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Iridium(III) Catalyzed Trifluoroacetoxylation of Aromatic Hydrocarbons

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General Considerations: All manipulations were carried out using an argon filled MBraun glovebox and standard Schlenk techniques using oven dried glassware (>1 h at 110 °C under vacuum, -30 mm Hg). Methylenechloride was HPLC grade from Fisher and distilled from P_2O_5 under argon prior to use. Benzene was HPLC grade from EMD and distilled from Na/benzophenone under argon prior to use. Pentane was HPLC grade from EMD and distilled over NaH under argon prior to use. Reagent-grade chemicals and solvents were purchased from Sigma Aldrich or Alfa Aesar and used as is unless otherwise specified. Deuterated solvents were purchased from Cambridge Isotope Inc. and degassed and stored under argon before use. Deuterated methylenechloride was further stored over 4Å molecular sieves. Deuterated

trifluoroacetic acid was degassed before use by several freeze-pump-thaw cycles and stored under argon. Trifluoroacetic acid was HPLC grade from Fisher and degassed before use by several freeze-pump-thaw cycles and stored under argon. $IrCl_3•(H_2O)_x$ was obtained from Pressure Chemical. Elemental. Analyses were performed by Columbia Analytical Services; Tucson, Arizona or Atlantic Microlab, Inc. of Norcross, Georgia. Electrospray Ionization (ESI) mass spectroscopy was performed at the University of Illinois Mass Spec Facility; Urbana, Illinois. Liquid phase organic products were analyzed with a Shimadzu GC-MS QP2010S equipped with cross-linked methyl silicone gum capillary column, RTX-5. Gas measurements were performed using an Agilent GasPro column. The retention times of the products were confirmed by known standards. NMR spectra were obtained on a Bruker Digital Avance III 400 (400.132 MHz for ^{1}H, 100.623 MHz for ^{13}C, and 376.461 MHz for ^{19}F) spectrometer. Chemical shifts are given in ppm relative to residual solvent proton resonances or to a stated internal or external standard. Liquid phase flash chromatography was performed on a Teledyne Isco Combiflash Rf and all solvents were of HPLC grade and obtained from Fisher or EMD. X-ray crystallography was performed at the Center for Nanostructured Materials located at the University of Texas at Arlington.

Figure 1. GC-MS calibration curve for PhOH.

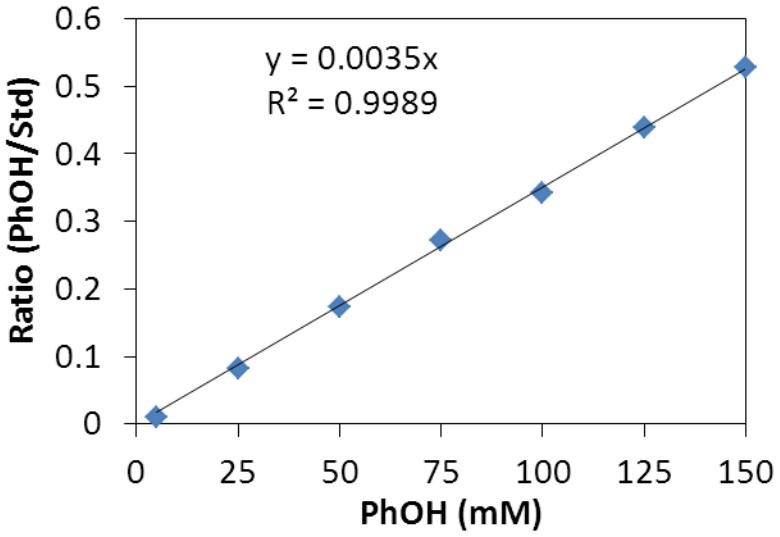
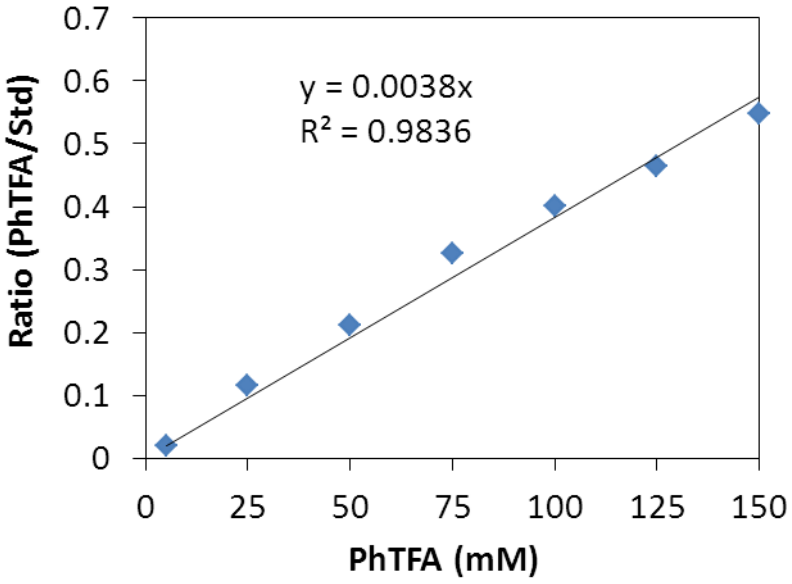


Figure 2. GC-MS calibration curve for PhTFA.



General Procedure for Arene Oxidation: A 4 mL Schlenk flask with a resealable PTFE valve and a magnetic stir bar was charged with 50 eq (to catalyst, unless otherwise specified) of oven dried $Cu(TFA)_2$ in the glovebox. With the flask removed from the glovebox and connected to a Schlenk line, 1.0 mL of a 8:2 (v/v%) CF_3COOD:benzene solution containing 8 mM catalyst (unless otherwise mentioned) was added. The reaction was sealed under argon or molecular

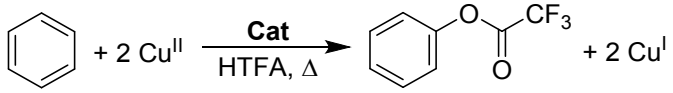
oxygen and heated in a well stirred oil bath. Upon completion of the reaction, the flasks were removed from the oil bath and rapidly cooled in an ice bath. Reaction work up was done by addition to a known amount of Na_2CO_3 ~1.8M in DI H_2O (~10 mL) on ice and then extracted with 2 mL of an ether solution containing n-decane or n-dodecane as the standard and analyzed via GC-MS.

Oxidation of Benzene: Given the fast CH activation between benzene and DTFA catalyzed by **2** and the known oxidation capabilities of the (^{t}Bu-NNC)Ir motif, we examined whether **2** would facilitate the oxy-functionalization of benzene as we had previously not explored oxidation reactions of aromatics with **1**. Initial reactions involved heating **2** in HTFA/benzene solutions with several equivalents of oxidants such as $NaIO_4$, $NaIO_3$, H_2O_2, Pd^{II}, KNO_3, CAN, $PhI(TFA)_2$, and Cu^{II} salts. Unfortunately, many of the oxidants only generated trace levels of oxygenated products or halogenated arenes as detected by GC-MS following a standardized workup procedure (see experimental section for details). For example, iodine containing reagents led to good yields of phenyl-iodide as the major product which we will explore in future work with these systems. We repeated work with **1** and observed similar levels of reactivity with many of the reagents yielding halogenated arenes. This was not surpising as we had previously observed similar results with methane oxidation reactions.

We have been interested in air regenerable oxidants that are capable of a Wacker – like oxidation scheme for hydrocarbons and gravitated towards species such as Cu^{II}, as they showed some product formation (Table 1, Entry 3)[1] This led us to question if the low yields of oxidized benzene could be increased through further reaction optimization. After examination of various reaction parameters (Table 1), running the oxidation reaction at 180 °C, with 1 atm of O_2 and 75 eq of $Cu(TFA)_2$ in a HTFA/$O(TFA)_2$/benzene solution generated ~7 TON of a PhOH/PhTFA

mixture (~1:10 PhOH to PhTFA) after 16 h. The derivation of the product from benzene was confirmed using $^{13}C_6H_6$. GC-MS analysis revealed an $[M+6]^+$ peak characteristic of incorporating the ^{13}C labeled benzene into the product. Interestingly, both the Ir and Cu components are required for the overall reaction to proceed based on control reactions. It was also observed that addition of O_2 and trifluoroacetic anhydride helped to improve yield of PhTFA/PhOH.

Table 1. Oxidation of C_6H_6 by **1** or **2** in HTFA using Cu^{II} oxidant.



Entry	Catalyst	Time (h)	Temp (°C)	Cu^{II} Species	Atm.	Additive[a]	Product	TON[b]
1	None	3	150	$Cu(TFA)_2$	Air	–	–	0.0
2	**2**	3	150	None	Air	–	–	0.0
3	**2**	3	150	$Cu(TFA)_2$	Argon	–	PhTFA	0.3
4	**2**	3	150	$Cu(TFA)_2$	O_2[e]	–	PhTFA	0.6
5	**2**	3	150	CuO	Air	–	PhTFA	0.5
6	**2**	3	150	$Cu(OTf)_2$	Air	–	PhTFA	0.5
7	**2**	3	180	$Cu(TFA)_2$[c]	O_2[d]	$O(TFA)_2$	PhTFA	2.2
8	**2**	6	180	$Cu(TFA)_2$[c]	O_2[d]	$O(TFA)_2$	PhTFA	3.8
9	None	16	180	$Cu(TFA)_2$[c]	O_2[d]	$O(TFA)_2$	PhTFA	<1
10	**2**	16	180	$Cu(TFA)_2$[c]	O_2[d]	$O(TFA)_2$	PhTFA	6.9
11	**2**	16	180	–	O_2[d]	$O(TFA)_2$	PhTFA	<1
12	**1**	16	180	$Cu(TFA)_2$[c]	O_2[d]	$O(TFA)_2$	PhTFA	<7

[a]100 eq of additive. [b]TON = mol of product/mol of catalyst. [c]75 eq of oxidant. [d]1 atm of O_2. PhOH and PhTFA were both observed and are considered to be PhTFA for simplicity at the ratio was about 1:10 PhOH:PhTFA.

Confirmation of PhTFA being Derived from C_6H_6: A 4 mL Schlenk flask with a resealable PTFE valve and a magnetic stir bar was charged with 50 eq (to catalyst) of oven dried $Cu(TFA)_2$ in the glovebox. With the flask removed from the glovebox and connected to a Schlenk line, 1.0 mL of a 8:2 (v/v%) CF_3COOD:$^{13}C_6H_6$ solution containing 8 mM catalyst was added. The reaction was sealed under O_2 and heated in a well stirred oil bath at 180 °C. Upon completion of the reaction, the flask was removed from the oil bath and rapidly cooled in an ice bath. Reaction work up was done by addition to a known amount of Na_2CO_3 ~1.8M in DI H_2O (~10 mL) on ice and then extracted with 2 mL of an ether solution containing n-dodecane as the standard and analyzed via GC-MS. GC-MS analysis revealed an $[M+6]^+$ characteristic of incorporating the ^{13}C labeled benzene into the product. The normal m/z for PhTFA gives an $[M]^+ = 190$; while the reaction with $^{13}C_6H_6$ produces PhTFA where $[M]^+ = 196$.

Figure 3. Fragmentation pattern of PhTFA from the reaction of $^{12}C_6H_6$ and **2** in HTFA with $Cu(TFA)_2$.

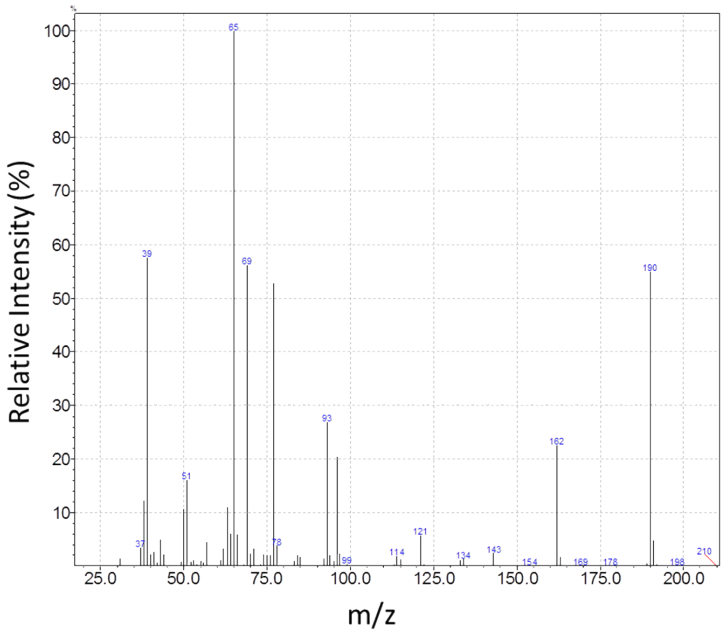
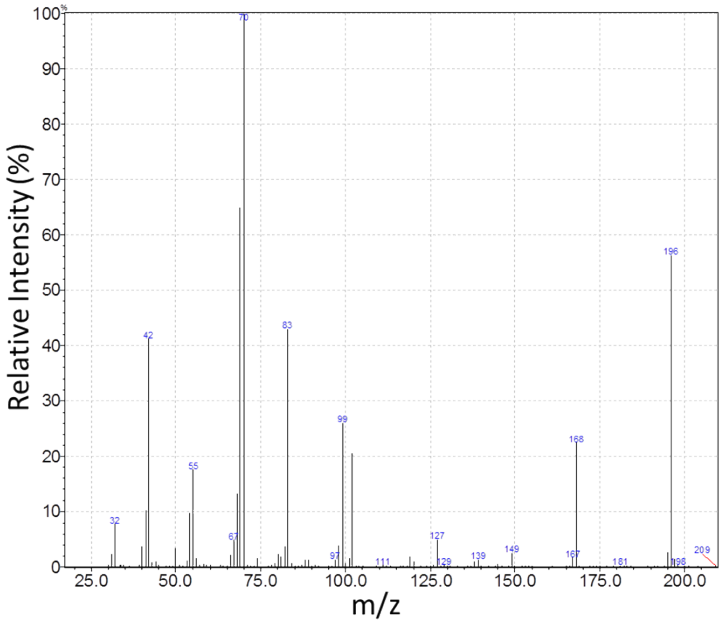


Figure 4. Fragmentation pattern of PhTFA from the reaction of $^{13}C_6H_6$ and **2** in HTFA with $Cu(TFA)_2$.



Loss of Ethylene from 4: The loss of ethylene from **4** was monitored by preparing a 15 mM solution in HTFA in a sealed J-Young NMR tube under argon. The NMR tube contained a coaxial insert containing 1,3,5-trimethoxybenzene in DMSO-D_6. The NMR tube was heated at 100 °C and monitored over 5 d by ^{1}H NMR (Figure 5). Monitoring of the ethylene signal at 4.9 ppm revealed that loss of the ethylene was in fact much slower than originally proposed. A $T_{1/2}$ = ~1000 min was observed for the loss of the Ir-ethylene signal (Figure 6).

Figure 5. ^{1}H NMR (400 MHz) monitoring C_2H_4 loss of **4** at 100 °C for 5 d.

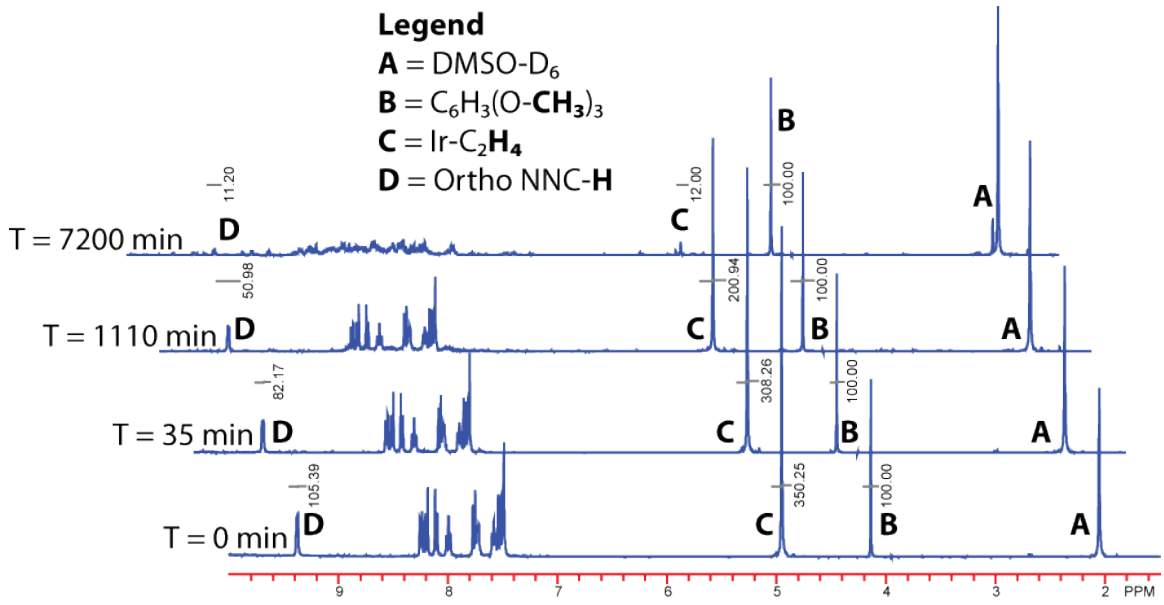
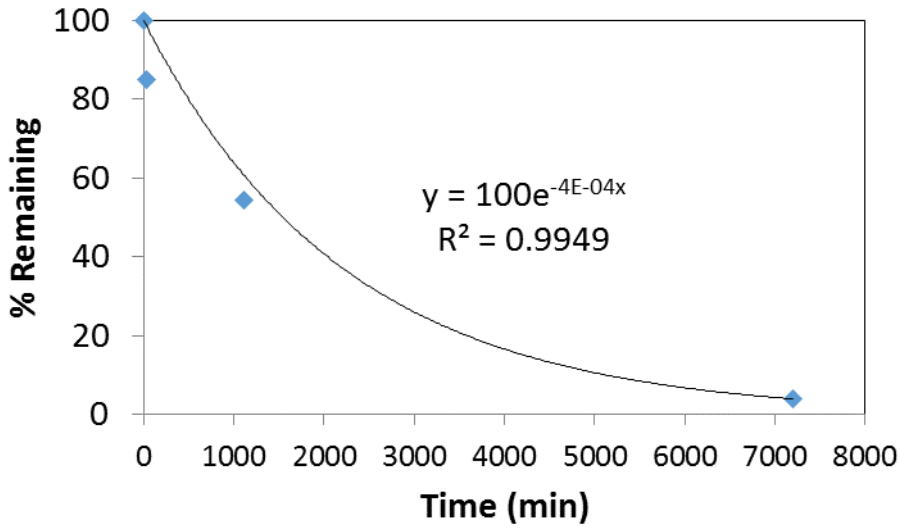


Figure 6. Loss of ethylene from **4** in HTFA over 5 d at 100 °C.



Stability of 11 in DTFA: A J-Young NMR tube or 4 mL vial with a PTFE cap containing a septum and magnetic stir bar was prepared with a 15 mM solution of **11** in DTFA under argon. The reaction was then analyzed by ^{1}H NMR or GC-MS after 30 min at RT. Mixtures for GC-MS analysis were poured on to ice-chilled solution of Na_2CO_3 followed by extraction with 2 mL of a standardized ether solution containing n-decane. Formation of benzene was observed in all cases

and was quantitatively formed, based on >90% mass balance, from **11**. It was found that rapid protonation of the Ir-Ph group occurs prior to reaching the NMR with the generation of predominetly $C_6D_1H_5$ (^{1}H NMR and GC-MS, Figure 7 and Figure 8, respectively). Furthermore, higher isotoplogues such as $C_6D_5H_1$ and C_6D_6 are also observed in low yield.

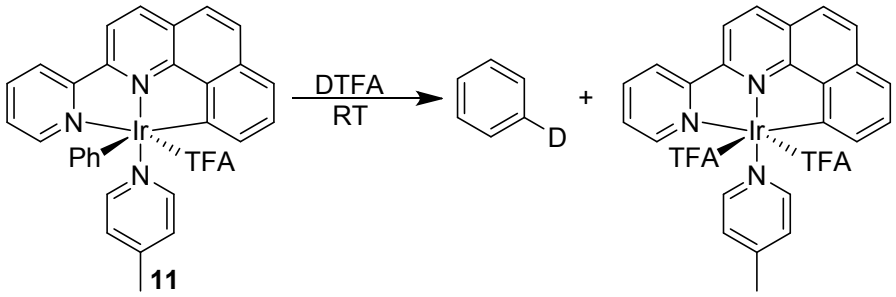


Figure 7. ^{1}H NMR (400 MHz) analysis of DTFA protonolysis of **11**.

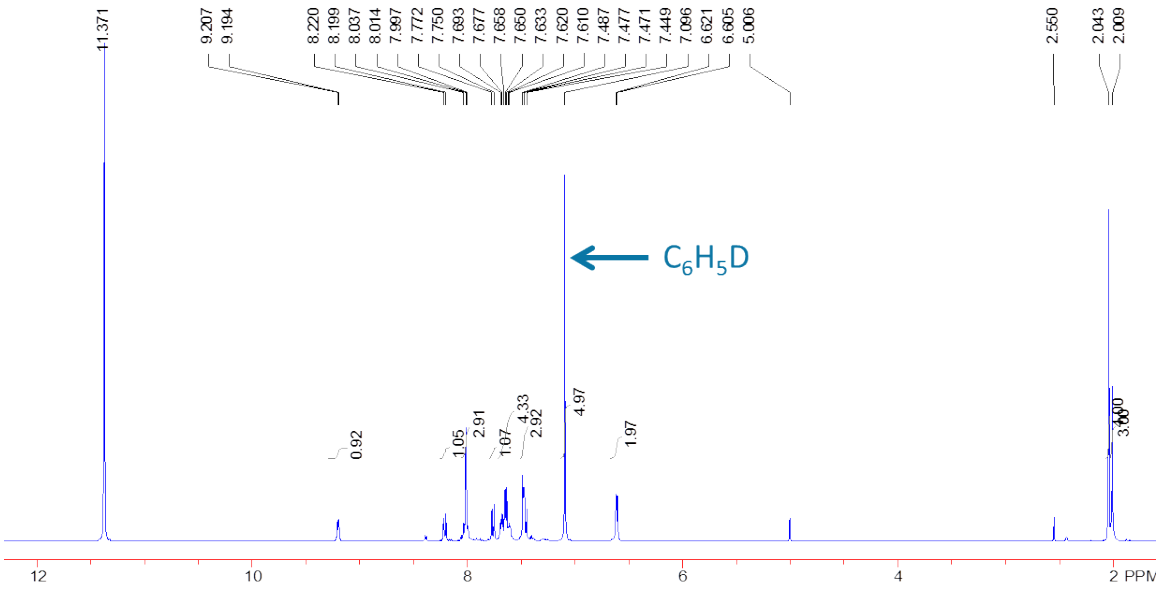
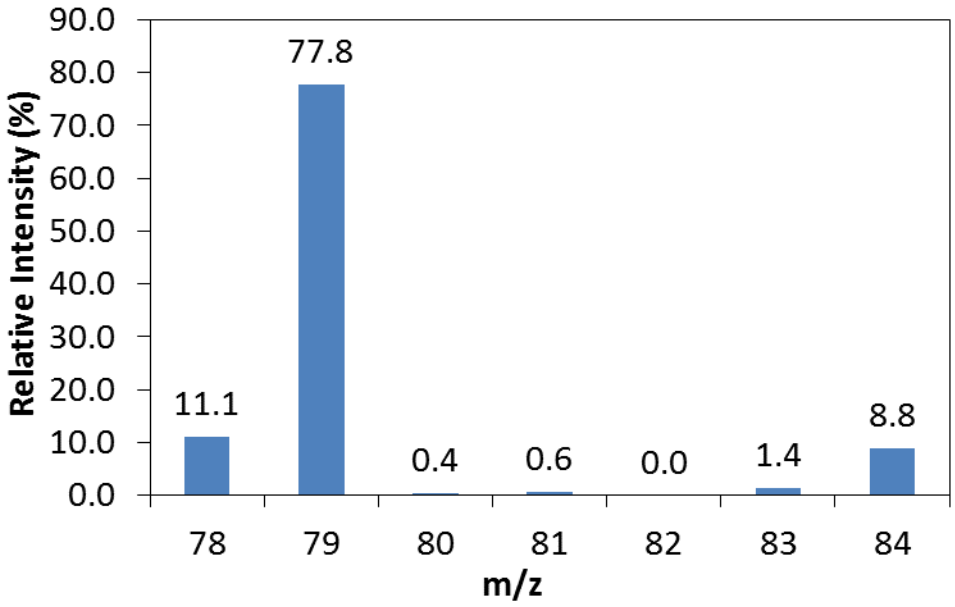


Figure 8. GC-MS analysis of the fragmentation pattern of benzene generated from DTFA protonolysis of **11.**



As shown in Figure 8, protonolysis of **11** led to predominant formation of $C_6D_1H_5$; however, multiple deuterium incorporations were also observed suggesting that the barrier for CH cleavage is lower than that of CH bond coordination. If the system was following a typical Schultz-Flory pathway, the only product observed should be $C_6D_1H_5$. Given that the more substituted isotopogogues are observed suggests that multiple CH activation events occur from **11** prior to benzene loss.

Stoichiometric Functionalization Studies with 11: In a sealed NMR tube, 10 mM solutions of **11** were prepared in dry THF-D_8 or CD_3CN with 3 eq. of added $Cu(TFA)_2$. The solution was heated to either 60 °C or 100 °C and monitored over time by ^1H NMR. Unfortunately, no PhOH or PhTFA was observed after heating at 60 °C for 2 h. Continued heating overnight at 100 °C did not lead to significant product formation; however, traces of phenol (<5%) were observed in the ^1H NMR spectrum. Benzene was still observed as the major product (>50%) resulting from protonolysis of the phenyl group. This is caused by generation of HTFA from the reaction of

$Cu(TFA)_2$ with H_2O contamination in the reaction solvents, even after rigoursouly drying the solvents by established methods.[2]

General Considerations for X-ray Diffraction: X-ray diffraction studies were performed at the University of Texas at Arlington in the Center for Nanostructured Materials. Crystals grown from concentrated solutions at -35 °C were moved from a scintillation vial to a microscope slide containing Paratone N. Samples were selected and mounted on a nylon loop using Paratone. The data collections were carried out at a sample temperature of 100 K on a Bruker SMART APEX diffractometer equipped with a CCD detector. The data were processed and reduced utilizing the program SAINTPLUS supplied by Bruker AXS. The structures were solved by direct methods (SHELXTL v5.1, Bruker AXS) in conjunction with standard difference Fourier techniques.

Figure 9. ORTEP structure of (NNC-tb)Ir(Et)(C_2H_4)(Cl) with ellipsoids at 50%.

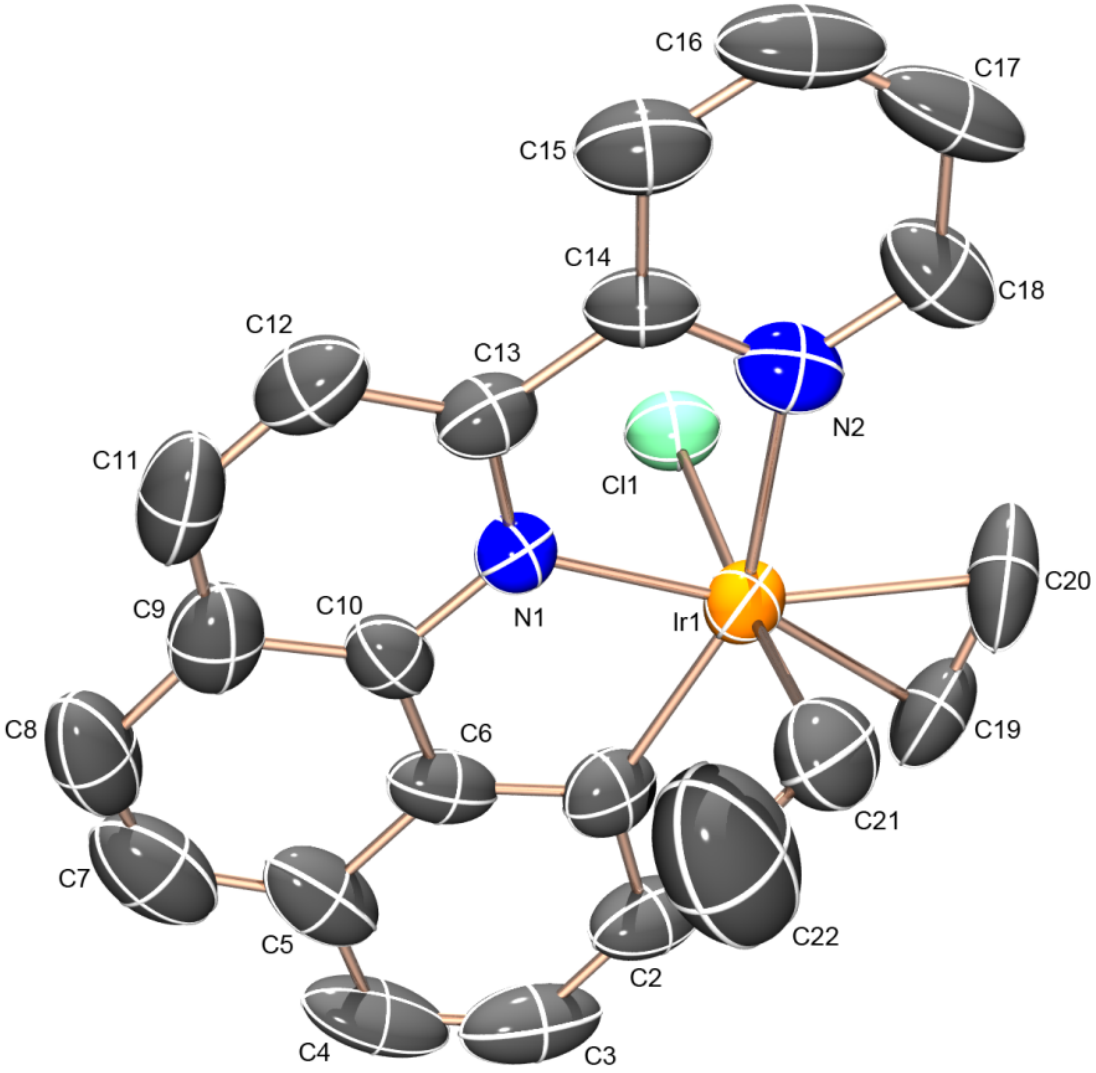


Table 2. Crystal data and structure refinement for (NNC-tb)Ir(Et)(C_2H_4)(Cl).

Identification code	periana20	
Empirical formula	C44 H39 Cl2 Ir2 N4	
Formula weight	1079.09	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.846(2) Å	α= 85.002(3)°.
	b = 10.265(3) Å	β= 89.516(3)°.
	c = 21.098(5) Å	γ = 79.875(3)°.
Volume	1878.7(8) $Å^3$	
Z	2	
Density (calculated)	1.908 Mg/m^3	
Absorption coefficient	7.256 mm^{-1}	
F(000)	1038	
Crystal size	0.35 x 0.02 x 0.02 mm^3	
Theta range for data collection	1.94 to 28.42°.	
Index ranges	-11<=h<=11, -13<=k<=13, -27<=l<=28	
Reflections collected	18115	
Independent reflections	9228 [R(int) = 0.0438]	
Completeness to theta = 28.42°	97.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.745 and 0.513	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9228 / 6 / 499	
Goodness-of-fit on F^2	1.048	
Final R indices [I>2sigma(I)]	R1 = 0.0532, wR2 = 0.1005	
R indices (all data)	R1 = 0.1019, wR2 = 0.1141	

Largest diff. peak and hole	2.589 and -1.183 e.Å^{-3}

Table 3. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for (NNC-tb)Ir(Et)(C_2H_4)(Cl). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ir(1)	1846(1)	2858(1)	1609(1)	47(1)
Cl(1)	2420(2)	5171(2)	1458(1)	55(1)
N(1)	1726(7)	2796(6)	663(3)	41(2)
N(2)	-560(7)	3646(7)	1367(4)	57(2)
C(1)	4097(9)	2079(8)	1430(4)	55(2)
C(2)	5395(10)	1627(9)	1799(5)	71(3)
C(3)	6829(12)	1147(10)	1539(7)	88(4)
C(4)	7008(11)	1053(10)	908(8)	89(4)
C(5)	5784(11)	1505(9)	500(6)	75(3)
C(6)	4332(9)	1965(7)	775(5)	57(2)
C(7)	5807(14)	1498(10)	-173(7)	92(4)
C(8)	4593(15)	1823(10)	-568(6)	83(3)
C(9)	3106(13)	2259(9)	-300(5)	67(3)
C(10)	3037(9)	2363(7)	365(4)	49(2)
C(11)	1707(14)	2642(9)	-626(5)	77(3)
C(12)	378(11)	3089(9)	-327(5)	65(2)
C(13)	407(9)	3184(8)	338(4)	51(2)
C(14)	-881(9)	3676(8)	730(4)	52(2)
C(15)	-2350(11)	4138(8)	489(5)	69(3)
C(16)	-3505(11)	4594(9)	889(7)	87(4)
C(17)	-3201(11)	4554(10)	1526(7)	86(4)
C(18)	-1721(10)	4088(9)	1755(5)	72(3)
C(19)	2750(14)	2711(12)	2575(5)	75(3)
C(20)	1198(15)	3242(14)	2584(5)	91(4)
C(21)	1288(11)	975(10)	1782(5)	81(3)
C(22)	1658(18)	18(15)	1434(8)	164(6)
Ir(2)	8343(1)	7514(1)	3364(1)	48(1)
Cl(2)	7835(2)	5181(2)	3401(1)	55(1)
N(3)	8662(7)	7276(6)	4313(3)	45(2)
N(4)	10787(8)	6734(7)	3489(4)	63(2)
C(23)	6135(8)	8253(7)	3676(4)	51(2)
C(24)	4778(10)	8853(8)	3369(5)	65(2)
C(25)	3429(10)	9245(9)	3724(6)	72(3)
C(26)	3416(12)	9004(10)	4371(7)	85(4)
C(27)	4731(11)	8422(9)	4717(5)	66(3)
C(28)	6068(9)	8083(8)	4338(4)	52(2)
C(29)	4824(13)	8160(10)	5384(5)	75(3)

C(30)	6157(15)	7642(10)	5691(5)	85(3)
C(31)	7542(12)	7356(9)	5349(4)	64(3)
C(32)	7438(10)	7557(8)	4678(4)	50(2)
C(33)	9014(14)	6852(9)	5608(5)	74(3)
C(34)	10216(13)	6560(9)	5225(5)	76(3)
C(35)	10072(10)	6751(8)	4562(4)	54(2)
C(36)	11255(10)	6473(9)	4100(5)	62(2)
C(37)	12794(10)	5944(9)	4267(6)	80(3)
C(38)	13841(12)	5677(11)	3793(8)	99(4)
C(39)	13371(12)	5955(11)	3169(7)	96(4)
C(40)	11865(10)	6464(10)	3039(5)	78(3)
C(41)	8738(15)	7467(13)	2354(4)	78(3)
C(42)	7202(14)	7881(12)	2444(5)	75(3)
C(43)	8833(10)	9449(8)	3284(5)	67(3)
C(44)	8984(12)	10095(11)	3830(6)	103(4)

Table 4. Bond lengths [Å] and angles [°] for (NNC-tb)Ir(Et)(C_2H_4)(Cl).

Ir(1)-N(1)	2.008(6)
Ir(1)-C(1)	2.056(9)
Ir(1)-C(21)	2.079(10)
Ir(1)-C(19)	2.179(10)
Ir(1)-C(20)	2.180(10)
Ir(1)-N(2)	2.188(7)
Ir(1)-Cl(1)	2.507(2)
N(1)-C(10)	1.338(9)
N(1)-C(13)	1.339(9)
N(2)-C(18)	1.349(10)
N(2)-C(14)	1.372(11)
C(1)-C(2)	1.379(11)
C(1)-C(6)	1.409(12)
C(2)-C(3)	1.403(14)
C(2)-H(2)	0.9300
C(3)-C(4)	1.349(15)
C(3)-H(3)	0.9300
C(4)-C(5)	1.378(15)
C(4)-H(4)	0.9300
C(5)-C(7)	1.420(15)
C(5)-C(6)	1.425(12)
C(6)-C(10)	1.421(12)
C(7)-C(8)	1.341(15)
C(7)-H(7)	0.9300
C(8)-C(9)	1.442(13)
C(8)-H(8)	0.9300
C(9)-C(11)	1.399(13)
C(9)-C(10)	1.416(12)
C(11)-C(12)	1.356(12)
C(11)-H(11)	0.9300
C(12)-C(13)	1.414(12)
C(12)-H(12)	0.9300
C(13)-C(14)	1.447(12)
C(14)-C(15)	1.387(11)
C(15)-C(16)	1.364(14)
C(15)-H(15)	0.9300
C(16)-C(17)	1.370(15)
C(16)-H(16)	0.9300
C(17)-C(18)	1.388(13)
C(17)-H(17)	0.9300
C(18)-H(18)	0.9300
C(19)-C(20)	1.386(16)
C(19)-H(19A)	1.04(8)

C(19)-H(19B)	0.96(6)
C(20)-H(20A)	0.84(8)
C(21)-C(22)	1.273(15)
C(21)-H(21A)	0.9700
C(21)-H(21B)	0.9700
C(22)-H(22A)	0.9600
C(22)-H(22B)	0.9600
C(22)-H(22C)	0.9600
Ir(2)-N(3)	2.011(6)
Ir(2)-C(23)	2.089(8)
Ir(2)-C(43)	2.099(8)
Ir(2)-C(41)	2.161(9)
Ir(2)-C(42)	2.168(9)
Ir(2)-N(4)	2.179(7)
Ir(2)-Cl(2)	2.508(2)
N(3)-C(32)	1.328(10)
N(3)-C(35)	1.359(10)
N(4)-C(36)	1.346(11)
N(4)-C(40)	1.351(11)
C(23)-C(24)	1.387(11)
C(23)-C(28)	1.396(11)
C(24)-C(25)	1.421(13)
C(24)-H(24)	0.9300
C(25)-C(26)	1.365(14)
C(25)-H(25)	0.9300
C(26)-C(27)	1.392(14)
C(26)-H(26)	0.9300
C(27)-C(29)	1.409(13)
C(27)-C(28)	1.431(11)
C(28)-C(32)	1.410(11)
C(29)-C(30)	1.352(14)
C(29)-H(29)	0.9300
C(30)-C(31)	1.416(13)
C(30)-H(30)	0.9300
C(31)-C(33)	1.409(13)
C(31)-C(32)	1.414(12)
C(33)-C(34)	1.338(13)
C(33)-H(33)	0.9300
C(34)-C(35)	1.399(12)
C(34)-H(34)	0.9300
C(35)-C(36)	1.433(12)
C(36)-C(37)	1.410(12)
C(37)-C(38)	1.369(15)
C(37)-H(37)	0.9300
C(38)-C(39)	1.374(16)
C(38)-H(38)	0.9300

C(39)-C(40)	1.364(14)
C(39)-H(39)	0.9300
C(40)-H(40)	0.9300
C(41)-C(42)	1.368(15)
C(41)-H(41A)	1.11(8)
C(41)-H(41B)	1.05(13)
C(42)-H(42A)	1.15(7)
C(42)-H(42B)	1.04(7)
C(43)-C(44)	1.396(12)
C(43)-H(43A)	0.9700
C(43)-H(43B)	0.9700
C(44)-H(44A)	0.9600
C(44)-H(44B)	0.9600
C(44)-H(44C)	0.9600

N(1)-Ir(1)-C(1)	80.3(3)
N(1)-Ir(1)-C(21)	92.4(3)
C(1)-Ir(1)-C(21)	91.9(3)
N(1)-Ir(1)-C(19)	161.6(4)
C(1)-Ir(1)-C(19)	81.4(4)
C(21)-Ir(1)-C(19)	89.7(4)
N(1)-Ir(1)-C(20)	161.2(4)
C(1)-Ir(1)-C(20)	118.4(4)
C(21)-Ir(1)-C(20)	88.9(5)
C(19)-Ir(1)-C(20)	37.1(4)
N(1)-Ir(1)-N(2)	75.5(3)
C(1)-Ir(1)-N(2)	155.8(3)
C(21)-Ir(1)-N(2)	89.2(3)
C(19)-Ir(1)-N(2)	122.9(4)
C(20)-Ir(1)-N(2)	85.8(4)
N(1)-Ir(1)-Cl(1)	90.40(17)
C(1)-Ir(1)-Cl(1)	90.8(2)
C(21)-Ir(1)-Cl(1)	176.5(3)
C(19)-Ir(1)-Cl(1)	88.4(3)
C(20)-Ir(1)-Cl(1)	87.8(4)
N(2)-Ir(1)-Cl(1)	89.33(17)
C(10)-N(1)-C(13)	121.0(7)
C(10)-N(1)-Ir(1)	116.9(5)
C(13)-N(1)-Ir(1)	122.1(6)
C(18)-N(2)-C(14)	117.9(8)
C(18)-N(2)-Ir(1)	128.7(7)
C(14)-N(2)-Ir(1)	113.5(5)
C(2)-C(1)-C(6)	113.9(8)
C(2)-C(1)-Ir(1)	135.2(8)
C(6)-C(1)-Ir(1)	110.9(6)
C(1)-C(2)-C(3)	122.8(10)

C(1)-C(2)-H(2)	118.6
C(3)-C(2)-H(2)	118.6
C(4)-C(3)-C(2)	121.5(10)
C(4)-C(3)-H(3)	119.2
C(2)-C(3)-H(3)	119.2
C(3)-C(4)-C(5)	119.9(10)
C(3)-C(4)-H(4)	120.1
C(5)-C(4)-H(4)	120.1
C(4)-C(5)-C(7)	126.2(11)
C(4)-C(5)-C(6)	117.5(11)
C(7)-C(5)-C(6)	116.2(11)
C(1)-C(6)-C(10)	117.4(7)
C(1)-C(6)-C(5)	124.2(9)
C(10)-C(6)-C(5)	118.4(10)
C(8)-C(7)-C(5)	126.5(11)
C(8)-C(7)-H(7)	116.7
C(5)-C(7)-H(7)	116.7
C(7)-C(8)-C(9)	118.3(11)
C(7)-C(8)-H(8)	120.9
C(9)-C(8)-H(8)	120.9
C(11)-C(9)-C(10)	115.3(9)
C(11)-C(9)-C(8)	127.3(10)
C(10)-C(9)-C(8)	117.4(10)
N(1)-C(10)-C(9)	122.5(8)
N(1)-C(10)-C(6)	114.4(8)
C(9)-C(10)-C(6)	123.0(8)
C(12)-C(11)-C(9)	122.4(9)
C(12)-C(11)-H(11)	118.8
C(9)-C(11)-H(11)	118.8
C(11)-C(12)-C(13)	119.0(9)
C(11)-C(12)-H(12)	120.5
C(13)-C(12)-H(12)	120.5
N(1)-C(13)-C(12)	119.8(8)
N(1)-C(13)-C(14)	113.7(8)
C(12)-C(13)-C(14)	126.6(8)
N(2)-C(14)-C(15)	121.5(8)
N(2)-C(14)-C(13)	115.3(7)
C(15)-C(14)-C(13)	123.1(9)
C(16)-C(15)-C(14)	119.8(10)
C(16)-C(15)-H(15)	120.1
C(14)-C(15)-H(15)	120.1
C(15)-C(16)-C(17)	119.2(9)
C(15)-C(16)-H(16)	120.4
C(17)-C(16)-H(16)	120.4
C(16)-C(17)-C(18)	119.9(10)
C(16)-C(17)-H(17)	120.0

C(18)-C(17)-H(17)	120.0
N(2)-C(18)-C(17)	121.7(11)
N(2)-C(18)-H(18)	119.1
C(17)-C(18)-H(18)	119.1
C(20)-C(19)-Ir(1)	71.5(6)
C(20)-C(19)-H(19A)	125(5)
Ir(1)-C(19)-H(19A)	104(5)
C(20)-C(19)-H(19B)	117(4)
Ir(1)-C(19)-H(19B)	92(3)
H(19A)-C(19)-H(19B)	118(6)
C(19)-C(20)-Ir(1)	71.4(6)
C(19)-C(20)-H(20A)	127(7)
Ir(1)-C(20)-H(20A)	106(6)
C(22)-C(21)-Ir(1)	125.5(9)
C(22)-C(21)-H(21A)	105.9
Ir(1)-C(21)-H(21A)	105.9
C(22)-C(21)-H(21B)	105.9
Ir(1)-C(21)-H(21B)	105.9
H(21A)-C(21)-H(21B)	106.3
C(21)-C(22)-H(22A)	109.5
C(21)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5
C(21)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5
N(3)-Ir(2)-C(23)	79.2(3)
N(3)-Ir(2)-C(43)	93.6(3)
C(23)-Ir(2)-C(43)	89.6(3)
N(3)-Ir(2)-C(41)	162.1(4)
C(23)-Ir(2)-C(41)	118.6(4)
C(43)-Ir(2)-C(41)	88.0(4)
N(3)-Ir(2)-C(42)	160.6(4)
C(23)-Ir(2)-C(42)	81.9(4)
C(43)-Ir(2)-C(42)	90.4(4)
C(41)-Ir(2)-C(42)	36.8(4)
N(3)-Ir(2)-N(4)	75.6(3)
C(23)-Ir(2)-N(4)	154.6(3)
C(43)-Ir(2)-N(4)	89.3(3)
C(41)-Ir(2)-N(4)	86.7(4)
C(42)-Ir(2)-N(4)	123.5(4)
N(3)-Ir(2)-Cl(2)	88.98(17)
C(23)-Ir(2)-Cl(2)	92.9(2)
C(43)-Ir(2)-Cl(2)	176.8(3)
C(41)-Ir(2)-Cl(2)	88.9(3)
C(42)-Ir(2)-Cl(2)	87.8(3)
N(4)-Ir(2)-Cl(2)	89.38(19)

C(32)-N(3)-C(35)	122.0(8)
C(32)-N(3)-Ir(2)	117.8(5)
C(35)-N(3)-Ir(2)	120.1(6)
C(36)-N(4)-C(40)	116.8(8)
C(36)-N(4)-Ir(2)	114.5(6)
C(40)-N(4)-Ir(2)	128.6(7)
C(24)-C(23)-C(28)	115.7(8)
C(24)-C(23)-Ir(2)	133.9(8)
C(28)-C(23)-Ir(2)	110.3(5)
C(23)-C(24)-C(25)	120.4(10)
C(23)-C(24)-H(24)	119.8
C(25)-C(24)-H(24)	119.8
C(26)-C(25)-C(24)	121.2(9)
C(26)-C(25)-H(25)	119.4
C(24)-C(25)-H(25)	119.4
C(25)-C(26)-C(27)	122.1(10)
C(25)-C(26)-H(26)	119.0
C(27)-C(26)-H(26)	119.0
C(26)-C(27)-C(29)	125.7(10)
C(26)-C(27)-C(28)	114.4(10)
C(29)-C(27)-C(28)	119.9(9)
C(23)-C(28)-C(32)	118.2(8)
C(23)-C(28)-C(27)	126.0(8)
C(32)-C(28)-C(27)	115.8(9)
C(30)-C(29)-C(27)	122.5(10)
C(30)-C(29)-H(29)	118.8
C(27)-C(29)-H(29)	118.8
C(29)-C(30)-C(31)	120.6(11)
C(29)-C(30)-H(30)	119.7
C(31)-C(30)-H(30)	119.7
C(33)-C(31)-C(32)	116.4(9)
C(33)-C(31)-C(30)	126.6(10)
C(32)-C(31)-C(30)	117.0(10)
N(3)-C(32)-C(28)	114.2(8)
N(3)-C(32)-C(31)	121.6(8)
C(28)-C(32)-C(31)	124.2(9)
C(34)-C(33)-C(31)	120.3(9)
C(34)-C(33)-H(33)	119.9
C(31)-C(33)-H(33)	119.9
C(33)-C(34)-C(35)	121.9(10)
C(33)-C(34)-H(34)	119.0
C(35)-C(34)-H(34)	119.0
N(3)-C(35)-C(34)	117.7(9)
N(3)-C(35)-C(36)	114.7(8)
C(34)-C(35)-C(36)	127.6(9)
N(4)-C(36)-C(37)	122.0(9)

N(4)-C(36)-C(35)	115.0(8)
C(37)-C(36)-C(35)	122.9(10)
C(38)-C(37)-C(36)	118.9(11)
C(38)-C(37)-H(37)	120.5
C(36)-C(37)-H(37)	120.5
C(37)-C(38)-C(39)	119.2(10)
C(37)-C(38)-H(38)	120.4
C(39)-C(38)-H(38)	120.4
C(40)-C(39)-C(38)	119.0(11)
C(40)-C(39)-H(39)	120.5
C(38)-C(39)-H(39)	120.5
N(4)-C(40)-C(39)	124.0(11)
N(4)-C(40)-H(40)	118.0
C(39)-C(40)-H(40)	118.0
C(42)-C(41)-Ir(2)	71.9(5)
C(42)-C(41)-H(41A)	118(4)
Ir(2)-C(41)-H(41A)	110(4)
C(42)-C(41)-H(41B)	138(8)
Ir(2)-C(41)-H(41B)	127(7)
H(41A)-C(41)-H(41B)	92(8)
C(41)-C(42)-Ir(2)	71.3(6)
C(41)-C(42)-H(42A)	120(4)
Ir(2)-C(42)-H(42A)	105(4)
C(41)-C(42)-H(42B)	116(4)
Ir(2)-C(42)-H(42B)	107(4)
H(42A)-C(42)-H(42B)	121(6)
C(44)-C(43)-Ir(2)	120.1(7)
C(44)-C(43)-H(43A)	107.3
Ir(2)-C(43)-H(43A)	107.3
C(44)-C(43)-H(43B)	107.3
Ir(2)-C(43)-H(43B)	107.3
H(43A)-C(43)-H(43B)	106.9
C(43)-C(44)-H(44A)	109.5
C(43)-C(44)-H(44B)	109.5
H(44A)-C(44)-H(44B)	109.5
C(43)-C(44)-H(44C)	109.5
H(44A)-C(44)-H(44C)	109.5
H(44B)-C(44)-H(44C)	109.5

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+1 #2 x,-y+3/2,z

Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for (NNC-tb)Ir(Et)(C_2H_4)(Cl). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

	U^{11}	U^{22}	U^{33}	U^{23}
Ir(1)	38(1)	57(1)	46(1)	5(1)
Cl(1)	47(1)	55(1)	63(1)	-2(1)
N(1)	38(4)	42(3)	44(4)	-1(3)
N(2)	41(4)	53(4)	78(6)	-5(4)
C(1)	51(5)	52(5)	64(6)	8(5)
C(2)	40(5)	70(6)	97(8)	27(6)
C(3)	53(7)	58(7)	148(12)	27(8)
C(4)	28(5)	56(6)	181(14)	-10(8)
C(5)	58(7)	52(6)	119(10)	-19(6)
C(6)	39(5)	38(4)	94(8)	5(5)
C(7)	83(9)	61(7)	138(13)	-31(8)
C(8)	106(9)	66(7)	85(8)	-16(6)
C(9)	90(8)	54(6)	61(7)	-3(5)
C(10)	50(5)	37(4)	61(6)	-6(4)
C(11)	127(10)	69(7)	43(6)	-8(5)
C(12)	76(7)	57(6)	61(6)	1(5)
C(13)	43(5)	50(5)	60(6)	8(4)
C(14)	44(5)	40(4)	72(6)	-4(4)
C(15)	55(6)	55(5)	97(8)	0(5)
C(16)	39(6)	53(6)	164(13)	12(7)
C(17)	37(6)	57(6)	161(13)	-18(7)
C(18)	51(6)	69(6)	101(8)	-22(6)
C(19)	90(9)	86(8)	47(6)	20(5)
C(20)	82(9)	163(12)	37(6)	-1(6)
C(21)	63(6)	95(8)	82(8)	5(6)
C(22)	172(10)	147(9)	185(10)	-31(8)
Ir(2)	42(1)	58(1)	45(1)	3(1)
Cl(2)	43(1)	58(1)	64(1)	-4(1)
N(3)	40(4)	49(4)	47(4)	-3(3)
N(4)	44(4)	68(5)	83(6)	-13(4)
C(23)	29(4)	44(5)	78(7)	0(4)
C(24)	57(6)	55(5)	81(7)	5(5)
C(25)	45(6)	46(5)	122(10)	-14(6)
C(26)	52(7)	69(7)	140(12)	-40(8)
C(27)	56(6)	63(6)	88(8)	-32(6)
C(28)	51(5)	42(5)	64(6)	-5(4)
C(29)	96(8)	68(7)	69(8)	-27(6)

C(30)	114(10)	83(8)	64(7)	-9(6)
C(31)	90(8)	58(6)	51(6)	-9(5)
C(32)	58(6)	42(5)	54(5)	-2(4)
C(33)	113(9)	68(6)	45(6)	5(5)
C(34)	91(8)	67(6)	72(8)	5(6)
C(35)	53(5)	50(5)	61(6)	4(4)
C(36)	50(5)	55(5)	86(8)	-15(5)
C(37)	40(5)	58(6)	142(11)	-5(6)
C(38)	41(6)	67(7)	192(15)	-28(9)
C(39)	49(7)	89(8)	163(13)	-43(9)
C(40)	49(6)	86(7)	105(9)	-31(6)
C(41)	96(9)	107(9)	32(5)	-15(5)
C(42)	98(9)	85(8)	44(6)	18(5)
C(43)	66(6)	54(5)	84(7)	7(5)
C(44)	96(9)	76(7)	145(12)	-30(8)

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for (NNC-tb)Ir(Et)(C_2H_4)(Cl).

	x	y	z	U(eq)
H(2)	5316	1640	2238	85
H(3)	7677	889	1808	106
H(4)	7955	682	748	107
H(7)	6758	1242	-358	111
H(8)	4709	1768	-1004	100
H(11)	1687	2589	-1063	93
H(12)	-538	3328	-555	78
H(15)	-2547	4136	57	83
H(16)	-4487	4929	730	104
H(17)	-3986	4838	1805	103
H(18)	-1524	4081	2188	87
H(19A)	3220(90)	1710(80)	2670(40)	60(30)
H(19B)	3430(60)	3320(60)	2460(30)	16(15)
H(20A)	470(100)	2830(90)	2670(40)	70(30)
H(21A)	1687	644	2204	98
H(21B)	178	1109	1815	98
H(22A)	1814	365	1005	247
H(22B)	851	-498	1439	247
H(22C)	2590	-535	1594	247
H(24)	4751	8999	2927	78
H(25)	2537	9674	3513	86
H(26)	2501	9235	4586	102
H(29)	3934	8350	5621	90
H(30)	6165	7471	6132	102
H(33)	9149	6722	6047	89
H(34)	11176	6220	5404	91
H(37)	13094	5780	4692	96
H(38)	14858	5311	3892	118
H(39)	14069	5798	2841	116
H(40)	11562	6637	2615	93
H(41A)	9120(80)	6430(80)	2230(40)	60(20)
H(41B)	9630(150)	7800(130)	2100(60)	180(60)
H(42A)	6360(80)	7130(70)	2450(30)	50(20)
H(42B)	6810(80)	8900(80)	2370(30)	50(20)
H(43A)	9782	9426	3049	80
H(43B)	8031	10003	3023	80
H(44A)	8103	10058	4096	154
H(44B)	9056	11006	3710	154
H(44C)	9895	9666	4059	154

Figure 10. ORTEP structure of (NNC-tb)Ir(Et)(C_2H_4)(TFA) with ellipsoids at 50%.

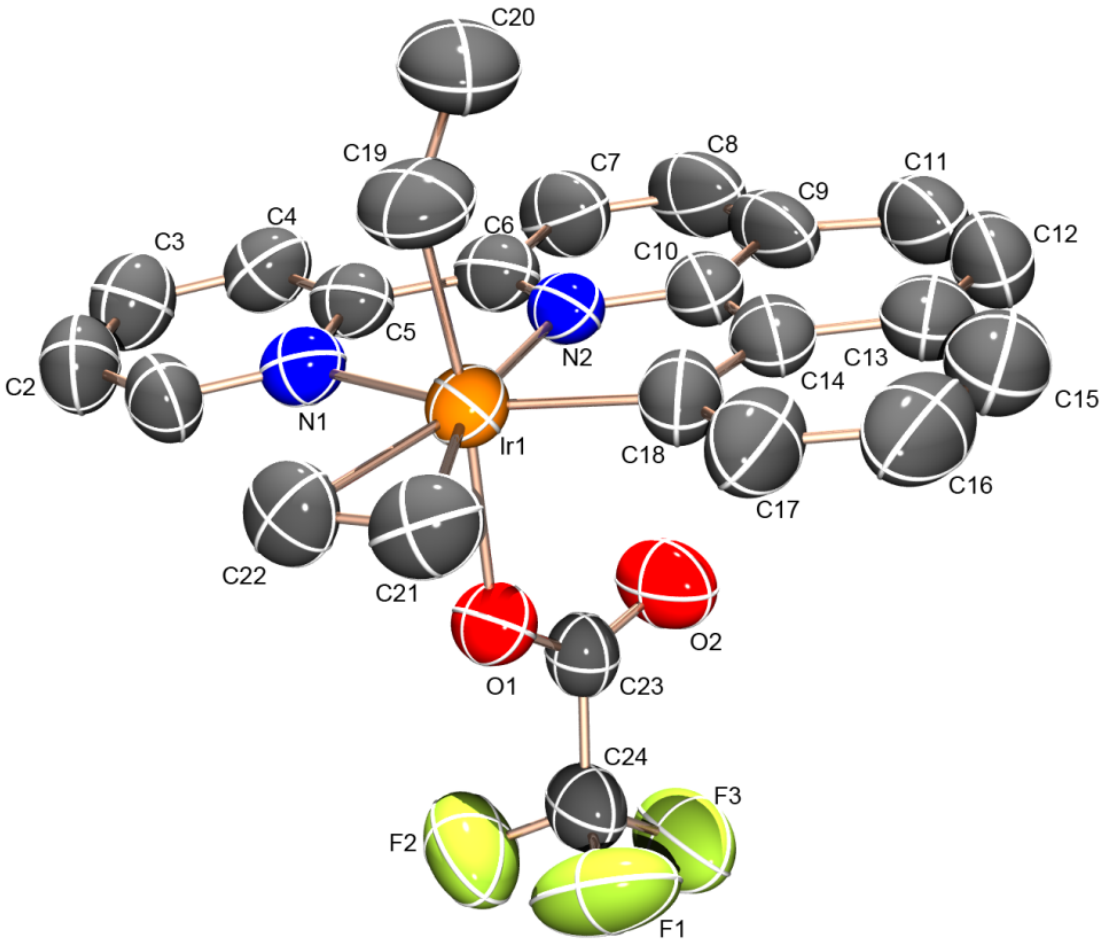


Table 7. Crystal data and structure refinement for (NNC-tb)Ir(Et)(C_2H_4)(TFA).

Identification code	periana21	
Empirical formula	C24 H20 F3 Ir N2 O2	
Formula weight	617.62	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.394(3) Å	α= 85.562(3)°.
	b = 10.594(3) Å	β= 82.141(3)°.
	c = 11.122(3) Å	γ = 77.186(3)°.
Volume	1067.9(5) $Å^3$	
Z	2	
Density (calculated)	1.921 Mg/m^3	
Absorption coefficient	6.301 mm^{-1}	
F(000)	596	
Crystal size	0.16 x 0.12 x 0.02 mm^3	
Theta range for data collection	1.85 to 28.32°.	
Index ranges	-12<=h<=12, -13<=k<=14, -14<=l<=14	
Reflections collected	10555	
Independent reflections	5239 [R(int) = 0.0333]	
Completeness to theta = 28.32°	98.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.745 and 0.527	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5239 / 30 / 290	
Goodness-of-fit on F^2	1.031	
Final R indices [I>2sigma(I)]	R1 = 0.0468, wR2 = 0.1116	
R indices (all data)	R1 = 0.0700, wR2 = 0.1223	

Largest diff. peak and hole	1.688 and -0.658 e.Å^{-3}

Table 8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for (NNC-tb)Ir(Et)(C_2H_4)(TFA). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ir(1)	6847(1)	7619(1)	6795(1)	65(1)
F(1)	8933(9)	11314(7)	5462(6)	143(3)
F(2)	6867(7)	12309(6)	6159(7)	133(2)
F(3)	8667(8)	12132(6)	7148(6)	131(2)
O(1)	7220(6)	9619(5)	6399(5)	73(1)
O(2)	7978(7)	10011(6)	8121(5)	95(2)
N(1)	4528(7)	8442(6)	7357(5)	65(2)
N(2)	6682(7)	7711(6)	8624(5)	64(2)
C(1)	3424(9)	8810(8)	6666(7)	72(2)
C(2)	1991(10)	9284(9)	7144(9)	85(2)
C(3)	1638(10)	9410(9)	8347(8)	85(2)
C(4)	2742(9)	9045(8)	9092(7)	78(2)
C(5)	4176(9)	8564(7)	8585(6)	66(2)
C(6)	5420(10)	8166(8)	9271(7)	71(2)
C(7)	5340(11)	8249(10)	10552(7)	89(3)
C(8)	6581(13)	7856(10)	11085(8)	99(3)
C(9)	7903(13)	7391(10)	10424(9)	98(3)
C(10)	7920(9)	7320(7)	9135(8)	76(2)
C(11)	9379(13)	6938(10)	10793(11)	105(3)
C(12)	10546(12)	6528(10)	10053(11)	102(3)
C(13)	10572(11)	6419(9)	8797(11)	98(3)
C(14)	9199(10)	6846(7)	8300(9)	84(3)
C(15)	11755(13)	5936(11)	7959(13)	115(3)
C(16)	11656(12)	5902(9)	6729(14)	114(4)
C(17)	10271(10)	6325(9)	6299(12)	99(3)
C(18)	8994(9)	6845(8)	7071(9)	82(2)
C(19)	6405(12)	5760(8)	6931(9)	95(3)
C(20)	6056(15)	5160(10)	8160(13)	139(5)
C(21)	7758(11)	7333(10)	4918(8)	91(3)
C(22)	6269(10)	7830(9)	4954(7)	80(2)
C(23)	7690(7)	10255(7)	7079(6)	61(2)
C(24)	8021(10)	11512(8)	6491(7)	77(2)

Table 9. Bond lengths [Å] and angles [°] for (NNC-tb)Ir(Et)(C_2H_4)(TFA).

Ir(1)-N(2)	2.028(6)
Ir(1)-C(18)	2.056(8)
Ir(1)-C(19)	2.091(8)
Ir(1)-C(21)	2.163(9)
Ir(1)-C(22)	2.172(8)
Ir(1)-N(1)	2.183(6)
Ir(1)-O(1)	2.224(5)
F(1)-C(24)	1.333(10)
F(2)-C(24)	1.296(9)
F(3)-C(24)	1.306(8)
O(1)-C(23)	1.235(8)
O(2)-C(23)	1.221(8)
N(1)-C(1)	1.346(9)
N(1)-C(5)	1.370(9)
N(2)-C(6)	1.312(10)
N(2)-C(10)	1.333(10)
C(1)-C(2)	1.376(12)
C(1)-H(1)	0.9300
C(2)-C(3)	1.344(12)
C(2)-H(2)	0.9300
C(3)-C(4)	1.385(11)
C(3)-H(3)	0.9300
C(4)-C(5)	1.387(11)
C(4)-H(4)	0.9300
C(5)-C(6)	1.450(11)
C(6)-C(7)	1.425(11)
C(7)-C(8)	1.349(13)
C(7)-H(7)	0.9300
C(8)-C(9)	1.364(14)
C(8)-H(8)	0.9300
C(9)-C(10)	1.439(13)
C(9)-C(11)	1.466(14)
C(10)-C(14)	1.435(13)
C(11)-C(12)	1.290(15)
C(11)-H(11)	0.9300
C(12)-C(13)	1.407(15)
C(12)-H(12)	0.9300
C(13)-C(15)	1.380(16)
C(13)-C(14)	1.439(12)
C(14)-C(18)	1.407(13)
C(15)-C(16)	1.387(17)
C(15)-H(15)	0.9300
C(16)-C(17)	1.414(14)

C(16)-H(16)	0.9300
C(17)-C(18)	1.407(13)
C(17)-H(17)	0.9300
C(19)-C(20)	1.489(15)
C(19)-H(19A)	0.9700
C(19)-H(19B)	0.9700
C(20)-H(20A)	0.9600
C(20)-H(20B)	0.9600
C(20)-H(20C)	0.9600
C(21)-C(22)	1.377(13)
C(21)-H(21A)	0.9700
C(21)-H(21B)	0.9700
C(22)-H(22A)	0.9700
C(22)-H(22B)	0.9700
C(23)-C(24)	1.514(11)

N(2)-Ir(1)-C(18)	79.9(4)
N(2)-Ir(1)-C(19)	92.5(3)
C(18)-Ir(1)-C(19)	90.2(4)
N(2)-Ir(1)-C(21)	161.7(3)
C(18)-Ir(1)-C(21)	81.9(4)
C(19)-Ir(1)-C(21)	89.6(4)
N(2)-Ir(1)-C(22)	161.1(3)
C(18)-Ir(1)-C(22)	118.9(4)
C(19)-Ir(1)-C(22)	88.6(4)
C(21)-Ir(1)-C(22)	37.0(3)
N(2)-Ir(1)-N(1)	75.0(2)
C(18)-Ir(1)-N(1)	154.9(3)
C(19)-Ir(1)-N(1)	90.2(3)
C(21)-Ir(1)-N(1)	123.2(3)
C(22)-Ir(1)-N(1)	86.1(3)
N(2)-Ir(1)-O(1)	94.7(2)
C(18)-Ir(1)-O(1)	93.9(3)
C(19)-Ir(1)-O(1)	172.2(3)
C(21)-Ir(1)-O(1)	84.4(3)
C(22)-Ir(1)-O(1)	83.6(3)
N(1)-Ir(1)-O(1)	89.0(2)
C(23)-O(1)-Ir(1)	126.2(5)
C(1)-N(1)-C(5)	117.1(7)
C(1)-N(1)-Ir(1)	128.7(5)
C(5)-N(1)-Ir(1)	114.2(5)
C(6)-N(2)-C(10)	121.6(7)
C(6)-N(2)-Ir(1)	121.5(5)
C(10)-N(2)-Ir(1)	116.9(6)
N(1)-C(1)-C(2)	122.8(7)
N(1)-C(1)-H(1)	118.6

C(2)-C(1)-H(1)	118.6
C(3)-C(2)-C(1)	120.4(8)
C(3)-C(2)-H(2)	119.8
C(1)-C(2)-H(2)	119.8
C(2)-C(3)-C(4)	118.7(8)
C(2)-C(3)-H(3)	120.6
C(4)-C(3)-H(3)	120.6
C(3)-C(4)-C(5)	119.7(8)
C(3)-C(4)-H(4)	120.2
C(5)-C(4)-H(4)	120.2
N(1)-C(5)-C(4)	121.3(7)
N(1)-C(5)-C(6)	114.1(7)
C(4)-C(5)-C(6)	124.6(7)
N(2)-C(6)-C(7)	120.4(8)
N(2)-C(6)-C(5)	115.1(7)
C(7)-C(6)-C(5)	124.5(8)
C(8)-C(7)-C(6)	118.9(10)
C(8)-C(7)-H(7)	120.5
C(6)-C(7)-H(7)	120.5
C(7)-C(8)-C(9)	121.4(8)
C(7)-C(8)-H(8)	119.3
C(9)-C(8)-H(8)	119.3
C(8)-C(9)-C(10)	117.4(8)
C(8)-C(9)-C(11)	131.1(10)
C(10)-C(9)-C(11)	111.6(11)
N(2)-C(10)-C(14)	114.4(8)
N(2)-C(10)-C(9)	120.4(9)
C(14)-C(10)-C(9)	125.2(8)
C(12)-C(11)-C(9)	124.2(11)
C(12)-C(11)-H(11)	117.9
C(9)-C(11)-H(11)	117.9
C(11)-C(12)-C(13)	124.8(10)
C(11)-C(12)-H(12)	117.6
C(13)-C(12)-H(12)	117.6
C(15)-C(13)-C(12)	128.4(10)
C(15)-C(13)-C(14)	114.4(11)
C(12)-C(13)-C(14)	117.2(10)
C(18)-C(14)-C(10)	116.9(7)
C(18)-C(14)-C(13)	126.1(10)
C(10)-C(14)-C(13)	117.0(9)
C(13)-C(15)-C(16)	123.4(11)
C(13)-C(15)-H(15)	118.3
C(16)-C(15)-H(15)	118.3
C(15)-C(16)-C(17)	119.4(12)
C(15)-C(16)-H(16)	120.3
C(17)-C(16)-H(16)	120.3

C(18)-C(17)-C(16)	122.0(11)
C(18)-C(17)-H(17)	119.0
C(16)-C(17)-H(17)	119.0
C(17)-C(18)-C(14)	114.5(8)
C(17)-C(18)-Ir(1)	133.7(8)
C(14)-C(18)-Ir(1)	111.7(7)
C(20)-C(19)-Ir(1)	118.7(7)
C(20)-C(19)-H(19A)	107.6
Ir(1)-C(19)-H(19A)	107.6
C(20)-C(19)-H(19B)	107.6
Ir(1)-C(19)-H(19B)	107.6
H(19A)-C(19)-H(19B)	107.1
C(19)-C(20)-H(20A)	109.5
C(19)-C(20)-H(20B)	109.5
H(20A)-C(20)-H(20B)	109.5
C(19)-C(20)-H(20C)	109.5
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
C(22)-C(21)-Ir(1)	71.8(5)
C(22)-C(21)-H(21A)	116.4
Ir(1)-C(21)-H(21A)	116.4
C(22)-C(21)-H(21B)	116.4
Ir(1)-C(21)-H(21B)	116.4
H(21A)-C(21)-H(21B)	113.4
C(21)-C(22)-Ir(1)	71.1(5)
C(21)-C(22)-H(22A)	116.5
Ir(1)-C(22)-H(22A)	116.5
C(21)-C(22)-H(22B)	116.5
Ir(1)-C(22)-H(22B)	116.5
H(22A)-C(22)-H(22B)	113.5
O(2)-C(23)-O(1)	130.5(7)
O(2)-C(23)-C(24)	115.7(6)
O(1)-C(23)-C(24)	113.8(6)
F(2)-C(24)-F(3)	108.2(7)
F(2)-C(24)-F(1)	103.9(8)
F(3)-C(24)-F(1)	104.6(8)
F(2)-C(24)-C(23)	113.0(7)
F(3)-C(24)-C(23)	114.7(7)
F(1)-C(24)-C(23)	111.6(7)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+1 #2 x,-y+3/2,z

Table 10. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for (NNC-tb)Ir(Et)(C_2H_4)(TFA). The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

	U^{11}	U^{22}	U^{33}	U^{23}
Ir(1)	71(1)	61(1)	68(1)	-4(1)
F(1)	197(7)	148(6)	101(4)	7(4)
F(2)	148(5)	84(3)	179(5)	17(4)
F(3)	189(6)	101(4)	141(5)	16(4)
O(1)	88(3)	72(3)	69(3)	0(3)
O(2)	138(5)	92(4)	70(3)	2(3)
N(1)	76(4)	67(4)	56(3)	-7(3)
N(2)	79(4)	61(3)	62(3)	8(3)
C(1)	75(5)	76(5)	66(4)	1(4)
C(2)	72(5)	91(6)	94(6)	-6(5)
C(3)	79(5)	88(6)	91(6)	-19(5)
C(4)	78(5)	86(6)	75(5)	-10(4)
C(5)	85(5)	65(4)	57(4)	-3(3)
C(6)	88(6)	71(5)	63(4)	1(4)
C(7)	117(7)	105(7)	59(4)	4(4)
C(8)	141(9)	110(8)	60(5)	15(5)
C(9)	139(9)	88(6)	88(6)	33(5)
C(10)	80(5)	60(4)	99(6)	17(4)
C(11)	123(7)	99(6)	108(6)	28(5)
C(12)	98(6)	86(6)	132(7)	20(5)
C(13)	91(5)	68(5)	140(7)	25(5)
C(14)	92(6)	50(4)	123(7)	14(4)
C(15)	103(6)	88(6)	160(8)	16(6)
C(16)	85(7)	64(6)	189(13)	-8(7)
C(17)	75(6)	70(6)	153(10)	-6(6)
C(18)	70(5)	66(5)	116(7)	4(5)
C(19)	124(7)	59(5)	110(7)	-8(5)
C(20)	166(12)	81(7)	186(13)	32(8)
C(21)	108(7)	93(6)	76(5)	-16(5)
C(22)	93(6)	96(6)	58(4)	-18(4)
C(23)	59(4)	71(4)	53(4)	-7(3)
C(24)	93(6)	73(5)	74(5)	-1(4)

Table 11. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for (NNC-tb)Ir(Et)(C_2H_4)(TFA).

	x	y	z	U(eq)
H(1)	3639	8740	5829	86
H(2)	1262	9520	6631	102
H(3)	672	9736	8674	102
H(4)	2523	9121	9928	94
H(7)	4447	8569	11013	107
H(8)	6535	7904	11922	119
H(11)	9480	6947	11612	126
H(12)	11436	6286	10375	122
H(15)	12665	5617	8232	138
H(16)	12493	5604	6193	137
H(17)	10204	6257	5481	119
H(19A)	5584	5786	6480	114
H(19B)	7252	5184	6521	114
H(20A)	6926	4944	8562	208
H(20B)	5707	4388	8083	208
H(20C)	5309	5761	8628	208
H(21A)	8400	7901	4560	109
H(21B)	8104	6442	4687	109
H(22A)	5681	7250	4749	97
H(22B)	5976	8709	4621	97

Figure 11. ORTEP structure of (NNC-tb)Ir(C_2H_4)$(TFA)_2$ with ellipsoids at 50%.

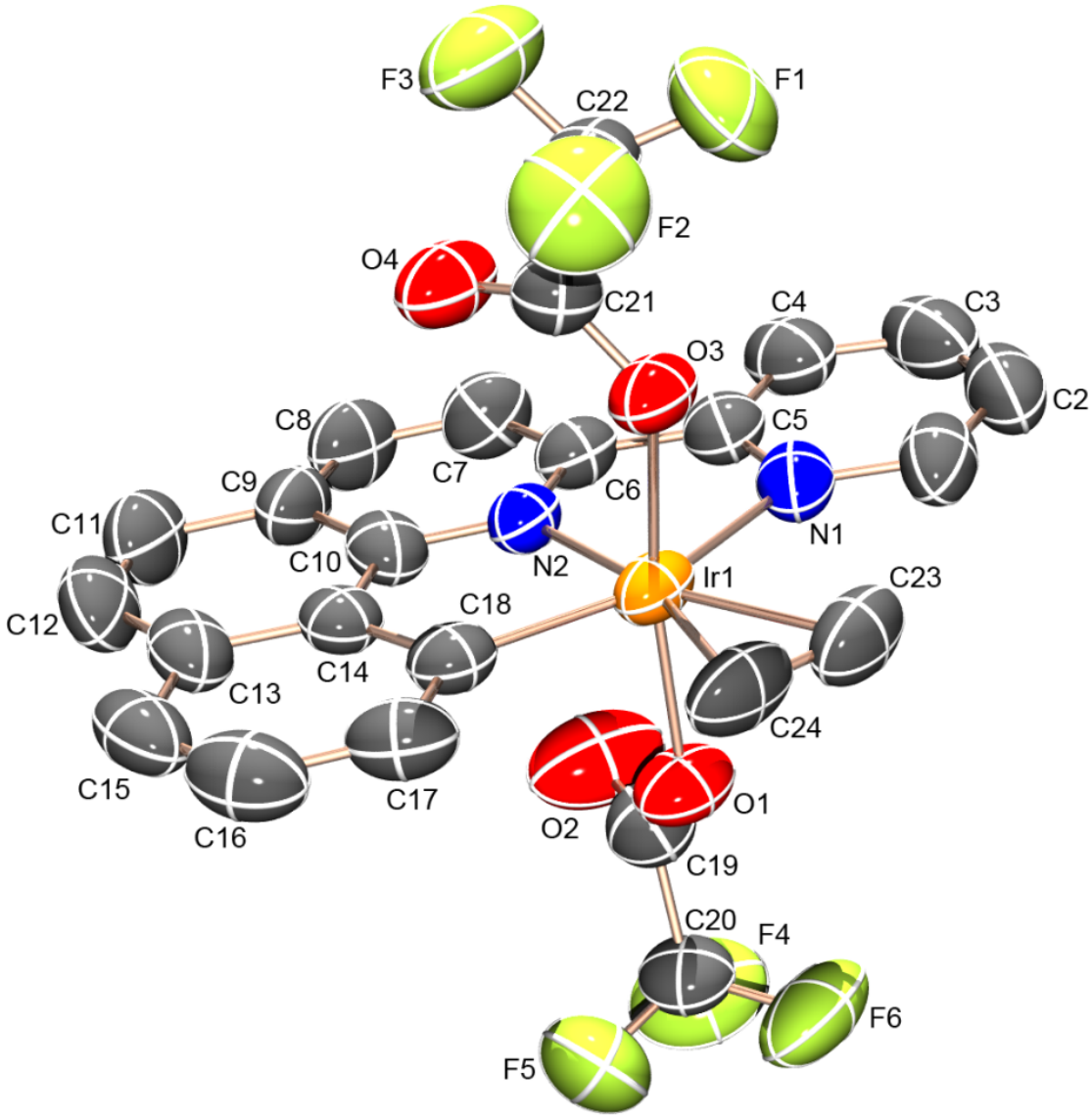


Table 12. Crystal data and structure refinement for (NNC-tb)Ir(C_2H_4)$(TFA)_2$.

Identification code	periana22	
Empirical formula	C24 H14 F6 Ir N2 O4	
Formula weight	700.57	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.2114(12) Å	α= 99.972(2)°.
	b = 9.5161(12) Å	β= 104.037(2)°.
	c = 14.2557(18) Å	γ= 98.975(2)°.
Volume	1168.1(3) $Å^3$	
Z	2	
Density (calculated)	1.992 Mg/m^3	
Absorption coefficient	5.798 mm^{-1}	
F(000)	670	
Crystal size	0.20 x 0.13 x 0.05 mm^3	
Theta range for data collection	1.51 to 28.42°.	
Index ranges	-12<=h<=11, -12<=k<=12, -19<=l<=18	
Reflections collected	11624	
Independent reflections	5759 [R(int) = 0.0196]	
Completeness to theta = 28.42°	98.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.745 and 0.578	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5759 / 78 / 401	
Goodness-of-fit on F^2	1.053	
Final R indices [I>2sigma(I)]	R1 = 0.0418, wR2 = 0.1060	
R indices (all data)	R1 = 0.0488, wR2 = 0.1113	

Largest diff. peak and hole	2.536 and -0.628 e.Å^{-3}

Table 13. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for (NNC-tb)Ir(C_2H_4)$(TFA)_2$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ir(1)	9363(1)	7538(1)	6917(1)	55(1)
F(1)	10981(14)	12628(13)	7359(15)	125(4)
F(2)	12632(18)	11800(14)	6796(10)	127(4)
F(3)	13184(16)	12672(13)	8307(10)	121(4)
F(4)	6040(19)	2411(13)	6826(10)	130(4)
F(5)	7824(17)	2258(15)	6145(14)	135(5)
F(6)	5896(13)	3126(11)	5540(9)	123(4)
F(1A)	11330(20)	12877(17)	8024(15)	98(5)
F(2A)	11580(30)	12120(20)	6711(13)	113(6)
F(3A)	13490(20)	12390(20)	7842(19)	104(6)
F(4A)	5500(30)	2810(30)	6130(20)	148(8)
F(5A)	7150(30)	2130(20)	6970(18)	138(7)
F(6A)	7440(30)	2500(20)	5692(14)	104(6)
O(1)	8155(6)	5430(5)	6485(4)	76(1)
O(2)	7948(10)	4794(8)	7874(5)	125(2)
O(3)	10407(6)	9657(5)	7079(4)	69(1)
O(4)	12174(7)	10154(5)	8542(4)	83(1)
N(1)	7566(6)	8367(6)	7488(5)	65(1)
N(2)	9974(5)	7524(5)	8361(4)	51(1)
C(1)	6345(9)	8856(8)	7002(7)	78(2)
C(2)	5364(9)	9421(9)	7461(8)	89(2)
C(3)	5597(9)	9531(9)	8451(9)	93(3)
C(4)	6834(9)	9056(8)	8982(7)	80(2)
C(5)	7796(7)	8464(7)	8481(5)	63(2)
C(6)	9118(7)	7948(7)	8968(5)	60(1)
C(7)	9573(9)	7865(9)	9968(5)	76(2)
C(8)	10852(10)	7361(9)	10318(6)	81(2)
C(9)	11780(8)	6985(7)	9704(5)	69(2)
C(10)	11223(7)	7026(6)	8714(5)	61(1)
C(11)	13211(10)	6493(9)	9998(7)	86(2)
C(12)	13943(9)	6140(8)	9308(7)	84(2)
C(13)	13405(8)	6184(7)	8309(6)	70(2)
C(14)	12029(7)	6653(6)	7993(5)	55(1)
C(15)	14141(9)	5766(7)	7572(7)	80(2)
C(16)	13504(11)	5855(9)	6622(8)	89(2)
C(17)	12165(9)	6357(8)	6330(6)	74(2)
C(18)	11366(7)	6778(6)	7014(5)	59(1)

C(19)	7762(9)	4595(8)	6975(6)	74(2)
C(20)	6898(9)	3075(8)	6379(6)	73(2)
C(21)	11493(8)	10415(7)	7763(5)	61(1)
C(22)	12020(9)	11929(8)	7560(6)	72(2)
C(23)	8102(11)	7800(11)	5473(6)	83(2)
C(24)	9372(11)	7348(10)	5367(5)	83(2)

Table 14. Bond lengths [Å] and angles [°] for (NNC-tb)Ir(C_2H_4)$(TFA)_2$.

Ir(1)-N(2)	2.000(5)
Ir(1)-O(3)	2.042(4)
Ir(1)-O(1)	2.045(5)
Ir(1)-C(18)	2.068(7)
Ir(1)-C(23)	2.182(8)
Ir(1)-C(24)	2.188(8)
Ir(1)-N(1)	2.206(6)
F(1)-C(22)	1.254(13)
F(2)-C(22)	1.338(14)
F(3)-C(22)	1.316(13)
F(4)-C(20)	1.287(12)
F(5)-C(20)	1.304(15)
F(6)-C(20)	1.336(13)
F(1A)-C(22)	1.355(17)
F(2A)-C(22)	1.233(17)
F(3A)-C(22)	1.29(2)
F(4A)-C(20)	1.22(2)
F(5A)-C(20)	1.34(2)
F(6A)-C(20)	1.28(2)
O(1)-C(19)	1.214(9)
O(2)-C(19)	1.228(10)
O(3)-C(21)	1.237(8)
O(4)-C(21)	1.224(8)
N(1)-C(5)	1.363(9)
N(1)-C(1)	1.363(9)
N(2)-C(10)	1.332(8)
N(2)-C(6)	1.357(8)
C(1)-C(2)	1.360(13)
C(1)-H(1)	0.9300
C(2)-C(3)	1.358(14)
C(2)-H(2)	0.9300
C(3)-C(4)	1.392(12)
C(3)-H(3)	0.9300
C(4)-C(5)	1.392(10)
C(4)-H(4)	0.9300
C(5)-C(6)	1.449(9)
C(6)-C(7)	1.406(10)
C(7)-C(8)	1.357(11)
C(7)-H(7)	0.9300
C(8)-C(9)	1.406(11)
C(8)-H(8)	0.9300
C(9)-C(10)	1.389(10)
C(9)-C(11)	1.458(10)

C(10)-C(14)	1.436(9)
C(11)-C(12)	1.346(12)
C(11)-H(11)	0.9300
C(12)-C(13)	1.401(12)
C(12)-H(12)	0.9300
C(13)-C(14)	1.406(9)
C(13)-C(15)	1.417(11)
C(14)-C(18)	1.414(9)
C(15)-C(16)	1.362(13)
C(15)-H(15)	0.9300
C(16)-C(17)	1.388(13)
C(16)-H(16)	0.9300
C(17)-C(18)	1.399(10)
C(17)-H(17)	0.9300
C(19)-C(20)	1.527(10)
C(21)-C(22)	1.546(9)
C(23)-C(24)	1.341(14)
C(23)-H(23A)	0.95(7)
C(23)-H(23B)	0.98(10)
C(24)-H(24A)	0.96(8)

N(2)-Ir(1)-O(3)	95.83(19)
N(2)-Ir(1)-O(1)	94.8(2)
O(3)-Ir(1)-O(1)	169.32(18)
N(2)-Ir(1)-C(18)	80.0(2)
O(3)-Ir(1)-C(18)	93.8(2)
O(1)-Ir(1)-C(18)	89.3(2)
N(2)-Ir(1)-C(23)	162.0(3)
O(3)-Ir(1)-C(23)	83.6(3)
O(1)-Ir(1)-C(23)	85.9(3)
C(18)-Ir(1)-C(23)	118.0(3)
N(2)-Ir(1)-C(24)	162.2(3)
O(3)-Ir(1)-C(24)	84.2(3)
O(1)-Ir(1)-C(24)	86.1(3)
C(18)-Ir(1)-C(24)	82.2(3)
C(23)-Ir(1)-C(24)	35.7(4)
N(2)-Ir(1)-N(1)	75.6(2)
O(3)-Ir(1)-N(1)	87.8(2)
O(1)-Ir(1)-N(1)	93.6(2)
C(18)-Ir(1)-N(1)	155.6(3)
C(23)-Ir(1)-N(1)	86.4(3)
C(24)-Ir(1)-N(1)	122.1(3)
C(19)-O(1)-Ir(1)	130.3(5)
C(21)-O(3)-Ir(1)	128.3(4)
C(5)-N(1)-C(1)	117.5(7)
C(5)-N(1)-Ir(1)	113.3(4)

C(1)-N(1)-Ir(1)	129.1(6)
C(10)-N(2)-C(6)	119.9(5)
C(10)-N(2)-Ir(1)	118.3(4)
C(6)-N(2)-Ir(1)	121.7(4)
C(2)-C(1)-N(1)	123.0(8)
C(2)-C(1)-H(1)	118.5
N(1)-C(1)-H(1)	118.5
C(3)-C(2)-C(1)	119.7(8)
C(3)-C(2)-H(2)	120.1
C(1)-C(2)-H(2)	120.1
C(2)-C(3)-C(4)	119.4(8)
C(2)-C(3)-H(3)	120.3
C(4)-C(3)-H(3)	120.3
C(3)-C(4)-C(5)	119.0(9)
C(3)-C(4)-H(4)	120.5
C(5)-C(4)-H(4)	120.5
N(1)-C(5)-C(4)	121.3(7)
N(1)-C(5)-C(6)	115.5(6)
C(4)-C(5)-C(6)	123.2(7)
N(2)-C(6)-C(7)	119.3(6)
N(2)-C(6)-C(5)	113.8(6)
C(7)-C(6)-C(5)	127.0(6)
C(8)-C(7)-C(6)	120.1(7)
C(8)-C(7)-H(7)	119.9
C(6)-C(7)-H(7)	119.9
C(7)-C(8)-C(9)	120.8(7)
C(7)-C(8)-H(8)	119.6
C(9)-C(8)-H(8)	119.6
C(10)-C(9)-C(8)	115.8(6)
C(10)-C(9)-C(11)	117.9(7)
C(8)-C(9)-C(11)	126.2(7)
N(2)-C(10)-C(9)	123.8(6)
N(2)-C(10)-C(14)	113.6(6)
C(9)-C(10)-C(14)	122.4(6)
C(12)-C(11)-C(9)	118.7(7)
C(12)-C(11)-H(11)	120.6
C(9)-C(11)-H(11)	120.6
C(11)-C(12)-C(13)	124.2(7)
C(11)-C(12)-H(12)	117.9
C(13)-C(12)-H(12)	117.9
C(12)-C(13)-C(14)	119.0(7)
C(12)-C(13)-C(15)	124.5(7)
C(14)-C(13)-C(15)	116.5(7)
C(13)-C(14)-C(18)	125.1(6)
C(13)-C(14)-C(10)	117.8(6)
C(18)-C(14)-C(10)	117.1(6)

C(16)-C(15)-C(13)	119.1(7)
C(16)-C(15)-H(15)	120.4
C(13)-C(15)-H(15)	120.4
C(15)-C(16)-C(17)	123.5(8)
C(15)-C(16)-H(16)	118.3
C(17)-C(16)-H(16)	118.3
C(16)-C(17)-C(18)	120.6(8)
C(16)-C(17)-H(17)	119.7
C(18)-C(17)-H(17)	119.7
C(17)-C(18)-C(14)	115.2(6)
C(17)-C(18)-Ir(1)	134.0(6)
C(14)-C(18)-Ir(1)	110.9(4)
O(1)-C(19)-O(2)	129.6(8)
O(1)-C(19)-C(20)	115.0(7)
O(2)-C(19)-C(20)	115.4(7)
F(4A)-C(20)-F(6A)	112.0(18)
F(4A)-C(20)-F(4)	54.1(15)
F(6A)-C(20)-F(4)	127.3(12)
F(4A)-C(20)-F(5)	128.8(15)
F(6A)-C(20)-F(5)	33.8(10)
F(4)-C(20)-F(5)	109.3(12)
F(4A)-C(20)-F(6)	49.3(15)
F(6A)-C(20)-F(6)	75.6(13)
F(4)-C(20)-F(6)	102.4(10)
F(5)-C(20)-F(6)	107.7(11)
F(4A)-C(20)-F(5A)	99.4(18)
F(6A)-C(20)-F(5A)	98.2(15)
F(4)-C(20)-F(5A)	48.3(11)
F(5)-C(20)-F(5A)	67.5(14)
F(6)-C(20)-F(5A)	138.0(13)
F(4A)-C(20)-C(19)	119.2(14)
F(6A)-C(20)-C(19)	116.1(10)
F(4)-C(20)-C(19)	113.2(8)
F(5)-C(20)-C(19)	111.7(9)
F(6)-C(20)-C(19)	112.0(7)
F(5A)-C(20)-C(19)	108.0(12)
O(4)-C(21)-O(3)	130.6(6)
O(4)-C(21)-C(22)	117.5(6)
O(3)-C(21)-C(22)	111.9(6)
F(2A)-C(22)-F(1)	60.6(13)
F(2A)-C(22)-F(3A)	106.4(16)
F(1)-C(22)-F(3A)	129.6(12)
F(2A)-C(22)-F(3)	130.1(12)
F(1)-C(22)-F(3)	112.5(11)
F(3A)-C(22)-F(3)	35.7(9)
F(2A)-C(22)-F(2)	47.6(12)

F(1)-C(22)-F(2)	106.7(11)
F(3A)-C(22)-F(2)	67.9(12)
F(3)-C(22)-F(2)	102.8(10)
F(2A)-C(22)-F(1A)	99.1(15)
F(1)-C(22)-F(1A)	40.2(9)
F(3A)-C(22)-F(1A)	110.6(14)
F(3)-C(22)-F(1A)	79.5(12)
F(2)-C(22)-F(1A)	138.5(11)
F(2A)-C(22)-C(21)	118.7(10)
F(1)-C(22)-C(21)	114.8(8)
F(3A)-C(22)-C(21)	113.5(11)
F(3)-C(22)-C(21)	108.8(8)
F(2)-C(22)-C(21)	110.6(8)
F(1A)-C(22)-C(21)	107.5(9)
C(24)-C(23)-Ir(1)	72.4(5)
C(24)-C(23)-H(23A)	120(4)
Ir(1)-C(23)-H(23A)	118(4)
C(24)-C(23)-H(23B)	115(6)
Ir(1)-C(23)-H(23B)	111(6)
H(23A)-C(23)-H(23B)	115(7)
C(23)-C(24)-Ir(1)	71.9(5)
C(23)-C(24)-H(24A)	115(5)
Ir(1)-C(24)-H(24A)	104(5)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+1 #2 x,-y+3/2,z

Table 15. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for (NNC-tb)Ir(C_2H_4)$(TFA)_2$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2} U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

	U^{11}	U^{22}	U^{33}	U^{23}
Ir(1)	59(1)	48(1)	49(1)	13(1)
F(1)	121(7)	83(6)	176(10)	54(6)
F(2)	136(8)	134(7)	125(7)	56(6)
F(3)	130(8)	89(6)	104(7)	23(5)
F(4)	141(8)	113(7)	130(7)	33(5)
F(5)	134(8)	100(6)	160(10)	-2(6)
F(6)	115(6)	103(6)	103(6)	17(5)
F(1A)	133(10)	66(6)	110(9)	21(6)
F(2A)	147(11)	103(9)	82(8)	47(7)
F(3A)	85(8)	95(9)	134(11)	36(8)
F(4A)	124(11)	154(12)	153(12)	32(9)
F(5A)	159(12)	109(9)	138(11)	51(8)
F(6A)	126(10)	89(8)	88(8)	2(6)
O(1)	84(3)	57(2)	73(3)	15(2)
O(2)	170(6)	97(4)	92(4)	23(3)
O(3)	70(3)	51(2)	69(3)	16(2)
O(4)	98(4)	66(3)	67(3)	20(2)
N(1)	55(3)	54(3)	81(4)	22(3)
N(2)	52(2)	47(2)	49(2)	11(2)
C(1)	65(4)	68(4)	94(5)	36(4)
C(2)	64(4)	69(4)	131(8)	32(5)
C(3)	72(5)	69(5)	136(8)	11(5)
C(4)	70(4)	64(4)	100(6)	8(4)
C(5)	57(3)	53(3)	73(4)	11(3)
C(6)	65(3)	55(3)	53(3)	10(3)
C(7)	81(5)	86(5)	59(4)	13(3)
C(8)	97(6)	87(5)	54(4)	26(3)
C(9)	71(4)	63(4)	60(4)	14(3)
C(10)	62(3)	44(3)	78(4)	20(3)
C(11)	90(5)	80(5)	82(5)	33(4)
C(12)	75(5)	68(4)	110(6)	39(4)
C(13)	65(4)	47(3)	100(5)	25(3)
C(14)	60(3)	39(2)	62(3)	11(2)
C(15)	72(4)	49(3)	126(7)	19(4)
C(16)	101(6)	65(4)	107(7)	12(4)
C(17)	85(5)	60(4)	74(4)	4(3)

C(18)	64(3)	44(3)	60(3)	8(2)
C(19)	72(4)	73(4)	77(5)	17(4)
C(20)	73(4)	56(4)	86(5)	14(3)
C(21)	68(4)	58(3)	59(3)	12(3)
C(22)	72(4)	56(3)	79(5)	21(3)
C(23)	87(6)	85(5)	59(4)	23(4)
C(24)	106(6)	80(5)	43(3)	13(3)

Table 16. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for (NNC-tb)Ir(C_2H_4)$(TFA)_2$.

	x	y	z	U(eq)
H(1)	6175	8801	6325	94
H(2)	4538	9731	7099	107
H(3)	4936	9921	8772	111
H(4)	7015	9132	9662	96
H(7)	8996	8155	10391	91
H(8)	11117	7262	10973	97
H(11)	13611	6425	10649	103
H(12)	14866	5848	9508	100
H(15)	15049	5435	7735	96
H(16)	13991	5565	6144	106
H(17)	11795	6415	5673	89
H(23A)	7130(80)	7170(70)	5160(50)	60(17)
H(23B)	8130(110)	8830(120)	5480(70)	120(30)
H(24A)	9250(90)	6320(90)	5120(60)	80(20)

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