C(17)	38.0(18)	36.0(17)	24.1(16)	1.7(13)	-3.0(13)	3.0(14)
C(18)	26.5(15)	25.0(14)	20.7(14)	0.9(11)	3.2(11)	0.5(12)
C(19)	20.1(13)	20.7(12)	16.8(13)	1.6(10)	2.6(10)	4.0(10)
C(20)	24.0(15)	28.1(14)	28.5(16)	-4.6(12)	7.1(12)	-3.2(12)
C(21)	23.8(15)	43.6(18)	32.7(17)	-2.3(14)	11.9(13)	-1.1(14)
C(22)	33.7(17)	37.2(17)	28.0(16)	-0.2(13)	11.7(13)	12.4(14)
C(23)	46(2)	24.4(15)	39.3(19)	-7.9(13)	16.6(15)	2.2(14)
C(24)	29.3(16)	25.4(14)	35.5(17)	-4.5(13)	11.8(13)	-2.8(12)

Table S6. Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 2a.

	x	Y	z	U
H(1A)	5343	8278	3863	32
H(1B)	3778	8290	3950	32
H(1C)	4398	9038	3515	32
H(4A)	-404	5584	2668	63
H(4B)	-820	6501	2859	63
H(4C)	107	5970	3560	63
H(4XA)	-726	5791	2543	63
H(4XB)	-620	6411	3302	63
H(4XC)	476	5693	3322	63
H(5A)	-18	8935	3194	82
H(5B)	1538	8893	3091	82
H(5C)	1105	8561	3914	82
H(5XA)	599	8973	3748	82
H(5XB)	-6	8490	2920	82
H(5XC)	1476	8891	3037	82
H(6A)	3548	6611	4985	66
H(6B)	4246	6752	4202	66
H(6C)	3202	6010	4210	66
H(6XA)	3672	6059	4672	66
H(6XB)	3027	6948	4746	66
H(6XC)	4220	6831	4233	66
H(14)	4528	5397	2502	38
H(15)	5245	4625	3684	51
H(16)	6953	5124	4723	48
H(17)	7878	6414	4594	41
H(18)	7151	7199	3417	29
H(20)	7868	7361	1395	32
H(21)	9383	6688	708	39
H(22)	8881	5377	173	39
H(23)	6838	4753	301	42
H(24)	5325	5416	991	35

Table S7. Crystal data, structure solution and refinement for 3a.

Identification code	sd26
Chemical formula	$C_{23}H_{20}Fe_2O_9P_2$
Formula weight	614.03
Temperature	160(2) K
Radiation and wavelength	MoKα, 0.71073 Å
Crystal system, space group	monoclinic, $P2_1/n$
Unit cell dimensions	a = 11.859(4) Å α = 90°
	b = 11.105(4) Å β = 104.74(4)°
	c = 19.825(6) Å γ = 90°
Volume	2524.9(15) Å^3
Z	4
Density (calculated)	1.615 g/cm^3
Absorption coefficient μ	1.326 mm^{-1}
F(000)	1248
Reflections for cell refinement	53 (θ range 10.47 to 12.46°)
Crystal colour	orange
Crystal size	0.34 × 0.29 × 0.15 mm
Data collection method	Stoe-Siemens diffractometer,
	ω/θ scans with on-line profile fitting
θ range for data collection	2.55 to 25.00°
Index ranges	$-14 \le h \le 14$, $-13 \le k \le 13$, $-23 \le l \le 23$
Standard reflections	5 every 60 minutes
Intensity decay	0%
Reflections collected	6974
Independent reflections	4433 (R_{int} = 0.0316)
Reflections with I>2σ(I)	3549
Absorption correction	semi-empirical from ψ-scans
Max. and min. transmission	0.905 and 0.744
Structure solution	direct methods
Refinement method	full-matrix least-squares on F^2
Weighting parameters a, b	0.0391, 1.4310
Data / restraints / parameters	4429 / 0 / 331
Final R indices [I>2σ(I)]	R1 = 0.0362, wR2 = 0.0837
R indices (all data)	R1 = 0.0517, wR2 = 0.0947
Goodness-of-fit on F^2	1.056
Largest and mean shift/esd	-0.003 and 0.000
Largest diff. peak and hole	0.385 and -0.388 eÅ^{-3}

Table S8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for 3a. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Fe(1)	-1555.2(4)	7685.4(4)	1832.5(2)	18.05(13)
Fe(2)	32.8(4)	8444.6(4)	1254.4(2)	18.50(13)
C(1)	1054(3)	6703(3)	2451(2)	26.4(8)
C(2)	57(3)	6954(3)	1815(2)	18.3(7)
C(3)	-793(3)	6047(3)	1585(2)	20.0(7)
P(1)	-880.8(8)	4674.2(8)	2037.3(5)	21.4(2)
O(1)	-845(2)	4730(2)	2780.9(12)	28.7(6)
O(2)	146(2)	3924(2)	1856.7(14)	32.5(6)
C(4)	469(4)	2765(3)	2182(2)	42.8(11)
O(3)	-2050(2)	4042(2)	1621.9(12)	28.9(6)
C(5)	-2198(4)	3617(4)	914(2)	40.9(10)
C(6)	-1136(3)	7431(3)	2767(2)	26.2(8)
O(6)	-888(3)	7341(3)	3357.4(13)	40.5(7)
C(7)	-3045(3)	7189(3)	1696(2)	23.2(8)
O(7)	-3983(2)	6865(2)	1591.9(14)	35.0(6)
C(8)	-1581(3)	9265(3)	2015(2)	23.0(8)
O(8)	-1565(2)	10253(2)	2174.4(13)	34.2(6)
C(9)	1175(3)	9107(3)	1930(2)	28.2(8)
O(9)	1883(3)	9604(3)	2325.3(15)	49.3(8)
C(10)	881(3)	7727(3)	746(2)	25.1(8)
O(10)	1442(2)	7233(2)	448.4(14)	38.1(7)
C(11)	-167(3)	9926(3)	868(2)	23.5(8)
O(11)	-231(2)	10881(2)	645.2(13)	34.1(6)
P(2)	-1809.5(7)	8049.2(8)	686.7(4)	18.5(2)
C(12)	-2324(3)	6854(3)	43(2)	21.7(7)
C(13)	-3491(3)	6486(3)	-87(2)	29.2(8)
C(14)	-3935(4)	5638(4)	-600(2)	34.8(9)
C(15)	-3232(4)	5167(3)	-996(2)	36.6(10)
C(16)	-2088(4)	5536(4)	-881(2)	36.6(10)
C(17)	-1627(3)	6375(3)	-356(2)	27.9(8)
C(18)	-2701(3)	9290(3)	242(2)	20.6(7)
C(19)	-3333(3)	10054(3)	567(2)	23.0(7)
C(20)	-3958(3)	11003(3)	198(2)	30.4(9)
C(21)	-3979(3)	11196(3)	-491(2)	30.6(9)
C(22)	-3376(3)	10430(3)	-825(2)	29.9(8)
C(23)	-2751(3)	9481(3)	-464(2)	26.9(8)

Table S9. Bond lengths (Å) and angles (°) for 3a.

Fe(1)-C(8)	1.793(4)	Fe(1)-C(7)	1.804(4)
Fe(1)-C(6)	1.814(4)	Fe(1)-C(2)	2.086(3)
Fe(1)-C(3)	2.143(3)	Fe(1)-P(2)	2.2508(12)
Fe(1)-Fe(2)	2.5826(10)	Fe(2)-C(10)	1.783(4)
Fe(2)-C(9)	1.802(4)	Fe(2)-C(11)	1.805(4)
Fe(2)-C(2)	1.989(3)	Fe(2)-P(2)	2.2320(14)
C(1)-C(2)	1.519(5)	C(2)-C(3)	1.416(5)
C(3)-P(1)	1.785(3)	P(1)-O(1)	1.465(2)
P(1)-O(3)	1.585(3)	P(1)-O(2)	1.589(3)
O(2)-C(4)	1.447(4)	O(3)-C(5)	1.448(4)
C(6)-O(6)	1.136(4)	C(7)-O(7)	1.137(4)
C(8)-O(8)	1.141(4)	C(9)-O(9)	1.135(4)
C(10)-O(10)	1.137(4)	C(11)-O(11)	1.144(4)
P(2)-C(18)	1.822(3)	P(2)-C(12)	1.835(3)
C(12)-C(17)	1.388(5)	C(12)-C(13)	1.402(5)
C(13)-C(14)	1.389(5)	C(14)-C(15)	1.384(6)
C(15)-C(16)	1.380(6)	C(16)-C(17)	1.400(5)
C(18)-C(19)	1.394(5)	C(18)-C(23)	1.402(5)
C(19)-C(20)	1.385(5)	C(20)-C(21)	1.378(5)
C(21)-C(22)	1.382(5)	C(22)-C(23)	1.380(5)
C(8)-Fe(1)-C(7)	105.2(2)	C(8)-Fe(1)-C(6)	87.7(2)
C(7)-Fe(1)-C(6)	96.3(2)	C(8)-Fe(1)-C(2)	116.57(14)
C(7)-Fe(1)-C(2)	138.17(14)	C(6)-Fe(1)-C(2)	86.72(14)
C(8)-Fe(1)-C(3)	155.06(14)	C(7)-Fe(1)-C(3)	99.20(14)
C(6)-Fe(1)-C(3)	94.57(14)	C(2)-Fe(1)-C(3)	39.09(13)
C(8)-Fe(1)-P(2)	91.36(11)	C(7)-Fe(1)-P(2)	91.63(11)
C(6)-Fe(1)-P(2)	172.00(12)	C(2)-Fe(1)-P(2)	86.60(10)
C(3)-Fe(1)-P(2)	82.96(9)	C(8)-Fe(1)-Fe(2)	79.77(11)
C(7)-Fe(1)-Fe(2)	146.08(11)	C(6)-Fe(1)-Fe(2)	117.57(12)
C(2)-Fe(1)-Fe(2)	49.04(9)	C(3)-Fe(1)-Fe(2)	77.18(9)
P(2)-Fe(1)-Fe(2)	54.48(4)	C(10)-Fe(2)-C(9)	100.2(2)
C(10)-Fe(2)-C(11)	101.48(15)	C(9)-Fe(2)-C(11)	86.4(2)
C(10)-Fe(2)-C(2)	90.79(14)	C(9)-Fe(2)-C(2)	91.32(15)
C(11)-Fe(2)-C(2)	167.73(14)	C(10)-Fe(2)-P(2)	104.33(12)
C(9)-Fe(2)-P(2)	155.42(12)	C(11)-Fe(2)-P(2)	87.68(12)
C(2)-Fe(2)-P(2)	89.48(10)	C(10)-Fe(2)-Fe(1)	133.23(11)
C(9)-Fe(2)-Fe(1)	107.35(12)	C(11)-Fe(2)-Fe(1)	117.03(11)
C(2)-Fe(2)-Fe(1)	52.35(9)	P(2)-Fe(2)-Fe(1)	55.16(3)
C(3)-C(2)-C(1)	118.5(3)	C(3)-C(2)-Fe(2)	120.3(2)
C(1)-C(2)-Fe(2)	120.9(2)	C(3)-C(2)-Fe(1)	72.6(2)
C(1)-C(2)-Fe(1)	125.5(2)	Fe(2)-C(2)-Fe(1)	78.62(12)
C(2)-C(3)-P(1)	125.0(3)	C(2)-C(3)-Fe(1)	68.3(2)
P(1)-C(3)-Fe(1)	121.9(2)	O(1)-P(1)-O(3)	109.82(14)
O(1)-P(1)-O(2)	114.9(2)	O(3)-P(1)-O(2)	105.64(14)
O(1)-P(1)-C(3)	118.6(2)	O(3)-P(1)-C(3)	105.8(2)
O(2)-P(1)-C(3)	100.88(14)	C(4)-O(2)-P(1)	119.8(2)
C(5)-O(3)-P(1)	121.5(2)	O(6)-C(6)-Fe(1)	176.0(3)
O(7)-C(7)-Fe(1)	178.1(3)	O(8)-C(8)-Fe(1)	175.7(3)
O(9)-C(9)-Fe(2)	174.6(3)	O(10)-C(10)-Fe(2)	176.7(3)
O(11)-C(11)-Fe(2)	176.1(3)	C(18)-P(2)-C(12)	99.3(2)
C(18)-P(2)-Fe(2)	117.92(12)	C(12)-P(2)-Fe(2)	127.06(12)
C(18)-P(2)-Fe(1)	122.45(11)	C(12)-P(2)-Fe(1)	120.48(11)
Fe(2)-P(2)-Fe(1)	70.35(5)	C(17)-C(12)-C(13)	119.1(3)
C(17)-C(12)-P(2)	122.2(3)	C(13)-C(12)-P(2)	118.5(3)
C(14)-C(13)-C(12)	120.3(3)	C(15)-C(14)-C(13)	120.1(4)
C(16)-C(15)-C(14)	120.2(4)	C(15)-C(16)-C(17)	120.2(4)
C(12)-C(17)-C(16)	120.1(3)	C(19)-C(18)-C(23)	118.6(3)

C(19)-C(18)-P(2)	123.4(3)	C(23)-C(18)-P(2)	118.0(3)
C(20)-C(19)-C(18)	119.8(3)	C(21)-C(20)-C(19)	120.9(3)
C(20)-C(21)-C(22)	119.9(3)	C(23)-C(22)-C(21)	119.8(3)
C(22)-C(23)-C(18)	120.9(3)		

Table S10. Anisotropic displacement parameters ($Å^2 \times 10^3$) for 3a.

The anisotropic displacement factor exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12})$.

	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
Fe(1)	18.8(3)	20.0(3)	14.8(2)	1.1(2)	3.2(2)	1.0(2)
Fe(2)	19.1(3)	18.3(2)	17.7(2)	0.9(2)	3.8(2)	-1.2(2)
C(1)	21(2)	26(2)	28(2)	8(2)	0(2)	2(2)
C(2)	18(2)	18(2)	19(2)	2.6(13)	5.8(14)	1.9(14)
C(3)	24(2)	20(2)	15(2)	3.0(14)	5.6(14)	1.7(14)
P(1)	22.5(5)	18.7(4)	24.3(5)	2.4(4)	8.1(4)	-0.7(4)
O(1)	33.5(14)	27.9(13)	24.0(13)	5.3(11)	6.3(11)	-1.5(11)
O(2)	34(2)	21.8(13)	47(2)	9.9(12)	21.4(13)	6.8(11)
C(4)	50(3)	22(2)	58(3)	7(2)	17(2)	10(2)
O(3)	34.2(15)	28.7(14)	24.8(13)	-4.2(11)	9.5(11)	-11.7(12)
C(5)	51(3)	41(2)	28(2)	-5(2)	6(2)	-11(2)
C(6)	28(2)	24(2)	26(2)	1.8(15)	7(2)	5(2)
O(6)	52(2)	50(2)	17.6(14)	3.2(12)	6.2(13)	14.6(15)
C(7)	28(2)	26(2)	16(2)	2.9(14)	5.4(15)	4(2)
O(7)	23.0(15)	45(2)	36(2)	8.8(13)	5.0(12)	-3.0(13)
C(8)	22(2)	31(2)	16(2)	0.0(15)	4.4(14)	0(2)
O(8)	42(2)	26.0(15)	34.5(15)	-11.4(12)	10.3(13)	-3.1(12)
C(9)	30(2)	27(2)	26(2)	4(2)	4(2)	-6(2)
O(9)	53(2)	52(2)	33(2)	1.5(14)	-6.7(14)	-28(2)
C(10)	27(2)	21(2)	27(2)	4.6(15)	6(2)	-1(2)
O(10)	38(2)	40(2)	42(2)	-1.3(13)	21.3(14)	8.7(13)
C(11)	25(2)	27(2)	20(2)	-2.3(15)	7.9(15)	-3(2)
O(11)	44(2)	22.7(14)	33.3(15)	7.6(12)	5.5(13)	-1.3(12)
P(2)	19.5(4)	18.9(4)	16.0(4)	0.4(3)	2.8(3)	1.5(4)
C(12)	25(2)	24(2)	15(2)	0.5(14)	2.3(14)	0.4(15)
C(13)	26(2)	33(2)	27(2)	-4(2)	4(2)	4(2)
C(14)	32(2)	37(2)	29(2)	-3(2)	-2(2)	-8(2)
C(15)	52(3)	27(2)	28(2)	-8(2)	3(2)	-8(2)
C(16)	49(3)	34(2)	32(2)	-11(2)	20(2)	-5(2)
C(17)	32(2)	26(2)	26(2)	-5(2)	8(2)	-4(2)
C(18)	18(2)	19(2)	23(2)	1.0(14)	0.9(14)	0.4(14)
C(19)	19(2)	26(2)	22(2)	-1.8(15)	0.2(14)	0.1(15)
C(20)	27(2)	24(2)	36(2)	-7(2)	0(2)	5(2)
C(21)	24(2)	23(2)	37(2)	4(2)	-5(2)	2(2)
C(22)	31(2)	33(2)	22(2)	7(2)	-2(2)	1(2)
C(23)	30(2)	27(2)	24(2)	2(2)	7(2)	2(2)

Table S11. Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 3a.

	x	y	z	U
H(1A)	1248(13)	7427(6)	2720(7)	40
H(1B)	1722(7)	6432(20)	2303(2)	40
H(1C)	823(8)	6092(15)	2731(6)	40
H(3)	-1103(31)	5954(32)	1136(19)	24
H(4A)	1093(18)	2429(12)	2014(12)	64
H(4B)	-191(8)	2235(9)	2067(12)	64
H(4C)	720(24)	2864(5)	2678(3)	64
H(5A)	-3013(4)	3500(24)	700(5)	61
H(5B)	-1793(20)	2867(13)	920(2)	61
H(5C)	-1888(22)	4201(12)	653(4)	61
H(13)	-3969(3)	6812(3)	172(2)	35
H(14)	-4706(4)	5386(4)	-678(2)	42
H(15)	-3532(4)	4600(3)	-1340(2)	44
H(16)	-1622(4)	5226(4)	-1152(2)	44
H(17)	-852(3)	6613(3)	-275(2)	34
H(19)	-3334(3)	9927(3)	1031(2)	28
H(20)	-4369(3)	11517(3)	420(2)	37
H(21)	-4398(3)	11839(3)	-732(2)	37
H(22)	-3393(3)	10555(3)	-1291(2)	36
H(23)	-2357(3)	8961(3)	-693(2)	32