

Table 1. Crystal data and structure refinement for **3a**.

Empirical formula	$\text{C}_{60}\text{H}_{61}\text{NCIF}_6\text{P}_5\text{Ru}$	
Formula weight	1202.48	
Temperature	183(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	$a = 13.167(3)$ Å	alpha = 90 deg.
	$b = 21.127(4)$ Å	beta = 96.94(3) deg.
	$c = 20.644(4)$ Å	gamma = 90 deg.
Volume	$5701(2)$ Å ³	
Z	4	
Density (calculated)	1.401 Mg/m ³	
Absorption coefficient	0.521 mm ⁻¹	
F(000)	2476	
Crystal size	0.35 x 0.25 x 0.2 mm	
Theta range for data collection	1.74 to 23.00 deg.	
Index ranges	$-15 \leq h \leq 15$, $-13 \leq k \leq 25$, $-24 \leq l \leq 24$	
Reflections collected	9685	
Independent reflections	7910 [R(int) = 0.0514]	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	7698 / 0 / 608	
Goodness-of-fit on F ²	3.932	
Final R indices [I > 2sigma(I)]	R1 = 0.1832, wR2 = 0.3055	
R indices (all data)	R1 = 0.2092, wR2 = 0.4194	
Largest diff. peak and hole	1.508 and -2.199 e·Å ⁻³	

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

	x	y	z	U(eq)
Ru	9094(1)	2368(1)	2351(1)	34(1)
Cl	10035(4)	2184(2)	3439(2)	49(1)
C(1)	8405(13)	2541(8)	1440(8)	34(5)
C(2)	8087(14)	2654(9)	923(9)	41(5)
C(3)	7452(17)	2730(8)	270(8)	41(5)
C(4)	6508(19)	2488(11)	124(12)	88(9)
N(5)	7970(14)	3093(7)	-199(6)	51(5)
C(511)	7939(32)	3787(11)	-91(13)	140(17)
C(512)	6665(23)	3995(12)	-86(16)	131(15)
C(521)	9133(16)	2988(14)	-132(11)	92(10)
C(522)	9780(20)	3262(18)	-616(12)	154(17)
C(531)	7608(17)	2926(11)	-907(9)	66(7)
C(532)	7784(28)	2261(15)	-1055(15)	153(16)
P(1)	7742(4)	2657(3)	2963(2)	46(2)
C(11)	7570(15)	1838(9)	3262(9)	55(6)
P(2)	8137(4)	1460(2)	2578(2)	40(1)
C(111)	6425(13)	2865(8)	2603(8)	31(5)
C(112)	5664(16)	2949(10)	3006(10)	60(7)
C(113)	4663(18)	3047(11)	2711(13)	76(8)
C(114)	4426(17)	3059(8)	2100(12)	54(6)
C(115)	5168(16)	2947(9)	1675(10)	48(5)
C(116)	6121(15)	2855(8)	1924(10)	44(5)
C(121)	7932(15)	3177(10)	3656(9)	45(5)
C(122)	7800(16)	3841(13)	3560(9)	63(7)
C(123)	8007(19)	4244(14)	4098(12)	89(9)
C(124)	8382(22)	4039(18)	4640(12)	99(11)
C(125)	8550(20)	3426(18)	4786(13)	97(11)
C(126)	8333(18)	2961(12)	4281(8)	72(8)
C(211)	7047(15)	1168(10)	2069(10)	51(6)
C(212)	6922(16)	1268(9)	1390(9)	54(6)
C(213)	6071(17)	1020(11)	1010(10)	66(7)
C(214)	5401(20)	608(10)	1265(12)	77(8)
C(215)	5549(18)	481(14)	1949(11)	85(9)
C(216)	6359(16)	744(11)	2281(10)	62(7)
C(221)	8748(15)	733(9)	2906(9)	45(5)
C(222)	8841(15)	238(10)	2468(9)	52(6)
C(223)	9436(19)	-318(12)	2648(11)	83(8)
C(224)	9857(18)	-354(12)	3286(11)	70(7)
C(225)	9719(15)	131(10)	3750(11)	63(7)
C(226)	9178(15)	675(10)	3559(10)	56(6)
P(3)	10490(4)	2053(2)	1829(2)	31(1)
C(31)	10922(14)	2860(8)	1685(8)	39(5)

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P(4)	10064(4)	3254(2)	2195(2)	31(1)
C(311)	11603(15)	1625(10)	2208(10)	48(6)
C(312)	12560(16)	1727(10)	2100(9)	52(6)
C(313)	13396(18)	1372(11)	2371(12)	75(8)
C(314)	13218(18)	884(11)	2759(12)	64(7)
C(315)	12234(18)	707(9)	2889(11)	57(6)
C(316)	11421(16)	1115(8)	2616(9)	42(5)
C(321)	10317(13)	1651(8)	1013(9)	32(4)
C(322)	10941(16)	1766(11)	567(10)	63(7)
C(323)	10830(18)	1462(13)	-37(11)	75(8)
C(324)	10059(21)	1014(14)	-161(13)	87(9)
C(325)	9434(21)	885(12)	331(13)	89(9)
C(326)	9550(18)	1225(10)	895(10)	59(6)
C(411)	10924(13)	3661(8)	2828(7)	30(4)
C(412)	11808(15)	3932(9)	2724(9)	48(5)
C(413)	12419(17)	4346(10)	3198(10)	62(6)
C(414)	12071(17)	4425(10)	3772(10)	61(6)
C(415)	11174(16)	4121(10)	3923(10)	58(6)
C(416)	10572(16)	3761(9)	3446(9)	53(6)
C(421)	9518(15)	3942(8)	1717(7)	32(5)
C(422)	8490(13)	4083(8)	1763(8)	33(5)
C(423)	8093(21)	4634(10)	1437(11)	72(8)
C(424)	8699(24)	4989(10)	1075(11)	68(9)
C(425)	9681(21)	4826(11)	1029(10)	67(7)
C(426)	10102(16)	4306(9)	1369(8)	43(5)
P	2702(5)	3798(3)	406(2)	51(2)
F(1)	2763(11)	3922(6)	1165(5)	85(4)
F(2)	2594(10)	3690(7)	-362(5)	88(5)
F(3)	1594(13)	3701(16)	375(8)	205(14)
F(4)	3818(13)	3832(16)	438(7)	200(13)
F(5)	2830(26)	3117(9)	517(10)	211(13)
F(6)	2565(33)	4466(8)	272(7)	271(21)

Table 3. Bond lengths [Å] and angles [deg] for **3a**.

Ru-C(1)	2.02(2)
Ru-P(4)	2.310(5)
Ru-P(3)	2.337(5)
Ru-P(2)	2.373(5)
Ru-P(1)	2.383(5)
Ru-Cl	2.460(5)
C(1)-C(2)	1.12(2)
C(2)-C(3)	1.51(2)
C(3)-C(4)	1.34(3)
C(3)-N(5)	1.47(2)
N(5)-C(511)	1.48(3)
N(5)-C(531)	1.52(2)
N(5)-C(521)	1.54(3)
C(511)-C(512)	1.73(4)
C(521)-C(522)	1.51(3)
C(531)-C(532)	1.46(3)
P(1)-C(121)	1.80(2)
P(1)-C(111)	1.85(2)
P(1)-C(11)	1.86(2)
C(11)-P(2)	1.86(2)
P(2)-C(211)	1.78(2)
P(2)-C(221)	1.82(2)
C(111)-C(112)	1.39(2)
C(111)-C(116)	1.41(2)
C(112)-C(113)	1.40(3)
C(113)-C(114)	1.26(3)
C(114)-C(115)	1.41(3)
C(115)-C(116)	1.31(2)
C(121)-C(126)	1.41(2)
C(121)-C(122)	1.42(3)
C(122)-C(123)	1.40(3)
C(123)-C(124)	1.25(3)
C(124)-C(125)	1.34(4)
C(125)-C(126)	1.44(4)
C(211)-C(216)	1.38(3)
C(211)-C(212)	1.41(2)
C(212)-C(213)	1.39(3)
C(213)-C(214)	1.39(3)
C(214)-C(215)	1.43(3)
C(215)-C(216)	1.32(3)
C(221)-C(226)	1.40(2)
C(221)-C(222)	1.40(2)
C(222)-C(223)	1.44(3)
C(223)-C(224)	1.37(3)
C(224)-C(225)	1.43(3)

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C(225)-C(226)	1.38(3)
P(3)-C(311)	1.82(2)
P(3)-C(31)	1.83(2)
P(3)-C(321)	1.88(2)
C(31)-P(4)	1.84(2)
P(4)-C(411)	1.84(2)
P(4)-C(421)	1.85(2)
C(311)-C(312)	1.32(2)
C(311)-C(316)	1.41(2)
C(312)-C(313)	1.39(3)
C(313)-C(314)	1.34(3)
C(314)-C(315)	1.41(3)
C(315)-C(316)	1.44(2)
C(321)-C(322)	1.33(2)
C(321)-C(326)	1.35(2)
C(322)-C(323)	1.39(3)
C(323)-C(324)	1.39(3)
C(324)-C(325)	1.41(3)
C(325)-C(326)	1.36(3)
C(411)-C(412)	1.34(2)
C(411)-C(416)	1.42(2)
C(412)-C(413)	1.48(3)
C(413)-C(414)	1.33(3)
C(414)-C(415)	1.41(3)
C(415)-C(416)	1.41(2)
C(421)-C(426)	1.35(2)
C(421)-C(422)	1.40(2)
C(422)-C(423)	1.41(3)
C(423)-C(424)	1.38(3)
C(424)-C(425)	1.35(3)
C(425)-C(426)	1.38(3)
P-F(6)	1.45(2)
P-F(5)	1.46(2)
P-F(3)	1.47(2)
P-F(4)	1.47(2)
P-F(1)	1.581(11)
P-F(2)	1.591(11)
C(1)-Ru-P(4)	85.3(5)
C(1)-Ru-P(3)	84.9(5)
P(4)-Ru-P(3)	71.8(2)
C(1)-Ru-P(2)	98.1(5)
P(4)-Ru-P(2)	176.6(2)
P(3)-Ru-P(2)	109.0(2)
C(1)-Ru-P(1)	99.6(5)
P(4)-Ru-P(1)	109.1(2)
P(3)-Ru-P(1)	175.4(2)
P(2)-Ru-P(1)	69.8(2)
C(1)-Ru-Cl	176.3(5)

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P(4)-Ru-Cl	91.7(2)
P(3)-Ru-Cl	92.2(2)
P(2)-Ru-Cl	85.0(2)
P(1)-Ru-Cl	83.3(2)
C(2)-C(1)-Ru	175(2)
C(1)-C(2)-C(3)	167(2)
C(4)-C(3)-N(5)	123(2)
C(4)-C(3)-C(2)	124(2)
N(5)-C(3)-C(2)	113(2)
C(3)-N(5)-C(511)	113(2)
C(3)-N(5)-C(531)	113(2)
C(511)-N(5)-C(531)	111(2)
C(3)-N(5)-C(521)	114(2)
C(511)-N(5)-C(521)	100(2)
C(531)-N(5)-C(521)	104(2)
N(5)-C(511)-C(512)	107(2)
C(522)-C(521)-N(5)	122(2)
C(532)-C(531)-N(5)	113(2)
C(121)-P(1)-C(111)	102.0(9)
C(121)-P(1)-C(11)	108.4(9)
C(111)-P(1)-C(11)	102.0(9)
C(121)-P(1)-Ru	122.4(7)
C(111)-P(1)-Ru	124.8(6)
C(11)-P(1)-Ru	93.8(6)
P(2)-C(11)-P(1)	94.1(9)
C(211)-P(2)-C(221)	102.4(9)
C(211)-P(2)-C(11)	103.2(9)
C(221)-P(2)-C(11)	106.2(9)
C(211)-P(2)-Ru	124.9(6)
C(221)-P(2)-Ru	122.2(7)
C(11)-P(2)-Ru	94.2(7)
C(112)-C(111)-C(116)	117(2)
C(112)-C(111)-P(1)	119.8(13)
C(116)-C(111)-P(1)	122.2(14)
C(111)-C(112)-C(113)	118(2)
C(114)-C(113)-C(112)	123(2)
C(113)-C(114)-C(115)	121(2)
C(116)-C(115)-C(114)	119(2)
C(115)-C(116)-C(111)	122(2)
C(126)-C(121)-C(122)	118(2)
C(126)-C(121)-P(1)	122(2)
C(122)-C(121)-P(1)	119(2)
C(123)-C(122)-C(121)	119(2)
C(124)-C(123)-C(122)	121(3)
C(123)-C(124)-C(125)	125(3)
C(124)-C(125)-C(126)	119(3)
C(121)-C(126)-C(125)	118(2)
C(216)-C(211)-C(212)	114(2)
C(216)-C(211)-P(2)	123(2)

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C(212)-C(211)-P(2)	122(2)
C(213)-C(212)-C(211)	120(2)
C(214)-C(213)-C(212)	122(2)
C(213)-C(214)-C(215)	118(2)
C(216)-C(215)-C(214)	116(2)
C(215)-C(216)-C(211)	129(2)
C(226)-C(221)-C(222)	120(2)
C(226)-C(221)-P(2)	123(2)
C(222)-C(221)-P(2)	117.3(14)
C(221)-C(222)-C(223)	122(2)
C(224)-C(223)-C(222)	116(2)
C(223)-C(224)-C(225)	123(2)
C(226)-C(225)-C(224)	120(2)
C(225)-C(226)-C(221)	119(2)
C(311)-P(3)-C(31)	106.4(9)
C(311)-P(3)-C(321)	99.5(9)
C(31)-P(3)-C(321)	106.4(8)
C(311)-P(3)-Ru	125.6(7)
C(31)-P(3)-Ru	95.0(6)
C(321)-P(3)-Ru	121.8(6)
P(4)-C(31)-P(3)	95.9(8)
C(31)-P(4)-C(411)	104.5(8)
C(31)-P(4)-C(421)	106.0(8)
C(411)-P(4)-C(421)	100.3(8)
C(31)-P(4)-Ru	95.9(6)
C(411)-P(4)-Ru	125.7(6)
C(421)-P(4)-Ru	121.6(6)
C(312)-C(311)-C(316)	117(2)
C(312)-C(311)-P(3)	126(2)
C(316)-C(311)-P(3)	117(2)
C(311)-C(312)-C(313)	125(2)
C(314)-C(313)-C(312)	118(2)
C(313)-C(314)-C(315)	123(2)
C(314)-C(315)-C(316)	115(2)
C(311)-C(316)-C(315)	122(2)
C(322)-C(321)-C(326)	120(2)
C(322)-C(321)-P(3)	121(2)
C(326)-C(321)-P(3)	118.1(14)
C(321)-C(322)-C(323)	122(2)
C(324)-C(323)-C(322)	118(2)
C(323)-C(324)-C(325)	118(2)
C(326)-C(325)-C(324)	120(3)
C(321)-C(326)-C(325)	121(2)
C(412)-C(411)-C(416)	118(2)
C(412)-C(411)-P(4)	124.1(14)
C(416)-C(411)-P(4)	117.8(14)
C(411)-C(412)-C(413)	124(2)
C(414)-C(413)-C(412)	117(2)
C(413)-C(414)-C(415)	122(2)

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C(416)-C(415)-C(414)	120(2)
C(415)-C(416)-C(411)	119(2)
C(426)-C(421)-C(422)	122(2)
C(426)-C(421)-P(4)	121(2)
C(422)-C(421)-P(4)	116.5(14)
C(421)-C(422)-C(423)	117(2)
C(424)-C(423)-C(422)	120(2)
C(425)-C(424)-C(423)	121(2)
C(424)-C(425)-C(426)	120(3)
C(421)-C(426)-C(425)	120(2)
F(6)-P-F(5)	178.0(10)
F(6)-P-F(3)	92(2)
F(5)-P-F(3)	88(2)
F(6)-P-F(4)	94(2)
F(5)-P-F(4)	87(2)
F(3)-P-F(4)	175(2)
F(6)-P-F(1)	91.1(8)
F(5)-P-F(1)	90.9(10)
F(3)-P-F(1)	89.9(8)
F(4)-P-F(1)	90.9(9)
F(6)-P-F(2)	87.5(8)
F(5)-P-F(2)	90.5(10)
F(3)-P-F(2)	88.2(8)
F(4)-P-F(2)	91.2(8)
F(1)-P-F(2)	177.5(9)

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **3a**. The anisotropic displacement factor exponent takes the form: $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
Ru	34(1)	39(1)	27(1)	20(1)	2(1)	10(1)
Cl	46(3)	52(4)	46(3)	18(2)	-2(3)	4(3)
C(1)	33(11)	31(12)	36(11)	0(9)	-1(9)	-7(9)
C(2)	44(12)	43(12)	35(11)	2(10)	-1(10)	-13(11)
C(3)	74(15)	21(11)	25(10)	-3(8)	-13(10)	-6(11)
C(4)	79(18)	71(20)	109(20)	56(16)	-10(16)	-21(16)
N(5)	95(15)	46(11)	12(7)	9(8)	5(9)	-23(11)
C(511)	316(53)	28(15)	76(19)	-2(13)	21(27)	-75(25)
C(512)	130(27)	34(17)	199(34)	3(18)	-99(26)	23(17)
C(521)	39(14)	155(28)	79(17)	32(19)	-10(13)	-31(18)
C(522)	86(22)	291(50)	82(19)	71(26)	-6(17)	-90(28)
C(531)	62(16)	86(18)	44(12)	17(13)	-20(12)	-28(15)
C(532)	200(40)	135(32)	138(28)	-95(25)	77(28)	-34(29)
P(1)	38(3)	74(4)	27(3)	15(3)	7(2)	12(3)
C(11)	37(13)	69(16)	62(14)	-17(12)	11(11)	13(12)
P(2)	35(3)	37(3)	48(3)	23(3)	2(3)	2(3)
C(111)	23(10)	30(11)	39(11)	15(9)	-2(9)	2(9)
C(112)	50(14)	72(16)	58(14)	53(13)	1(12)	24(13)
C(113)	50(16)	77(19)	105(21)	63(17)	28(15)	13(14)
C(114)	48(15)	12(11)	97(18)	20(12)	-16(14)	12(11)
C(115)	45(14)	33(12)	64(14)	6(11)	-7(12)	11(11)
C(116)	38(13)	26(11)	65(14)	-8(10)	0(11)	13(10)
C(121)	41(13)	56(15)	42(12)	3(10)	22(10)	12(12)
C(122)	54(15)	115(22)	22(11)	-7(13)	8(10)	-10(16)
C(123)	80(20)	136(26)	52(16)	-27(17)	18(15)	21(19)
C(124)	89(23)	175(35)	29(15)	-31(18)	-15(15)	-8(25)
C(125)	62(18)	170(34)	61(18)	35(22)	15(15)	32(22)
C(126)	99(19)	110(20)	8(10)	15(12)	10(11)	62(17)
C(211)	47(14)	53(14)	56(13)	37(11)	14(11)	21(12)
C(212)	53(14)	46(14)	57(14)	28(11)	-17(12)	-9(12)
C(213)	70(17)	83(19)	39(12)	41(12)	-14(12)	4(15)
C(214)	91(20)	43(15)	89(18)	33(13)	-24(16)	-20(15)
C(215)	39(15)	143(26)	73(18)	27(18)	8(14)	1(17)
C(216)	41(14)	96(19)	46(13)	41(13)	-4(12)	-10(14)
C(225)	40(14)	57(15)	82(16)	48(13)	-39(12)	-3(12)
C(226)	56(15)	37(13)	70(15)	20(11)	-10(12)	-21(12)
P(3)	35(3)	18(3)	39(3)	7(2)	4(2)	-1(2)
P(4)	41(3)	38(3)	14(2)	2(2)	-1(2)	7(3)
C(311)	34(12)	46(14)	64(14)	15(11)	3(11)	1(11)
C(312)	51(14)	56(15)	51(13)	14(11)	12(11)	5(13)
C(313)	53(16)	44(16)	118(21)	9(15)	-23(15)	33(14)

*Supporting Information*H¹ 10

C(314)	48(16)	49(16)	89(19)	-31(14)	-19(14)	-6(13)
C(315)	70(17)	10(11)	90(17)	5(10)	-4(14)	3(12)
C(316)	52(13)	10(10)	62(13)	9(9)	3(11)	9(10)
C(321)	29(11)	15(10)	55(12)	5(9)	14(10)	0(9)
C(322)	51(15)	65(16)	78(16)	-35(13)	33(13)	1(13)
C(323)	56(16)	108(22)	63(16)	-32(15)	17(14)	-16(16)
C(324)	79(21)	117(26)	63(17)	-3(17)	4(16)	7(19)
C(325)	101(23)	74(19)	85(19)	-23(16)	-17(18)	-30(18)
C(326)	86(18)	46(14)	45(13)	-10(11)	6(13)	-13(14)
C(421)	59(14)	17(10)	16(9)	-3(8)	-12(9)	18(10)
C(423)	88(20)	39(15)	75(17)	-33(13)	-50(16)	36(15)
C(424)	116(26)	26(14)	48(15)	-9(11)	-52(16)	10(16)
C(425)	85(21)	52(16)	55(14)	-8(12)	-27(15)	7(15)
C(426)	57(14)	46(13)	24(10)	11(10)	-2(10)	20(12)
P	59(5)	63(4)	30(3)	-4(3)	0(3)	-13(3)
F(1)	122(12)	95(10)	34(6)	-10(7)	-12(7)	-1(10)
F(2)	104(11)	136(13)	26(6)	-3(7)	14(7)	-13(10)
F(3)	77(13)	464(44)	76(11)	-78(19)	18(10)	-51(20)
F(4)	91(14)	442(42)	62(11)	-26(18)	-3(10)	-94(20)
F(5)	383(42)	85(15)	153(18)	-32(13)	-21(23)	4(21)
F(6)	677(64)	62(12)	51(10)	3(8)	-57(22)	-16(24)

Supporting Information

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Figure S1: Plot of the packing diagram of **3a** (view down the *a* axis).

Figure S2: EPR spectrum of electrochemically oxidized *trans*-[Cl(dppm)₂Ru-C≡C-C(NEt₃)=CH₂]²⁺.

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Pt₅₀-Cluster in CH₂Cl₂, GC1 -78°C

File	v [V/s]	Ep _f [V]	Ep _r [V]	E _{p2} [V]	Ep-Ep ₂ [mV]	ΔE _p [mV]	E _{1/2} [V]	i _{pf} [μA]	i _{pr} [μA]	i _h [μA]	r	i _p v ^{1/2} ΔEp
Cv326	0.1	-1.270	0.530/0.67	-1.15	120			410				
		1.312	1.231	-1.234	78			-84				
Cv30	0.4	-1.516	0.421/0.32	-1.376	140							
		1.174		1.090	84			14.6				
Cv28	0.4	1.174	-	1.090				14.0				
		-1.526	-					-				
Cv27	0.2	1.142	-1.204	1.058	84			-113				
		-1.419	-					-				
Cv33	0.1	-1.234	0.581/0.72	-1.10	124			+4.44				
		1.360	-	-1.290	70			-7.8				
Cv34	0.1	-1.310		-1.18	130			+4.5				-2.015
		0.704	0.630	0.649	55	74	0	-9.75	+9.75			0
		1.154										0.450

Weg der Folgepeaks: Peaks, die der Reduktion zugehörig sind: $\sim -0.19\text{ V}$, -0.05 V
 Peak, der Oxidation zugehörig ist: $\sim -1.70\text{ V}$

Fe



File: 02ruambu
Date: 4-JUL-1996

Comment: RUAMBU, CH₂Cl₂/TBAPF, 2.1V, g=2.3815, g=2.1547, g=1.9381
Time: 11:41:53

