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Randal W. Eveland, Casey C. Raymond, Duward F. Shriver

Department of Chemistry

Northwestern University

Evanston, Illinois 60208-3113

X-ray Structure Report for [PPN][$Fe_3(CO)_9(CCH)$] (Ia)

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Table 1. Crystal data and structure refinement for (PPN)[Fe3(CO)9(CCH)]-Ia.

Identification code	nu6m
Empirical formula	$C_{47}H_{31}Fe_3NO_9P_2$
Formula weight	983.22
Temperature	163(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 9.2601(1) Å alpha = 90°
	b = 29.7878(1) Å beta = 101.313(1)°
	c = 15.7931(2) Å gamma = 90°
Volume, Z	4271.69(7) $Å^3$, 4
Density (calculated)	1.529 Mg/m^3
Absorption coefficient	1.139 mm^{-1}
F(000)	2000
Crystal size	0.40 x 0.30 x 0.28 mm
θ range for data collection	1.37 to 28.27°
Limiting indices	$-11 \le h \le 12$, $-28 \le k \le 38$, $-20 \le l \le 20$
Reflections collected	28216
Independent reflections	10226 (R_{int} = 0.0271)
Absorption correction	SADABS (Sheldrick, 1996)
Max. and min. transmission	not reported and not reported
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	10225 / 0 / 559
Goodness-of-fit on F^2	1.033
Final R indices [I>2σ(I)]	R1 = 0.0305, wR2 = 0.0685
R indices (all data)	R1 = 0.0466, wR2 = 0.0756
Largest diff. peak and hole	0.356 and -0.342 $eÅ^{-3}$

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for (PPN)[Fe3(CO)9(CCH)]-Ia. U(eq) one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Fe(1)	4209(1)	1463(1)	2145(1)	25(1)
Fe(2)	5076(1)	620(1)	2469(1)	23(1)
Fe(3)	7015(1)	1213(1)	2686(1)	24(1)
C(1)	4094(2)	1489(1)	993(1)	30(1)
O(1)	3999(2)	1510(1)	260(1)	41(1)
C(2)	4316(3)	2047(1)	2347(2)	40(1)
O(2)	4359(3)	2426(1)	2500(1)	68(1)
C(3)	2282(2)	1396(1)	2092(2)	39(1)
O(3)	1039(2)	1358(1)	2067(1)	67(1)
O(4)	5074(2)	486(1)	625(1)	35(1)
C(4)	5060(2)	548(1)	1341(1)	26(1)
C(5)	3261(2)	403(1)	2464(1)	30(1)
O(5)	2095(2)	259(1)	2409(1)	42(1)
O(6)	6835(2)	-165(1)	3153(1)	43(1)
C(6)	6136(2)	139(1)	2869(1)	30(1)
C(7)	7266(2)	1253(1)	1597(1)	30(1)
O(7)	7502(2)	1271(1)	907(1)	43(1)
O(8)	9535(2)	627(1)	3295(1)	50(1)
C(8)	8560(2)	860(1)	3051(1)	32(1)
C(9)	7851(2)	1740(1)	3058(1)	33(1)
O(9)	8430(2)	2076(1)	3257(1)	50(1)
C(10)	5095(2)	1205(1)	3154(1)	27(1)
C(11)	5984(2)	930(1)	3631(1)	30(1)
N(1)	10072(2)	1405(1)	7444(1)	25(1)
P(11)	9471(1)	1356(1)	6445(1)	20(1)
P(12)	9720(1)	1324(1)	8373(1)	20(1)
C(111)	8254(2)	1811(1)	6023(1)	22(1)
C(112)	7455(2)	1800(1)	5174(1)	30(1)
C(113)	6542(2)	2154(1)	4850(1)	36(1)
C(114)	6402(3)	2519(1)	5367(1)	38(1)
C(115)	7178(3)	2533(1)	6207(1)	37(1)
C(116)	8115(2)	2182(1)	6540(1)	29(1)
C(121)	8462(2)	849(1)	6107(1)	22(1)
C(122)	9140(2)	470(1)	5837(1)	30(1)
C(123)	8320(3)	84(1)	5599(1)	37(1)
C(124)	6844(3)	71(1)	5642(1)	38(1)
C(125)	6174(2)	441(1)	5927(1)	35(1)
C(126)	6980(2)	829(1)	6161(1)	28(1)
C(131)	11039(2)	1368(1)	5921(1)	23(1)
C(132)	12428(2)	1466(1)	6406(1)	29(1)
C(133)	13635(2)	1489(1)	6006(2)	38(1)
C(134)	13471(2)	1401(1)	5134(2)	39(1)
C(135)	12098(3)	1297(1)	4650(1)	39(1)
C(136)	10875(2)	1286(1)	5039(1)	34(1)
C(211)	10413(2)	1798(1)	9036(1)	23(1)
C(212)	11446(2)	2079(1)	8771(1)	28(1)
C(213)	12078(2)	2427(1)	9301(2)	39(1)
C(214)	11690(3)	2492(1)	10098(2)	42(1)

C(215)	10659(3)	2218(1)	10358(1)	39(1)
C(216)	9999(2)	1873(1)	9828(1)	30(1)
C(221)	7800(2)	1255(1)	8425(1)	22(1)
C(222)	6876(2)	1628(1)	8394(1)	29(1)
C(223)	5406(2)	1570(1)	8437(1)	34(1)
C(224)	4849(2)	1144(1)	8511(1)	34(1)
C(225)	5753(2)	772(1)	8535(1)	35(1)
C(226)	7226(2)	826(1)	8491(1)	29(1)
C(231)	10639(2)	826(1)	8850(1)	22(1)
C(232)	10877(2)	758(1)	9744(1)	31(1)
C(233)	11497(2)	358(1)	10092(1)	36(1)
C(234)	11877(2)	28(1)	9566(1)	36(1)
C(235)	11642(3)	92(1)	8684(1)	39(1)
C(236)	11030(2)	492(1)	8326(1)	32(1)

Table 3. Bond lengths [Å] and angles [°] for (PPN)[Fe3(CO)9(CCH)]-Ia.

Fe(1)-C(2)	1.769(2)	Fe(1)-C(3)	1.781(2)
Fe(1)-C(1)	1.803(2)	Fe(1)-C(10)	1.814(2)
Fe(1)-Fe(2)	2.6569(4)	Fe(1)-Fe(3)	2.6769(4)
Fe(2)-C(6)	1.781(2)	Fe(2)-C(4)	1.791(2)
Fe(2)-C(5)	1.799(2)	Fe(2)-C(10)	2.049(2)
Fe(2)-C(11)	2.079(2)	Fe(2)-Fe(3)	2.4958(4)
Fe(3)-C(8)	1.778(2)	Fe(3)-C(7)	1.784(2)
Fe(3)-C(9)	1.797(2)	Fe(3)-C(10)	2.055(2)
Fe(3)-C(11)	2.099(2)	C(1)-O(1)	1.146(2)
C(2)-O(2)	1.153(3)	C(3)-O(3)	1.149(3)
O(4)-C(4)	1.149(2)	C(5)-O(5)	1.150(2)
O(6)-C(6)	1.151(3)	C(7)-O(7)	1.154(2)
O(8)-C(8)	1.145(3)	C(9)-O(9)	1.150(3)
C(10)-C(11)	1.293(3)	N(1)-P(11)	1.573(2)
N(1)-P(12)	1.583(2)	P(11)-C(121)	1.803(2)
P(11)-C(111)	1.804(2)	P(11)-C(131)	1.806(2)
P(12)-C(211)	1.800(2)	P(12)-C(231)	1.801(2)
P(12)-C(221)	1.807(2)	C(111)-C(116)	1.394(3)
C(111)-C(112)	1.399(3)	C(112)-C(113)	1.384(3)
C(113)-C(114)	1.382(3)	C(114)-C(115)	1.381(3)
C(115)-C(116)	1.393(3)	C(121)-C(126)	1.393(3)
C(121)-C(122)	1.399(3)	C(122)-C(123)	1.387(3)
C(123)-C(124)	1.382(3)	C(124)-C(125)	1.383(3)
C(125)-C(126)	1.386(3)	C(131)-C(132)	1.393(3)
C(131)-C(136)	1.393(3)	C(132)-C(133)	1.390(3)
C(133)-C(134)	1.381(3)	C(134)-C(135)	1.383(3)
C(135)-C(136)	1.391(3)	C(211)-C(212)	1.395(3)
C(211)-C(216)	1.396(3)	C(212)-C(213)	1.388(3)
C(213)-C(214)	1.389(3)	C(214)-C(215)	1.378(3)
C(215)-C(216)	1.390(3)	C(221)-C(226)	1.394(3)
C(221)-C(222)	1.397(3)	C(222)-C(223)	1.387(3)
C(223)-C(224)	1.385(3)	C(224)-C(225)	1.383(3)
C(225)-C(226)	1.388(3)	C(231)-C(236)	1.387(3)
C(231)-C(232)	1.400(3)	C(232)-C(233)	1.387(3)
C(233)-C(234)	1.376(3)	C(234)-C(235)	1.382(3)
C(235)-C(236)	1.390(3)		

C(2)-Fe(1)-C(3)	98.00(11)	C(2)-Fe(1)-C(1)	97.48(10)
C(3)-Fe(1)-C(1)	95.56(10)	C(2)-Fe(1)-C(10)	105.04(10)
C(3)-Fe(1)-C(10)	105.68(9)	C(1)-Fe(1)-C(10)	146.21(9)
C(2)-Fe(1)-Fe(2)	153.09(8)	C(3)-Fe(1)-Fe(2)	99.51(8)
C(1)-Fe(1)-Fe(2)	100.98(6)	C(10)-Fe(1)-Fe(2)	50.35(6)
C(2)-Fe(1)-Fe(3)	101.49(8)	C(3)-Fe(1)-Fe(3)	152.22(7)
C(1)-Fe(1)-Fe(3)	101.27(7)	C(10)-Fe(1)-Fe(3)	50.07(6)
Fe(2)-Fe(1)-Fe(3)	55.797(10)	C(6)-Fe(2)-C(4)	98.87(9)
C(6)-Fe(2)-C(5)	99.39(9)	C(4)-Fe(2)-C(5)	97.36(9)
C(6)-Fe(2)-C(10)	123.36(9)	C(4)-Fe(2)-C(10)	128.52(8)
C(5)-Fe(2)-C(10)	102.59(9)	C(6)-Fe(2)-C(11)	86.88(9)
C(4)-Fe(2)-C(11)	148.57(8)	C(5)-Fe(2)-C(11)	112.25(8)
C(10)-Fe(2)-C(11)	36.48(8)	C(6)-Fe(2)-Fe(3)	100.93(7)
C(4)-Fe(2)-Fe(3)	94.93(6)	C(5)-Fe(2)-Fe(3)	154.28(7)
C(10)-Fe(2)-Fe(3)	52.65(5)	C(11)-Fe(2)-Fe(3)	53.70(6)
C(6)-Fe(2)-Fe(1)	162.51(7)	C(4)-Fe(2)-Fe(1)	88.68(6)
C(5)-Fe(2)-Fe(1)	95.22(7)	C(10)-Fe(2)-Fe(1)	42.96(6)
C(11)-Fe(2)-Fe(1)	78.68(6)	Fe(3)-Fe(2)-Fe(1)	62.508(11)

C(8)-Fe(3)-C(7)	95.92(9)	C(8)-Fe(3)-C(9)	98.21(10)
C(7)-Fe(3)-C(9)	97.35(9)	C(8)-Fe(3)-C(10)	125.04(9)
C(7)-Fe(3)-C(10)	129.23(8)	C(9)-Fe(3)-C(10)	103.98(9)
C(8)-Fe(3)-C(11)	88.93(9)	C(7)-Fe(3)-C(11)	151.04(8)
C(9)-Fe(3)-C(11)	110.23(9)	C(10)-Fe(3)-C(11)	36.24(8)
C(8)-Fe(3)-Fe(2)	97.87(7)	C(7)-Fe(3)-Fe(2)	98.08(6)
C(9)-Fe(3)-Fe(2)	156.38(7)	C(10)-Fe(3)-Fe(2)	52.44(6)
C(11)-Fe(3)-Fe(2)	52.96(5)	C(8)-Fe(3)-Fe(1)	159.54(7)
C(7)-Fe(3)-Fe(1)	88.59(7)	C(9)-Fe(3)-Fe(1)	101.00(7)
C(10)-Fe(3)-Fe(1)	42.60(5)	C(11)-Fe(3)-Fe(1)	77.88(6)
Fe(2)-Fe(3)-Fe(1)	61.696(11)	O(1)-C(1)-Fe(1)	178.8(2)
O(2)-C(2)-Fe(1)	177.8(2)	O(3)-C(3)-Fe(1)	178.9(2)
O(4)-C(4)-Fe(2)	177.2(2)	O(5)-C(5)-Fe(2)	175.8(2)
O(6)-C(6)-Fe(2)	177.8(2)	O(7)-C(7)-Fe(3)	176.5(2)
O(8)-C(8)-Fe(3)	178.4(2)	O(9)-C(9)-Fe(3)	176.0(2)
C(11)-C(10)-Fe(1)	155.1(2)	C(11)-C(10)-Fe(2)	73.03(13)
Fe(1)-C(10)-Fe(2)	86.68(8)	C(11)-C(10)-Fe(3)	73.76(12)
Fe(1)-C(10)-Fe(3)	87.34(8)	Fe(2)-C(10)-Fe(3)	74.91(6)
C(10)-C(11)-Fe(2)	70.49(12)	C(10)-C(11)-Fe(3)	70.00(12)
Fe(2)-C(11)-Fe(3)	73.34(6)	P(11)-N(1)-P(12)	144.97(11)
N(1)-P(11)-C(121)	115.78(8)	N(1)-P(11)-C(111)	111.91(9)
C(121)-P(11)-C(111)	105.75(8)	N(1)-P(11)-C(131)	107.39(9)
C(121)-P(11)-C(131)	107.25(9)	C(111)-P(11)-C(131)	108.50(8)
N(1)-P(12)-C(211)	107.77(9)	N(1)-P(12)-C(231)	110.29(9)
C(211)-P(12)-C(231)	108.52(8)	N(1)-P(12)-C(221)	116.33(8)
C(211)-P(12)-C(221)	107.76(9)	C(231)-P(12)-C(221)	105.93(8)
C(116)-C(111)-C(112)	119.2(2)	C(116)-C(111)-P(11)	120.12(14)
C(112)-C(111)-P(11)	120.67(14)	C(113)-C(112)-C(111)	120.3(2)
C(114)-C(113)-C(112)	120.3(2)	C(115)-C(114)-C(113)	119.9(2)
C(114)-C(115)-C(116)	120.7(2)	C(115)-C(116)-C(111)	119.7(2)
C(126)-C(121)-C(122)	119.5(2)	C(126)-C(121)-P(11)	118.46(14)
C(122)-C(121)-P(11)	122.01(14)	C(123)-C(122)-C(121)	119.7(2)
C(124)-C(123)-C(122)	120.3(2)	C(123)-C(124)-C(125)	120.3(2)
C(124)-C(125)-C(126)	119.9(2)	C(125)-C(126)-C(121)	120.3(2)
C(132)-C(131)-C(136)	119.6(2)	C(132)-C(131)-P(11)	119.38(14)
C(136)-C(131)-P(11)	120.99(15)	C(133)-C(132)-C(131)	119.9(2)
C(134)-C(133)-C(132)	120.3(2)	C(133)-C(134)-C(135)	120.2(2)
C(134)-C(135)-C(136)	120.0(2)	C(135)-C(136)-C(131)	120.0(2)
C(212)-C(211)-C(216)	119.9(2)	C(212)-C(211)-P(12)	118.84(14)
C(216)-C(211)-P(12)	121.15(15)	C(213)-C(212)-C(211)	119.9(2)
C(212)-C(213)-C(214)	119.9(2)	C(215)-C(214)-C(213)	120.4(2)
C(214)-C(215)-C(216)	120.4(2)	C(215)-C(216)-C(211)	119.5(2)
C(226)-C(221)-C(222)	119.3(2)	C(226)-C(221)-P(12)	120.10(15)
C(222)-C(221)-P(12)	120.56(15)	C(223)-C(222)-C(221)	120.1(2)
C(224)-C(223)-C(222)	120.1(2)	C(225)-C(224)-C(223)	120.2(2)
C(224)-C(225)-C(226)	120.1(2)	C(225)-C(226)-C(221)	120.2(2)
C(236)-C(231)-C(232)	119.3(2)	C(236)-C(231)-P(12)	119.91(14)
C(232)-C(231)-P(12)	120.67(15)	C(233)-C(232)-C(231)	119.7(2)
C(234)-C(233)-C(232)	120.6(2)	C(233)-C(234)-C(235)	120.0(2)
C(234)-C(235)-C(236)	120.0(2)	C(231)-C(236)-C(235)	120.4(2)

Symmetry transformations used to generate equivalent atoms:

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Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for (PPN)[Fe3(CO)9(CCH)]-Ia.
The anisotropic displacement factor exponent takes $-2\pi^2 [(ha^*)^2U_{11} + ... + 2hka^*b^*U_{12}]$

	U11	U22	U33	U23	U13	U12
Fe(1)	25(1)	26(1)	25(1)	1(1)	6(1)	2(1)
Fe(2)	25(1)	23(1)	22(1)	1(1)	6(1)	-2(1)
Fe(3)	23(1)	26(1)	22(1)	-1(1)	4(1)	-2(1)
C(1)	29(1)	25(1)	35(1)	1(1)	8(1)	2(1)
O(1)	52(1)	44(1)	28(1)	5(1)	9(1)	6(1)
C(2)	47(1)	34(1)	38(1)	-3(1)	8(1)	5(1)
O(2)	97(2)	34(1)	70(1)	-12(1)	14(1)	4(1)
C(3)	33(1)	46(1)	39(1)	13(1)	10(1)	6(1)
O(3)	30(1)	94(2)	81(1)	34(1)	16(1)	5(1)
O(4)	38(1)	39(1)	28(1)	-6(1)	9(1)	1(1)
C(4)	25(1)	23(1)	30(1)	-1(1)	5(1)	-1(1)
C(5)	35(1)	32(1)	22(1)	2(1)	7(1)	-2(1)
O(5)	32(1)	58(1)	37(1)	6(1)	6(1)	-13(1)
O(6)	43(1)	36(1)	50(1)	14(1)	13(1)	9(1)
C(6)	31(1)	31(1)	31(1)	0(1)	10(1)	-5(1)
C(7)	25(1)	34(1)	31(1)	-1(1)	6(1)	-6(1)
O(7)	44(1)	57(1)	30(1)	-1(1)	14(1)	-12(1)
O(8)	38(1)	55(1)	53(1)	3(1)	0(1)	16(1)
C(8)	29(1)	36(1)	31(1)	-1(1)	5(1)	-3(1)
C(9)	37(1)	34(1)	25(1)	1(1)	4(1)	-5(1)
O(9)	68(1)	41(1)	40(1)	-5(1)	6(1)	-23(1)
C(10)	31(1)	30(1)	22(1)	-2(1)	10(1)	0(1)
C(11)	35(1)	35(1)	21(1)	-2(1)	7(1)	-4(1)
N(1)	26(1)	33(1)	18(1)	2(1)	5(1)	-2(1)
P(11)	21(1)	21(1)	18(1)	0(1)	5(1)	1(1)
P(12)	22(1)	22(1)	17(1)	-1(1)	5(1)	-1(1)
C(111)	23(1)	21(1)	22(1)	0(1)	7(1)	1(1)
C(112)	37(1)	28(1)	25(1)	-3(1)	4(1)	5(1)
C(113)	39(1)	40(1)	27(1)	6(1)	1(1)	9(1)
C(114)	43(1)	33(1)	39(1)	7(1)	10(1)	16(1)
C(115)	49(1)	27(1)	38(1)	-4(1)	13(1)	10(1)
C(116)	34(1)	27(1)	26(1)	-3(1)	7(1)	3(1)
C(121)	25(1)	22(1)	19(1)	1(1)	4(1)	-1(1)
C(122)	32(1)	27(1)	32(1)	-3(1)	6(1)	2(1)
C(123)	52(1)	25(1)	32(1)	-2(1)	4(1)	2(1)
C(124)	50(1)	31(1)	30(1)	6(1)	-3(1)	-14(1)
C(125)	31(1)	41(1)	32(1)	8(1)	2(1)	-11(1)
C(126)	27(1)	29(1)	28(1)	2(1)	6(1)	0(1)
C(131)	23(1)	24(1)	24(1)	2(1)	9(1)	1(1)
C(132)	26(1)	33(1)	27(1)	1(1)	5(1)	8(1)
C(133)	21(1)	47(1)	45(1)	2(1)	8(1)	7(1)
C(134)	33(1)	43(1)	47(1)	4(1)	23(1)	6(1)
C(135)	44(1)	49(1)	30(1)	-3(1)	21(1)	-1(1)
C(136)	30(1)	48(1)	26(1)	-3(1)	9(1)	-5(1)
C(211)	24(1)	22(1)	24(1)	-2(1)	3(1)	0(1)
C(212)	28(1)	26(1)	32(1)	-1(1)	9(1)	-1(1)

C(213)	36(1)	28(1)	56(1)	-5(1)	11(1)	-9(1)
C(214)	44(1)	30(1)	51(1)	-18(1)	4(1)	-5(1)
C(215)	47(1)	37(1)	34(1)	-14(1)	12(1)	3(1)
C(216)	34(1)	30(1)	29(1)	-7(1)	11(1)	-1(1)
C(221)	22(1)	26(1)	18(1)	1(1)	3(1)	-2(1)
C(222)	28(1)	26(1)	33(1)	2(1)	7(1)	-1(1)
C(223)	26(1)	38(1)	40(1)	3(1)	8(1)	6(1)
C(224)	23(1)	47(1)	32(1)	1(1)	5(1)	-6(1)
C(225)	32(1)	32(1)	39(1)	0(1)	6(1)	-11(1)
C(226)	28(1)	26(1)	32(1)	-1(1)	5(1)	-3(1)
C(231)	20(1)	23(1)	23(1)	1(1)	4(1)	0(1)
C(232)	38(1)	32(1)	22(1)	-1(1)	3(1)	2(1)
C(233)	40(1)	39(1)	26(1)	7(1)	0(1)	2(1)
C(234)	32(1)	35(1)	41(1)	13(1)	9(1)	9(1)
C(235)	45(1)	37(1)	39(1)	2(1)	15(1)	17(1)
C(236)	38(1)	34(1)	25(1)	2(1)	10(1)	10(1)

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Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for (PPN)[Fe3(CO)9(CCH)]-Ia.

	x	y	z	U(eq)
H(11A)	6254(2)	847(1)	4255(1)	36
H(11B)	7538(2)	1548(1)	4818(1)	36
H(11C)	6011(2)	2145(1)	4271(1)	43
H(11D)	5772(3)	2761(1)	5143(1)	46
H(11E)	7072(3)	2783(1)	6562(1)	45
H(11F)	8656(2)	2196(1)	7117(1)	35
H(12A)	10158(2)	476(1)	5816(1)	37
H(12B)	8775(3)	-172(1)	5406(1)	45
H(12C)	6288(3)	-194(1)	5475(1)	46
H(12D)	5162(2)	429(1)	5962(1)	42
H(12E)	6520(2)	1083(1)	6358(1)	33
H(13A)	12549(2)	1518(1)	7010(1)	34
H(13B)	14577(2)	1564(1)	6334(2)	45
H(13C)	14303(2)	1412(1)	4865(2)	46
H(13D)	11991(3)	1234(1)	4051(1)	47
H(13E)	9929(2)	1222(1)	4704(1)	41
H(21A)	11718(2)	2031(1)	8228(1)	34
H(21B)	12775(2)	2620(1)	9119(2)	47
H(21C)	12137(3)	2727(1)	10465(2)	51
H(21D)	10398(3)	2266(1)	10904(1)	46
H(21E)	9272(2)	1690(1)	10003(1)	36
H(22A)	7254(2)	1921(1)	8342(1)	34
H(22B)	4779(2)	1824(1)	8416(1)	41
H(22C)	3844(2)	1106(1)	8545(1)	41
H(22D)	5365(2)	480(1)	8581(1)	42
H(22E)	7844(2)	571(1)	8506(1)	35
H(23A)	10616(2)	984(1)	10110(1)	37
H(23B)	11659(2)	312(1)	10698(1)	43
H(23C)	12301(2)	-245(1)	9811(1)	43
H(23D)	11899(3)	-137(1)	8321(1)	47
H(23E)	10880(2)	537(1)	7719(1)	38

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Randal W. Eveland, Casey C. Raymond, Duward F. Shriver

Department of Chemistry

Northwestern University

Evanston, Illinois 60208-3113

X-ray Structure Report for [PPN][$Fe_3(CO)_9(CCH)$] (**Ib**)

Table 1. Crystal data and structure refinement for (PPN)[Fe3(CO)9(CCH)]-Ib.

Identification code	s47g2m
Empirical formula	$C_{47}H_{31}Fe_3NO_9P_2$
Formula weight	983.22
Temperature	153(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	*a* = 9.2707(9) Å alpha = 90°
	b = 29.716(3) Å beta = 101.608(2)°
	c = 15.8471(15) Å gamma = 90°
Volume, Z	4276.4(7) $Å^3$, 4
Density (calculated)	1.527 Mg/m^3
Absorption coefficient	1.138 mm^{-1}
F(000)	2000
Crystal size	0.32 x 0.09 x 0.03 mm
θ range for data collection	1.37 to 28.33°
Limiting indices	$-12 \le h \le 12$, $-39 \le k \le 39$, $-20 \le l \le 20$
Reflections collected	38331
Independent reflections	10181 (R_{int} = 0.0835)
Completeness to θ = 28.33°	95.4 %
Absorption correction	Analytical
Max. and min. transmission	0.968 and 0.754
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	10181 / 0 / 559
Goodness-of-fit on F^2	0.919
Final R indices [I>2σ(I)]	R1 = 0.0521, wR2 = 0.0905
R indices (all data)	R1 = 0.1160, wR2 = 0.1037
Largest diff. peak and hole	0.802 and -0.682 $eÅ^{-3}$

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for (PPN)[Fe3(CO)9(CCH)]-Ib. U(eq) one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Fe(1)	745(1)	1468(1)	2865(1)	36(1)
Fe(2)	-71(1)	624(1)	2542(1)	31(1)
Fe(3)	-2041(1)	1219(1)	2350(1)	40(1)
C(1)	967(4)	1494(1)	4014(3)	41(1)
O(1)	1091(3)	1512(1)	4743(2)	57(1)
C(2)	2619(5)	1393(2)	2851(3)	58(1)
O(2)	3859(4)	1353(1)	2845(3)	102(1)
C(3)	672(4)	2051(1)	2647(2)	47(1)
O(3)	645(4)	2428(1)	2483(2)	79(1)
C(4)	-71(3)	557(1)	3656(2)	32(1)
O(4)	-93(2)	498(1)	4375(2)	43(1)
C(5)	-1125(4)	140(1)	2143(2)	37(1)
O(5)	-1802(3)	-168(1)	1864(2)	51(1)
C(6)	1728(4)	405(1)	2562(2)	41(1)
O(6)	2896(3)	257(1)	2617(2)	54(1)
C(7)	-2364(5)	1235(1)	3394(3)	63(1)
O(7)	-2615(4)	1244(1)	4076(2)	99(1)
C(8)	-2884(4)	1738(1)	1970(2)	48(1)
O(8)	-3476(3)	2074(1)	1753(2)	67(1)
C(9)	-3472(4)	861(1)	1908(2)	47(1)
O(9)	-4441(3)	626(1)	1634(2)	88(1)
C(10)	-201(5)	1205(1)	1866(2)	49(1)
C(11)	-862(6)	930(2)	1361(3)	74(1)
N(1)	5012(3)	-1414(1)	2449(2)	32(1)
P(11)	4443(1)	-1359(1)	1454(1)	27(1)
P(12)	4688(1)	-1330(1)	3374(1)	28(1)
C(100)	7105(4)	-1309(1)	-305(2)	48(1)
C(101)	3073(4)	-2183(1)	1519(2)	37(1)
C(102)	1532(4)	-2150(1)	-166(2)	45(1)
C(103)	6663(4)	-2491(1)	5103(3)	52(1)
C(104)	5879(4)	-1296(1)	73(2)	43(1)
C(105)	-145(4)	-1138(1)	3537(2)	40(1)
C(106)	2146(4)	-2532(1)	1181(2)	45(1)
C(107)	1176(4)	-435(1)	932(2)	43(1)
C(108)	6877(4)	-34(1)	4550(2)	42(1)
C(109)	8467(4)	-1403(1)	192(3)	50(1)
C(110)	3317(4)	-84(1)	606(2)	45(1)
C(111)	394(4)	-1568(1)	3453(2)	40(1)
C(112)	2451(4)	-1797(1)	168(2)	37(1)
C(113)	1386(4)	-2515(1)	342(3)	46(1)
C(114)	6410(3)	-2085(1)	3783(2)	37(1)
C(115)	6485(4)	-361(1)	5072(2)	42(1)
C(116)	5631(4)	-2218(1)	5363(2)	46(1)
C(117)	1980(3)	-826(1)	1169(2)	35(1)
C(118)	6025(4)	-500(1)	3321(2)	40(1)
C(119)	7038(4)	-2431(1)	4313(3)	48(1)
C(120)	5857(4)	-756(1)	4728(2)	37(1)

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C(121)	6651(4)	-102(1)	3677(2)	45(1)
C(122)	1854(4)	-67(1)	651(2)	44(1)
C(123)	4124(4)	-472(1)	844(2)	38(1)
C(124)	8614(4)	-1486(1)	1056(3)	47(1)
C(125)	4980(4)	-1875(1)	4824(2)	36(1)
C(126)	1855(3)	-1626(1)	3401(2)	37(1)
C(127)	7400(3)	-1463(1)	1443(2)	37(1)
C(128)	2219(4)	-828(1)	3505(2)	36(1)
C(129)	752(4)	-771(1)	3554(2)	40(1)
C(130)	2783(3)	-1257(1)	3435(2)	28(1)
C(131)	6022(3)	-1371(1)	948(2)	29(1)
C(132)	3460(3)	-850(1)	1117(2)	27(1)
C(133)	3223(3)	-1811(1)	1014(2)	28(1)
C(134)	5380(3)	-1801(1)	4039(2)	30(1)
C(135)	5623(3)	-831(1)	3842(2)	28(1)

Table 3. Bond lengths [Å] and angles [°] for (PPN)[Fe3(CO)9(CCH)]-Ib.

Fe(1)-C(2)	1.756(4)	Fe(1)-C(3)	1.765(4)
Fe(1)-C(1)	1.793(4)	Fe(1)-C(10)	1.824(4)
Fe(1)-Fe(2)	2.6405(7)	Fe(1)-Fe(3)	2.6529(7)
Fe(2)-C(4)	1.776(4)	Fe(2)-C(5)	1.781(4)
Fe(2)-C(6)	1.785(4)	Fe(2)-C(10)	2.023(4)
Fe(2)-C(11)	2.078(4)	Fe(2)-Fe(3)	2.5169(7)
Fe(3)-C(9)	1.735(4)	Fe(3)-C(7)	1.740(5)
Fe(3)-C(8)	1.777(4)	Fe(3)-C(10)	2.007(4)
Fe(3)-C(11)	2.255(5)	C(1)-O(1)	1.138(4)
C(2)-O(2)	1.158(5)	C(3)-O(3)	1.149(4)
C(4)-O(4)	1.156(4)	C(5)-O(5)	1.149(4)
C(6)-O(6)	1.155(4)	C(7)-O(7)	1.150(5)
C(8)-O(8)	1.159(4)	C(9)-O(9)	1.152(4)
C(10)-C(11)	1.219(5)	N(1)-P(11)	1.567(3)
N(1)-P(12)	1.574(3)	P(11)-C(132)	1.793(3)
P(11)-C(133)	1.803(3)	P(11)-C(131)	1.805(3)
P(12)-C(134)	1.792(3)	P(12)-C(135)	1.799(3)
P(12)-C(130)	1.801(3)	C(100)-C(109)	1.376(5)
C(100)-C(104)	1.387(5)	C(101)-C(106)	1.384(5)
C(101)-C(133)	1.389(4)	C(102)-C(113)	1.374(5)
C(102)-C(112)	1.387(5)	C(103)-C(119)	1.378(5)
C(103)-C(116)	1.378(5)	C(104)-C(131)	1.384(5)
C(105)-C(129)	1.369(5)	C(105)-C(111)	1.387(5)
C(106)-C(113)	1.375(5)	C(107)-C(122)	1.378(5)
C(107)-C(117)	1.392(5)	C(108)-C(115)	1.372(5)
C(108)-C(121)	1.371(5)	C(109)-C(124)	1.370(5)
C(110)-C(122)	1.374(5)	C(110)-C(123)	1.384(5)
C(111)-C(126)	1.384(4)	C(112)-C(133)	1.387(4)
C(114)-C(119)	1.379(5)	C(114)-C(134)	1.395(4)
C(115)-C(120)	1.374(5)	C(116)-C(125)	1.388(5)
C(117)-C(132)	1.393(4)	C(118)-C(135)	1.383(4)
C(118)-C(121)	1.389(5)	C(120)-C(135)	1.394(4)
C(123)-C(132)	1.391(4)	C(124)-C(127)	1.387(5)
C(125)-C(134)	1.386(4)	C(126)-C(130)	1.388(4)
C(127)-C(131)	1.385(4)	C(128)-C(129)	1.388(4)
C(128)-C(130)	1.390(4)		
C(2)-Fe(1)-C(3)	97.06(19)	C(2)-Fe(1)-C(1)	96.12(18)
C(3)-Fe(1)-C(1)	98.51(16)	C(2)-Fe(1)-C(10)	103.79(19)
C(3)-Fe(1)-C(10)	104.98(17)	C(1)-Fe(1)-C(10)	146.80(17)
C(2)-Fe(1)-Fe(2)	96.98(14)	C(3)-Fe(1)-Fe(2)	153.70(12)
C(1)-Fe(1)-Fe(2)	101.97(11)	C(10)-Fe(1)-Fe(2)	49.89(12)
C(2)-Fe(1)-Fe(3)	149.87(14)	C(3)-Fe(1)-Fe(3)	102.52(13)
C(1)-Fe(1)-Fe(3)	103.28(11)	C(10)-Fe(1)-Fe(3)	49.11(14)
Fe(2)-Fe(1)-Fe(3)	56.779(18)	C(4)-Fe(2)-C(5)	98.80(15)
C(4)-Fe(2)-C(6)	97.37(15)	C(5)-Fe(2)-C(6)	98.93(16)
C(4)-Fe(2)-C(10)	127.61(16)	C(5)-Fe(2)-C(10)	122.06(16)
C(6)-Fe(2)-C(10)	106.05(17)	C(4)-Fe(2)-C(11)	150.92(17)
C(5)-Fe(2)-C(11)	87.95(18)	C(6)-Fe(2)-C(11)	109.55(18)
C(10)-Fe(2)-C(11)	34.56(15)	C(4)-Fe(2)-Fe(3)	93.09(10)
C(5)-Fe(2)-Fe(3)	101.06(11)	C(6)-Fe(2)-Fe(3)	155.70(12)
C(10)-Fe(2)-Fe(3)	51.08(12)	C(11)-Fe(2)-Fe(3)	57.83(14)
C(4)-Fe(2)-Fe(1)	88.37(11)	C(5)-Fe(2)-Fe(1)	161.99(11)
C(6)-Fe(2)-Fe(1)	96.48(12)	C(10)-Fe(2)-Fe(1)	43.60(11)

C(11)-Fe(2)-Fe(1)	78.11(13)	Fe(3)-Fe(2)-Fe(1)	61.86(2)
C(9)-Fe(3)-C(7)	97.8(2)	C(9)-Fe(3)-C(8)	98.50(17)
C(7)-Fe(3)-C(8)	98.69(18)	C(9)-Fe(3)-C(10)	118.18(17)
C(7)-Fe(3)-C(10)	133.34(18)	C(8)-Fe(3)-C(10)	103.80(17)
C(9)-Fe(3)-C(11)	85.61(18)	C(7)-Fe(3)-C(11)	150.64(16)
C(8)-Fe(3)-C(11)	109.69(16)	C(10)-Fe(3)-C(11)	32.60(15)
C(9)-Fe(3)-Fe(2)	95.40(13)	C(7)-Fe(3)-Fe(2)	99.38(13)
C(8)-Fe(3)-Fe(2)	155.43(13)	C(10)-Fe(3)-Fe(2)	51.64(10)
C(11)-Fe(3)-Fe(2)	51.27(11)	C(9)-Fe(3)-Fe(1)	155.94(13)
C(7)-Fe(3)-Fe(1)	92.51(15)	C(8)-Fe(3)-Fe(1)	101.36(13)
C(10)-Fe(3)-Fe(1)	43.39(11)	C(11)-Fe(3)-Fe(1)	75.02(13)
Fe(2)-Fe(3)-Fe(1)	61.36(2)	O(1)-C(1)-Fe(1)	179.2(4)
O(2)-C(2)-Fe(1)	178.4(4)	O(3)-C(3)-Fe(1)	177.7(4)
O(4)-C(4)-Fe(2)	177.6(3)	O(5)-C(5)-Fe(2)	178.2(3)
O(6)-C(6)-Fe(2)	176.6(3)	O(7)-C(7)-Fe(3)	178.3(5)
O(8)-C(8)-Fe(3)	176.6(4)	O(9)-C(9)-Fe(3)	178.0(4)
C(11)-C(10)-Fe(1)	161.3(4)	C(11)-C(10)-Fe(3)	84.9(3)
Fe(1)-C(10)-Fe(3)	87.50(17)	C(11)-C(10)-Fe(2)	75.2(3)
Fe(1)-C(10)-Fe(2)	86.51(15)	Fe(3)-C(10)-Fe(2)	77.28(15)
C(10)-C(11)-Fe(2)	70.3(3)	C(10)-C(11)-Fe(3)	62.5(3)
Fe(2)-C(11)-Fe(3)	70.90(14)	P(11)-N(1)-P(12)	146.26(18)
N(1)-P(11)-C(132)	115.81(14)	N(1)-P(11)-C(133)	111.77(15)
C(132)-P(11)-C(133)	105.79(14)	N(1)-P(11)-C(131)	107.79(14)
C(132)-P(11)-C(131)	107.00(15)	C(133)-P(11)-C(131)	108.40(14)
N(1)-P(12)-C(134)	108.11(15)	N(1)-P(12)-C(135)	110.39(15)
C(134)-P(12)-C(135)	108.35(14)	N(1)-P(12)-C(130)	116.23(14)
C(134)-P(12)-C(130)	107.56(15)	C(135)-P(12)-C(130)	105.95(14)
C(109)-C(100)-C(104)	119.7(4)	C(106)-C(101)-C(133)	120.0(3)
C(113)-C(102)-C(112)	119.9(3)	C(119)-C(103)-C(116)	120.9(4)
C(131)-C(104)-C(100)	120.4(3)	C(129)-C(105)-C(111)	120.4(3)
C(113)-C(106)-C(101)	120.3(3)	C(122)-C(107)-C(117)	119.8(3)
C(115)-C(108)-C(121)	119.9(3)	C(124)-C(109)-C(100)	120.2(3)
C(122)-C(110)-C(123)	120.1(3)	C(126)-C(111)-C(105)	119.7(3)
C(102)-C(112)-C(133)	120.3(3)	C(102)-C(113)-C(106)	120.3(3)
C(119)-C(114)-C(134)	120.2(3)	C(120)-C(115)-C(108)	120.6(3)
C(103)-C(116)-C(125)	119.3(4)	C(107)-C(117)-C(132)	120.4(3)
C(135)-C(118)-C(121)	120.3(3)	C(103)-C(119)-C(114)	119.9(4)
C(115)-C(120)-C(135)	120.2(3)	C(108)-C(121)-C(118)	120.2(4)
C(110)-C(122)-C(107)	120.4(3)	C(110)-C(123)-C(132)	120.6(3)
C(109)-C(124)-C(127)	120.4(3)	C(134)-C(125)-C(116)	120.5(3)
C(111)-C(126)-C(130)	120.4(3)	C(131)-C(127)-C(124)	119.9(3)
C(129)-C(128)-C(130)	120.3(3)	C(105)-C(129)-C(128)	120.0(3)
C(126)-C(130)-C(128)	119.2(3)	C(126)-C(130)-P(12)	120.6(3)
C(128)-C(130)-P(12)	120.2(2)	C(104)-C(131)-C(127)	119.3(3)
C(104)-C(131)-P(11)	121.3(2)	C(127)-C(131)-P(11)	119.4(3)
C(123)-C(132)-C(117)	118.7(3)	C(123)-C(132)-P(11)	123.0(2)
C(117)-C(132)-P(11)	118.2(3)	C(112)-C(133)-C(101)	119.2(3)
C(112)-C(133)-P(11)	120.9(3)	C(101)-C(133)-P(11)	119.9(2)
C(125)-C(134)-C(114)	119.2(3)	C(125)-C(134)-P(12)	121.6(3)
C(114)-C(134)-P(12)	119.2(3)	C(118)-C(135)-C(120)	118.8(3)
C(118)-C(135)-P(12)	120.3(2)	C(120)-C(135)-P(12)	120.8(3)

Symmetry transformations used to generate equivalent atoms:

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Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for (PPN)[Fe3(CO)9(CCH)]-Ib.

The anisotropic displacement factor exponent takes $-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
Fe(1)	35(1)	31(1)	39(1)	0(1)	5(1)	-2(1)
Fe(2)	32(1)	28(1)	34(1)	0(1)	9(1)	3(1)
Fe(3)	32(1)	31(1)	54(1)	4(1)	0(1)	2(1)
C(1)	45(2)	26(2)	55(3)	-2(2)	17(2)	-4(2)
O(1)	88(2)	47(2)	41(2)	-4(1)	24(2)	-10(2)
C(2)	57(3)	62(3)	61(3)	0(2)	29(2)	8(2)
O(2)	63(2)	111(3)	147(4)	-17(3)	60(2)	7(2)
C(3)	59(3)	41(3)	42(2)	3(2)	10(2)	-4(2)
O(3)	113(3)	39(2)	83(2)	12(2)	13(2)	-12(2)
C(4)	27(2)	27(2)	43(2)	0(2)	8(2)	0(1)
O(4)	46(2)	50(2)	33(2)	7(1)	11(1)	-3(1)
C(5)	38(2)	39(2)	37(2)	2(2)	13(2)	5(2)
O(5)	52(2)	39(2)	63(2)	-13(1)	17(1)	-7(1)
C(6)	43(2)	45(2)	36(2)	-5(2)	8(2)	1(2)
O(6)	40(2)	71(2)	49(2)	-10(1)	5(1)	17(1)
C(7)	83(3)	46(3)	52(3)	-5(2)	-6(3)	29(2)
O(7)	146(3)	105(3)	45(2)	2(2)	19(2)	74(3)
C(8)	54(2)	44(3)	39(2)	-6(2)	-4(2)	3(2)
O(8)	93(2)	51(2)	53(2)	5(2)	6(2)	33(2)
C(9)	55(2)	39(2)	43(2)	3(2)	0(2)	-1(2)
O(9)	79(2)	63(2)	99(3)	4(2)	-35(2)	-20(2)
C(10)	75(3)	27(2)	39(2)	3(2)	-4(2)	1(2)
C(11)	136(4)	45(3)	36(3)	10(2)	8(3)	8(3)
N(1)	34(2)	34(2)	26(2)	-1(1)	5(1)	2(1)
P(11)	27(1)	26(1)	29(1)	-1(1)	7(1)	-1(1)
P(12)	29(1)	28(1)	28(1)	1(1)	6(1)	1(1)
C(100)	46(2)	62(3)	42(2)	-2(2)	24(2)	-2(2)
C(101)	37(2)	37(2)	36(2)	0(2)	8(2)	-3(2)
C(102)	45(2)	50(3)	36(2)	-7(2)	3(2)	-9(2)
C(103)	50(2)	38(2)	65(3)	20(2)	8(2)	2(2)
C(104)	36(2)	56(3)	37(2)	4(2)	9(2)	5(2)
C(105)	28(2)	52(3)	39(2)	0(2)	6(2)	6(2)
C(106)	53(2)	31(2)	50(3)	4(2)	12(2)	-11(2)
C(107)	36(2)	51(3)	39(2)	-13(2)	1(2)	12(2)
C(108)	37(2)	40(2)	48(2)	-16(2)	9(2)	-9(2)
C(109)	35(2)	52(3)	68(3)	-10(2)	26(2)	-8(2)
C(110)	61(3)	28(2)	42(2)	1(2)	4(2)	-5(2)
C(111)	33(2)	40(2)	47(2)	-1(2)	9(2)	-7(2)
C(112)	41(2)	34(2)	34(2)	1(2)	5(2)	-7(2)
C(113)	46(2)	36(2)	57(3)	-11(2)	13(2)	-16(2)
C(114)	34(2)	34(2)	43(2)	-1(2)	10(2)	1(2)
C(115)	45(2)	47(2)	31(2)	-7(2)	-1(2)	-1(2)
C(116)	51(2)	44(2)	43(2)	17(2)	12(2)	-5(2)
C(117)	35(2)	37(2)	32(2)	-5(2)	8(2)	-1(2)
C(118)	45(2)	40(2)	35(2)	-5(2)	8(2)	-10(2)

 17

C(119)	38(2)	34(2)	70(3)	8(2)	12(2)	7(2)
C(120)	41(2)	38(2)	30(2)	3(2)	4(2)	-3(2)
C(121)	53(2)	36(2)	50(3)	-3(2)	18(2)	-15(2)
C(122)	55(2)	31(2)	40(2)	-7(2)	-4(2)	17(2)
C(123)	33(2)	38(2)	42(2)	4(2)	8(2)	-4(2)
C(124)	24(2)	52(3)	63(3)	-1(2)	7(2)	-6(2)
C(125)	37(2)	33(2)	39(2)	3(2)	11(2)	1(2)
C(126)	33(2)	32(2)	44(2)	-6(2)	6(2)	1(2)
C(127)	28(2)	40(2)	42(2)	-1(2)	2(2)	-7(2)
C(128)	37(2)	30(2)	38(2)	3(2)	3(2)	0(2)
C(129)	38(2)	38(2)	43(2)	-4(2)	7(2)	12(2)
C(130)	29(2)	31(2)	24(2)	0(2)	2(1)	2(2)
C(131)	30(2)	27(2)	33(2)	-4(2)	9(2)	0(1)
C(132)	28(2)	28(2)	24(2)	-2(1)	3(1)	0(1)
C(133)	26(2)	25(2)	35(2)	-1(2)	9(2)	2(1)
C(134)	29(2)	29(2)	31(2)	0(2)	7(2)	-4(1)
C(135)	22(2)	30(2)	32(2)	-1(2)	6(1)	2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for (PPN)[Fe3(CO)9(CCH)]-Ib.

	x	y	z	U(eq)
H(11A)	-1051	857	732	89
H(10A)	7005	-1252	-904	58
H(10B)	3608	-2198	2096	44
H(10C)	1004	-2139	-745	53
H(10D)	7121	-2724	5474	62
H(10E)	4936	-1236	-271	51
H(10F)	-1143	-1099	3583	48
H(10G)	2033	-2784	1531	54
H(10H)	165	-421	963	52
H(10I)	7304	240	4793	50
H(10J)	9309	-1411	-64	59
H(11B)	3776	171	411	54
H(11C)	-237	-1821	3431	48
H(11D)	2553	-1545	-184	44
H(11E)	757	-2757	113	55
H(11F)	6679	-2041	3241	44
H(11G)	6649	-314	5676	50
H(11H)	5368	-2265	5906	55
H(11I)	1516	-1079	1367	42
H(11J)	5873	-546	2716	48
H(11K)	7729	-2626	4133	57
H(12A)	5582	-979	5095	44
H(12B)	6924	125	3316	54
H(12C)	1305	200	488	52
H(12D)	5140	-479	821	45
H(12E)	9553	-1560	1392	56
H(12F)	4255	-1689	4995	43
H(12G)	2226	-1920	3342	44
H(12H)	7513	-1510	2045	45
H(12I)	2841	-573	3520	43
H(12J)	371	-477	3600	48

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Randal W. Eveland, Casey C. Raymond, Duward F. Shriver

Department of Chemistry

Northwestern University

Evanston, Illinois 60208-3113

X-ray Structure Report for [$Fe_3(CO)_9$ CCOTi$(THF)_4$Cl] (III)

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Table 1. Crystal data and structure refinement for Fe3(CO)9{CCOTi(THF)4Cl}-III

Identification code	s34g
Empirical formula	$C_{27}H_{32}ClFe_3O_{14}Ti$
Formula weight	831.43
Temperature	153(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	a = 10.5178(11) Å alpha = 90°
	b = 17.9033(17) Å beta = 102.915(2)°
	c = 18.4631(18) Å gamma = 90°
Volume, Z	3388.7(6) Å^3, 4
Density (calculated)	1.630 Mg/m^3
Absorption coefficient	1.628 mm^{-1}
F(000)	1692
Crystal size	0.375 x 0.300 x 0.025 mm
θ range for data collection	1.60 to 28.32°
Limiting indices	$-13 \le h \le 13$, $-23 \le k \le 22$, $-24 \le l \le 15$
Reflections collected	21692
Independent reflections	8004 (R_{int} = 0.0543)
Completeness to θ = 28.32°	95.0 %
Absorption correction	SADABS (Sheldrick, 1996)
Max. and min. transmission	Not reported and Not reported
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8004 / 0 / 415
Goodness-of-fit on F^2	0.850
Final R indices [I>2σ(I)]	R1 = 0.0447, wR2 = 0.0838
R indices (all data)	R1 = 0.0964, wR2 = 0.0948
Largest diff. peak and hole	0.623 and -0.330 eÅ^{-3}

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for Fe3(CO)9{CCOTi(THF)4Cl}-III. U one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Fe(1)	2862(1)	4266(1)	719(1)	29(1)
Fe(2)	4321(1)	4888(1)	1923(1)	28(1)
Fe(3)	3774(1)	3534(1)	1966(1)	26(1)
C(1)	3991(4)	4063(2)	142(2)	41(1)
O(1)	4698(3)	3920(2)	-223(2)	62(1)
C(2)	1652(4)	3607(2)	342(2)	35(1)
O(2)	838(3)	3180(2)	124(2)	53(1)
C(3)	2139(4)	5080(2)	241(2)	42(1)
O(3)	1625(3)	5596(2)	-58(2)	65(1)
C(4)	5664(4)	4737(2)	1493(2)	34(1)
O(4)	6539(3)	4648(2)	1237(2)	48(1)
C(5)	3916(4)	5831(2)	1653(2)	38(1)
O(5)	3669(3)	6441(2)	1490(2)	61(1)
C(6)	5170(3)	5100(2)	2842(2)	38(1)
O(6)	5646(3)	5246(2)	3448(2)	62(1)
C(7)	4710(3)	3076(2)	1406(2)	32(1)
O(7)	5342(2)	2736(1)	1089(2)	44(1)
C(8)	4891(4)	3421(2)	2831(2)	36(1)
O(8)	5618(3)	3344(2)	3397(2)	54(1)
C(9)	2635(4)	2806(2)	1988(2)	32(1)
O(9)	1916(3)	2328(1)	2016(2)	48(1)
C(10)	2546(3)	4409(2)	1637(2)	24(1)
C(11)	2821(3)	4432(2)	2362(2)	25(1)
O(10)	2356(2)	4554(1)	2952(1)	26(1)
Ti(1)	895(1)	4612(1)	3375(1)	24(1)
Cl(1)	-965(1)	4703(1)	3871(1)	39(1)
O(20)	-38(2)	3768(1)	2651(1)	33(1)
C(20)	-643(4)	3101(2)	2878(2)	52(1)
C(21)	-1700(5)	2909(3)	2248(2)	68(2)
C(22)	-1257(4)	3181(2)	1576(2)	41(1)
C(23)	-517(3)	3879(2)	1856(2)	35(1)
O(30)	1876(2)	5424(1)	4112(1)	29(1)
C(30)	1418(3)	6181(2)	4174(2)	36(1)
C(31)	2395(4)	6525(2)	4801(2)	45(1)
C(32)	3635(4)	6146(2)	4748(2)	48(1)
C(33)	3186(3)	5355(2)	4559(2)	47(1)
O(40)	1677(2)	3783(1)	4149(1)	29(1)
C(40)	1429(4)	3713(2)	4897(2)	39(1)
C(41)	2565(4)	3287(2)	5331(2)	40(1)
C(42)	2823(4)	2743(2)	4752(2)	46(1)
C(43)	2621(4)	3213(2)	4054(2)	36(1)
C(50)	-1216(4)	5814(2)	2460(2)	44(1)
O(50)	90(2)	5482(1)	2600(1)	29(1)
C(51)	-1319(4)	6246(2)	1743(2)	60(1)
C(52)	52(4)	6490(2)	1781(3)	64(1)

C(53)	833(4)	5840(2)	2121(2)	43(1)

Table 3. Bond lengths [Å] and angles [°] for Fe3(CO)9{CCOTi(THF)4Cl}-II

Fe(1)-C(2)	1.760(4)	Fe(1)-C(3)	1.784(4)
Fe(1)-C(1)	1.800(4)	Fe(1)-C(10)	1.818(3)
Fe(1)-Fe(3)	2.6360(7)	Fe(1)-Fe(2)	2.6490(7)
Fe(2)-C(6)	1.773(4)	Fe(2)-C(5)	1.785(4)
Fe(2)-C(4)	1.789(4)	Fe(2)-C(10)	2.015(3)
Fe(2)-C(11)	2.094(3)	Fe(2)-Fe(3)	2.4963(7)
Fe(3)-C(8)	1.770(4)	Fe(3)-C(9)	1.777(4)
Fe(3)-C(7)	1.779(4)	Fe(3)-C(10)	2.034(3)
Fe(3)-C(11)	2.109(3)	C(1)-O(1)	1.139(4)
C(2)-O(2)	1.152(4)	C(3)-O(3)	1.147(4)
C(4)-O(4)	1.137(4)	C(5)-O(5)	1.146(4)
C(6)-O(6)	1.150(4)	C(7)-O(7)	1.153(4)
C(8)-O(8)	1.157(4)	C(9)-O(9)	1.151(4)
C(10)-C(11)	1.304(4)	C(11)-O(10)	1.310(4)
O(10)-Ti(1)	1.875(2)	Ti(1)-O(40)	2.096(2)
Ti(1)-O(30)	2.099(2)	Ti(1)-O(20)	2.107(2)
Ti(1)-O(50)	2.155(2)	Ti(1)-Cl(1)	2.3436(11)
O(20)-C(23)	1.456(4)	O(20)-C(20)	1.458(4)
C(20)-C(21)	1.460(5)	C(21)-C(22)	1.501(5)
C(22)-C(23)	1.503(5)	O(30)-C(33)	1.444(4)
O(30)-C(30)	1.451(4)	C(30)-C(31)	1.498(5)
C(31)-C(32)	1.493(5)	C(32)-C(33)	1.508(5)
O(40)-C(43)	1.462(4)	O(40)-C(40)	1.468(4)
C(40)-C(41)	1.491(5)	C(41)-C(42)	1.515(5)
C(42)-C(43)	1.513(5)	C(50)-O(50)	1.466(4)
C(50)-C(51)	1.516(5)	O(50)-C(53)	1.455(4)
C(51)-C(52)	1.493(6)	C(52)-C(53)	1.481(5)
C(2)-Fe(1)-C(3)	99.29(17)	C(2)-Fe(1)-C(1)	98.38(17)
C(3)-Fe(1)-C(1)	97.92(17)	C(2)-Fe(1)-C(10)	101.28(16)
C(3)-Fe(1)-C(10)	101.47(16)	C(1)-Fe(1)-C(10)	149.61(15)
C(2)-Fe(1)-Fe(3)	95.86(11)	C(3)-Fe(1)-Fe(3)	150.36(12)
C(1)-Fe(1)-Fe(3)	104.90(11)	C(10)-Fe(1)-Fe(3)	50.33(10)
C(2)-Fe(1)-Fe(2)	147.72(12)	C(3)-Fe(1)-Fe(2)	100.01(12)
C(1)-Fe(1)-Fe(2)	104.27(12)	C(10)-Fe(1)-Fe(2)	49.46(10)
Fe(3)-Fe(1)-Fe(2)	56.370(18)	C(6)-Fe(2)-C(5)	95.91(17)
C(6)-Fe(2)-C(4)	100.30(16)	C(5)-Fe(2)-C(4)	100.46(17)
C(6)-Fe(2)-C(10)	125.49(16)	C(5)-Fe(2)-C(10)	100.39(15)
C(4)-Fe(2)-C(10)	126.63(15)	C(6)-Fe(2)-C(11)	88.59(15)
C(5)-Fe(2)-C(11)	108.88(15)	C(4)-Fe(2)-C(11)	148.31(15)
C(10)-Fe(2)-C(11)	36.93(12)	C(6)-Fe(2)-Fe(3)	104.35(12)
C(5)-Fe(2)-Fe(3)	152.23(12)	C(4)-Fe(2)-Fe(3)	94.47(12)
C(10)-Fe(2)-Fe(3)	52.27(9)	C(11)-Fe(2)-Fe(3)	53.84(9)
C(6)-Fe(2)-Fe(1)	165.20(12)	C(5)-Fe(2)-Fe(1)	96.08(12)
C(4)-Fe(2)-Fe(1)	86.00(11)	C(10)-Fe(2)-Fe(1)	43.28(10)
C(11)-Fe(2)-Fe(1)	79.34(9)	Fe(3)-Fe(2)-Fe(1)	61.551(19)
C(8)-Fe(3)-C(9)	102.11(16)	C(8)-Fe(3)-C(7)	97.46(16)
C(9)-Fe(3)-C(7)	97.96(16)	C(8)-Fe(3)-C(10)	127.36(15)
C(9)-Fe(3)-C(10)	100.64(14)	C(7)-Fe(3)-C(10)	125.34(15)
C(8)-Fe(3)-C(11)	92.65(15)	C(9)-Fe(3)-C(11)	100.12(14)
C(7)-Fe(3)-C(11)	156.93(15)	C(10)-Fe(3)-C(11)	36.63(12)
C(8)-Fe(3)-Fe(2)	91.66(12)	C(9)-Fe(3)-Fe(2)	150.95(11)
C(7)-Fe(3)-Fe(2)	105.54(11)	C(10)-Fe(3)-Fe(2)	51.61(9)
C(11)-Fe(3)-Fe(2)	53.28(9)	C(8)-Fe(3)-Fe(1)	152.10(12)
C(9)-Fe(3)-Fe(1)	105.58(11)	C(7)-Fe(3)-Fe(1)	82.04(11)

C(10)-Fe(3)-Fe(1)	43.49(10)	C(11)-Fe(3)-Fe(1)	79.39(9)
Fe(2)-Fe(3)-Fe(1)	62.078(19)	O(1)-C(1)-Fe(1)	178.7(4)
O(2)-C(2)-Fe(1)	176.9(4)	O(3)-C(3)-Fe(1)	177.1(4)
O(4)-C(4)-Fe(2)	178.2(3)	O(5)-C(5)-Fe(2)	178.9(4)
O(6)-C(6)-Fe(2)	175.7(4)	O(7)-C(7)-Fe(3)	174.3(3)
O(8)-C(8)-Fe(3)	179.7(4)	O(9)-C(9)-Fe(3)	178.4(3)
C(11)-C(10)-Fe(1)	156.4(3)	C(11)-C(10)-Fe(2)	74.8(2)
Fe(1)-C(10)-Fe(2)	87.26(14)	C(11)-C(10)-Fe(3)	74.8(2)
Fe(1)-C(10)-Fe(3)	86.18(13)	Fe(2)-C(10)-Fe(3)	76.12(11)
C(10)-C(11)-O(10)	145.2(3)	C(10)-C(11)-Fe(2)	68.3(2)
O(10)-C(11)-Fe(2)	135.3(2)	C(10)-C(11)-Fe(3)	68.5(2)
O(10)-C(11)-Fe(3)	135.3(2)	Fe(2)-C(11)-Fe(3)	72.88(11)
C(11)-O(10)-Ti(1)	148.0(2)	O(10)-Ti(1)-O(40)	90.64(9)
O(10)-Ti(1)-O(30)	88.74(9)	O(40)-Ti(1)-O(30)	89.25(9)
O(10)-Ti(1)-O(20)	90.34(9)	O(40)-Ti(1)-O(20)	88.92(9)
O(30)-Ti(1)-O(20)	177.94(9)	O(10)-Ti(1)-O(50)	90.08(9)
O(40)-Ti(1)-O(50)	178.63(9)	O(30)-Ti(1)-O(50)	89.60(9)
O(20)-Ti(1)-O(50)	92.24(9)	O(10)-Ti(1)-Cl(1)	178.30(7)
O(40)-Ti(1)-Cl(1)	91.00(7)	O(30)-Ti(1)-Cl(1)	91.75(7)
O(20)-Ti(1)-Cl(1)	89.23(7)	O(50)-Ti(1)-Cl(1)	88.29(7)
C(23)-O(20)-C(20)	109.1(2)	C(23)-O(20)-Ti(1)	123.26(19)
C(20)-O(20)-Ti(1)	125.2(2)	O(20)-C(20)-C(21)	105.5(3)
C(20)-C(21)-C(22)	105.1(3)	C(21)-C(22)-C(23)	102.6(3)
O(20)-C(23)-C(22)	105.6(3)	C(33)-O(30)-C(30)	109.0(2)
C(33)-O(30)-Ti(1)	125.4(2)	C(30)-O(30)-Ti(1)	125.05(19)
O(30)-C(30)-C(31)	105.2(3)	C(32)-C(31)-C(30)	102.7(3)
C(31)-C(32)-C(33)	102.1(3)	O(30)-C(33)-C(32)	105.2(3)
C(43)-O(40)-C(40)	108.8(2)	C(43)-O(40)-Ti(1)	125.88(19)
C(40)-O(40)-Ti(1)	125.23(19)	O(40)-C(40)-C(41)	105.0(3)
C(40)-C(41)-C(42)	101.4(3)	C(43)-C(42)-C(41)	103.3(3)
O(40)-C(43)-C(42)	104.8(3)	O(50)-C(50)-C(51)	104.1(3)
C(53)-O(50)-C(50)	108.8(3)	C(53)-O(50)-Ti(1)	122.50(19)
C(50)-O(50)-Ti(1)	128.7(2)	C(52)-C(51)-C(50)	103.1(3)
C(53)-C(52)-C(51)	103.3(3)	O(50)-C(53)-C(52)	106.4(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [$Å^2 \times 10^3$] for $Fe3(CO)9\{CCOTi(THF)4Cl\}$-III.
The anisotropic displacement factor exponent t $-2\pi^2$ [$(ha^*)^2U_{11}$ + ... + $2hka^*b^*U_{12}$]

	U11	U22	U33	U23	U13	U12
Fe(1)	35(1)	33(1)	19(1)	1(1)	7(1)	3(1)
Fe(2)	31(1)	28(1)	26(1)	0(1)	8(1)	-3(1)
Fe(3)	29(1)	26(1)	22(1)	0(1)	6(1)	3(1)
C(1)	50(3)	52(3)	21(2)	6(2)	9(2)	9(2)
O(1)	65(2)	86(2)	44(2)	12(2)	31(2)	20(2)
C(2)	37(2)	46(2)	22(2)	3(2)	4(2)	10(2)
O(2)	46(2)	55(2)	52(2)	-9(2)	-2(2)	-9(2)
C(3)	52(3)	51(3)	26(2)	-1(2)	14(2)	5(2)
O(3)	91(2)	58(2)	47(2)	19(2)	19(2)	30(2)
C(4)	32(2)	37(2)	32(2)	4(2)	3(2)	-2(2)
O(4)	35(2)	63(2)	52(2)	0(2)	21(1)	-4(1)
C(5)	42(2)	35(2)	42(2)	-3(2)	20(2)	-6(2)
O(5)	94(2)	28(2)	67(2)	6(2)	34(2)	7(2)
C(6)	35(2)	44(2)	34(2)	-3(2)	4(2)	-9(2)
O(6)	58(2)	83(2)	40(2)	-20(2)	-1(2)	-12(2)
C(7)	32(2)	33(2)	28(2)	3(2)	1(2)	-3(2)
O(7)	44(2)	44(2)	48(2)	-9(1)	20(1)	7(1)
C(8)	39(2)	34(2)	35(2)	0(2)	8(2)	8(2)
O(8)	58(2)	61(2)	33(2)	4(1)	-11(2)	10(2)
C(9)	38(2)	31(2)	28(2)	2(2)	10(2)	12(2)
O(9)	49(2)	38(2)	62(2)	2(1)	20(2)	-7(1)
C(10)	22(2)	28(2)	24(2)	3(2)	6(2)	1(1)
C(11)	26(2)	23(2)	26(2)	3(2)	5(2)	1(1)
O(10)	33(1)	27(1)	18(1)	0(1)	9(1)	2(1)
Ti(1)	27(1)	25(1)	19(1)	-1(1)	4(1)	1(1)
Cl(1)	36(1)	43(1)	42(1)	0(1)	17(1)	4(1)
O(20)	41(2)	30(1)	23(1)	-2(1)	1(1)	-7(1)
C(20)	75(3)	36(2)	39(3)	2(2)	3(2)	-18(2)
C(21)	76(3)	80(3)	45(3)	-1(3)	5(3)	-50(3)
C(22)	46(2)	41(2)	31(2)	-12(2)	0(2)	-9(2)
C(23)	38(2)	41(2)	24(2)	-2(2)	4(2)	-7(2)
O(30)	33(1)	28(1)	23(1)	-6(1)	2(1)	-1(1)
C(30)	43(2)	25(2)	42(2)	-5(2)	12(2)	0(2)
C(31)	61(3)	31(2)	43(2)	-12(2)	8(2)	-13(2)
C(32)	42(2)	56(3)	41(2)	-10(2)	1(2)	-12(2)
C(33)	37(2)	51(3)	43(3)	-11(2)	-9(2)	5(2)
O(40)	39(1)	31(1)	21(1)	3(1)	12(1)	10(1)
C(40)	55(3)	41(2)	23(2)	5(2)	17(2)	8(2)
C(41)	56(3)	40(2)	23(2)	6(2)	9(2)	1(2)
C(42)	62(3)	35(2)	37(2)	9(2)	5(2)	16(2)
C(43)	43(2)	36(2)	29(2)	0(2)	11(2)	14(2)
C(50)	40(2)	47(2)	41(2)	-1(2)	1(2)	17(2)
O(50)	32(1)	28(1)	27(1)	4(1)	6(1)	4(1)
C(51)	62(3)	51(3)	55(3)	19(2)	-12(2)	10(2)
C(52)	77(3)	50(3)	57(3)	19(2)	1(3)	5(2)

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C(53)	51(3)	40(2)	38(2)	13(2)	9(2)	5(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for Fe3(CO)9{CCOTi(THF)4Cl}-III.

	x	y	z	U(eq)
H(20A)	-988	3204	3325	62
H(20B)	-3	2688	2991	62
H(21A)	-2521	3159	2289	82
H(21B)	-1846	2362	2222	82
H(22A)	-2009	3291	1161	49
H(22B)	-686	2810	1408	49
H(23A)	219	3954	1609	42
H(23B)	-1096	4321	1758	42
H(30A)	539	6178	4284	43
H(30B)	1378	6460	3706	43
H(31A)	2454	7072	4734	54
H(31B)	2172	6424	5285	54
H(32A)	4286	6169	5226	57
H(32B)	4012	6369	4353	57
H(33A)	3760	5103	4277	56
H(33B)	3190	5065	5016	56
H(40A)	605	3440	4882	46
H(40B)	1372	4212	5120	46
H(41A)	2341	3024	5757	47
H(41B)	3325	3617	5514	47
H(42A)	3725	2547	4889	55
H(42B)	2203	2320	4687	55
H(43A)	3450	3444	4003	43
H(43B)	2274	2905	3608	43
H(50A)	-1304	6151	2871	53
H(50B)	-1896	5423	2402	53
H(51A)	-1641	5924	1304	72
H(51B)	-1908	6681	1720	72
H(52A)	197	6596	1280	76
H(52C)	268	6941	2095	76
H(53C)	974	5490	1732	52
H(53A)	1693	6006	2414	52