

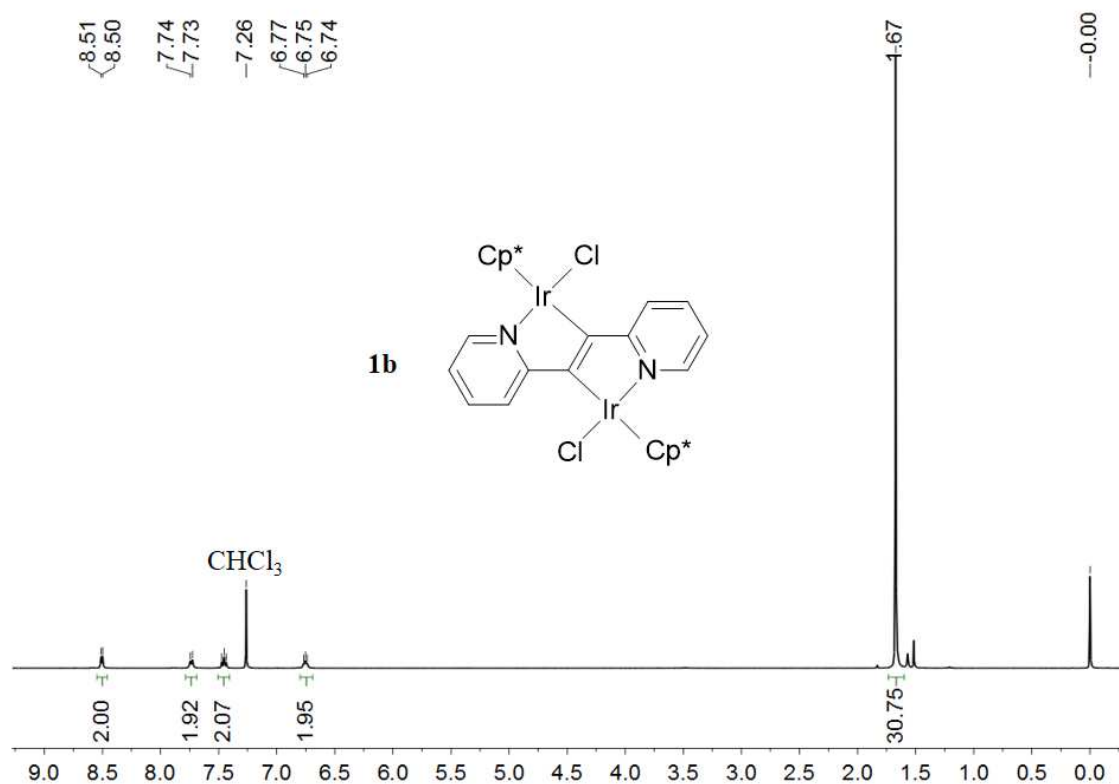
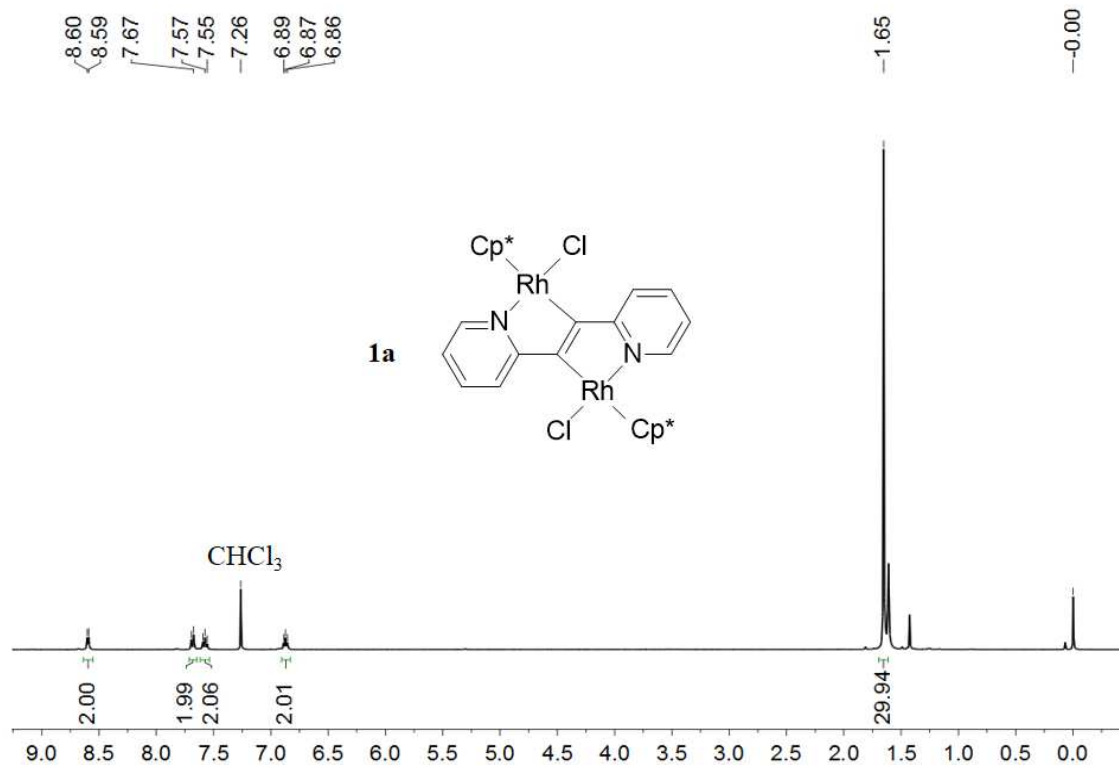
Supporting Information

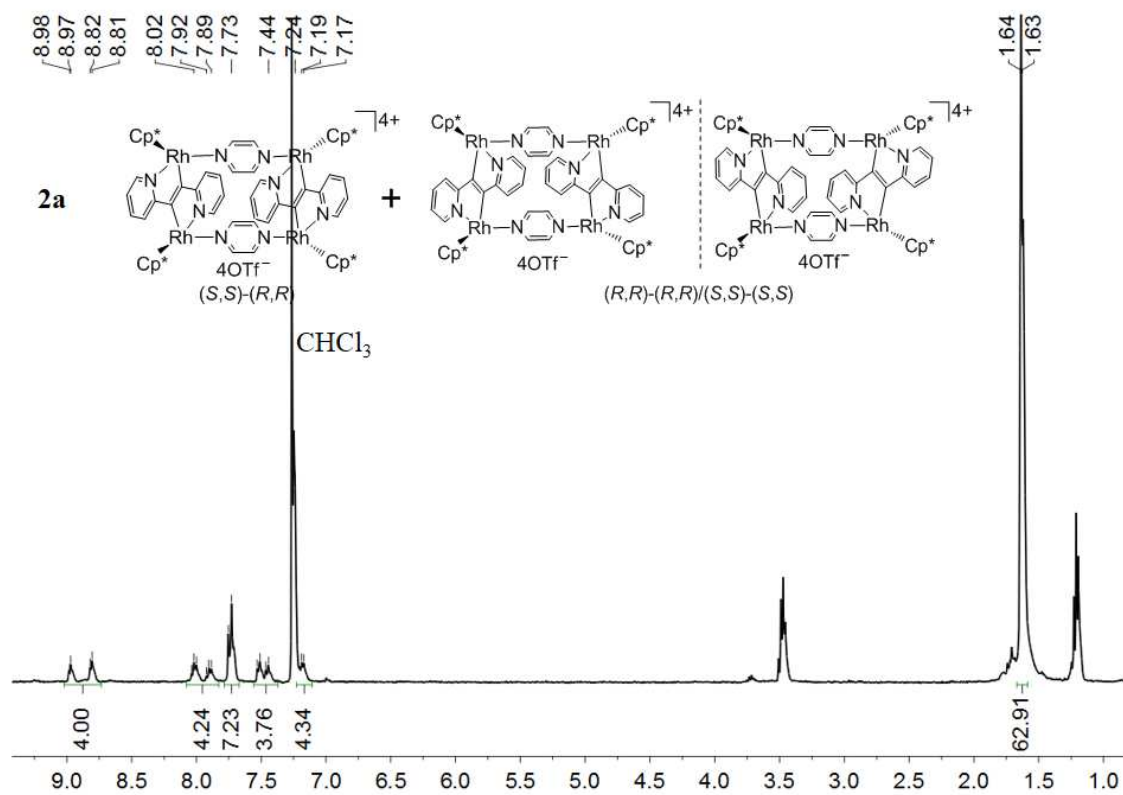
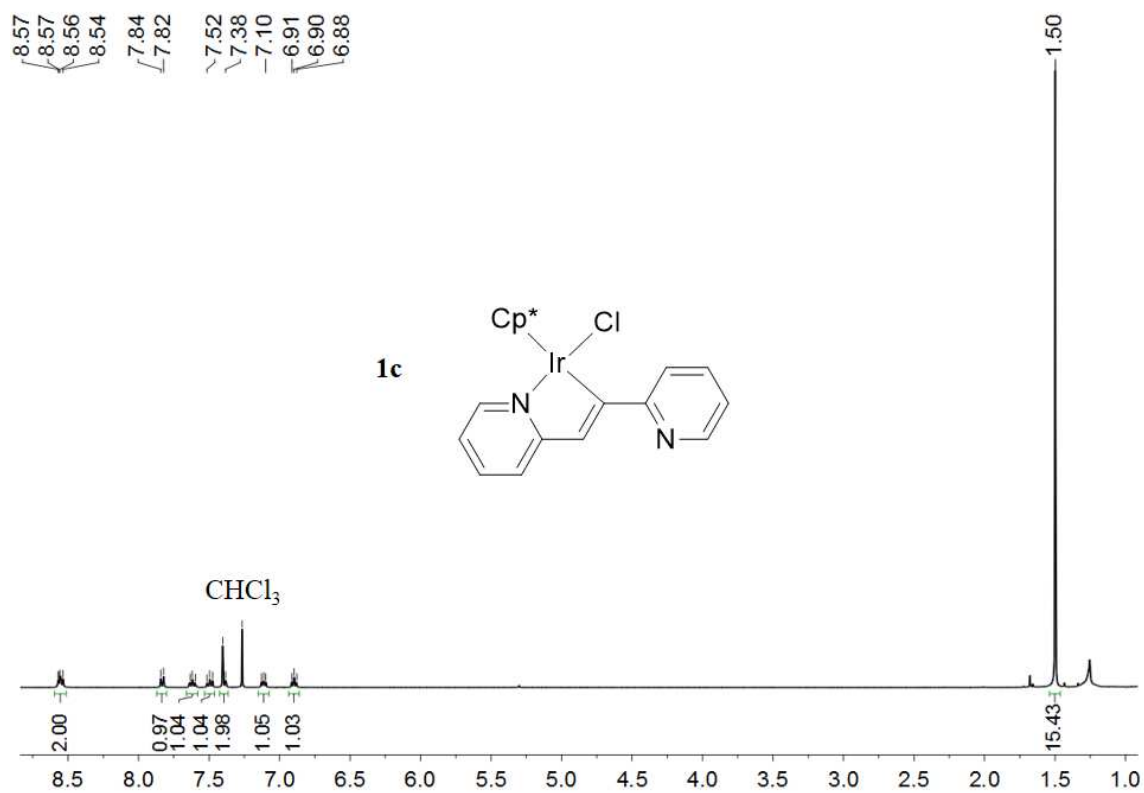
Isomers of Cyclometalated Macrocycles Constructed through Olefinic C–H Activation

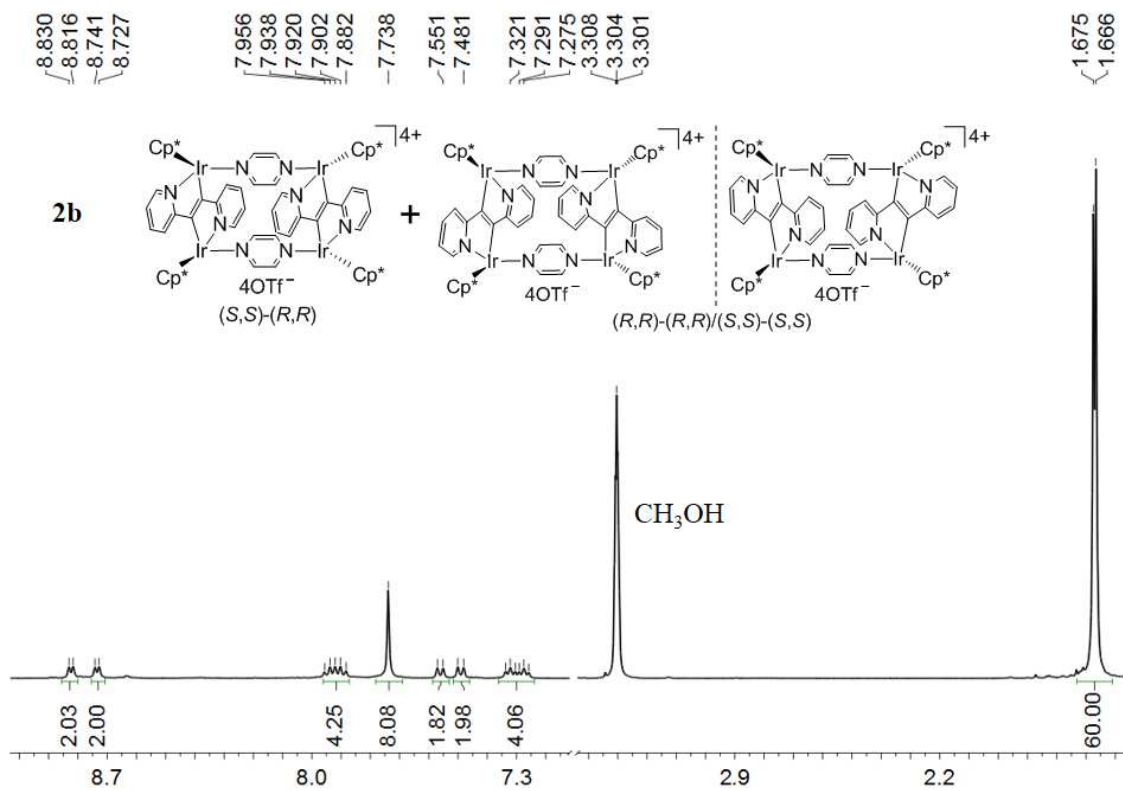
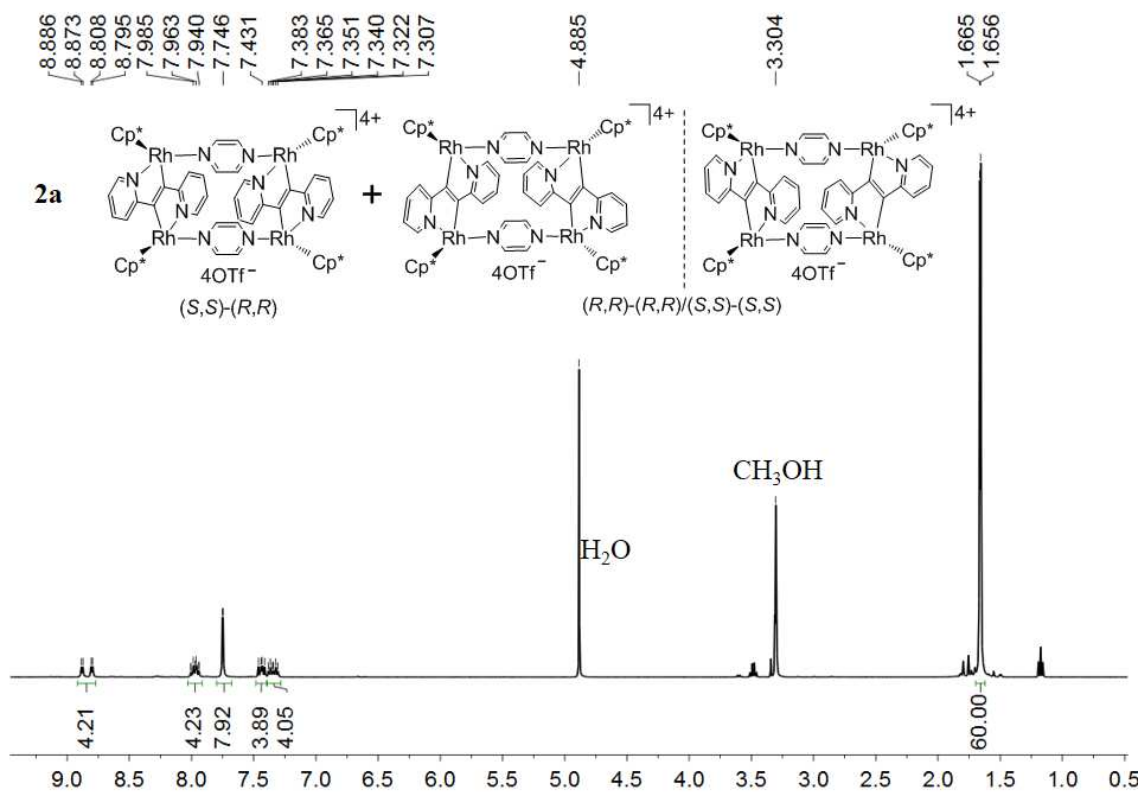
Long Zhang, Hao Li, Lin-Hong Weng, and Guo-Xin Jin*

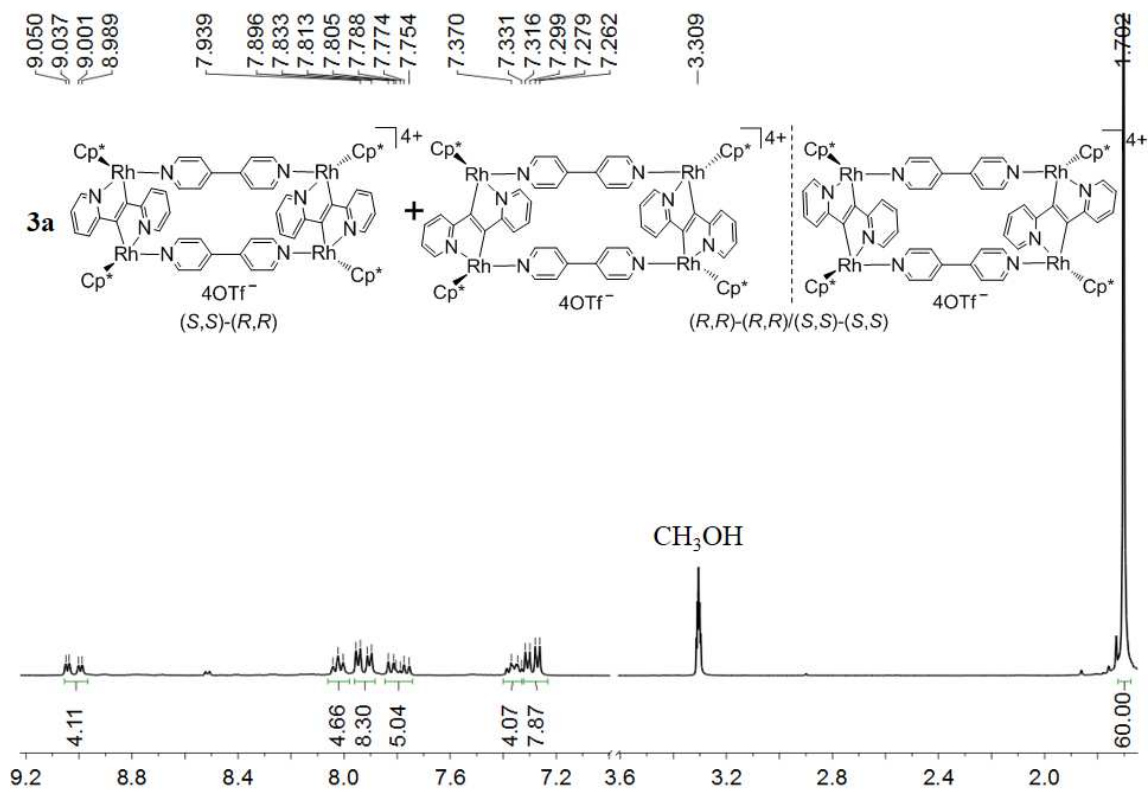
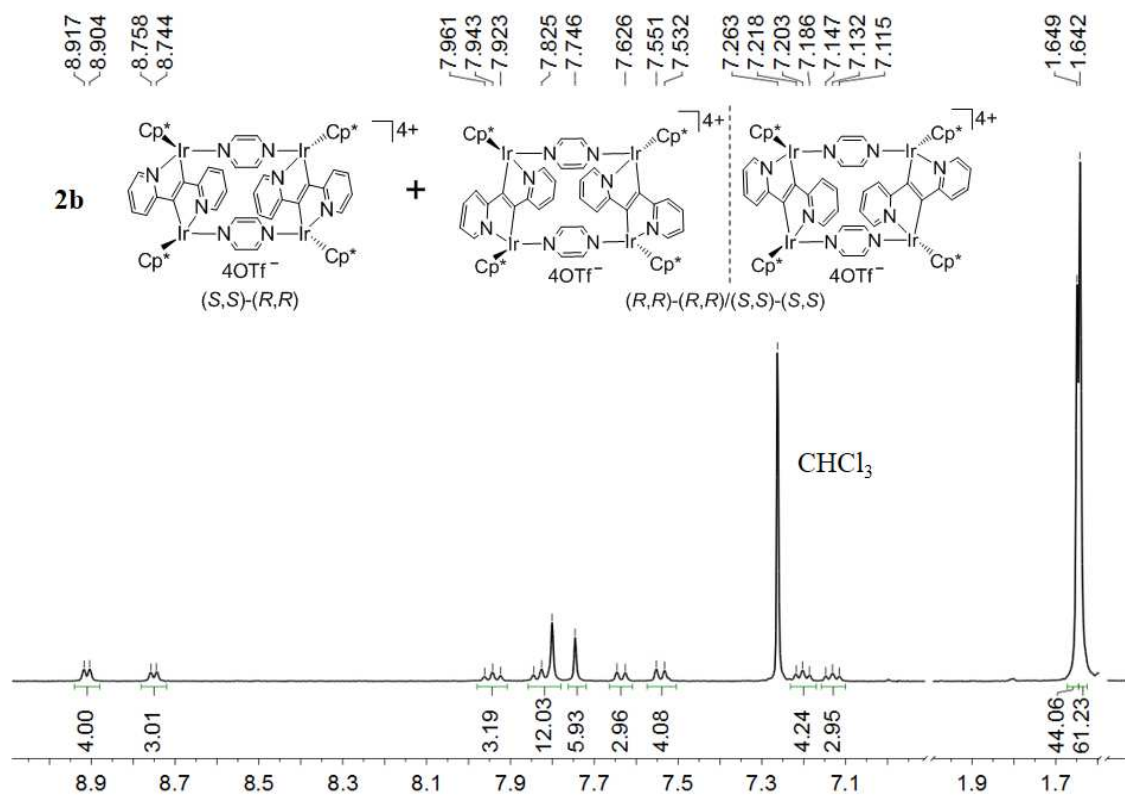
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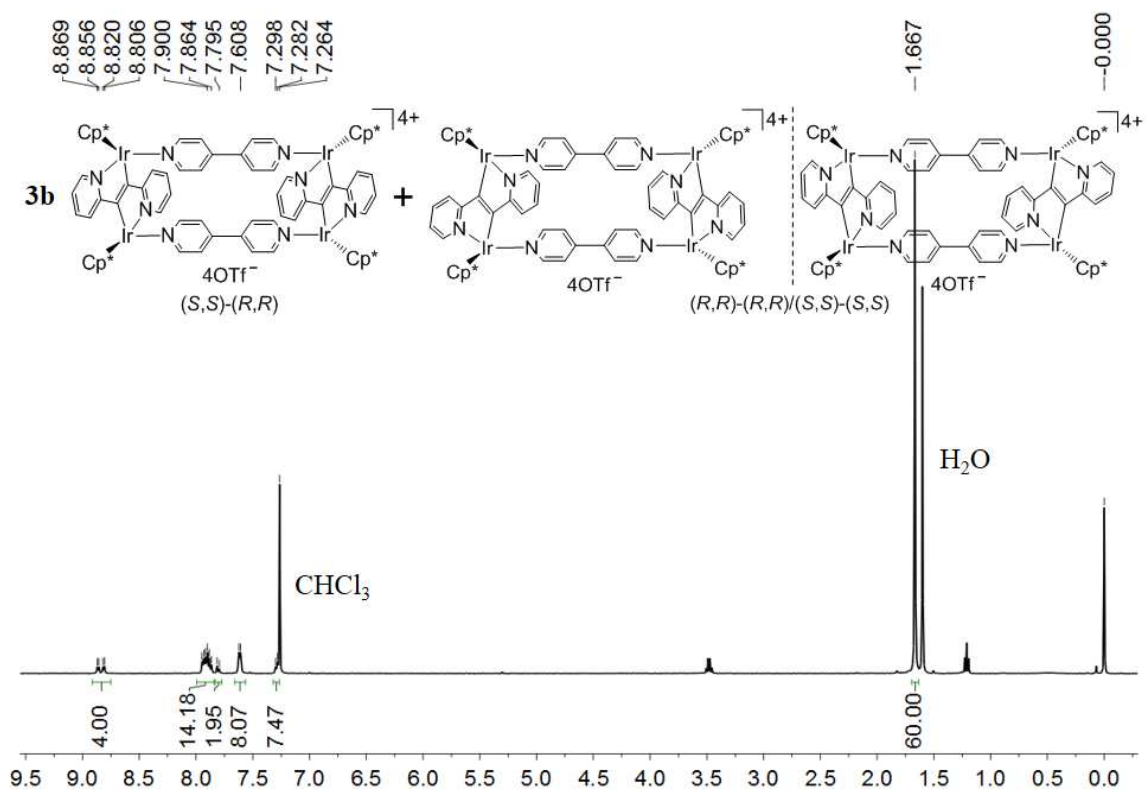
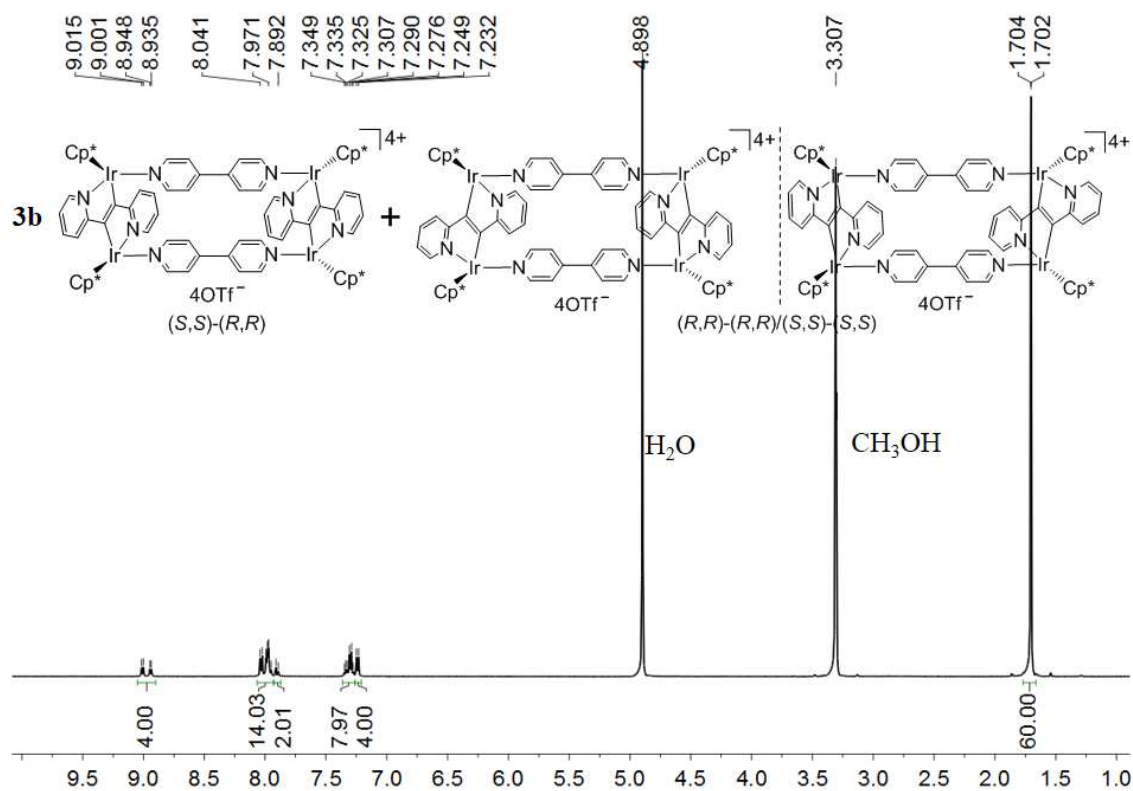
Part I. ^1H NMR Spectra	S2-S9
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Part III. Crystallographic data.....	S13

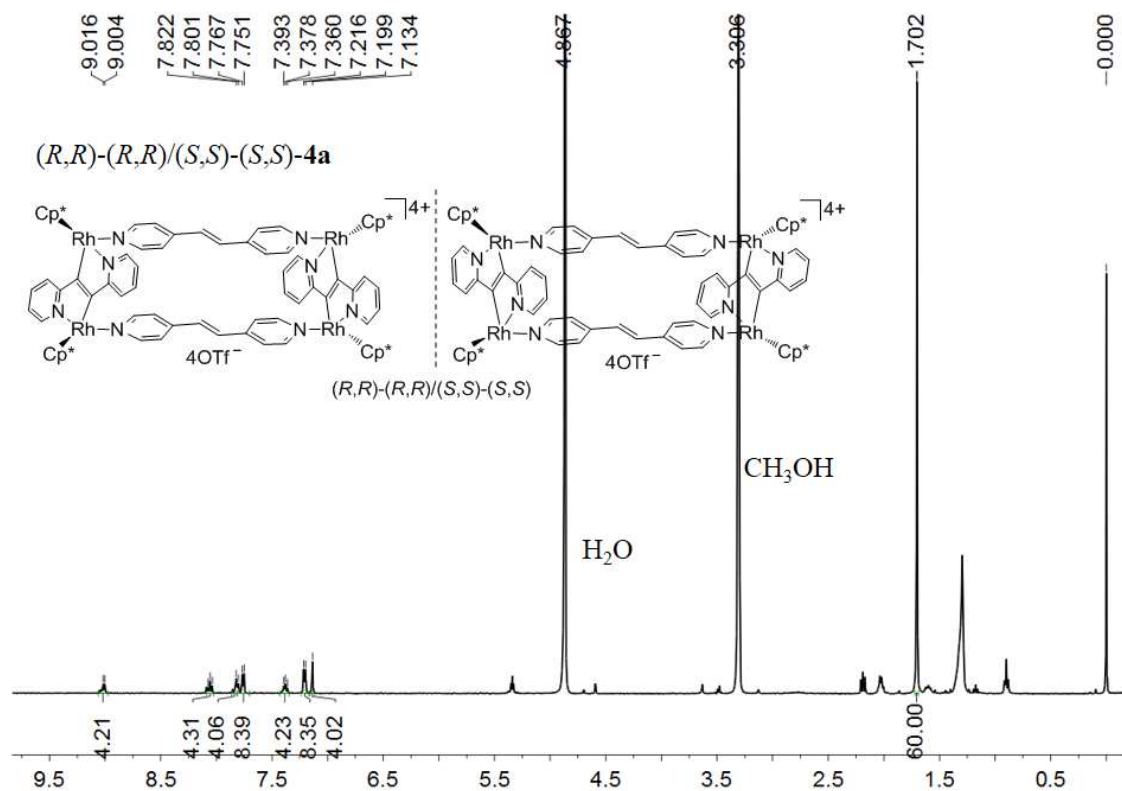
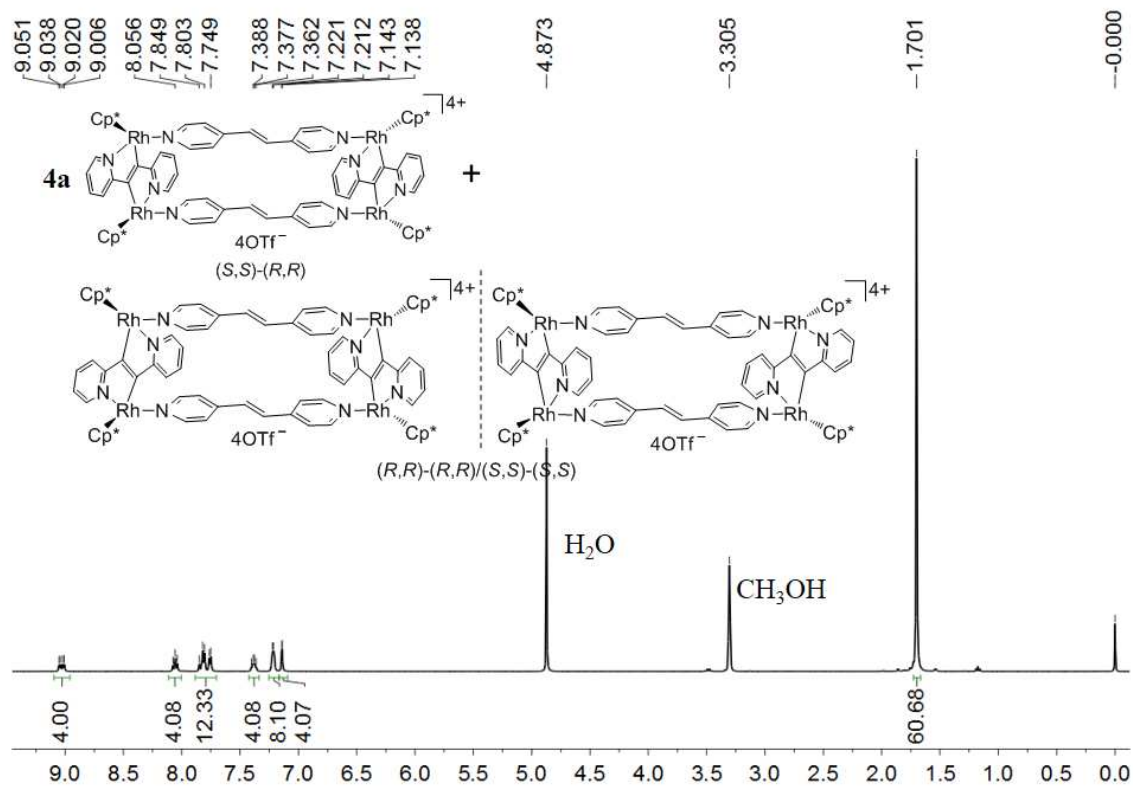
Part I. ^1H NMR Spectra

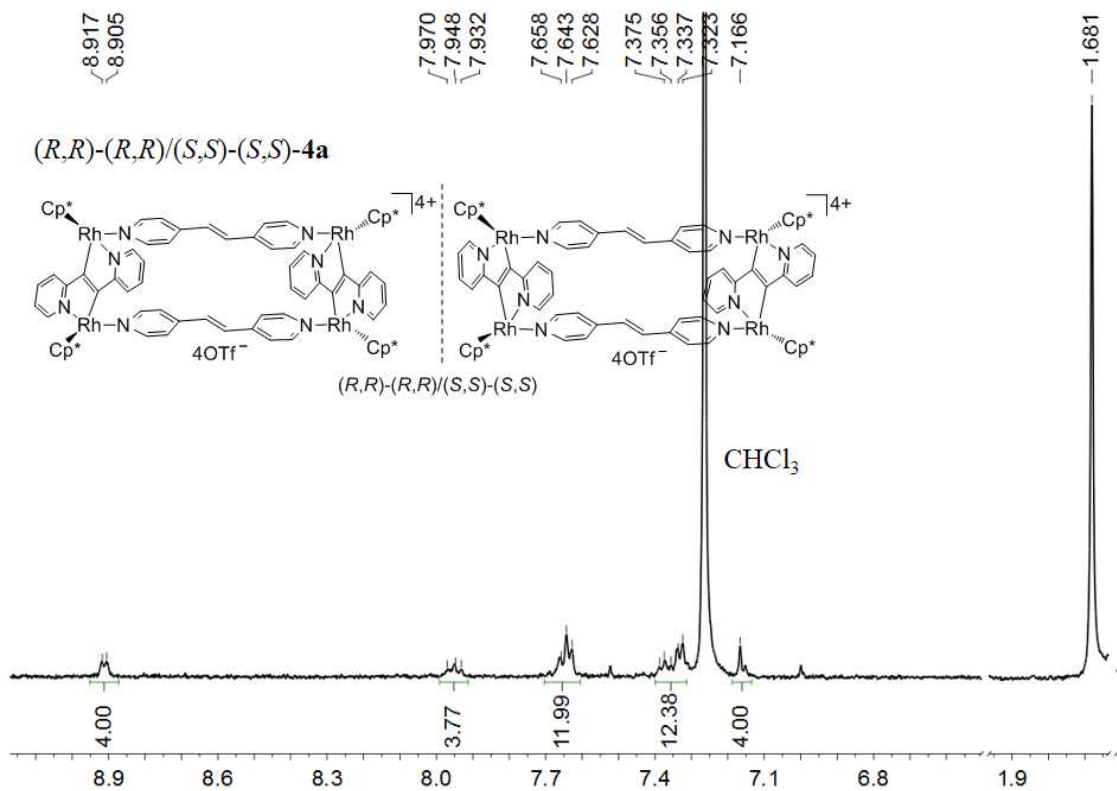
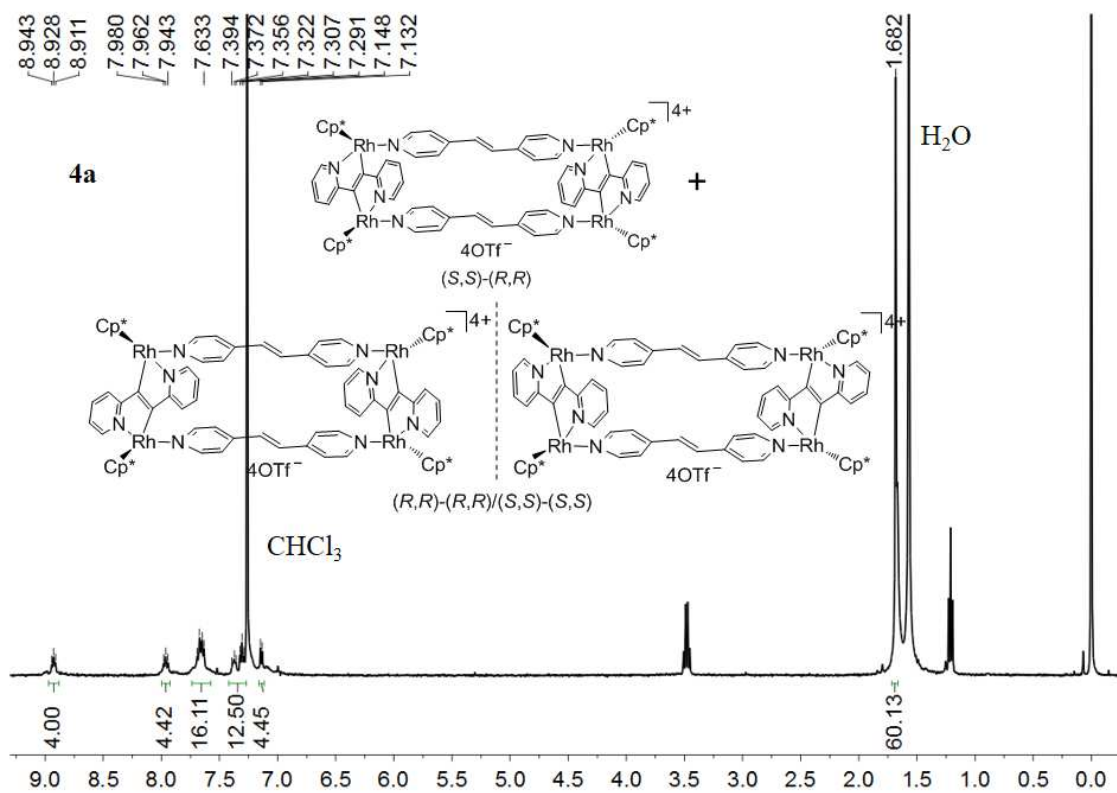


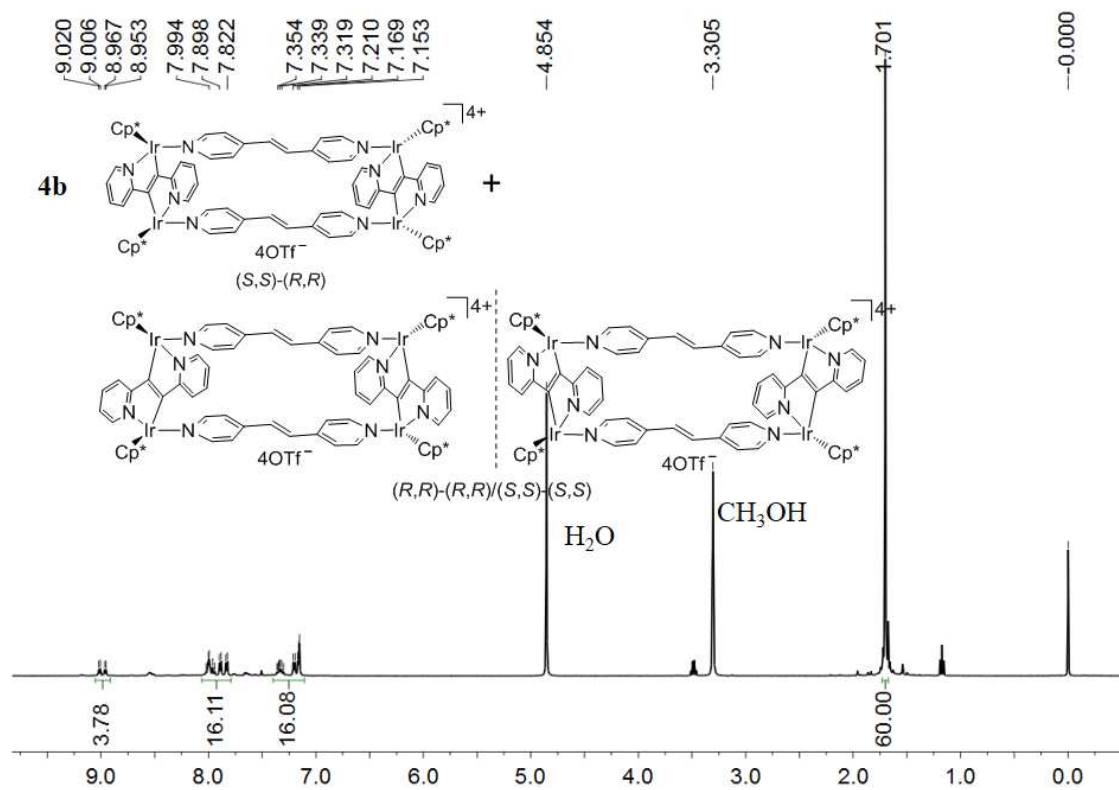




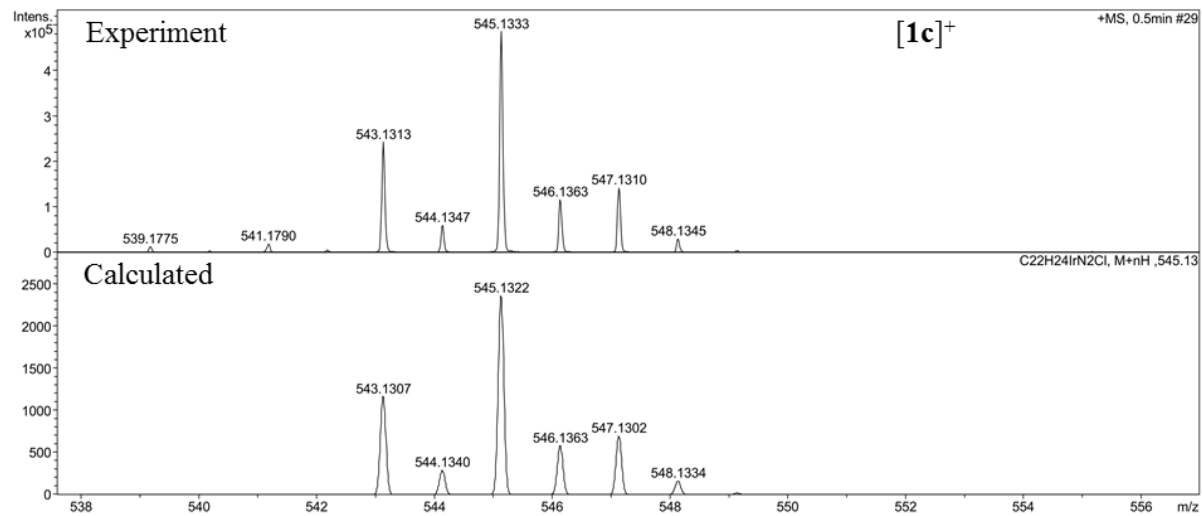
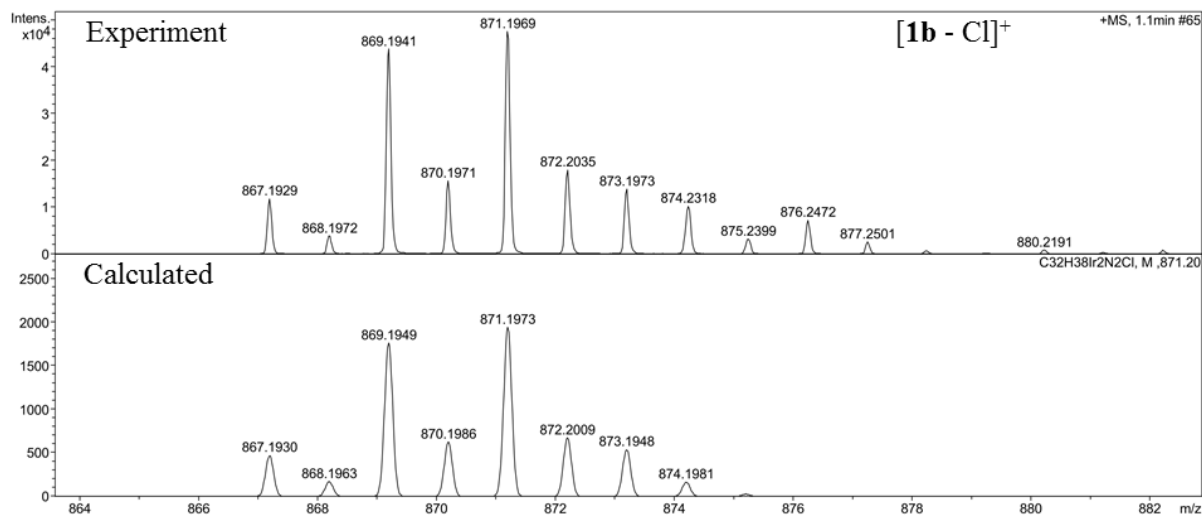
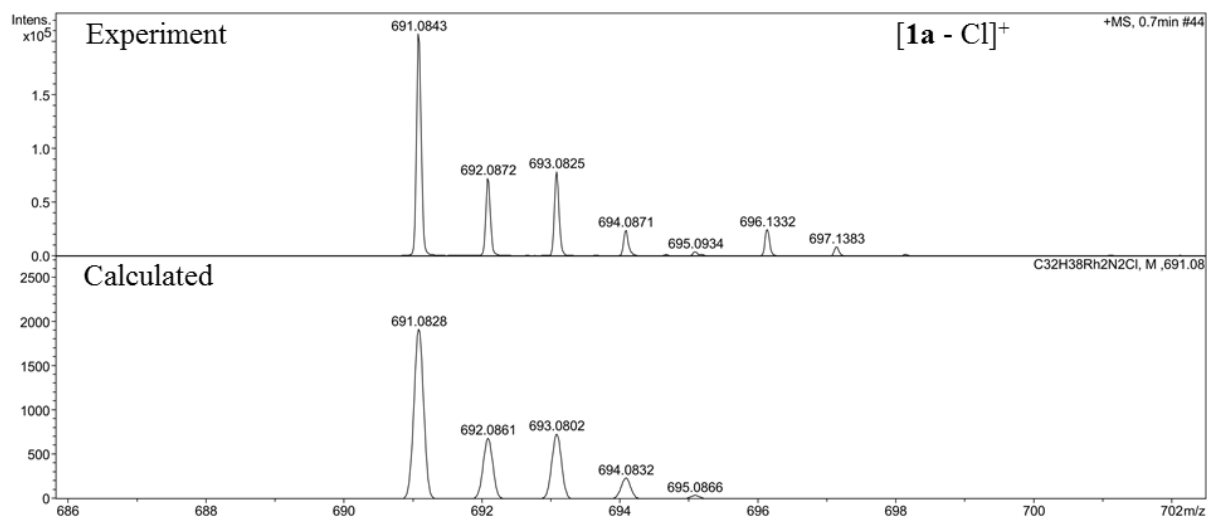


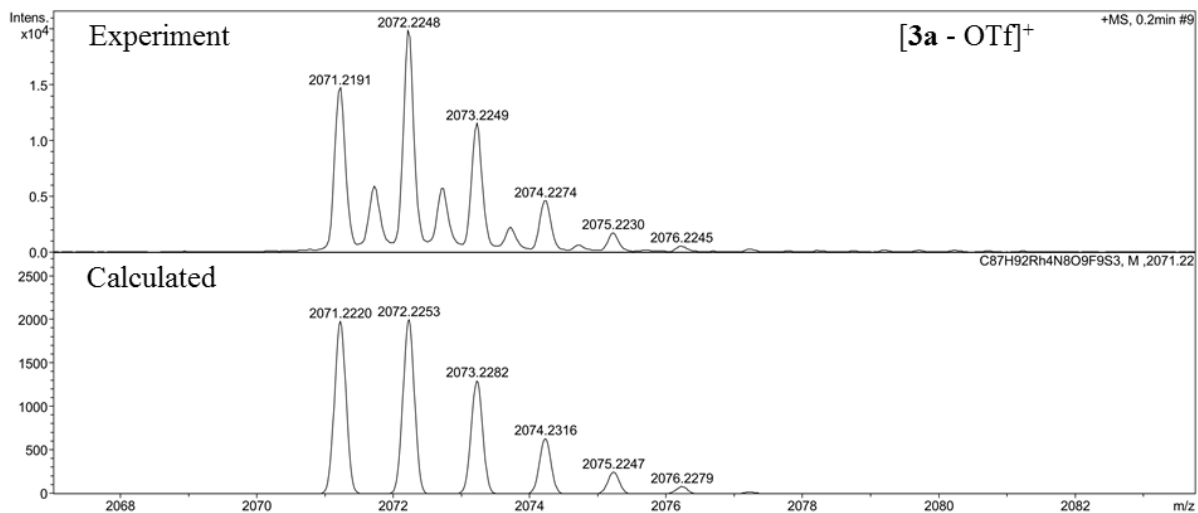
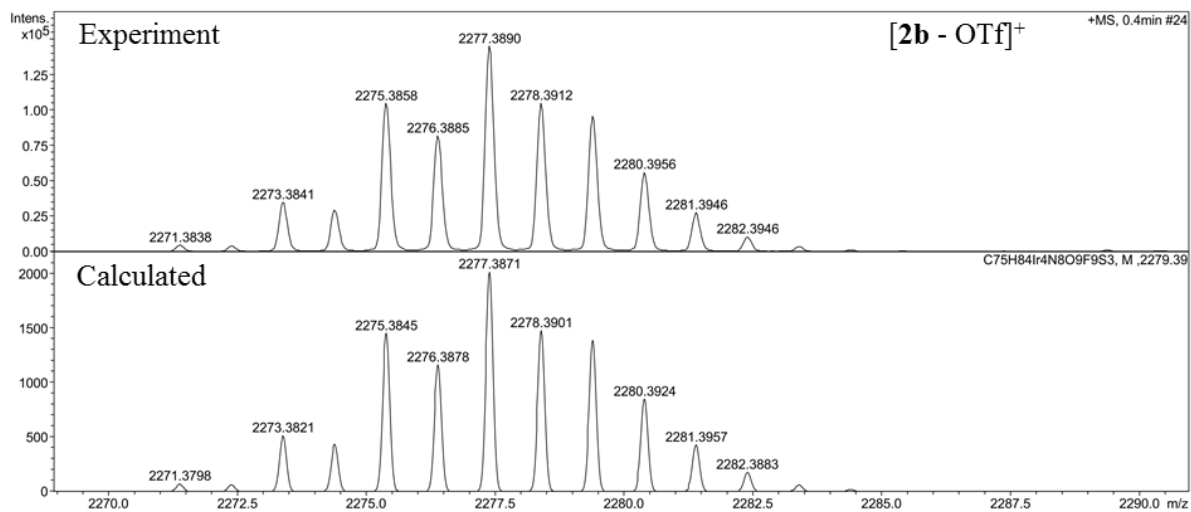
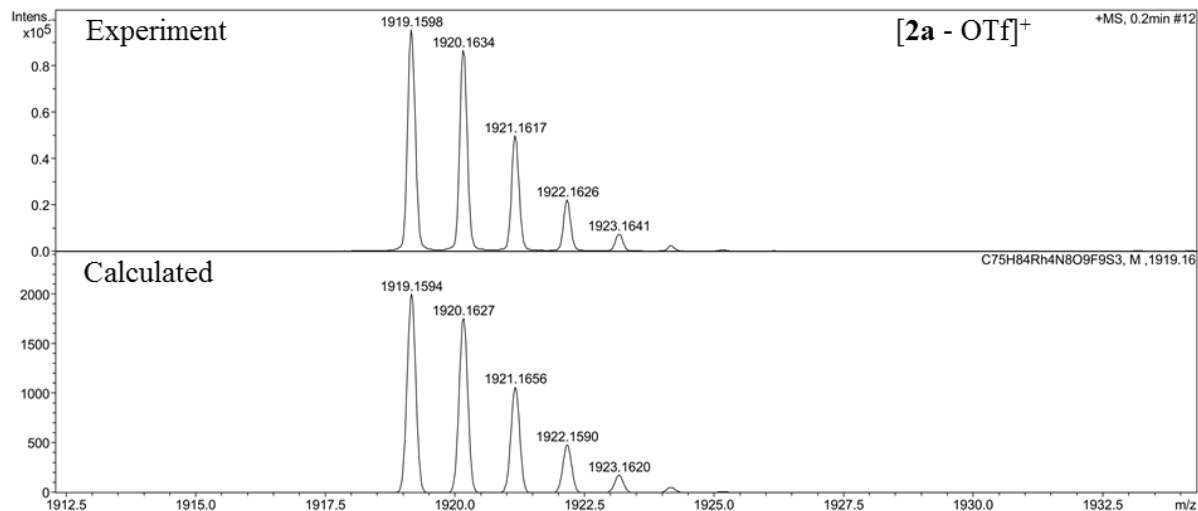


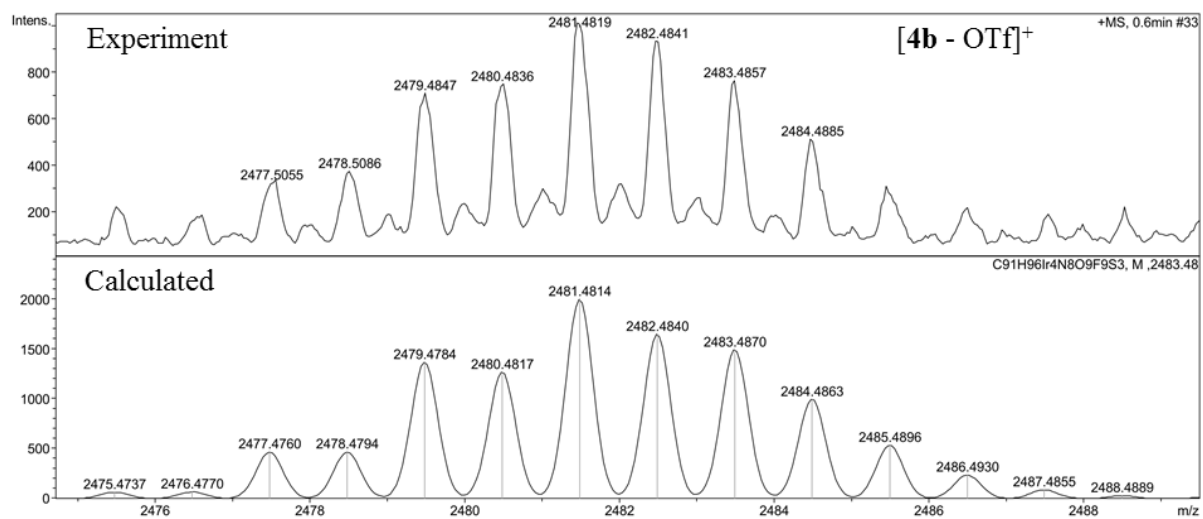
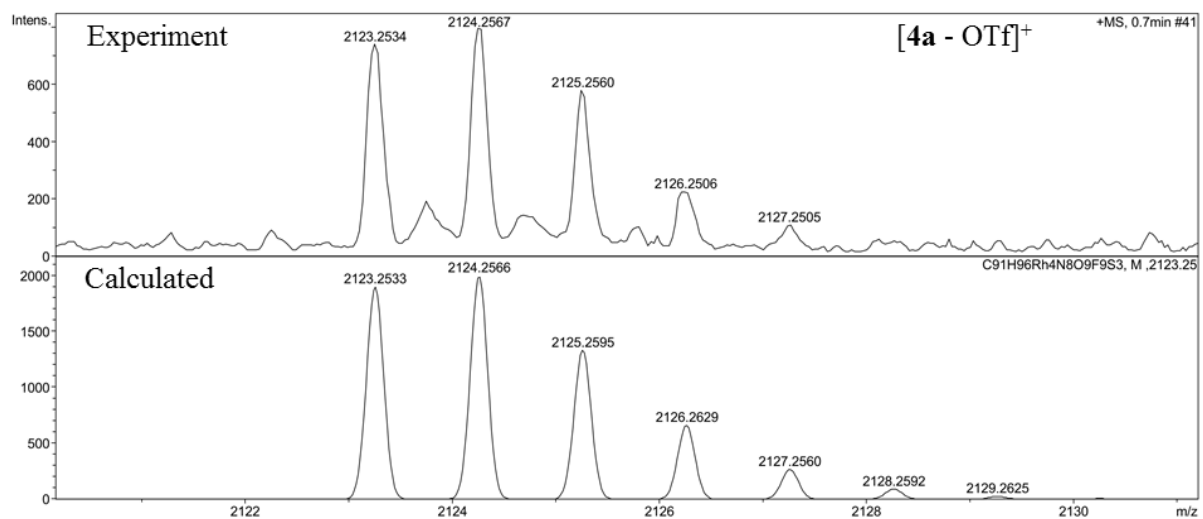
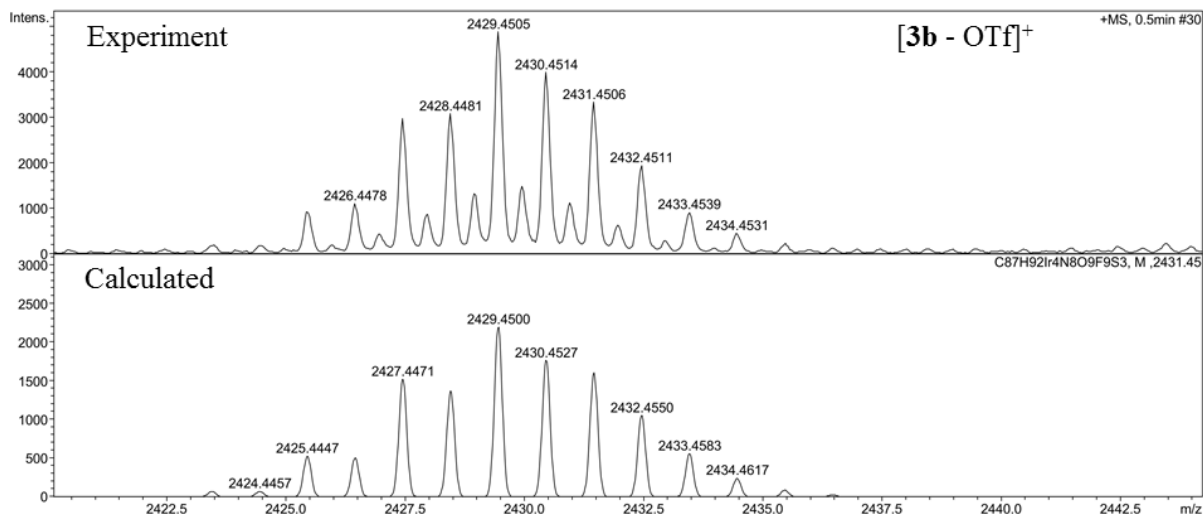




Part II. ESI-MS Spectra







Part III. Crystallographic data

Complex	1a	1b ·H ₂ O	1c	(<i>R,R</i>)-(<i>S,S</i>)-3b ·4CH ₃ OH·2H ₂ O	(<i>R,R</i>)-(<i>S,S</i>)-4a ·4CH ₃ CH ₂ OCH ₂ CH ₃ ·2H ₂ O	(<i>R,R</i>)-(<i>R,R</i>)/(<i>S,S</i>)-(<i>S,S</i>)-4a ·3H ₂ O	(<i>R,R</i>)-(<i>S,S</i>)-4b ·CH ₃ OH·3H ₂ O
Empirical formula	C ₃₂ H ₃₈ Rh ₂ N ₂ Cl ₂	C ₃₂ H ₄₀ Cl ₂ Ir ₂ N ₂ O	C ₂₂ H ₂₄ ClIrN ₂	C ₉₂ H ₁₁₂ F ₁₂ Ir ₄ N ₈ O ₁₈ S ₄	C ₁₀₈ H ₁₄₀ F ₁₂ N ₈ O ₁₈ Rh ₄ S ₄	C ₉₂ H ₁₀₂ F ₁₂ N ₈ O ₁₅ Rh ₄ S ₄	C ₉₃ H ₁₀₆ F ₁₂ Ir ₄ N ₈ O ₁₆ S ₄
M _w	727.36	923.96	544.08	2742.94	2606.15	2327.69	2716.89
Crystal system	Triclinic	Monoclinic	Monoclinic	Triclinic	Monoclinic	Monoclinic	Triclinic
Space group	P-1	C2/c	P 21/c	P-1	P2(1)/c	P2(1)/c	P-1
<i>a</i> [Å]	8.5691(7)	21.325(4)	7.547(3)	11.6837(13)	23.317(3)	15.987(6)	15.0697(14)
<i>b</i> [Å]	11.5133(10)	9.702(2)	14.682(6)	12.1287(13)	14.8917(16)	34.473(13)	16.4871(15)
<i>c</i> [Å]	16.3558(14)	15.081(3)	17.714(7)	18.142(2)	16.6240(18)	19.523(8)	21.966(2)
<i>α</i> [°]	100.5780(10)	90	90	77.590(2)	90	90	97.147(2)
<i>β</i> [°]	95.4880(10)	97.507(3)	98.653(5)	80.454(2)	98.888(2)	101.423(6)	90.421(2)
<i>γ</i> [°]	106.7520(10)	90	90	89.950(2)	90	90	90
<i>V</i> [Å ³]	1499.9(2)	3093.6(11)	1940.5(13)	2474.4(5)	5703.0(11)	10547(7)	5145.0(8)
<i>Z</i>	2	4	4	1	2	4	2
<i>ρ</i> [g cm ⁻³]	1.611	1.984	1.862	1.841	1.518	1.466	1.754
absorption coefficient [mm ⁻¹]	1.302	8.795	7.026	5.537	0.729	0.776	5.324
<i>F</i> [000]	736	1768	1056	1344	2680	4728	2656
<i>T</i> [K]	173(2)	173(2)	293(2)	173(2)	173(2)	293(2)	203(2)
θ range [°]	1.28-28.01	1.93-27.99	1.810-26.007	1.17-27.68	0.88-27.00	1.43-25.10	0.98-25.10
reflections/unique [R(int)]	11502 / 7123	11248 / 3724	8588 / 3804	18234 / 11348	35418 / 12421	43562/18684	31663/18113
completeness to θ [°]	28.01(98.2 %)	25.24(99.8 %)	25.24(99.6 %)	25.24(98.6 %)	25.24(99.6 %)	25.24(97.9 %)	25.24(97.4 %)
data/restraints/parameters	7123/0/353	3724/1/186	3804/0/240	11348/39/645	12421/0/605	18684/45/1179	18113/192/1205
GOF on <i>F</i> ²	1.162	1.289	0.794	1.015	1.091	0.817	0.998
final <i>R</i> indices [I>2σ(I)] <i>R</i> ₁ , <i>wR</i> ₂	0.0315	0.0395	0.0305	0.0527	0.0531	0.0627	0.0658
<i>R</i> indices (all data) <i>R</i> ₁ , <i>wR</i> ₂	0.1491	0.0893	0.0430	0.1843	0.1707	0.1539	0.2177