Supporting Information for Organometallics Communication

Syntheses and Structural Characterizations of *cis*-$[M(NO)_2(CNXyl)_4]^+$ (M = Nb, Ta; Xyl = 2,6-$Me_2C_6H_3$), the First Dinitrosyls of Niobium and Tantalum[1]

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Additional Analytical Data for 1 and 2

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Molecular structure of $[Ta((NO)_2(CNXyl)_4]^+$

Additional Analytical Data for 1 and 2

$[Nb(NO)_2(CNXyl)_4][BF_4]$ (1). Anal. Calcd. (%) for $C_{36}H_{36}N_6BF_4NbO_2$: C, 56.57; H 4.75; N, 10.99. Found (%): C, 56.35; H, 4.71; N, 10.81.

$[Ta(NO)_2(CNXyl)_4][BF_4]$ (2). Anal. Calcd. (%) for $C_{36}H_{36}N_6BF_4O_2Ta$: C, 50.72; H 4.26; N, 9.86. Found (%): C, 50.48; H, 4.10; N, 9.72.

Table S.1. Crystal data, data collection, solution and refinement for (1).

Crystal Data	
Reference number	99034
Empirical formula	$C_{36}H_{36}BF_4N_6NbO_2$
Crystal habit, color	Block, red-ochre
Crystal size	0.30 × 0.24 × 0.24 mm
Crystal system	Monoclinic
Space group	$P2_1/c$
	a = 15.8513(1) Å α = 90°
	b = 18.4832(3) Å β = 92.681(1)°
	c = 12.5908(2) Å γ = 90°
Volume	3684.85(9) Å^3
Z	4
Formula weight	764.43
Density (calculated)	1.378 Mg/m^3
Absorption coefficient	0.387 mm^{-1}
F(000)	1568
Data Collection	
Diffractometer	Siemens SMART Platform CCD
Wavelength	0.71073 Å (Mo Kα)
Temperature	173 (2) K
θ range for data collection	1.29 to 25.08°
Index ranges	$-18 \le h \le 18$, $0 \le k \le 22$, $0 \le l \le 14$
Reflections collected	19157
Independent reflections	6378 (R_{int} = 0.0349)
Solution and Refinement	
System used	SHELXTL–V5.0
Solution	Direct methods
Refinement method	Full-matrix least-squares on F^2
Weighting scheme	$w = [\sigma^2(F_o^2) + (AP)^2 + (BP)]^{-1}$, where P = $(F_o^2 + 2F_c^2)/3$, A = 0.0261 and B = 2.4101
Absorption correction	SADABS (Sheldrick, 1996)
Max. and min. transmission	1.0000 and 0.904936
Data / restraints / parameters	6378 / 0 / 495
R indices (I > 2σ(I) = 5372)	R1 = 0.0352, wR2 = 0.0761
R indices (all data)	R1 = 0.0479, wR2 = 0.0811
Goodness-of-fit on F^2	1.113
Largest diff. peak and hole	0.408 and -0.347 eÅ^3

Table S.2. Atomic coordinates (× 10^4) and equivalent isotropic displacement parameters (Å^2 × 10^3) for (1). U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U(eq)	SOF
Nb(1)	3042(1)	2038(1)	2768(1)	26(1)	1
N(1)	2851(2)	2957(1)	2148(2)	39(1)	1
O(1)	2759(2)	3533(1)	1752(2)	72(1)	1
N(2)	3465(1)	2489(1)	4031(2)	35(1)	1
O(2)	3739(1)	2769(1)	4821(2)	58(1)	1
C(1)	4342(2)	1964(1)	2136(2)	28(1)	1
N(3)	5028(1)	1945(1)	1865(2)	27(1)	1
C(2)	5872(2)	1932(1)	1560(2)	26(1)	1
C(3)	6080(2)	1447(1)	760(2)	31(1)	1
C(4)	6920(2)	1425(2)	484(2)	40(1)	1
C(5)	7508(2)	1883(2)	977(3)	47(1)	1
C(6)	7276(2)	2368(2)	1737(2)	42(1)	1
C(7)	6447(2)	2407(1)	2054(2)	31(1)	1
C(8)	5421(2)	971(2)	220(2)	43(1)	1
C(9)	6197(2)	2936(2)	2897(2)	41(1)	1
C(10)	3381(2)	910(2)	3458(2)	31(1)	1
N(4)	3642(1)	376(1)	3814(2)	30(1)	1
C(11)	3989(2)	-270(1)	4228(2)	28(1)	1
C(12)	3621(2)	-573(1)	5110(2)	33(1)	1
C(13)	3983(2)	-1210(2)	5508(2)	42(1)	1
C(14)	4674(2)	-1514(2)	5051(2)	43(1)	1
C(15)	5018(2)	-1201(2)	4181(2)	41(1)	1
C(16)	4679(2)	-568(2)	3732(2)	33(1)	1
C(17)	2876(2)	-229(2)	5602(3)	48(1)	1
C(18)	5011(2)	-226(2)	2751(2)	48(1)	1
C(19)	1727(2)	1906(2)	3355(2)	35(1)	1
N(5)	1055(1)	1842(1)	3649(2)	36(1)	1
C(20)	251(2)	1765(2)	4064(2)	34(1)	1
C(21)	-403(2)	2208(2)	3657(2)	37(1)	1
C(22)	-1179(2)	2134(2)	4123(2)	46(1)	1
C(23)	-1290(2)	1650(2)	4931(3)	46(1)	1
C(24)	-631(2)	1219(2)	5307(2)	46(1)	1
C(25)	157(2)	1266(2)	4882(2)	41(1)	1
C(26)	-276(2)	2727(2)	2761(3)	57(1)	1
C(27)	891(2)	801(2)	5261(3)	68(1)	1
C(28)	2465(2)	1474(1)	1268(2)	32(1)	1
N(6)	2104(1)	1211(1)	553(2)	33(1)	1
C(29)	1631(2)	857(2)	-263(2)	36(1)	1
C(30)	751(2)	910(2)	-263(2)	47(1)	1
C(31)	299(2)	538(2)	-1054(3)	66(1)	1
C(32)	708(3)	146(2)	-1801(3)	76(1)	1
C(33)	1578(3)	108(2)	-1785(3)	64(1)	1
C(34)	2065(2)	468(2)	-1012(2)	44(1)	1
C(35)	330(2)	1344(2)	570(3)	63(1)	1

C(36)	3016(2)	448(2)	-965(3)	54(1)	1
B(1)	2103(2)	-517(2)	2129(3)	35(1)	1
F(1)	1750(1)	-1032(1)	1450(2)	67(1)	1
F(2)	1541(1)	51(1)	2243(2)	60(1)	1
F(3)	2278(1)	-822(1)	3112(2)	67(1)	1
F(4)	2849(1)	-267(1)	1717(1)	47(1)	1

Table S.3. Bond lengths (Å) for (1).

Nb(1)-N(1)	1.887(2)	C(15)-C(16)	1.397(4)
Nb(1)-N(2)	1.889(2)	C(16)-C(18)	1.504(4)
Nb(1)-C(1)	2.249(3)	C(19)-N(5)	1.150(3)
Nb(1)-C(19)	2.258(3)	N(5)-C(20)	1.406(3)
Nb(1)-C(28)	2.309(3)	C(20)-C(25)	1.396(4)
Nb(1)-C(10)	2.312(3)	C(20)-C(21)	1.399(4)
N(1)-O(1)	1.183(3)	C(21)-C(22)	1.394(4)
N(2)-O(2)	1.186(3)	C(21)-C(26)	1.501(4)
C(1)-N(3)	1.155(3)	C(22)-C(23)	1.373(4)
N(3)-C(2)	1.409(3)	C(23)-C(24)	1.381(4)
C(2)-C(7)	1.392(3)	C(24)-C(25)	1.385(4)
C(2)-C(3)	1.400(4)	C(25)-C(27)	1.506(4)
C(3)-C(4)	1.392(4)	C(28)-N(6)	1.152(3)
C(3)-C(8)	1.503(4)	N(6)-C(29)	1.405(3)
C(4)-C(5)	1.385(4)	C(29)-C(34)	1.392(4)
C(5)-C(6)	1.374(4)	C(29)-C(30)	1.399(4)
C(6)-C(7)	1.393(4)	C(30)-C(31)	1.382(5)
C(7)-C(9)	1.509(4)	C(30)-C(35)	1.501(5)
C(10)-N(4)	1.153(3)	C(31)-C(32)	1.374(6)
N(4)-C(11)	1.405(3)	C(32)-C(33)	1.381(5)
C(11)-C(12)	1.397(4)	C(33)-C(34)	1.384(4)
C(11)-C(16)	1.397(4)	C(34)-C(36)	1.506(4)
C(12)-C(13)	1.391(4)	B(1)-F(3)	1.377(4)
C(12)-C(17)	1.500(4)	B(1)-F(1)	1.380(4)
C(13)-C(14)	1.381(4)	B(1)-F(2)	1.389(4)
C(14)-C(15)	1.373(4)	B(1)-F(4)	1.392(3)

Table S.4. Bond angles (°) for (1).

N(1)-Nb(1)-N(2)	89.78(10)	N(1)-Nb(1)-C(19)	95.75(10)
N(1)-Nb(1)-C(1)	92.34(10)	N(2)-Nb(1)-C(19)	93.97(10)
N(2)-Nb(1)-C(1)	91.72(9)	C(1)-Nb(1)-C(19)	170.12(9)

N(1)-Nb(1)-C(28)	91.04(10)	C(15)-C(16)-C(18)	122.8(3)
N(2)-Nb(1)-C(28)	177.16(9)	C(11)-C(16)-C(18)	120.9(3)
C(1)-Nb(1)-C(28)	90.96(9)	N(5)-C(19)-Nb(1)	179.5(2)
C(19)-Nb(1)-C(28)	83.24(10)	C(19)-N(5)-C(20)	177.0(3)
N(1)-Nb(1)-C(10)	175.23(10)	C(25)-C(20)-C(21)	123.7(3)
N(2)-Nb(1)-C(10)	90.79(9)	C(25)-C(20)-N(5)	118.0(2)
C(1)-Nb(1)-C(10)	82.91(9)	C(21)-C(20)-N(5)	118.2(2)
C(19)-Nb(1)-C(10)	88.94(9)	C(22)-C(21)-C(20)	116.2(3)
C(28)-Nb(1)-C(10)	88.62(9)	C(22)-C(21)-C(26)	122.0(3)
O(1)-N(1)-Nb(1)	177.7(2)	C(20)-C(21)-C(26)	121.7(3)
O(2)-N(2)-Nb(1)	179.3(2)	C(23)-C(22)-C(21)	121.5(3)
N(3)-C(1)-Nb(1)	175.9(2)	C(22)-C(23)-C(24)	120.6(3)
C(1)-N(3)-C(2)	178.6(2)	C(23)-C(24)-C(25)	120.9(3)
C(7)-C(2)-C(3)	123.7(2)	C(24)-C(25)-C(20)	117.1(3)
C(7)-C(2)-N(3)	118.5(2)	C(24)-C(25)-C(27)	122.6(3)
C(3)-C(2)-N(3)	117.8(2)	C(20)-C(25)-C(27)	120.3(3)
C(4)-C(3)-C(2)	117.3(2)	N(6)-C(28)-Nb(1)	173.5(2)
C(4)-C(3)-C(8)	121.6(3)	C(28)-N(6)-C(29)	175.6(3)
C(2)-C(3)-C(8)	121.2(2)	C(34)-C(29)-C(30)	124.1(3)
C(5)-C(4)-C(3)	120.2(3)	C(34)-C(29)-N(6)	118.2(2)
C(6)-C(5)-C(4)	121.0(3)	C(30)-C(29)-N(6)	117.7(3)
C(5)-C(6)-C(7)	121.3(3)	C(31)-C(30)-C(29)	116.6(3)
C(2)-C(7)-C(6)	116.5(3)	C(31)-C(30)-C(35)	122.5(3)
C(2)-C(7)-C(9)	122.4(2)	C(29)-C(30)-C(35)	120.9(3)
C(6)-C(7)-C(9)	121.1(2)	C(32)-C(31)-C(30)	120.7(3)
N(4)-C(10)-Nb(1)	172.3(2)	C(31)-C(32)-C(33)	121.3(3)
C(10)-N(4)-C(11)	177.8(3)	C(32)-C(33)-C(34)	120.6(4)
C(12)-C(11)-C(16)	124.2(2)	C(33)-C(34)-C(29)	116.6(3)
C(12)-C(11)-N(4)	117.8(2)	C(33)-C(34)-C(36)	122.6(3)
C(16)-C(11)-N(4)	118.0(2)	C(29)-C(34)-C(36)	120.7(3)
C(13)-C(12)-C(11)	116.5(3)	F(3)-B(1)-F(1)	109.5(3)
C(13)-C(12)-C(17)	122.0(3)	F(3)-B(1)-F(2)	108.6(3)
C(11)-C(12)-C(17)	121.6(2)	F(1)-B(1)-F(2)	110.1(2)
C(14)-C(13)-C(12)	121.0(3)	F(3)-B(1)-F(4)	109.4(2)
C(15)-C(14)-C(13)	120.9(3)	F(1)-B(1)-F(4)	109.0(2)
C(14)-C(15)-C(16)	121.1(3)	F(2)-B(1)-F(4)	110.3(2)
C(15)-C(16)-C(11)	116.3(3)		

Table S.5. Anisotropic displacement parameters ($Å^2 \times 10^3$) for (1). The anisotropic displacement factor exponent takes the form: $-2\pi^2[(ha^*)^2U_{11} + \ldots + 2hka^*b^*U_{12}]$.

Atom	U11	U22	U33	U23	U13	U12
Nb(1)	21(1)	26(1)	31(1)	3(1)	2(1)	0(1)
N(1)	42(1)	31(1)	44(1)	5(1)	-3(1)	1(1)

O(1)	81(2)	42(2)	93(2)	26(1)	1(2)	6(1)
N(2)	31(1)	34(1)	38(1)	-3(1)	0(1)	6(1)
O(2)	56(1)	65(2)	52(1)	-25(1)	-10(1)	15(1)
C(1)	31(2)	26(1)	26(1)	0(1)	-2(1)	-1(1)
N(3)	27(1)	28(1)	27(1)	2(1)	2(1)	-1(1)
C(2)	24(1)	31(1)	24(1)	6(1)	3(1)	0(1)
C(3)	37(2)	31(2)	25(1)	3(1)	4(1)	1(1)
C(4)	44(2)	42(2)	34(2)	4(1)	15(1)	9(1)
C(5)	28(2)	54(2)	58(2)	12(2)	13(1)	0(1)
C(6)	30(2)	48(2)	47(2)	11(2)	-3(1)	-11(1)
C(7)	31(2)	32(2)	29(1)	7(1)	-2(1)	-5(1)
C(8)	50(2)	42(2)	37(2)	-7(1)	0(1)	-1(2)
C(9)	51(2)	35(2)	35(2)	-4(1)	-2(1)	-7(1)
C(10)	29(1)	30(2)	33(1)	1(1)	0(1)	-2(1)
N(4)	31(1)	29(1)	31(1)	-1(1)	-3(1)	-2(1)
C(11)	28(1)	25(1)	29(1)	-2(1)	-6(1)	1(1)
C(12)	37(2)	29(2)	32(1)	-3(1)	1(1)	3(1)
C(13)	58(2)	34(2)	34(2)	4(1)	-4(1)	4(1)
C(14)	50(2)	32(2)	45(2)	-1(1)	-14(2)	11(1)
C(15)	32(2)	41(2)	50(2)	-15(1)	-6(1)	9(1)
C(16)	29(1)	35(2)	36(2)	-10(1)	-3(1)	-4(1)
C(17)	56(2)	40(2)	51(2)	1(2)	19(2)	8(2)
C(18)	44(2)	56(2)	45(2)	-8(2)	12(2)	-9(2)
C(19)	30(2)	35(2)	40(2)	3(1)	0(1)	4(1)
N(5)	26(1)	40(1)	42(1)	0(1)	5(1)	1(1)
C(20)	24(1)	39(2)	40(2)	-4(1)	6(1)	-1(1)
C(21)	32(2)	40(2)	40(2)	-4(1)	5(1)	7(1)
C(22)	29(2)	59(2)	49(2)	-3(2)	4(1)	10(1)
C(23)	30(2)	56(2)	53(2)	-11(2)	14(1)	-5(1)
C(24)	43(2)	48(2)	48(2)	4(2)	14(2)	-6(2)
C(25)	34(2)	43(2)	45(2)	2(1)	5(1)	2(1)
C(26)	51(2)	60(2)	60(2)	16(2)	11(2)	18(2)
C(27)	49(2)	79(3)	75(3)	32(2)	2(2)	11(2)
C(28)	26(1)	30(2)	39(2)	7(1)	5(1)	-1(1)
N(6)	28(1)	35(1)	36(1)	5(1)	2(1)	0(1)
C(29)	33(2)	41(2)	32(1)	5(1)	-5(1)	-6(1)
C(30)	33(2)	59(2)	47(2)	12(2)	-5(1)	-5(1)
C(31)	41(2)	102(3)	52(2)	19(2)	-13(2)	-23(2)
C(32)	80(3)	107(3)	39(2)	6(2)	-13(2)	-51(3)
C(33)	83(3)	74(3)	36(2)	-9(2)	15(2)	-27(2)
C(34)	49(2)	46(2)	37(2)	2(1)	7(1)	-12(1)
C(35)	35(2)	75(3)	79(3)	8(2)	7(2)	7(2)
C(36)	50(2)	50(2)	66(2)	0(2)	26(2)	0(2)
B(1)	29(2)	35(2)	41(2)	-2(1)	2(1)	-3(1)
F(1)	45(1)	72(1)	84(1)	-37(1)	11(1)	-23(1)
F(2)	46(1)	50(1)	83(1)	-3(1)	6(1)	13(1)
F(3)	65(1)	79(2)	56(1)	26(1)	8(1)	6(1)
F(4)	38(1)	52(1)	51(1)	-4(1)	10(1)	-13(1)

Table S.6. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($Å^2 \times 10^3$) for (1).

Atom	x	y	z	U(eq)	SOF
H(4A)	7090(2)	1096(2)	-45(2)	34(7)	1
H(5A)	8081(2)	1862(2)	787(3)	63(10)	1
H(6A)	7690(2)	2683(2)	2053(2)	48(9)	1
H(8A)	5674(3)	696(8)	-349(10)	70(11)	1
H(8B)	4960(6)	1272(2)	-83(13)	52(9)	1
H(8C)	5197(9)	635(7)	740(4)	59(10)	1
H(9A)	6469(11)	3403(4)	2780(11)	83(12)	1
H(9B)	6379(13)	2750(6)	3600(2)	98(14)	1
H(9C)	5583(2)	2997(9)	2860(12)	75(12)	1
H(13A)	3750(2)	-1439(2)	6103(2)	52(9)	1
H(14A)	4915(2)	-1945(2)	5342(2)	49(9)	1
H(15A)	5493(2)	-1420(2)	3879(2)	55(9)	1
H(17A)	2707(8)	-522(6)	6205(10)	79(12)	1
H(17B)	2405(4)	-197(10)	5072(5)	57(10)	1
H(17C)	3030(4)	258(4)	5852(14)	62(10)	1
H(18A)	4557(4)	-196(10)	2198(6)	81(12)	1
H(18B)	5474(9)	-521(6)	2495(10)	71(11)	1
H(18C)	5220(12)	261(5)	2922(5)	58(10)	1
H(22A)	-1641(2)	2426(2)	3875(2)	52(9)	1
H(23A)	-1826(2)	1612(2)	5234(3)	50(9)	1
H(24A)	-718(2)	886(2)	5866(2)	46(9)	1
H(26A)	-727(8)	3089(7)	2740(12)	89(13)	1
H(26B)	-289(14)	2461(3)	2086(3)	94(14)	1
H(26C)	272(7)	2968(9)	2870(9)	64(11)	1
H(27A)	695(3)	434(8)	5755(15)	80(12)	1
H(27B)	1322(7)	1104(3)	5625(16)	67(11)	1
H(27C)	1133(9)	562(10)	4651(4)	111(17)	1
H(31A)	-301(2)	554(2)	-1081(3)	70(11)	1
H(32A)	385(3)	-103(2)	-2341(3)	88(13)	1
H(33A)	1846(3)	-169(2)	-2309(3)	72(11)	1
H(35A)	-283(2)	1331(9)	433(10)	90(13)	1
H(35B)	527(10)	1847(3)	546(11)	54(10)	1
H(35C)	472(10)	1139(7)	1273(3)	64(11)	1
H(36A)	3209(2)	87(8)	-1469(13)	78(12)	1
H(36B)	3223(2)	320(12)	-244(5)	74(12)	1
H(36C)	3234(2)	925(4)	-1152(17)	78(12)	1

Table S.7. Crystal data, data collection, solution and refinement for (2).

Crystal Data	
Reference number	98298c
Empirical formula	$C_{36}H_{36}BF_4N_6O_2Ta$
Crystal habit, color	Irregular, orange
Crystal size	0.35 × 0.25 × 0.20 mm
Crystal system	Monoclinic
Space group	$P2_1/c$
	a = 15.8829(3) Å α = 90°
	b = 18.6094(2) Å β = 92.556(1)°
	c = 12.7125(2) Å γ = 90°
Volume	3753.7(1) Å^3
Z	4
Formula weight	852.47
Density (calculated)	1.508 Mg/m^3
Absorption coefficient	2.987 mm^{-1}
F(000)	1696
Data Collection	
Diffractometer	Siemens SMART Platform CCD
Wavelength	0.71073 Å (Mo Kα)
Temperature	173 (2) K
θ range for data collection	1.28 to 24.93°
Index ranges	$-18 \le h \le 18$, $0 \le k \le 21$, $0 \le l \le 15$
Reflections collected	17323
Independent reflections	6420 (R_{int} = 0.0322)
Solution and Refinement	
System used	SHELXTL–V5.0
Solution	Direct methods
Refinement method	Full-matrix least-squares on F^2
Weighting scheme	$w = [\sigma^2(F_o^2) + (AP)^2 + (BP)]^{-1}$, where $P = (F_o^2 + 2F_c^2)/3$, A = 0.0471 and B = 2.6401
Absorption correction	SADABS (Sheldrick, 1996)
Max. and min. transmission	1.0000 and 0.804293
Data / restraints / parameters	6420 / 0 / 459
R indices (I > 2σ(I) = 5206)	R1 = 0.0340, wR2 = 0.0788
R indices (all data)	R1 = 0.0496, wR2 = 0.0918
Goodness-of-fit on F^2	1.057
Largest diff. peak and hole	1.387 (near B) and –0.503 eÅ^3

Table S.8. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for (2). U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U(eq)	SOF
Ta(1)	3047(1)	2038(1)	2791(1)	28(1)	1
N(1)	2852(3)	2962(2)	2168(4)	40(1)	1
O(1)	2759(3)	3533(2)	1772(4)	68(1)	1
N(2)	3464(3)	2474(2)	4064(4)	35(1)	1
O(2)	3731(3)	2742(3)	4863(3)	57(1)	1
C(1)	4332(3)	1963(3)	2161(4)	29(1)	1
N(3)	5019(3)	1944(2)	1881(3)	29(1)	1
C(2)	5862(3)	1927(3)	1560(4)	27(1)	1
C(3)	6065(3)	1440(3)	761(4)	33(1)	1
C(4)	6907(4)	1420(3)	477(5)	44(2)	1
C(5)	7498(4)	1876(4)	967(5)	49(2)	1
C(6)	7272(4)	2354(4)	1723(5)	47(2)	1
C(7)	6448(3)	2396(3)	2056(4)	35(1)	1
C(8)	5395(4)	974(3)	227(4)	45(2)	1
C(9)	6200(4)	2926(3)	2905(4)	41(1)	1
C(10)	3387(3)	917(3)	3461(4)	32(1)	1
N(4)	3641(3)	383(2)	3811(3)	32(1)	1
C(11)	3985(3)	-263(3)	4229(4)	31(1)	1
C(12)	3629(3)	-562(3)	5112(4)	35(1)	1
C(13)	3978(4)	-1195(3)	5505(4)	42(1)	1
C(14)	4674(4)	-1509(3)	5045(5)	43(1)	1
C(15)	5018(3)	-1200(3)	4174(5)	43(1)	1
C(16)	4675(3)	-563(3)	3733(4)	34(1)	1
C(17)	2876(4)	-219(3)	5603(5)	52(2)	1
C(18)	5015(4)	-227(4)	2746(5)	50(2)	1
C(19)	1744(3)	1903(3)	3359(4)	36(1)	1
N(5)	1064(3)	1837(2)	3658(4)	39(1)	1
C(20)	258(3)	1763(3)	4068(5)	40(1)	1
C(21)	-395(4)	2208(3)	3653(5)	46(2)	1
C(22)	-1174(4)	2138(4)	4129(5)	54(2)	1
C(23)	-1290(4)	1653(4)	4931(5)	51(2)	1
C(24)	-626(4)	1223(4)	5312(5)	53(2)	1
C(25)	161(4)	1265(3)	4882(5)	45(2)	1
C(26)	-261(4)	2725(4)	2772(6)	63(2)	1
C(27)	898(4)	808(4)	5264(6)	75(2)	1
C(28)	2476(3)	1481(3)	1303(5)	35(1)	1
N(6)	2122(3)	1219(2)	590(3)	35(1)	1
C(29)	1648(4)	869(3)	-225(4)	42(1)	1
C(30)	769(4)	921(4)	-227(5)	54(2)	1
C(31)	325(5)	552(5)	-1038(6)	78(3)	1
C(32)	724(5)	167(5)	-1774(6)	87(3)	1
C(33)	1593(5)	131(4)	-1760(5)	71(2)	1
C(34)	2089(4)	490(4)	-980(5)	48(2)	1
C(35)	332(4)	1355(4)	603(6)	72(2)	1

C(36)	3036(4)	465(4)	-939(5)	58(2)	1
B(1)	1999(4)	-508(3)	2150(4)	34(2)	1
F(1)	1749(2)	-1031(2)	1453(3)	76(1)	1
F(2)	1538(2)	49(2)	2246(3)	68(1)	1
F(3)	2268(2)	-821(2)	3111(3)	73(1)	1
F(4)	2847(2)	-260(2)	1723(3)	49(1)	1

Table S.9. Bond lengths (Å) for (2).

Ta(1)-N(2)	1.902(5)	C(15)-C(16)	1.410(8)
Ta(1)-N(1)	1.914(5)	C(16)-C(18)	1.523(8)
Ta(1)-C(1)	2.230(5)	C(19)-N(5)	1.166(7)
Ta(1)-C(19)	2.237(5)	N(5)-C(20)	1.411(7)
Ta(1)-C(28)	2.307(6)	C(20)-C(25)	1.404(8)
Ta(1)-C(10)	2.307(5)	C(20)-C(21)	1.411(8)
N(1)-O(1)	1.182(6)	C(21)-C(22)	1.408(8)
N(2)-O(2)	1.193(6)	C(21)-C(26)	1.499(9)
C(1)-N(3)	1.163(6)	C(22)-C(23)	1.381(9)
N(3)-C(2)	1.418(6)	C(23)-C(24)	1.394(9)
C(2)-C(7)	1.403(7)	C(24)-C(25)	1.389(8)
C(2)-C(3)	1.409(7)	C(25)-C(27)	1.509(8)
C(3)-C(4)	1.401(7)	C(28)-N(6)	1.153(7)
C(3)-C(8)	1.511(7)	N(6)-C(29)	1.412(7)
C(4)-C(5)	1.391(9)	C(29)-C(30)	1.399(8)
C(5)-C(6)	1.370(9)	C(29)-C(34)	1.403(8)
C(6)-C(7)	1.397(8)	C(30)-C(31)	1.403(10)
C(7)-C(9)	1.527(8)	C(30)-C(35)	1.521(10)
C(10)-N(4)	1.155(6)	C(31)-C(32)	1.359(12)
N(4)-C(11)	1.414(6)	C(32)-C(33)	1.381(10)
C(11)-C(12)	1.395(7)	C(33)-C(34)	1.408(9)
C(11)-C(16)	1.405(7)	C(34)-C(36)	1.502(8)
C(12)-C(13)	1.386(7)	B(1)-F(2)	1.278(7)
C(12)-C(17)	1.514(7)	B(1)-F(1)	1.363(7)
C(13)-C(14)	1.401(8)	B(1)-F(3)	1.403(6)
C(14)-C(15)	1.383(8)	B(1)-F(4)	1.545(8)

Table S.10. Bond angles (°) for (2).

N(2)-Ta(1)-N(1)	90.7(2)	N(1)-Ta(1)-C(1)	92.4(2)
N(2)-Ta(1)-C(1)	92.5(2)	N(2)-Ta(1)-C(19)	93.8(2)

N(1)-Ta(1)-C(19)	95.7(2)	C(11)-C(16)-C(15)	116.8(5)
C(1)-Ta(1)-C(19)	169.7(2)	C(11)-C(16)-C(18)	121.3(5)
N(2)-Ta(1)-C(28)	176.6(2)	C(15)-C(16)-C(18)	121.9(5)
N(1)-Ta(1)-C(28)	90.8(2)	N(5)-C(19)-Ta(1)	179.5(4)
C(1)-Ta(1)-C(28)	90.6(2)	C(19)-N(5)-C(20)	177.3(6)
C(19)-Ta(1)-C(28)	83.0(2)	C(25)-C(20)-C(21)	124.1(5)
N(2)-Ta(1)-C(10)	90.3(2)	C(25)-C(20)-N(5)	117.9(5)
N(1)-Ta(1)-C(10)	175.0(2)	C(21)-C(20)-N(5)	118.0(5)
C(1)-Ta(1)-C(10)	82.7(2)	C(22)-C(21)-C(20)	115.5(6)
C(19)-Ta(1)-C(10)	89.1(2)	C(22)-C(21)-C(26)	122.5(6)
C(28)-Ta(1)-C(10)	88.5(2)	C(20)-C(21)-C(26)	122.0(5)
O(1)-N(1)-Ta(1)	177.8(4)	C(23)-C(22)-C(21)	121.7(6)
O(2)-N(2)-Ta(1)	179.4(4)	C(22)-C(23)-C(24)	120.6(6)
N(3)-C(1)-Ta(1)	176.2(4)	C(25)-C(24)-C(23)	120.8(6)
C(1)-N(3)-C(2)	178.8(5)	C(24)-C(25)-C(20)	117.2(5)
C(7)-C(2)-C(3)	123.7(5)	C(24)-C(25)-C(27)	122.7(6)
C(7)-C(2)-N(3)	118.2(4)	C(20)-C(25)-C(27)	120.0(5)
C(3)-C(2)-N(3)	118.1(4)	N(6)-C(28)-Ta(1)	174.0(4)
C(4)-C(3)-C(2)	117.0(5)	C(28)-N(6)-C(29)	175.4(5)
C(4)-C(3)-C(8)	122.0(5)	C(30)-C(29)-C(34)	124.2(5)
C(2)-C(3)-C(8)	121.0(5)	C(30)-C(29)-N(6)	117.9(5)
C(5)-C(4)-C(3)	120.1(5)	C(34)-C(29)-N(6)	117.9(5)
C(6)-C(5)-C(4)	121.2(6)	C(29)-C(30)-C(31)	115.9(7)
C(5)-C(6)-C(7)	121.8(6)	C(29)-C(30)-C(35)	121.5(6)
C(6)-C(7)-C(2)	116.2(5)	C(31)-C(30)-C(35)	122.6(6)
C(6)-C(7)-C(9)	121.6(5)	C(32)-C(31)-C(30)	122.0(7)
C(2)-C(7)-C(9)	122.2(5)	C(31)-C(32)-C(33)	120.9(7)
N(4)-C(10)-Ta(1)	173.0(4)	C(32)-C(33)-C(34)	120.9(7)
C(10)-N(4)-C(11)	177.7(5)	C(29)-C(34)-C(33)	116.1(6)
C(12)-C(11)-C(16)	123.7(5)	C(29)-C(34)-C(36)	121.4(5)
C(12)-C(11)-N(4)	118.6(5)	C(33)-C(34)-C(36)	122.5(6)
C(16)-C(11)-N(4)	117.7(5)	F(2)-B(1)-F(1)	119.3(6)
C(13)-C(12)-C(11)	117.3(5)	F(2)-B(1)-F(3)	114.0(5)
C(13)-C(12)-C(17)	121.4(5)	F(1)-B(1)-F(3)	109.8(5)
C(11)-C(12)-C(17)	121.2(5)	F(2)-B(1)-F(4)	107.6(4)
C(12)-C(13)-C(14)	121.0(5)	F(1)-B(1)-F(4)	102.6(4)
C(15)-C(14)-C(13)	120.5(5)	F(3)-B(1)-F(4)	101.2(5)
C(14)-C(15)-C(16)	120.7(5)		

Table S.11. Anisotropic displacement parameters ($Å^2 \times 10^3$) for (2). The anisotropic displacement factor exponent takes the form: $-2\pi^2[(ha^*)^2U_{11} + \ldots + 2hka^*b^*U_{12}]$.

Atom	U11	U22	U33	U23	U13	U12
Ta(1)	25(1)	26(1)	32(1)	3(1)	3(1)	0(1)

N(1)	41(3)	30(3)	47(3)	3(2)	1(2)	4(2)
O(1)	88(4)	36(3)	81(3)	21(3)	2(3)	6(2)
N(2)	34(2)	30(3)	40(3)	-4(2)	2(2)	9(2)
O(2)	59(3)	64(3)	47(3)	-24(2)	-8(2)	15(2)
C(1)	35(3)	25(3)	28(3)	-2(2)	0(2)	-4(2)
N(3)	29(2)	31(3)	27(2)	2(2)	1(2)	-1(2)
C(2)	28(3)	30(3)	24(2)	7(2)	4(2)	2(2)
C(3)	35(3)	38(3)	25(3)	3(2)	6(2)	1(2)
C(4)	50(4)	45(4)	40(3)	1(3)	18(3)	8(3)
C(5)	38(3)	59(4)	52(4)	11(3)	11(3)	4(3)
C(6)	40(3)	51(4)	49(4)	12(3)	-2(3)	-11(3)
C(7)	37(3)	36(3)	30(3)	9(2)	0(2)	-8(2)
C(8)	54(4)	47(4)	35(3)	-12(3)	2(3)	-6(3)
C(9)	54(4)	36(3)	33(3)	-1(3)	-3(3)	-8(3)
C(10)	32(3)	29(3)	33(3)	-3(2)	1(2)	-4(2)
N(4)	36(2)	27(3)	32(2)	-1(2)	-2(2)	-1(2)
C(11)	34(3)	29(3)	28(3)	-3(2)	-10(2)	3(2)
C(12)	42(3)	32(3)	31(3)	-1(2)	-1(2)	3(2)
C(13)	55(4)	35(3)	37(3)	4(3)	-4(3)	1(3)
C(14)	48(4)	34(3)	44(3)	-5(3)	-14(3)	11(3)
C(15)	33(3)	45(4)	50(4)	-14(3)	-1(3)	10(3)
C(16)	29(3)	36(3)	35(3)	-12(2)	-4(2)	-3(2)
C(17)	63(4)	45(4)	51(4)	-3(3)	20(3)	9(3)
C(18)	49(4)	55(4)	46(3)	-3(3)	10(3)	-5(3)
C(19)	31(3)	34(3)	42(3)	3(3)	2(2)	3(2)
N(5)	34(3)	38(3)	45(3)	-2(2)	8(2)	5(2)
C(20)	30(3)	46(3)	44(3)	-5(3)	5(2)	-2(2)
C(21)	42(3)	51(4)	44(3)	-1(3)	5(3)	10(3)
C(22)	29(3)	76(5)	57(4)	-2(4)	6(3)	12(3)
C(23)	38(4)	57(4)	58(4)	-10(3)	17(3)	-8(3)
C(24)	54(4)	49(4)	58(4)	2(3)	20(3)	-8(3)
C(25)	39(3)	50(4)	47(3)	1(3)	4(3)	2(3)
C(26)	60(4)	62(5)	67(5)	20(4)	19(4)	21(3)
C(27)	51(4)	91(6)	83(5)	41(5)	5(4)	13(4)
C(28)	30(3)	32(3)	45(3)	10(3)	8(3)	3(2)
N(6)	33(2)	37(3)	34(3)	3(2)	2(2)	-5(2)
C(29)	41(3)	54(4)	33(3)	5(3)	-1(2)	-8(3)
C(30)	36(3)	79(5)	47(4)	14(3)	0(3)	-3(3)
C(31)	45(4)	129(8)	58(5)	20(5)	-15(4)	-25(4)
C(32)	85(6)	132(8)	43(4)	6(5)	-6(4)	-58(6)
C(33)	93(6)	87(6)	33(3)	-11(4)	14(4)	-28(5)
C(34)	52(4)	54(4)	38(3)	-1(3)	2(3)	-13(3)
C(35)	33(4)	96(6)	87(5)	5(5)	8(3)	10(4)
C(36)	59(4)	51(4)	65(4)	-8(3)	24(3)	-3(3)
B(1)	80(5)	8(3)	15(3)	1(2)	-4(3)	7(3)
F(1)	48(2)	83(3)	97(3)	-35(3)	10(2)	-24(2)
F(2)	51(2)	69(3)	85(3)	1(2)	9(2)	9(2)
F(3)	70(3)	82(3)	68(3)	26(2)	14(2)	2(2)
F(4)	45(2)	50(2)	52(2)	-5(2)	11(2)	-14(2)

Table S.12. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($Å^2 \times 10^3$) for (2).

Atom	x	y	z	U(eq)	SOF
H(4A)	7074(4)	1095(3)	-50(5)	53	1
H(5A)	8068(4)	1855(4)	773(5)	59	1
H(6A)	7688(4)	2666(4)	2030(5)	56	1
H(8A)	5642(7)	698(16)	-339(20)	68	1
H(8B)	4939(12)	1278(3)	-69(26)	68	1
H(8C)	5168(17)	643(14)	743(8)	68	1
H(9A)	5596(6)	3030(15)	2824(18)	61	1
H(9B)	6520(17)	3373(7)	2835(18)	61	1
H(9C)	6326(21)	2717(8)	3602(4)	61	1
H(13A)	3743(4)	-1419(3)	6096(4)	51	1
H(14A)	4911(4)	-1939(3)	5334(5)	51	1
H(15A)	5489(3)	-1418(3)	3869(5)	51	1
H(17A)	3026(9)	267(9)	5842(30)	79	1
H(17B)	2712(17)	-507(13)	6204(21)	79	1
H(17C)	2405(9)	-194(21)	5079(11)	79	1
H(18A)	5493(17)	-511(13)	2515(19)	75	1
H(18B)	5202(24)	265(8)	2900(10)	75	1
H(18C)	4571(9)	-219(21)	2186(11)	75	1
H(22A)	-1633(4)	2433(4)	3891(5)	65	1
H(23A)	-1827(4)	1613(4)	5227(5)	61	1
H(24A)	-714(4)	896(4)	5871(5)	63	1
H(26A)	-706(17)	3090(15)	2757(24)	94	1
H(26B)	-279(30)	2464(6)	2101(7)	94	1
H(26C)	289(13)	2958(19)	2881(20)	94	1
H(27A)	705(7)	448(19)	5762(32)	112	1
H(27B)	1327(15)	1114(6)	5615(36)	112	1
H(27C)	1139(21)	566(22)	4663(8)	112	1
H(31A)	-274(5)	573(5)	-1073(6)	93	1
H(32A)	401(5)	-80(5)	-2306(6)	104	1
H(33A)	1859(5)	-140(4)	-2284(5)	85	1
H(35A)	531(25)	1853(8)	591(28)	108	1
H(35B)	460(27)	1147(17)	1299(8)	108	1
H(35C)	-279(5)	1345(24)	452(25)	108	1
H(36A)	3241(4)	288(23)	-248(14)	87	1
H(36B)	3258(4)	948(6)	-1054(35)	87	1
H(36C)	3225(4)	142(19)	-1489(24)	87	1

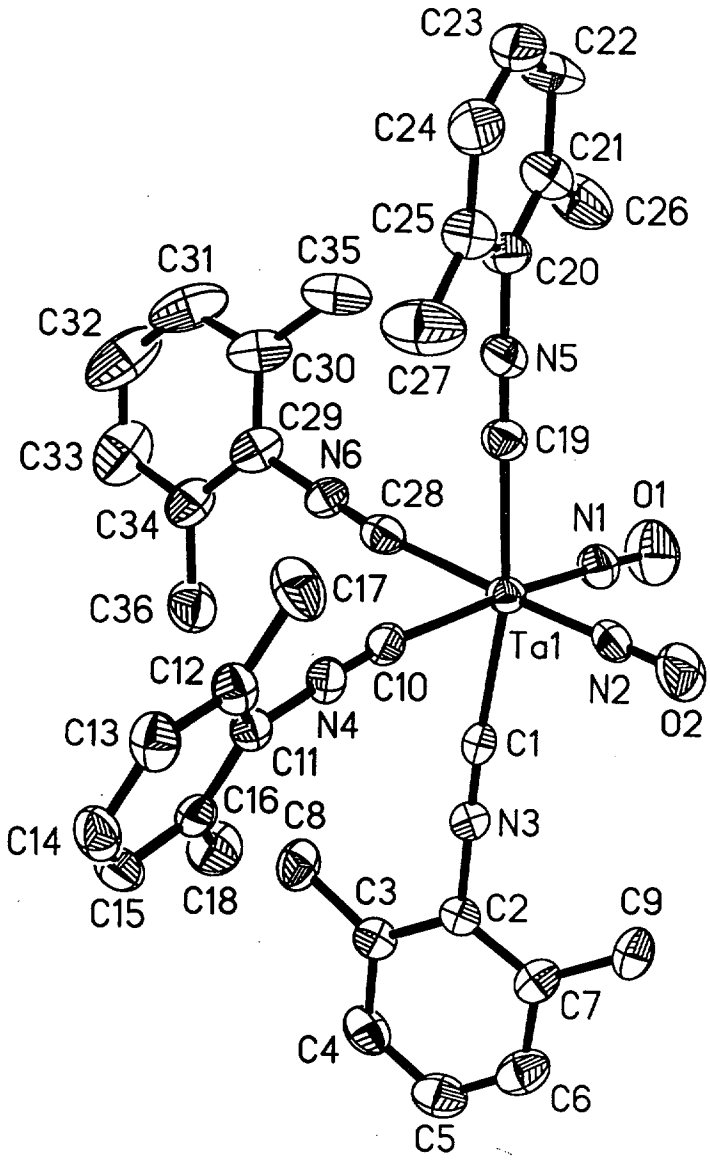

Figure S1. Molecular Structure of (**2**); 50% thermal ellipsoids. Hydrogens and $[BF_4]^-$ are omitted for clarity.