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OM9505470

Table 1. Crystal data and structure refinement for 4.

Identification code	tor1
Empirical formula	$C_{31}H_{30}CrFe_2O_{11}P_2$
Formula weight	804.19
Crystal colour	red
Crystal size	0.40 x 0.30 x 0.10 mm
Temperature	173(2) K
Wavelength	0.71073 Å (graphite monochromator)
Space group	$P\bar{1}$
Unit cell dimensions	a = 9.365(3) Å α = 77.30(3)°
	b = 13.117(4) Å β = 74.15(3)°
	c = 14.947(5) Å γ = 76.80(3)°
Volume	1695.1(9) $Å^3$
Z	2
Density (calculated)	1.576 Mg/m^3
Absorption coefficient	1.311 mm^{-1}
F(000)	820
θ range for data collection	1.62 to 27.56°
Index ranges	$0 \le h \le 12$, $-16 \le k \le 17$, $-18 \le \ell \le 19$
Reflections collected	8307
Independent reflections	7830 (R_{int} = 0.0332)
Absorption correction	Semi-empirical from ψ-scans
Data / restraints / parameters	7818 / 0 / 434
Goodness-of-fit on F^2	1.088
Final R indices [$I>2\sigma(I)$]	R1 = 0.0502 for 5286 reflections, wR2 = 0.0991
R indices (all data)	R1 = 0.0930, wR2 = 0.1207
Largest and mean Δ/σ	-0.001 and 0.000
Largest diff. peak and hole	0.5 and -0.5 $eÅ^{-3}$

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Table 2. Measurement and program parameters for 4.

Diffractometer used	Siemens P2(1) diffractometer
Scan type	Omega-scan
Scan range	1.2^{o} in ω arround $K\alpha_{1,2}$- maximum
Scan speed	3.9 - 29.3 ^{o}/min in ω
Standard reflections	3 of 100 measured reflections
Programs used	Siemens SHELXTL plus / SHELXL-93
Structure solution	direct
Structure refinement	Full-matrix least-squares on F^2
Structure factor source	International Tables Vol.C

Definition of R- values: $R1 = \sum||F_o|-|F_c||/\sum|F_o|$

$$wR2 = [\ \sum[w(F_o^2-F_c^2)^2]/\sum[w(F_o^2)^2]\ \ ]^{1/2}$$

Definition of the weighting scheme (CIF format):

calc w=1/[\s^2^(Fo^2^)+(0.0438P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3

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Table 4. Selected bond lengths [Å] and angles [°] for 4.

Fe(1)-C(12)	1.762(4)	Fe(1)-C(11)	1.766(4)
Fe(1)-C(3)	2.102(4)	Fe(1)-C(5)	2.106(4)
Fe(1)-C(4)	2.112(4)	Fe(1)-C(2)	2.140(4)
Fe(1)-C(1)	2.153(4)	Fe(1)-P(1)	2.3057(13)
Fe(2)-C(26)	1.763(4)	Fe(2)-C(25)	1.786(4)
Fe(2)-C(15)	2.101(4)	Fe(2)-C(19)	2.104(4)
Fe(2)-C(18)	2.116(4)	Fe(2)-C(17)	2.117(4)
Fe(2)-C(16)	2.146(4)	Fe(2)-P(2)	2.2296(12)
Cr(1)-C(31)	1.856(4)	Cr(1)-C(30)	1.887(4)
Cr(1)-C(29)	1.902(4)	Cr(1)-C(28)	1.903(4)
Cr(1)-C(27)	1.906(4)	Cr(1)-P(1)	2.4197(14)
P(1)-C(14)	1.902(4)	P(1)-C(13)	1.917(4)
P(2)-C(14)	1.812(4)	P(2)-C(13)	1.815(4)
O(1)-C(11)	1.152(4)	O(2)-C(12)	1.146(4)
O(3)-C(13)	1.201(4)	O(4)-C(14)	1.213(4)
O(5)-C(25)	1.138(4)	O(6)-C(26)	1.148(5)
O(7)-C(27)	1.139(5)	O(8)-C(28)	1.143(5)
O(9)-C(29)	1.143(5)	O(10)-C(30)	1.147(4)
O(11)-C(31)	1.152(5)	C(1)-C(2)	1.421(5)
C(1)-C(5)	1.435(5)	C(1)-C(6)	1.494(5)
C(2)-C(3)	1.432(5)	C(2)-C(7)	1.495(5)
C(3)-C(4)	1.415(5)	C(3)-C(8)	1.502(5)
C(4)-C(5)	1.437(6)	C(4)-C(9)	1.500(5)
C(5)-C(10)	1.505(5)	C(15)-C(16)	1.421(5)
C(15)-C(19)	1.442(5)	C(15)-C(20)	1.508(5)
C(16)-C(17)	1.434(5)	C(16)-C(21)	1.504(5)
C(17)-C(18)	1.444(6)	C(17)-C(22)	1.495(6)
C(18)-C(19)	1.431(6)	C(18)-C(23)	1.498(5)
C(19)-C(24)	1.497(5)		

C(12)-Fe(1)-C(11)	95.4(2)	C(12)-Fe(1)-C(3)	113.7(2)
C(11)-Fe(1)-C(3)	94.9(2)	C(12)-Fe(1)-C(5)	97.8(2)
C(11)-Fe(1)-C(5)	160.4(2)	C(3)-Fe(1)-C(5)	66.6(2)
C(12)-Fe(1)-C(4)	86.8(2)	C(11)-Fe(1)-C(4)	127.2(2)
C(3)-Fe(1)-C(4)	39.2(2)	C(5)-Fe(1)-C(4)	39.8(2)
C(12)-Fe(1)-C(2)	151.6(2)	C(11)-Fe(1)-C(2)	96.1(2)
C(3)-Fe(1)-C(2)	39.4(2)	C(5)-Fe(1)-C(2)	65.82(14)
C(4)-Fe(1)-C(2)	65.6(2)	C(12)-Fe(1)-C(1)	135.9(2)
C(11)-Fe(1)-C(1)	128.6(2)	C(3)-Fe(1)-C(1)	65.9(2)
C(5)-Fe(1)-C(1)	39.37(14)	C(4)-Fe(1)-C(1)	65.8(2)
C(2)-Fe(1)-C(1)	38.66(14)	C(12)-Fe(1)-P(1)	89.89(13)
C(11)-Fe(1)-P(1)	88.98(13)	C(3)-Fe(1)-P(1)	155.54(11)
C(5)-Fe(1)-P(1)	105.39(12)	C(4)-Fe(1)-P(1)	143.78(12)
C(2)-Fe(1)-P(1)	116.15(11)	C(1)-Fe(1)-P(1)	93.00(11)
C(26)-Fe(2)-C(25)	94.0(2)	C(26)-Fe(2)-C(15)	92.0(2)
C(25)-Fe(2)-C(15)	156.0(2)	C(26)-Fe(2)-C(19)	92.0(2)
C(25)-Fe(2)-C(19)	116.4(2)	C(15)-Fe(2)-C(19)	40.11(14)
C(26)-Fe(2)-C(18)	125.9(2)	C(25)-Fe(2)-C(18)	91.2(2)
C(15)-Fe(2)-C(18)	66.7(2)	C(19)-Fe(2)-C(18)	39.6(2)
C(26)-Fe(2)-C(17)	157.3(2)	C(25)-Fe(2)-C(17)	102.8(2)
C(15)-Fe(2)-C(17)	66.6(2)	C(19)-Fe(2)-C(17)	67.0(2)
C(18)-Fe(2)-C(17)	39.9(2)	C(26)-Fe(2)-C(16)	125.4(2)
C(25)-Fe(2)-C(16)	140.5(2)	C(15)-Fe(2)-C(16)	39.1(2)
C(19)-Fe(2)-C(16)	66.2(2)	C(18)-Fe(2)-C(16)	66.0(2)
C(17)-Fe(2)-C(16)	39.31(14)	C(26)-Fe(2)-P(2)	94.74(13)

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C(25)-Fe(2)-P(2)	90.60(13)	C(15)-Fe(2)-P(2)	112.03(11)
C(19)-Fe(2)-P(2)	151.64(11)	C(18)-Fe(2)-P(2)	139.09(13)
C(17)-Fe(2)-P(2)	100.17(11)	C(16)-Fe(2)-P(2)	87.72(10)
C(31)-Cr(1)-C(30)	87.6(2)	C(31)-Cr(1)-C(29)	90.5(2)
C(30)-Cr(1)-C(29)	87.0(2)	C(31)-Cr(1)-C(28)	88.1(2)
C(30)-Cr(1)-C(28)	175.7(2)	C(29)-Cr(1)-C(28)	92.7(2)
C(31)-Cr(1)-C(27)	92.3(2)	C(30)-Cr(1)-C(27)	90.4(2)
C(29)-Cr(1)-C(27)	176.1(2)	C(28)-Cr(1)-C(27)	90.1(2)
C(31)-Cr(1)-P(1)	175.71(14)	C(30)-Cr(1)-P(1)	95.25(12)
C(29)-Cr(1)-P(1)	86.43(13)	C(28)-Cr(1)-P(1)	89.02(12)
C(27)-Cr(1)-P(1)	90.86(13)	C(14)-P(1)-C(13)	79.1(2)
C(14)-P(1)-Fe(1)	109.14(12)	C(13)-P(1)-Fe(1)	115.24(13)
C(14)-P(1)-Cr(1)	106.56(12)	C(13)-P(1)-Cr(1)	111.63(12)
Fe(1)-P(1)-Cr(1)	124.90(5)	C(14)-P(2)-C(13)	84.3(2)
C(14)-P(2)-Fe(2)	130.19(13)	C(13)-P(2)-Fe(2)	130.13(13)
C(2)-C(1)-C(5)	107.8(3)	C(2)-C(1)-C(6)	124.9(3)
C(5)-C(1)-C(6)	127.0(4)	C(2)-C(1)-Fe(1)	70.2(2)
C(5)-C(1)-Fe(1)	68.6(2)	C(6)-C(1)-Fe(1)	131.8(3)
C(1)-C(2)-C(3)	108.3(3)	C(1)-C(2)-C(7)	125.8(4)
C(3)-C(2)-C(7)	125.2(4)	C(1)-C(2)-Fe(1)	71.1(2)
C(3)-C(2)-Fe(1)	68.8(2)	C(7)-C(2)-Fe(1)	133.1(3)
C(4)-C(3)-C(2)	108.0(3)	C(4)-C(3)-C(8)	126.1(4)
C(2)-C(3)-C(8)	125.6(4)	C(4)-C(3)-Fe(1)	70.8(2)
C(2)-C(3)-Fe(1)	71.7(2)	C(8)-C(3)-Fe(1)	127.8(3)
C(3)-C(4)-C(5)	108.2(3)	C(3)-C(4)-C(9)	126.6(4)
C(5)-C(4)-C(9)	125.0(4)	C(3)-C(4)-Fe(1)	70.0(2)
C(5)-C(4)-Fe(1)	69.9(2)	C(9)-C(4)-Fe(1)	129.7(3)
C(1)-C(5)-C(4)	107.6(3)	C(1)-C(5)-C(10)	126.8(4)
C(4)-C(5)-C(10)	125.3(4)	C(1)-C(5)-Fe(1)	72.1(2)
C(4)-C(5)-Fe(1)	70.3(2)	C(10)-C(5)-Fe(1)	128.5(3)
O(1)-C(11)-Fe(1)	178.0(3)	O(2)-C(12)-Fe(1)	176.4(3)
O(3)-C(13)-P(2)	132.5(3)	O(3)-C(13)-P(1)	130.7(3)
P(2)-C(13)-P(1)	96.7(2)	O(4)-C(14)-P(2)	132.9(3)
O(4)-C(14)-P(1)	129.7(3)	P(2)-C(14)-P(1)	97.3(2)
C(16)-C(15)-C(19)	108.2(3)	C(16)-C(15)-C(20)	125.8(4)
C(19)-C(15)-C(20)	125.6(4)	C(16)-C(15)-Fe(2)	72.2(2)
C(19)-C(15)-Fe(2)	70.1(2)	C(20)-C(15)-Fe(2)	128.2(3)
C(15)-C(16)-C(17)	108.5(3)	C(15)-C(16)-C(21)	125.5(4)
C(17)-C(16)-C(21)	125.9(4)	C(15)-C(16)-Fe(2)	68.8(2)
C(17)-C(16)-Fe(2)	69.3(2)	C(21)-C(16)-Fe(2)	130.7(3)
C(16)-C(17)-C(18)	107.4(3)	C(16)-C(17)-C(22)	125.7(4)
C(18)-C(17)-C(22)	126.8(4)	C(16)-C(17)-Fe(2)	71.4(2)
C(18)-C(17)-Fe(2)	70.0(2)	C(22)-C(17)-Fe(2)	127.5(3)
C(19)-C(18)-C(17)	108.2(3)	C(19)-C(18)-C(23)	125.3(4)
C(17)-C(18)-C(23)	126.4(4)	C(19)-C(18)-Fe(2)	69.8(2)
C(17)-C(18)-Fe(2)	70.1(2)	C(23)-C(18)-Fe(2)	128.3(3)
C(18)-C(19)-C(15)	107.6(3)	C(18)-C(19)-C(24)	126.5(4)
C(15)-C(19)-C(24)	125.8(4)	C(18)-C(19)-Fe(2)	70.6(2)
C(15)-C(19)-Fe(2)	69.8(2)	C(24)-C(19)-Fe(2)	127.7(3)
O(5)-C(25)-Fe(2)	177.2(4)	O(6)-C(26)-Fe(2)	174.9(3)
O(7)-C(27)-Cr(1)	177.4(4)	O(8)-C(28)-Cr(1)	176.7(4)
O(9)-C(29)-Cr(1)	175.5(3)	O(10)-C(30)-Cr(1)	174.7(4)
O(11)-C(31)-Cr(1)	178.8(4)		

Symmetry transformations used to generate equivalent atoms:

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Table 5. Anisotropic displacement [a)] parameters [$Å^2 \times 10^3$] for 4.

	U11	U22	U33	U23	U13	U12
Fe(1)	21(1)	19(1)	23(1)	-4(1)	-3(1)	-3(1)
Fe(2)	29(1)	17(1)	23(1)	-4(1)	-3(1)	-4(1)
Cr(1)	28(1)	19(1)	28(1)	-6(1)	-7(1)	-2(1)
P(1)	24(1)	17(1)	22(1)	-4(1)	-2(1)	-3(1)
P(2)	35(1)	19(1)	27(1)	-5(1)	-4(1)	-7(1)
O(1)	32(2)	47(2)	37(2)	-21(1)	1(1)	-1(1)
O(2)	49(2)	24(1)	37(2)	4(1)	-14(1)	-12(1)
O(3)	45(2)	36(2)	32(2)	-10(1)	13(1)	-17(1)
O(4)	33(2)	33(2)	39(2)	-5(1)	5(1)	-11(1)
O(5)	46(2)	55(2)	34(2)	-2(2)	3(2)	-20(2)
O(6)	39(2)	36(2)	45(2)	-10(1)	-11(2)	4(1)
O(7)	58(2)	59(2)	32(2)	6(2)	-6(2)	-16(2)
O(8)	64(2)	38(2)	45(2)	-15(2)	-18(2)	-12(2)
O(9)	31(2)	59(2)	47(2)	-20(2)	5(2)	-16(2)
O(10)	63(2)	32(2)	40(2)	-9(1)	-18(2)	-10(2)
O(11)	53(2)	40(2)	78(3)	-15(2)	-31(2)	13(2)
C(1)	20(2)	25(2)	32(2)	-2(2)	-3(2)	-11(2)
C(2)	22(2)	22(2)	37(2)	-7(2)	-5(2)	-6(2)
C(3)	23(2)	37(2)	32(2)	-10(2)	-2(2)	-14(2)
C(4)	22(2)	30(2)	40(2)	1(2)	-7(2)	-8(2)
C(5)	19(2)	25(2)	38(2)	-7(2)	-1(2)	-4(2)
C(6)	39(3)	38(2)	34(2)	4(2)	-7(2)	-15(2)
C(7)	35(2)	23(2)	59(3)	-10(2)	-5(2)	-5(2)
C(8)	41(3)	60(3)	37(3)	-16(2)	-3(2)	-25(2)
C(9)	34(3)	44(3)	63(3)	13(2)	-21(2)	-12(2)
C(10)	31(2)	34(2)	60(3)	-19(2)	8(2)	-6(2)
C(11)	33(2)	27(2)	24(2)	-5(2)	-9(2)	-9(2)
C(12)	27(2)	28(2)	23(2)	-3(2)	-6(2)	-3(2)
C(13)	29(2)	23(2)	27(2)	-8(2)	-5(2)	-4(2)
C(14)	29(2)	20(2)	27(2)	0(2)	-10(2)	-5(2)
C(15)	35(2)	21(2)	25(2)	-1(2)	-5(2)	-7(2)
C(16)	39(2)	16(2)	24(2)	0(2)	-4(2)	-6(2)
C(17)	33(2)	17(2)	32(2)	2(2)	-9(2)	-1(2)
C(18)	42(2)	16(2)	32(2)	-6(2)	1(2)	-3(2)
C(19)	44(2)	16(2)	26(2)	-4(2)	-5(2)	-9(2)
C(20)	37(3)	44(2)	34(2)	-7(2)	3(2)	-10(2)
C(21)	61(3)	32(2)	29(2)	-2(2)	-15(2)	-14(2)
C(22)	39(3)	36(2)	54(3)	2(2)	-11(2)	-1(2)
C(23)	58(3)	30(2)	37(3)	-12(2)	9(2)	-1(2)
C(24)	60(3)	27(2)	34(2)	-4(2)	-13(2)	-18(2)
C(25)	33(2)	29(2)	31(2)	-3(2)	-8(2)	-7(2)
C(26)	39(2)	21(2)	27(2)	-6(2)	0(2)	-5(2)
C(27)	37(2)	28(2)	35(2)	-3(2)	-13(2)	-4(2)
C(28)	38(2)	27(2)	28(2)	-2(2)	-10(2)	-4(2)
C(29)	25(2)	29(2)	42(3)	-17(2)	-6(2)	-2(2)
C(30)	37(2)	21(2)	27(2)	-1(2)	-9(2)	-3(2)
C(31)	41(3)	28(2)	45(3)	-10(2)	-16(2)	-1(2)

a) The anisotropic displacement factor exponent takes the form:

$$-2\pi^2 [(ha^*)^2 U_{11} + \ldots + 2hka^* b^* U_{12}]$$

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Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters [$Å^2$ x 10^3] for 4.

	x	y	z	U(eq)
H(6A)	5435(14)	9295(19)	6390(6)	56
H(6B)	3636(18)	9632(14)	6616(3)	56
H(6C)	4420(31)	8500(7)	6317(5)	56
H(7A)	3006(29)	10592(4)	7785(11)	60
H(7B)	4239(9)	10639(4)	8327(20)	60
H(7C)	2574(22)	10477(3)	8910(10)	60
H(8A)	4689(15)	9012(22)	10273(8)	64
H(8B)	3694(33)	8101(6)	10723(3)	64
H(8C)	2915(19)	9272(17)	10329(9)	64
H(9A)	4903(22)	6487(13)	10319(4)	71
H(9B)	6531(10)	6473(13)	9610(16)	71
H(9C)	5379(31)	5817(3)	9476(14)	71
H(10A)	5269(23)	5965(8)	7780(12)	65
H(10B)	6800(8)	6433(16)	7478(17)	65
H(10C)	5593(28)	6821(9)	6839(7)	65
H(20A)	-1977(5)	3738(22)	7670(4)	60
H(20B)	-1507(9)	4179(15)	8445(16)	60
H(20C)	-1525(9)	2939(9)	8567(14)	60
H(21A)	2358(18)	4055(20)	8797(5)	59
H(21B)	1554(32)	3055(4)	9302(5)	59
H(21C)	557(15)	4196(18)	9051(9)	59
H(22A)	5169(7)	3180(24)	6631(4)	68
H(22B)	4811(13)	2503(12)	7671(17)	68
H(22C)	4524(8)	3772(13)	7527(20)	68
H(23A)	3754(27)	1696(4)	5757(7)	69
H(23B)	4433(15)	2769(18)	5423(13)	69
H(23C)	3006(14)	2667(20)	5077(7)	69
H(24A)	-1047(13)	3267(14)	6189(16)	58
H(24B)	-271(27)	2043(8)	6368(13)	58
H(24C)	451(14)	2827(22)	5459(4)	58

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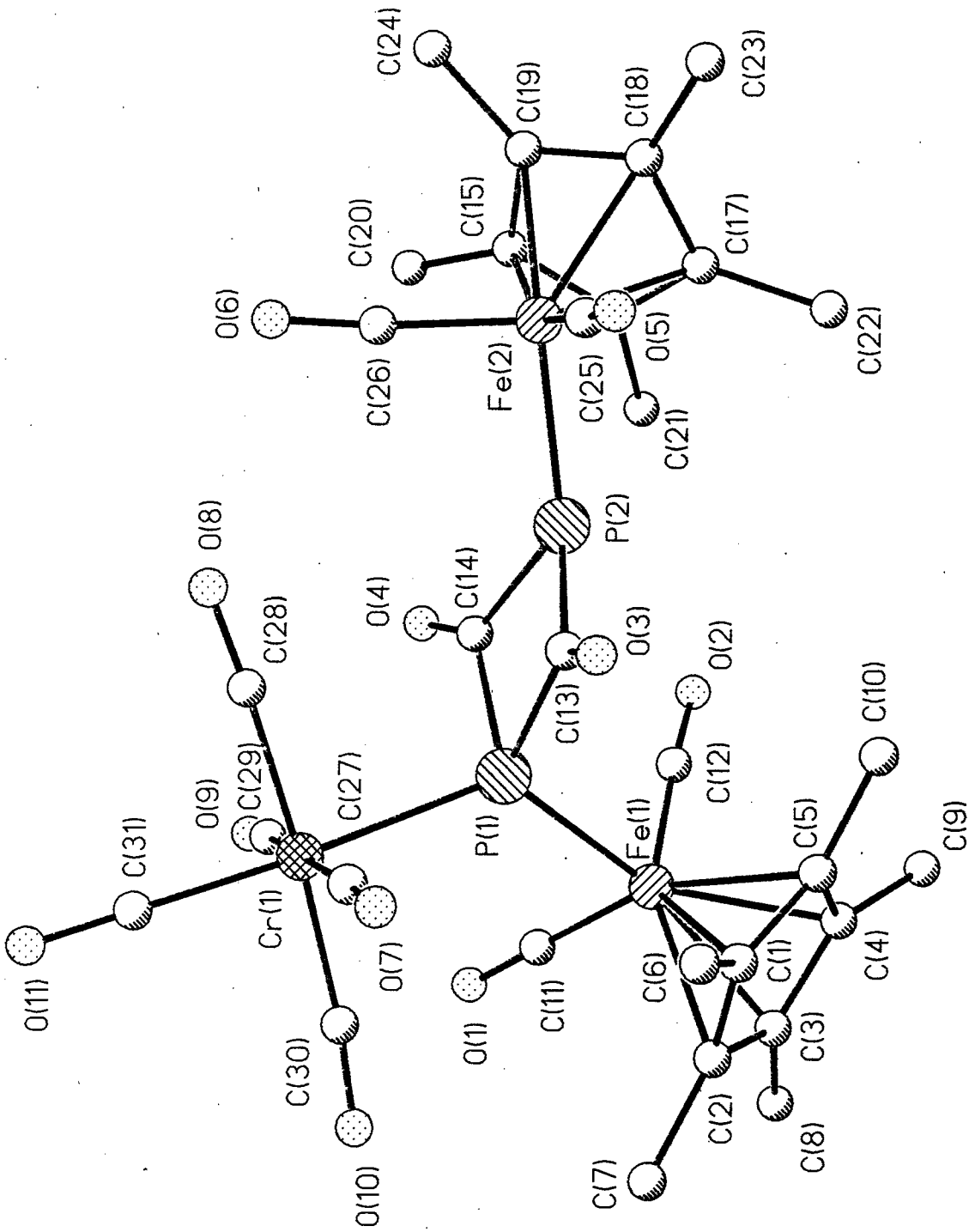
C(24)
C(19)
C(18)
C(23)
C(20)
C(15)
C(17)
O(6)
O(5)
C(22)
C(26)
Fe(2)
C(25)
C(21)
P(2)
O(8)
C(14)
O(4)
C(28)
O(3)
O(2)
C(13)
C(10)
C(12)
O(9)
C(29)
C(27)
P(1)
Fe(1)
C(5)
C(31)
C(9)
Cr(1)
O(7)
C(11)
C(6)
C(1)
C(4)
O(11)
O(1)
C(3)
C(30)
C(2)
C(8)
C(7)
O(10)