Iridium Polypyridyl Complexes

Team 1: Lisa Akai, Lara Cokin, JJ Donato, Adrieon Key

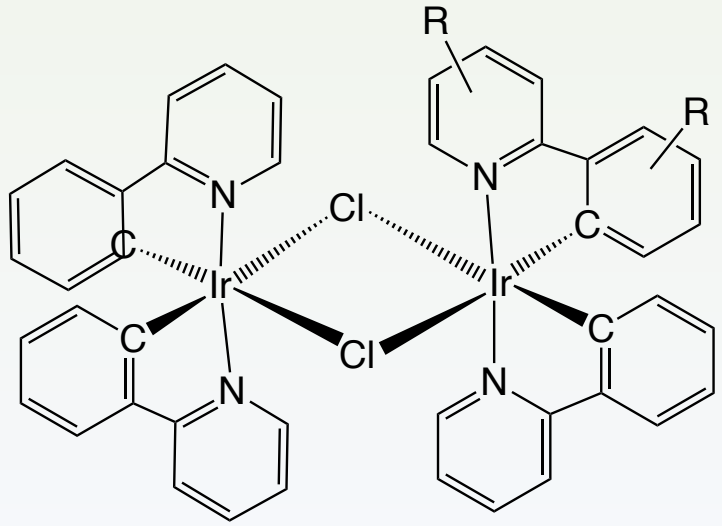
Garces Group: Tam AboNabout, Lisa Akai, Lara Cokin, JJ Donato, Adrieon Key, Brian Lam

Summer Research

Area of Research: Team 1

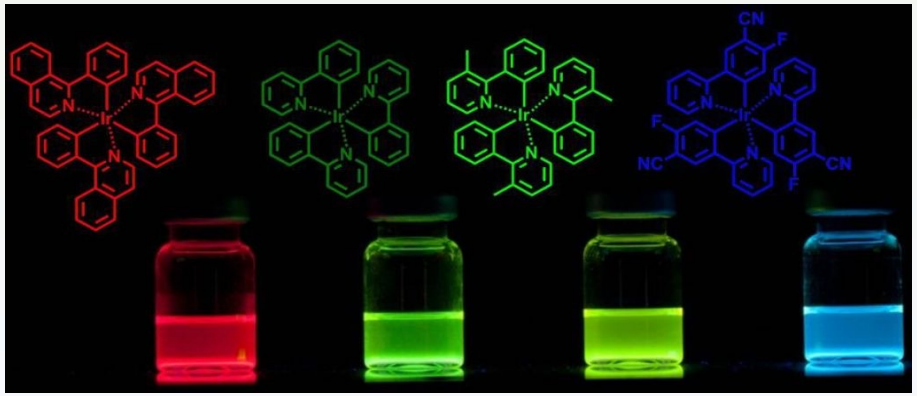
In a dynamic summer research initiative led by community college students, a series of Iridium orthometallation complexes with phenylpyridine derivatives were successfully synthesized and comprehensively characterized. Employing advanced techniques such as NMR spectroscopy and UV-VIS absorption, the structures and physical properties of these complexes were meticulously examined. Remarkably, proton and carbon resonance assignments were fully accomplished. Furthermore, crystallization yielded single crystals amenable to X-ray crystallography, providing valuable insights into their structural details.

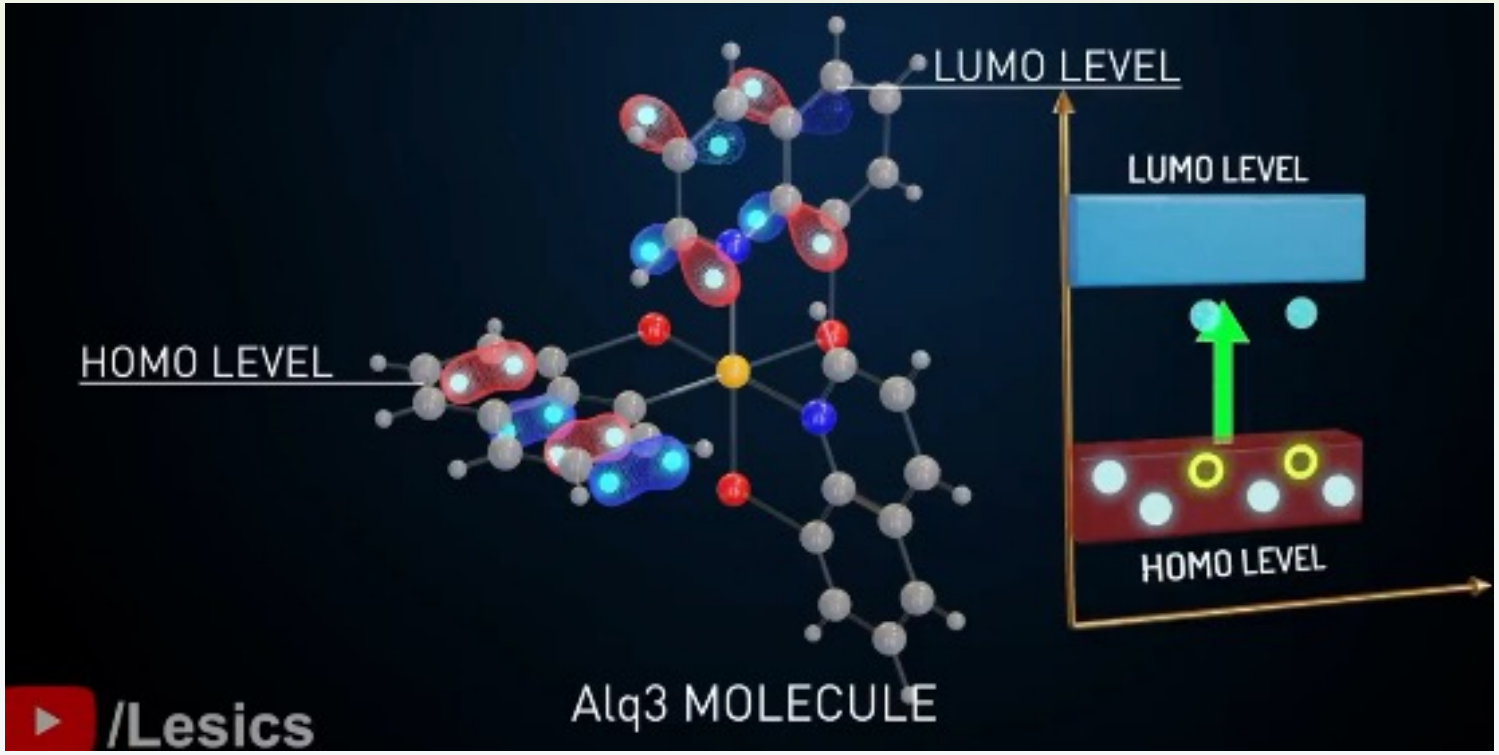
This collaborative effort by community college students underscores the potential for impactful research endeavors even within a short timeframe, shedding light on the exciting field of organometallic chemistry.



Applications: OLED

OLED –Organic Light Emitting Diodes is a display technology that uses organic compounds, i.e., $Ir(mppy)_3$ to emit light when an electric current passes through them. It offers vibrant colors, high contrast, and flexible form factors, but may be prone to burn-in and higher production costs compared to LCDs. Scan the QR code to see the movie about OLED and your iphone.

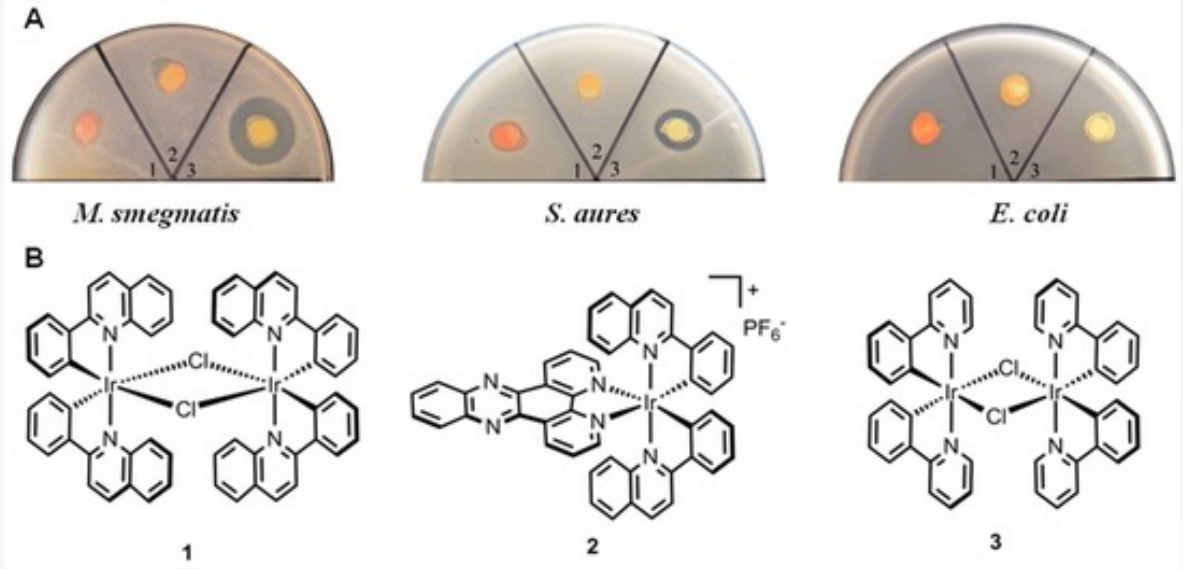




Anti Cancer, Bacterial Agent

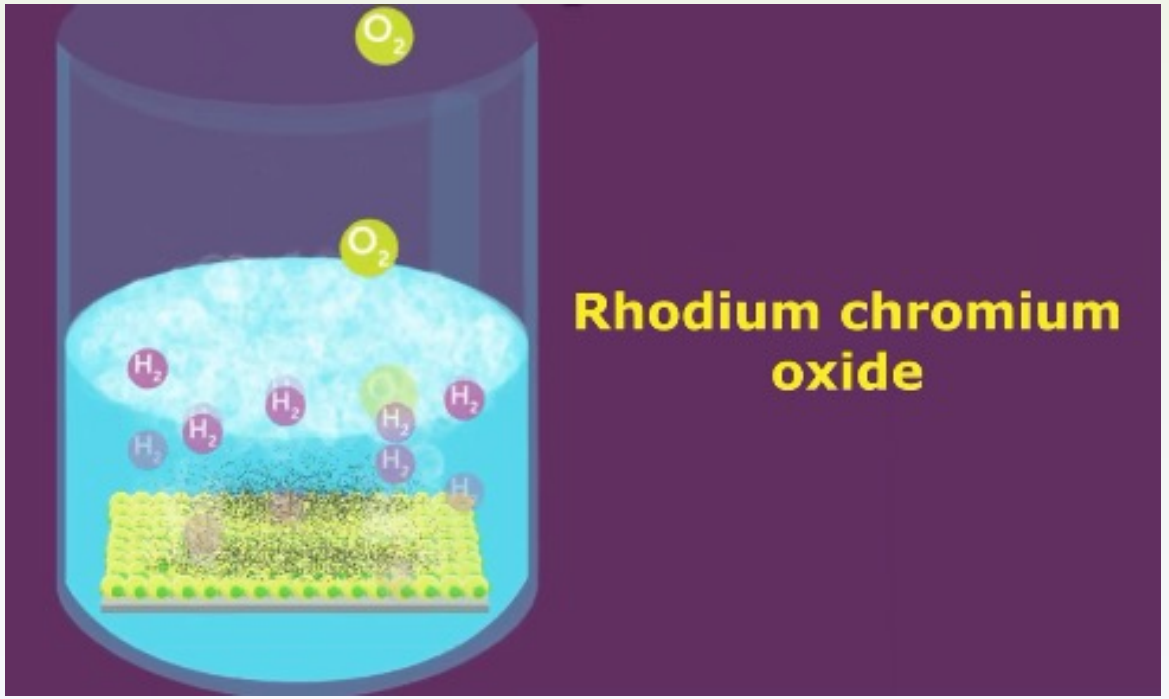
Anti Cancer (Bacterial) Agents –Iridium-based compounds have shown promise as anticancer agents, exhibiting potential for targeted cancer therapy. Additionally, iridium complexes have demonstrated antibacterial activity against drug-resistant bacteria, suggesting their potential application in combating bacterial infections. Further research is needed to explore their effectiveness and mechanisms of action. Scan the QR code to read more about the anticancer properties of cyclometallated iridium complexes.

Figure 1. Anti-bacterial activity of complexes **1–3** as determined by the disk diffusion assay. (**A**) Strains including *S. aureus* ATCC 33591(MRSA), *E. coli* ATCC25922 and *M. smegmatis* mc^2 155; (**B**) Chemical structures of iridium complex **1–3**.



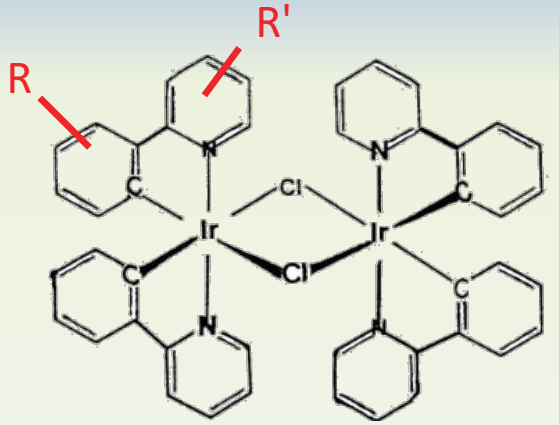
Photocatalysts

Photocatalyst - Iridium photocatalysts are being actively researched for their ability to harness light energy and facilitate various chemical reactions. These catalysts can drive important transformations such as water splitting, carbon dioxide reduction, and organic synthesis, offering potential advancements in renewable energy and sustainable chemistry. Further studies aim to optimize their efficiency and explore new applications. Scan the QR code to watch the movie on the promise of photocatalysts.

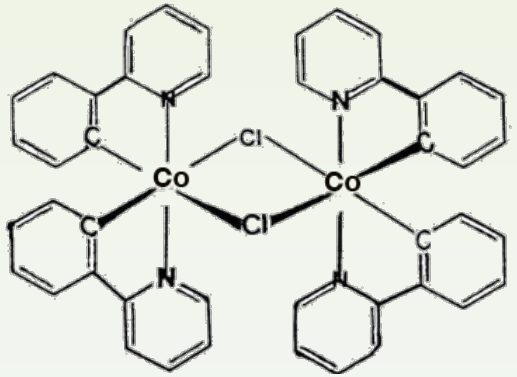


Area of Research, Summer 2023

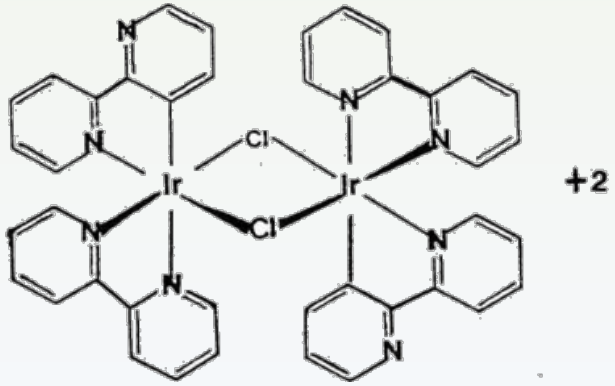
1. **Iridium 2-phenylpyridine derivatives**



~~2. Cobalt 2-phenylpyridine derivatives~~



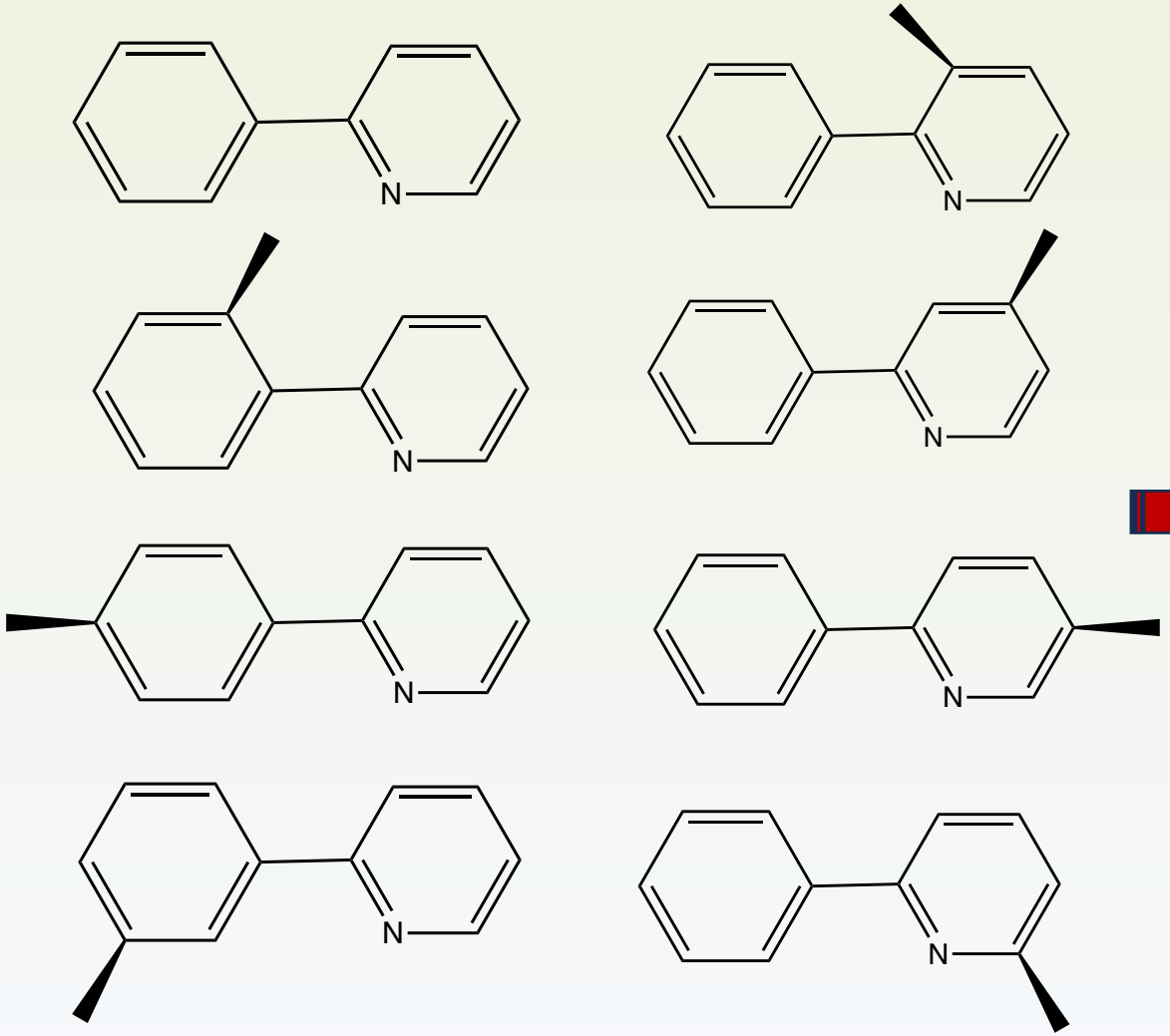
2. **Iridium C-bonded Bipyridine (Watts Dimer)**

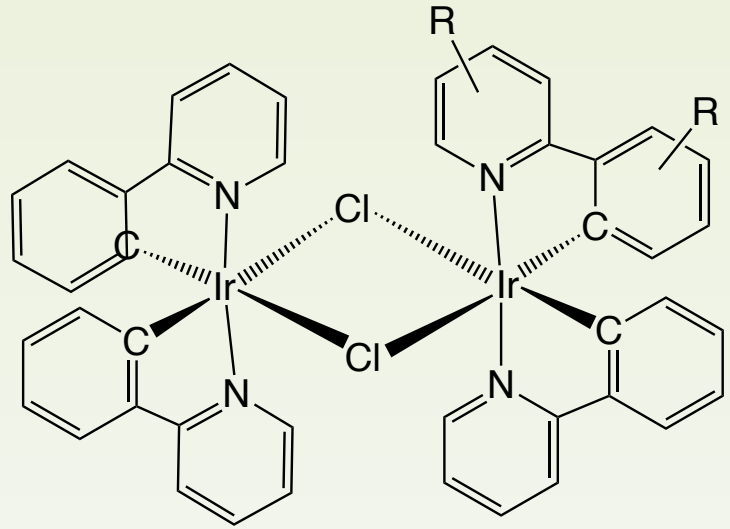


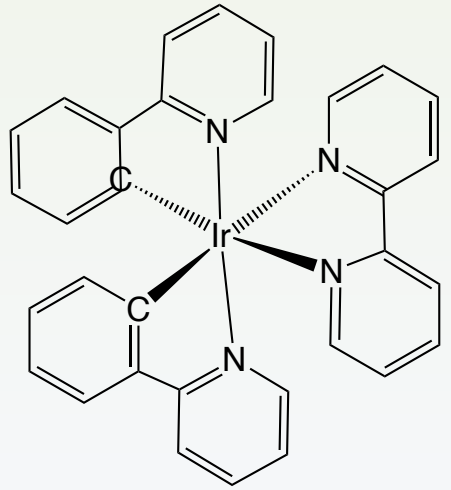
This summer: Team 1

Iridium 2-phenylpyridine derivatives

K_3IrCl_6 +





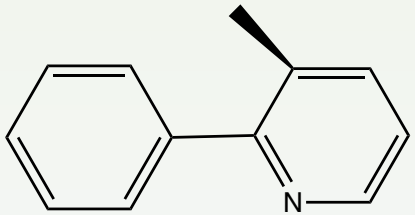


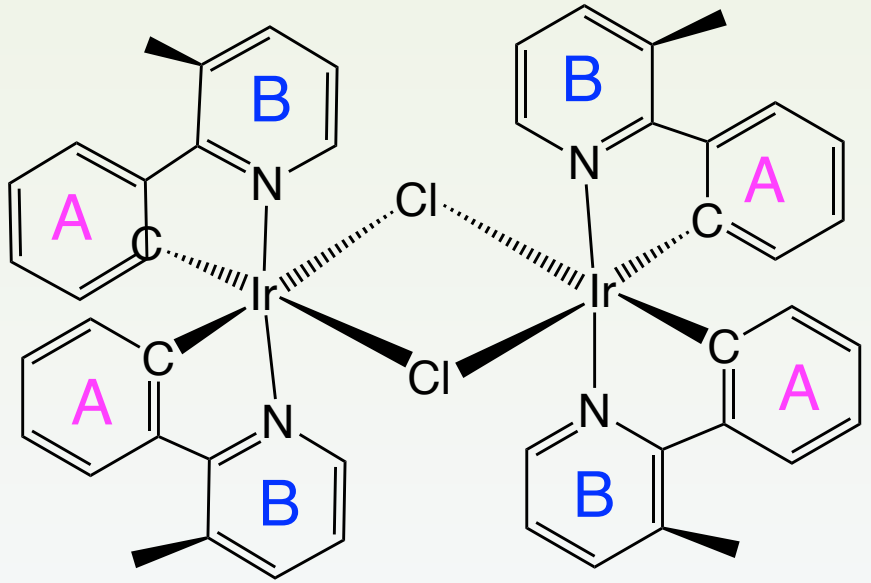
L.Cokic: 3-methyl-2-phenylpyridine (1)

Tetrakis (3-methyl-2-phenylpyridine-C^2,N')-μ-dichloroiridium

[Ir(3m2phpy)2Cl]2

K_3IrCl_6 +



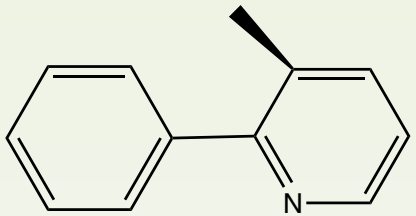


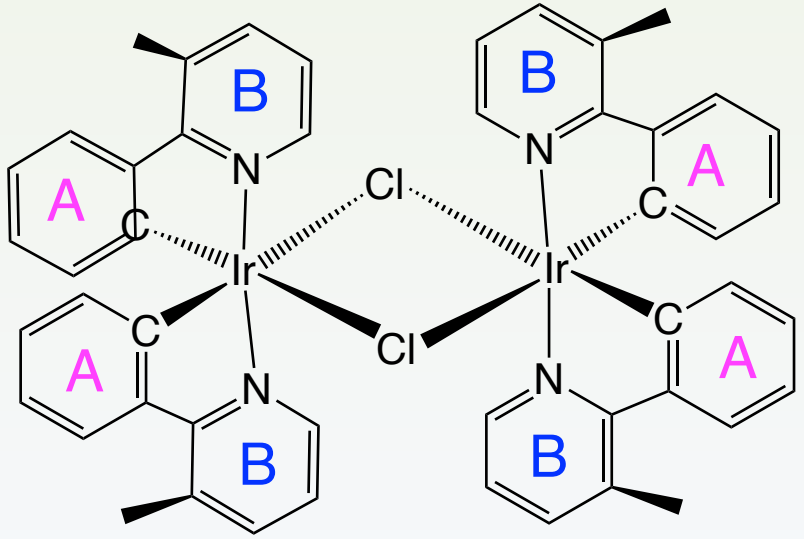
LCokin: 3-methyl-2-phenylpyridine (1)

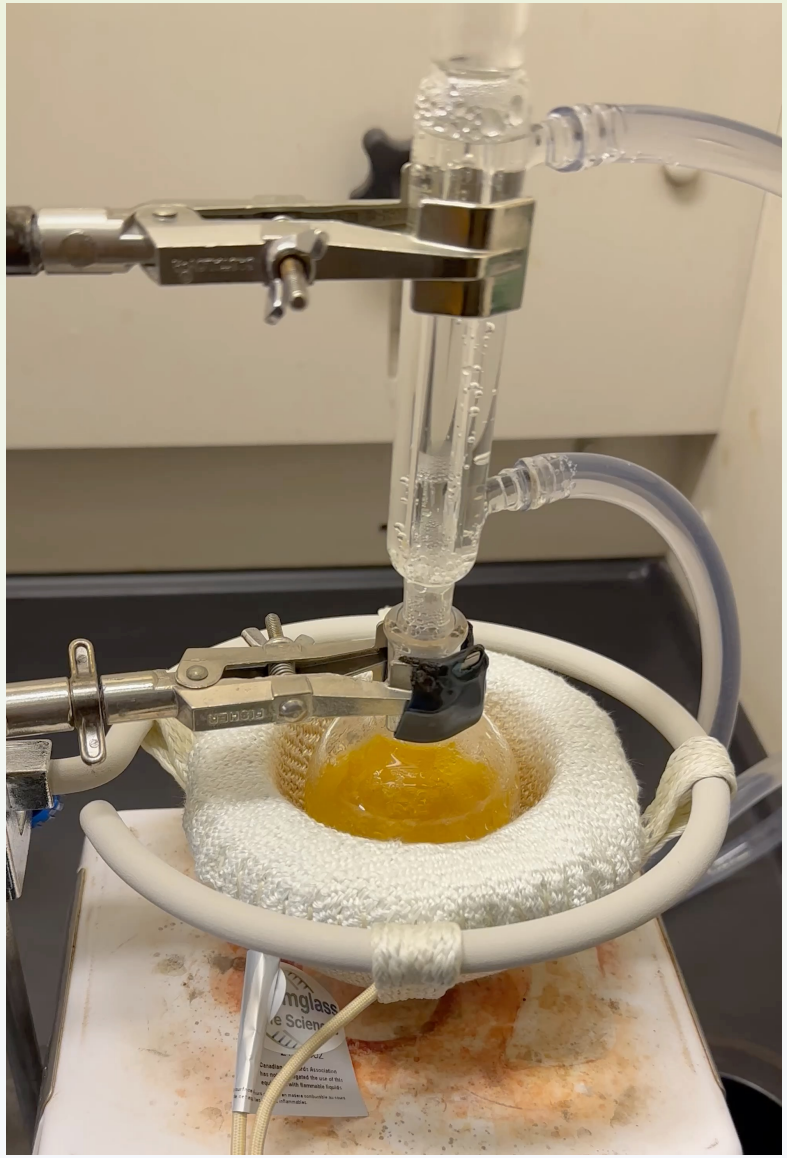
Tetrakis (3-methyl-2-phenylpyridine-C^2,N')-μ-dichloroiridium

[Ir(3mppy)2Cl]2

K_3IrCl_6 +



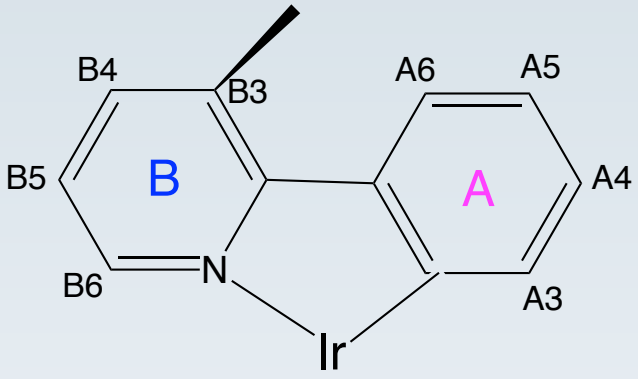


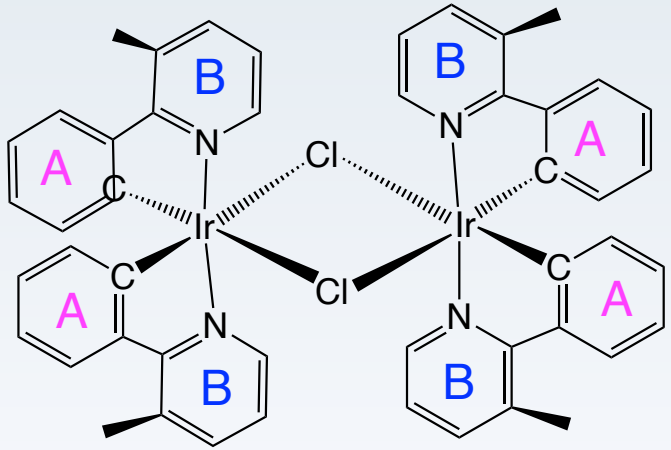


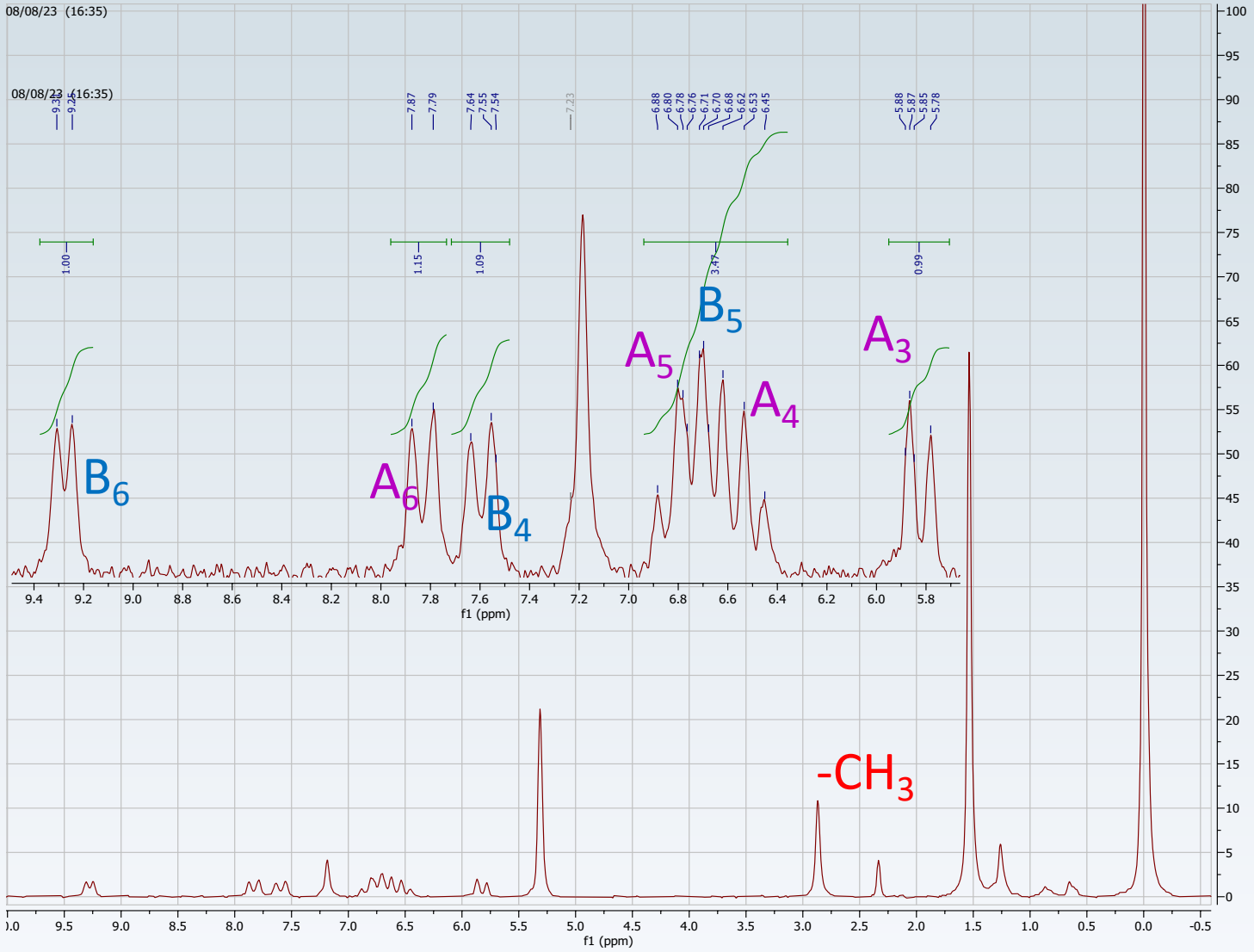
$[Ir(3\text{-methyl-2-phenylpyridine})_2Cl]_2$ (1)

^{1}N NMR Spectrum in DCM-d2

$[Ir(3m2phpy)_2Cl]_2$





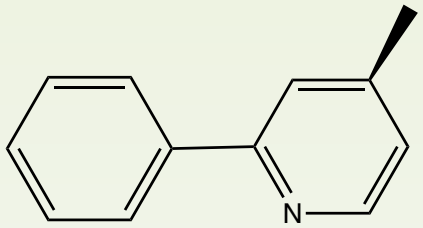


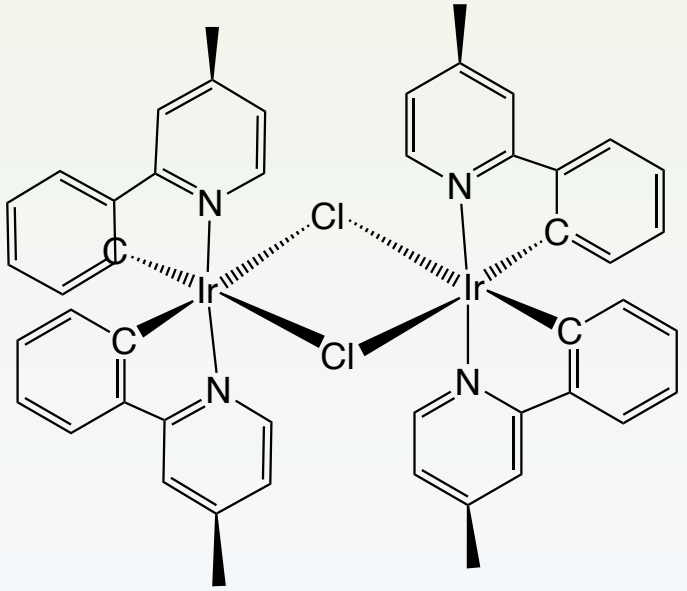
A.Key: $[Ir(4\text{-methyl-2-phenylpyridine})_2Cl]_2$ (2)

Tetrakis (4-methyl-2-phenylpyridine-C^2,N')-μ-dichloroiridium

[Ir(4mppy)2Cl]2

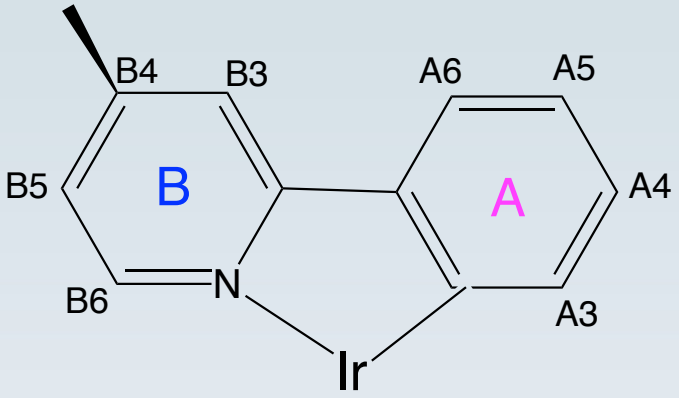
K_3IrCl_6 +

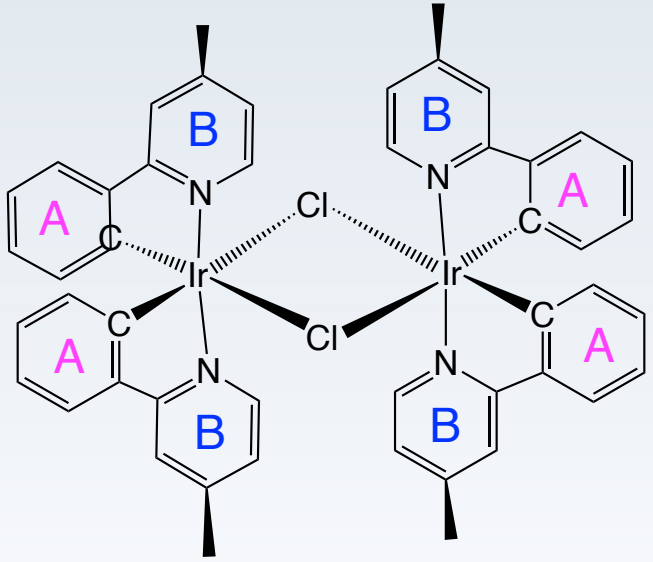


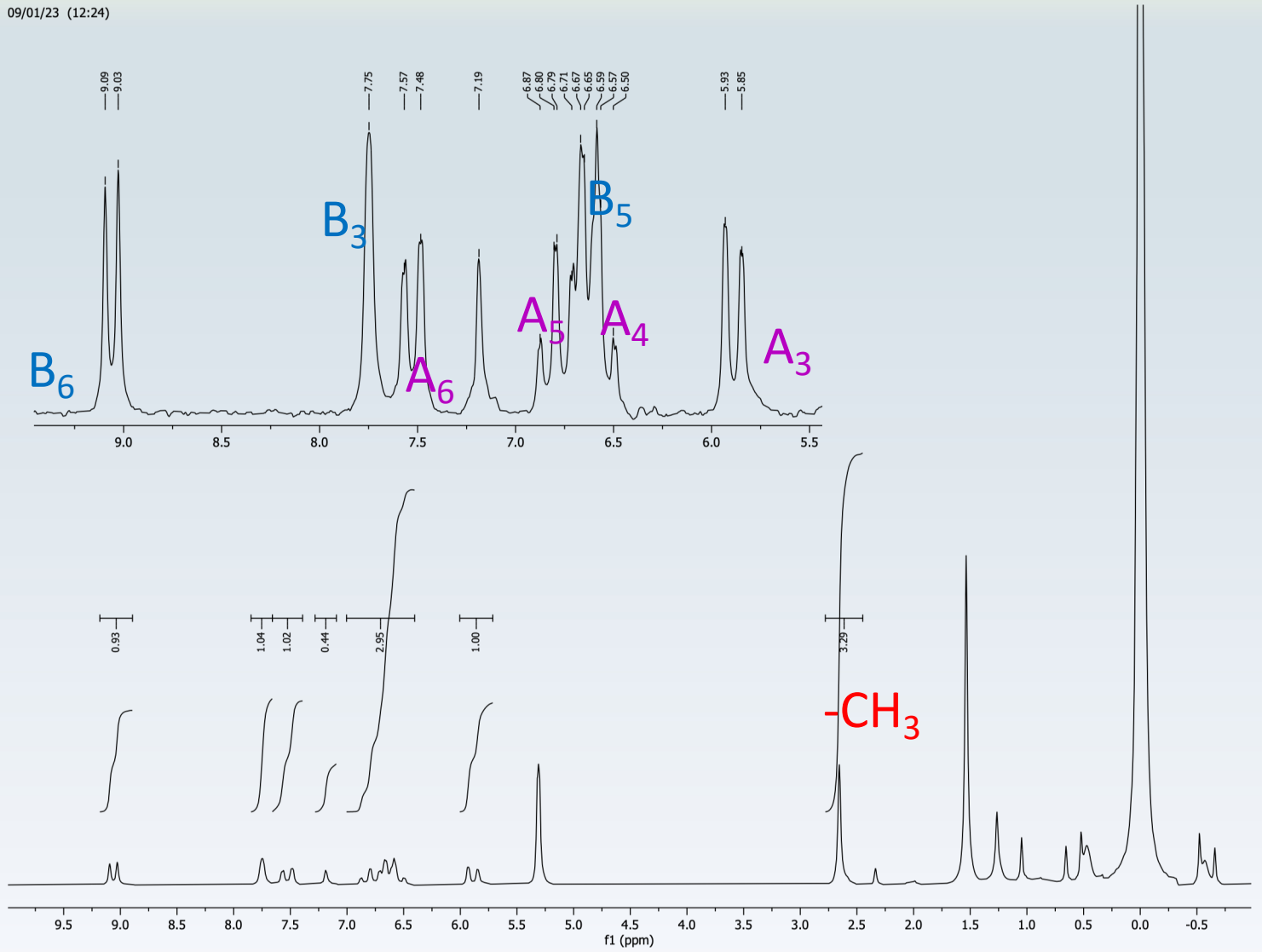


$[Ir(3\text{-methyl-2-phenylpyridine})_2Cl]_2$ (2)

^{1}H NMR Spectrum in DCM



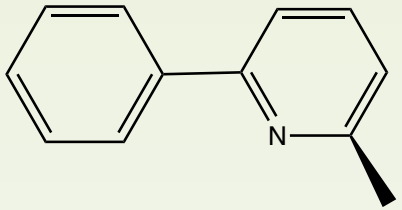


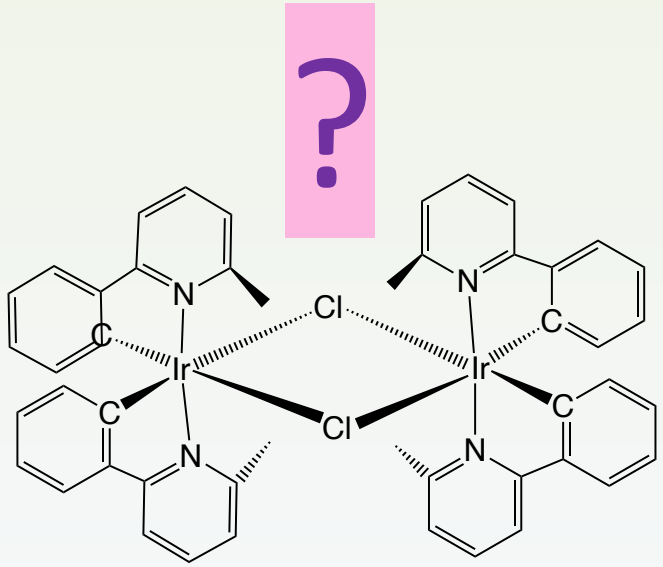


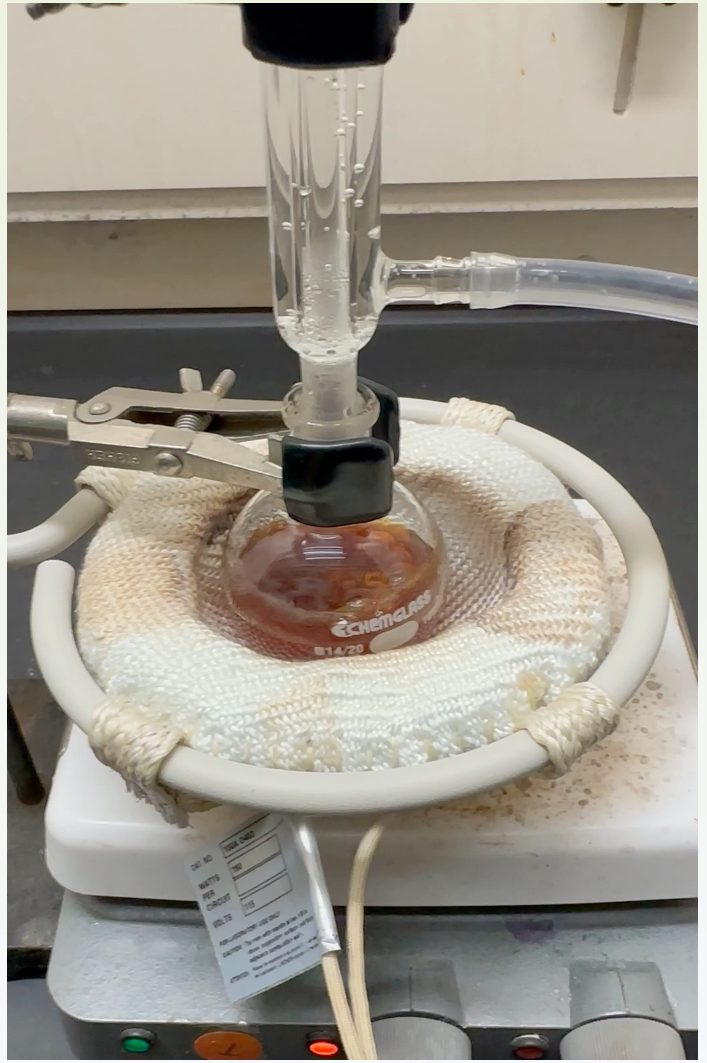
L.Akai: $[Ir(2\text{-methyl-5-phenylpyridine})_3Cl_3]$ (3)

Iridium 2-methyl-5-phenylpyridine derivatives: Synthesis

K_3IrCl_6 +



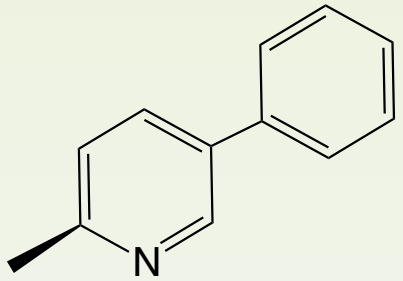


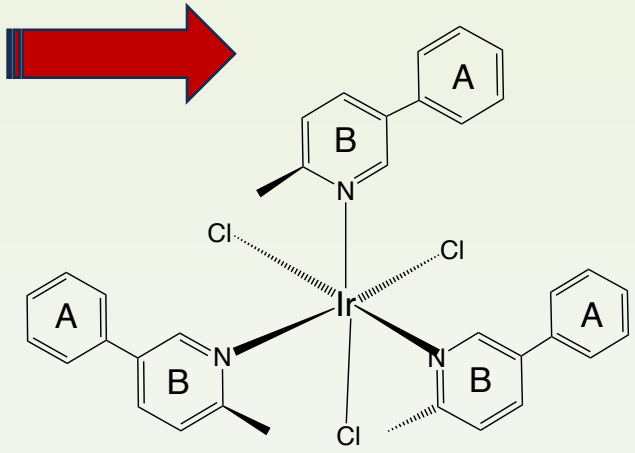


$[Ir(3\text{-methyl-5-phenylpyridine})_2Cl]_2$ (3)

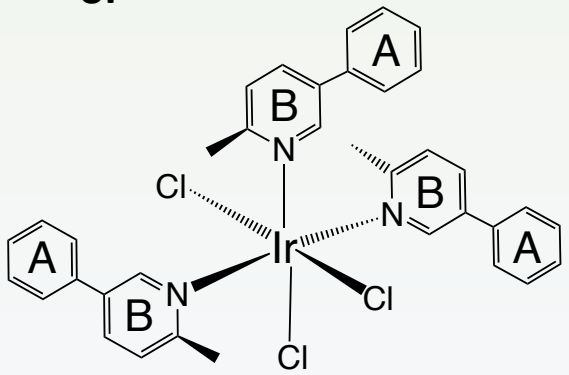
Iridium 6-methyl-2-phenylpyridine derivatives: Synthesis

K_3IrCl_6 +





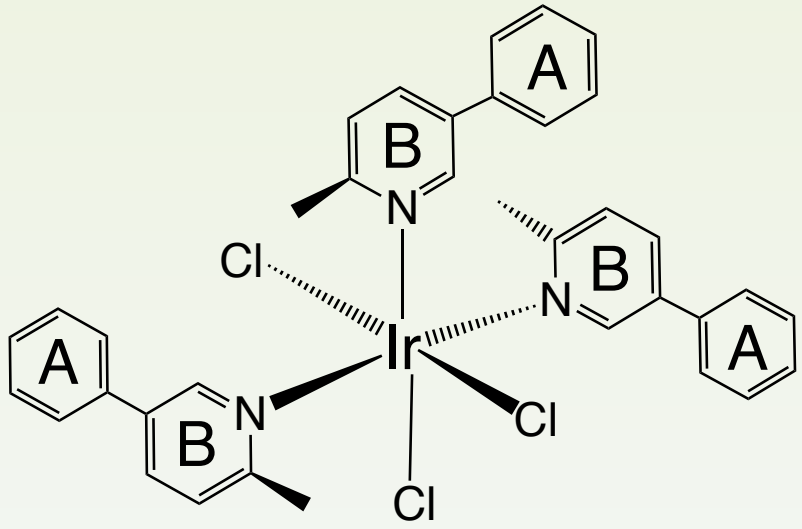
or



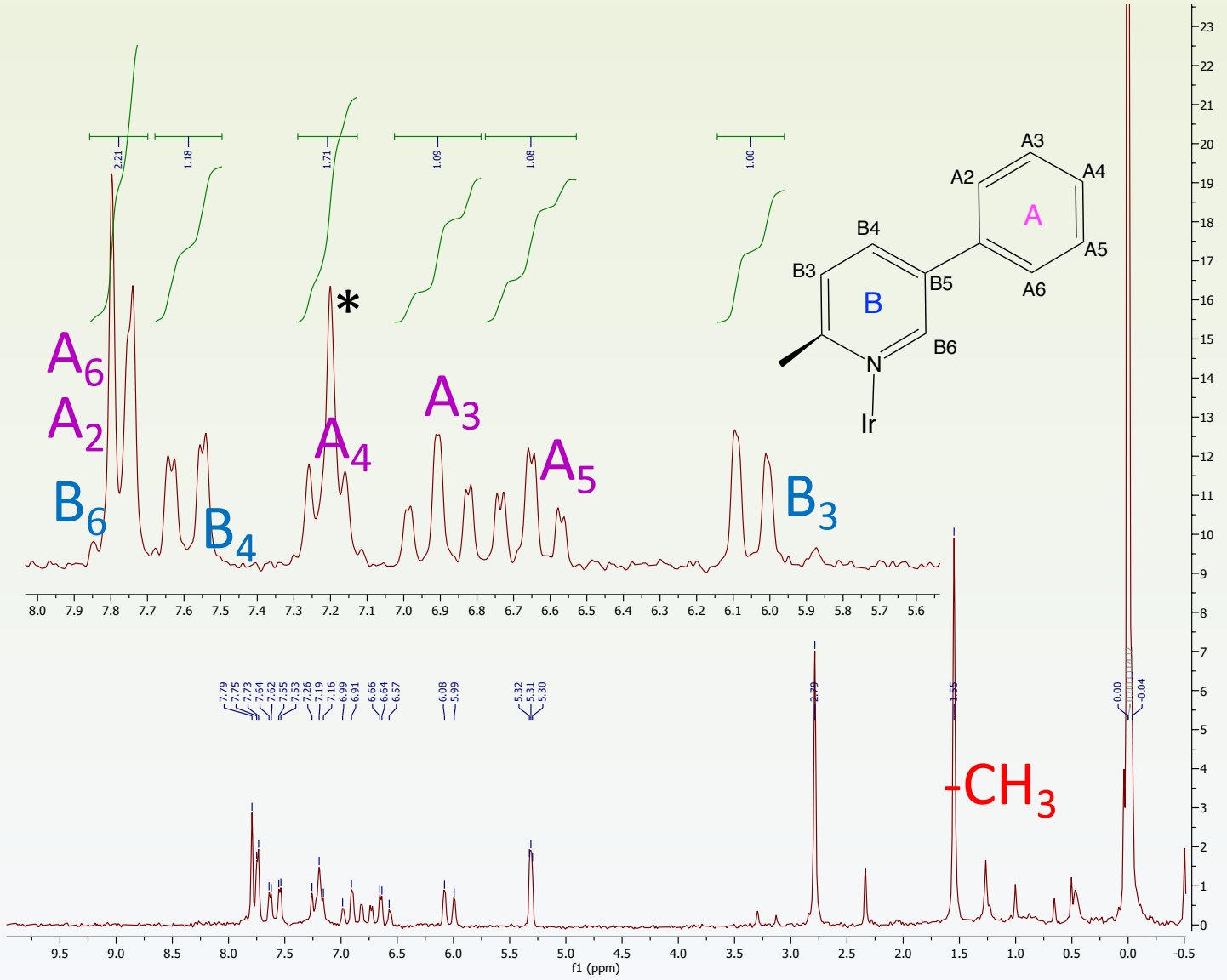
$[Ir(2\text{-methyl-5-phenylpyridine})_2Cl]_2$ (3)

1H NMR Spectrum in DCM

$[Ir(2\text{-methyl-5-phenylpyridine})_3Cl_3]$



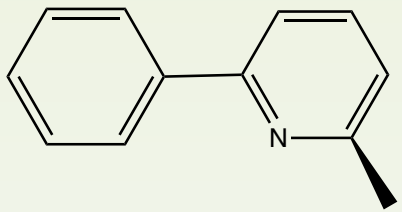
MolView

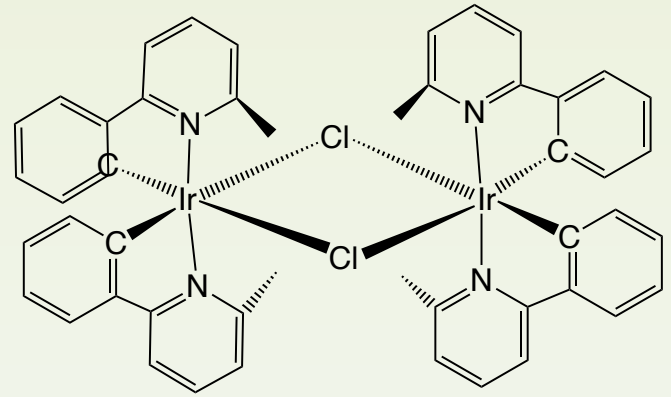


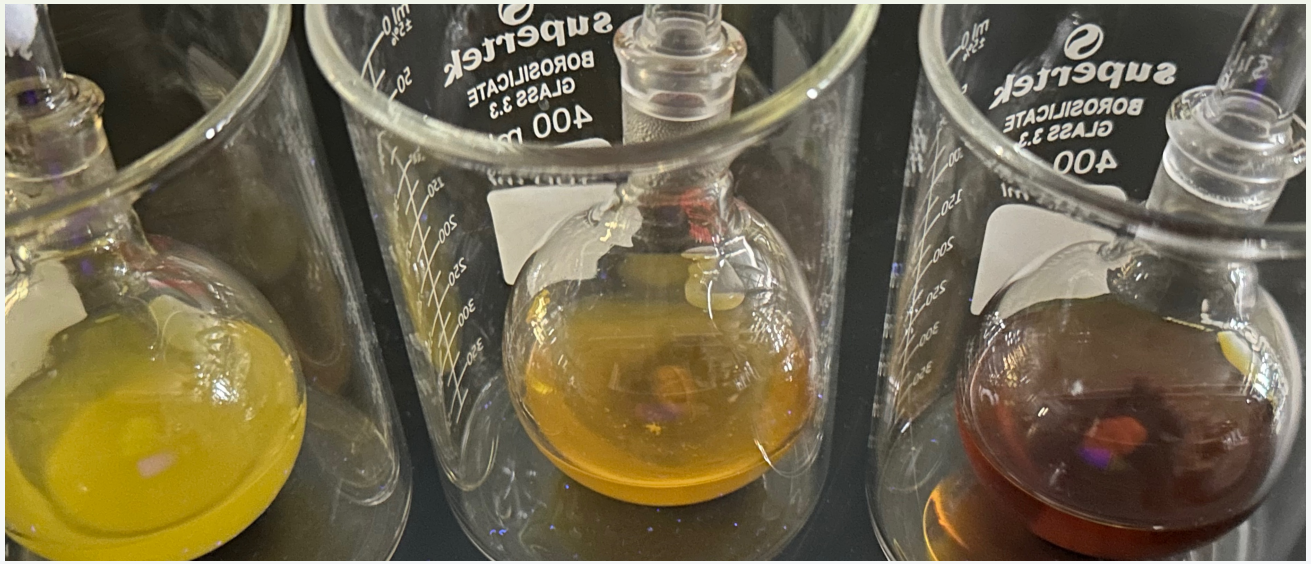
Iridium Complexes

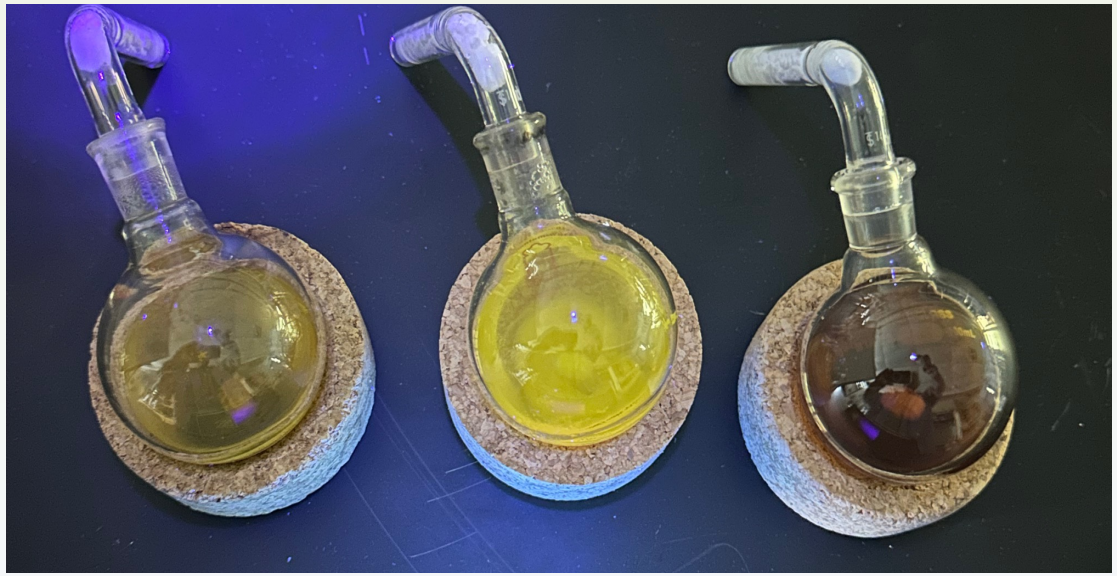
Iridium Complexes, in Solution

K_3IrCl_6 +





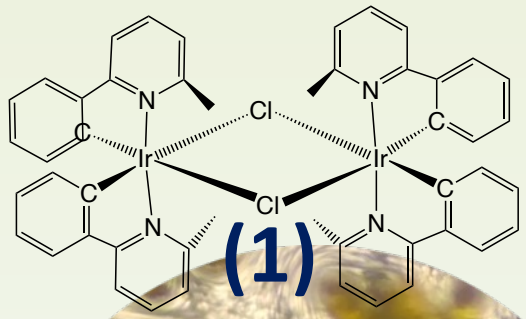




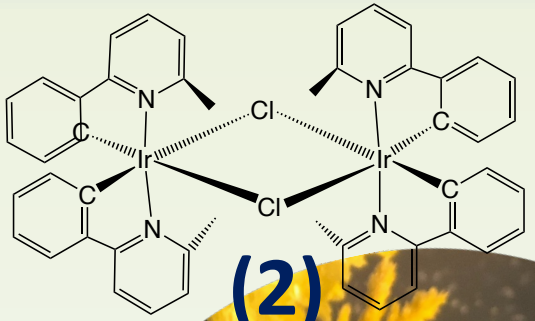
(2) (1) (3) (1) (2) (3)

Iridium Complexes: Crystals

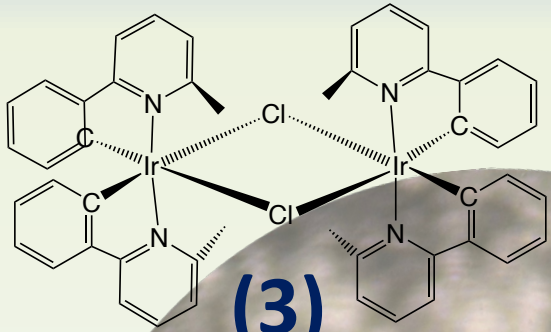
Iridium 6-methyl-2-phenylpyridine derivatives: Synthesis



(1)



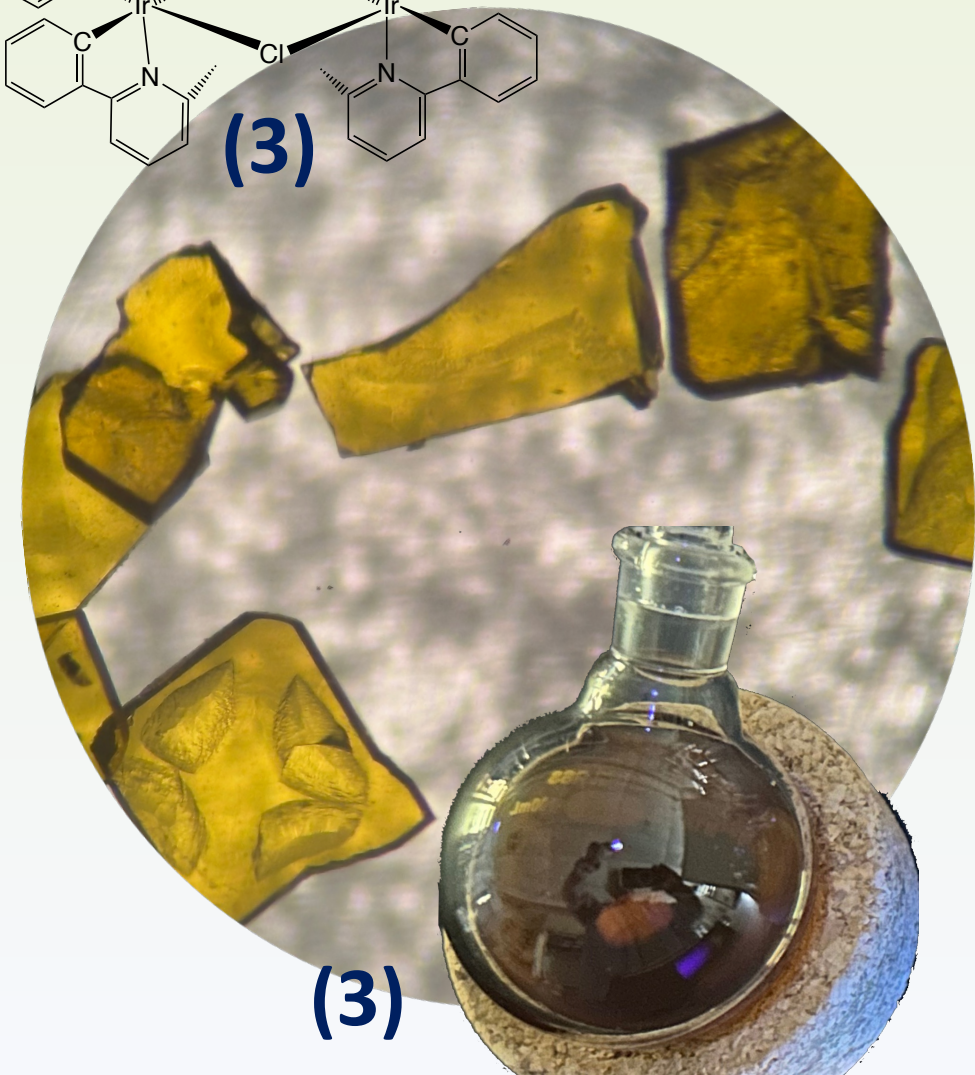
(2)



(3)

(1)

(2)

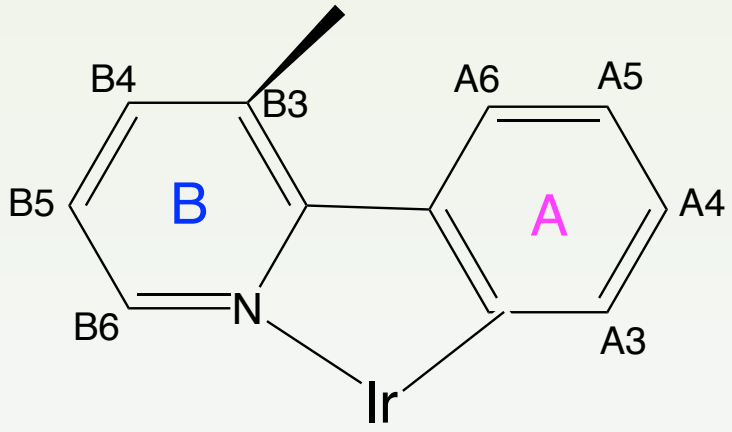


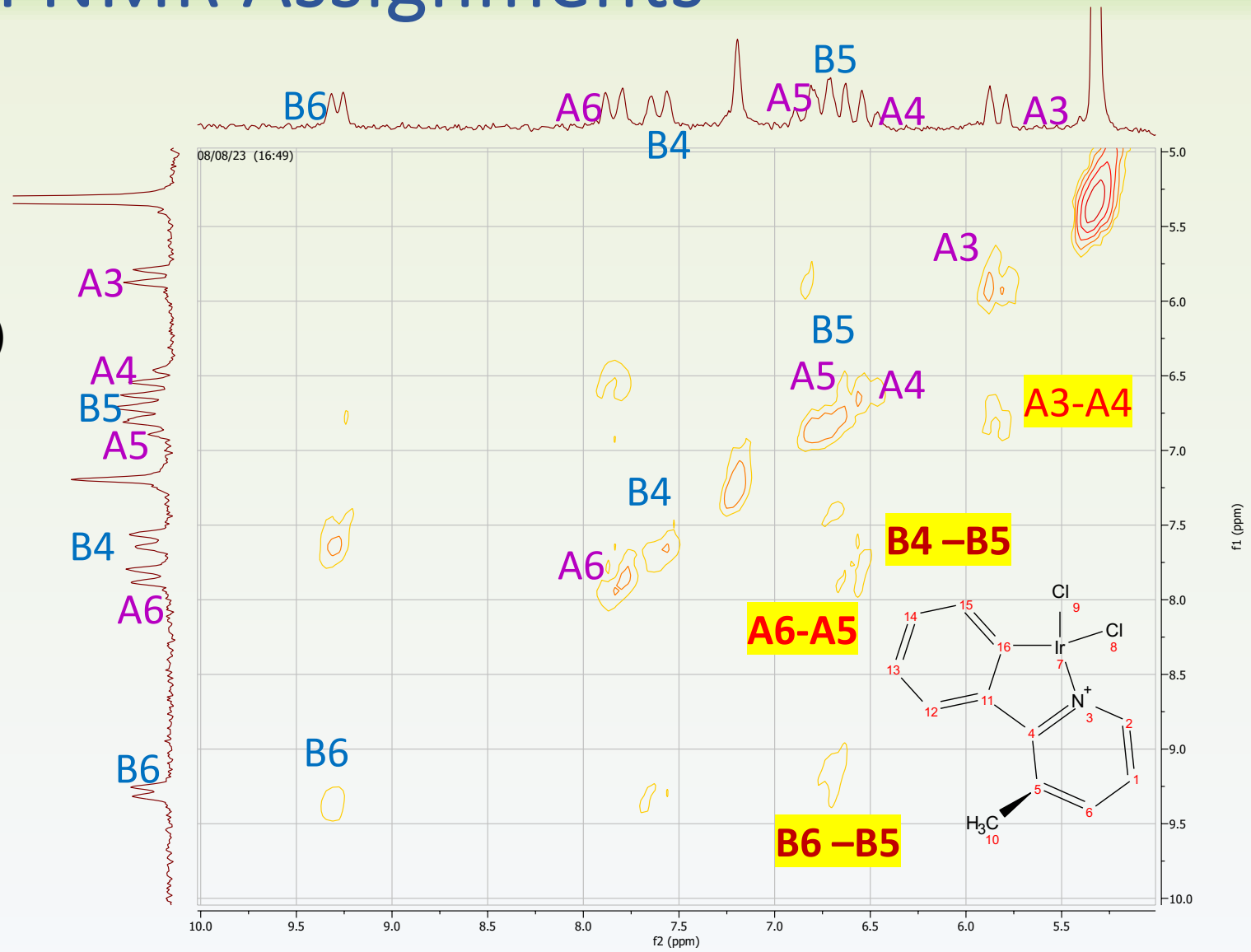
(3)

Correlation Spectroscopy (COSY): ^{1}H NMR Assignments

(1)

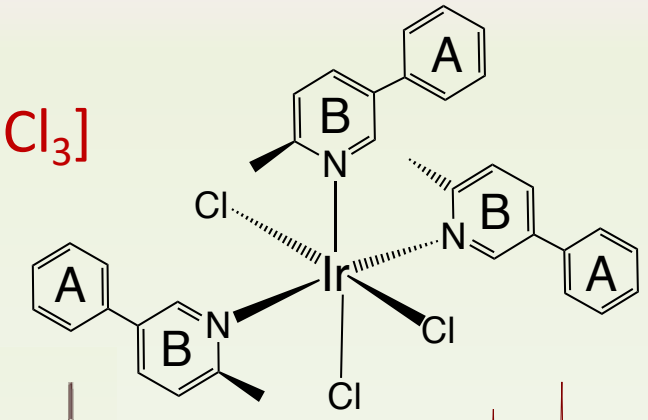
[Ir(3-methyl-2-phenylpyridine)$_2$Cl]$_2$ (1)

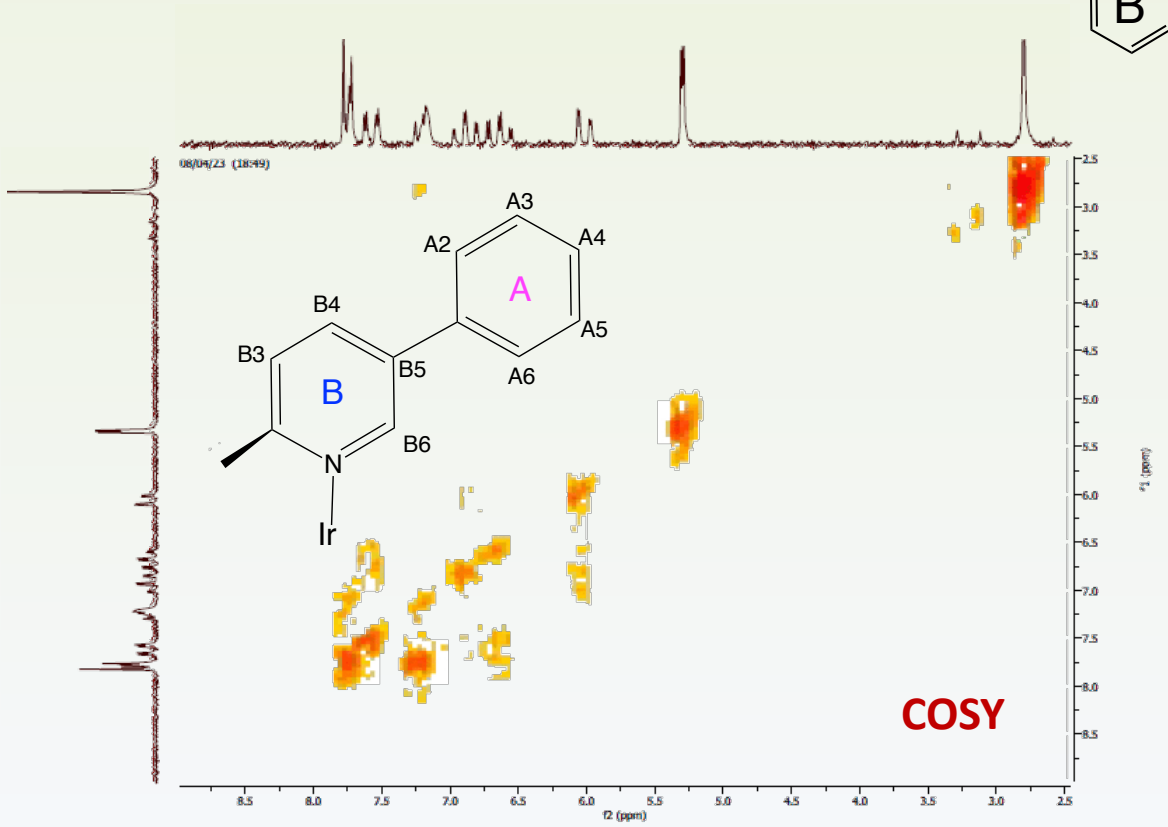


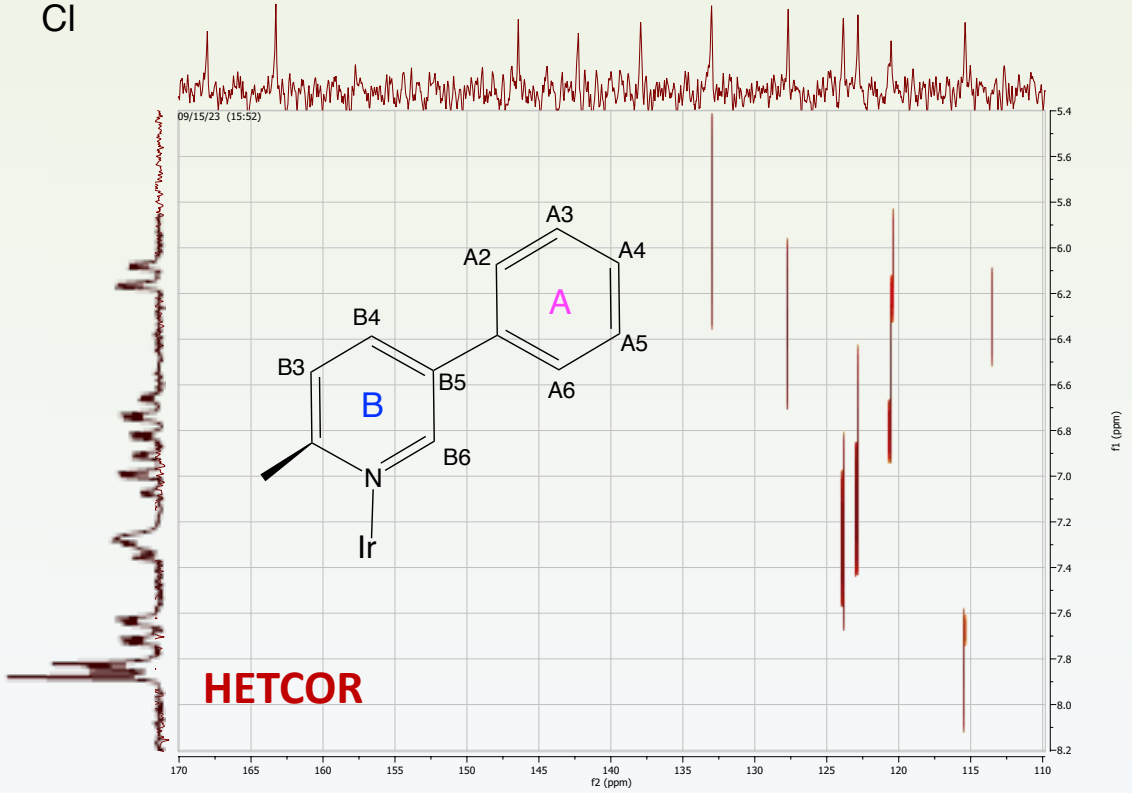


^{1}H-Correlation Spectrum & ^{1}H - ^{13}C Heteronuclear Correlation Spectrum

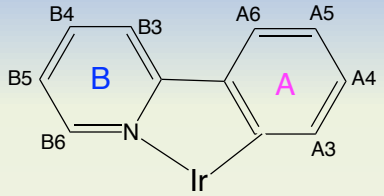
$[Ir(2\text{-methyl-5-phenylpyridine})_3Cl_3]$

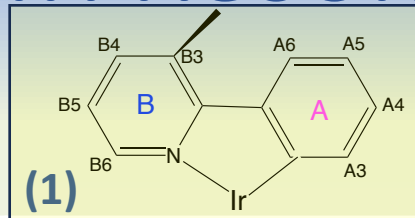


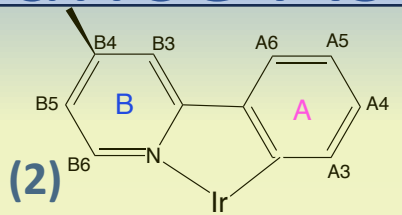


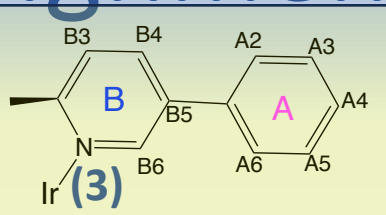


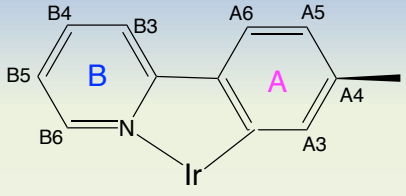
^{1}H NMR Resonance Assignments





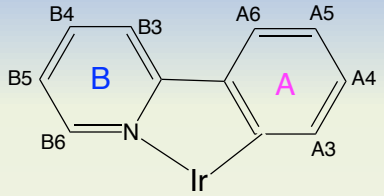


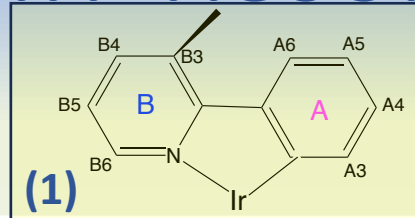


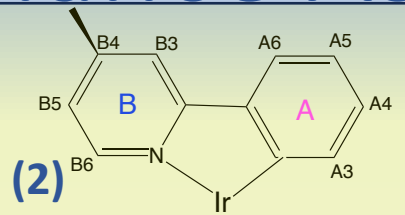


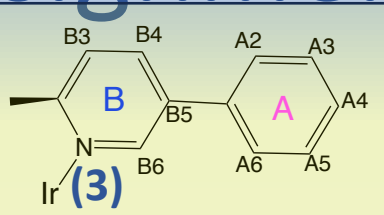
Resonance	2-phenylpyridine	3-me-2-phpyridine	4-me-2-phpyridine	5-me-2-phpyridine	2-(p-tolyl)pyridine
	Ligand / **Complex**	Ligand / **Complex**	Ligand / **Complex**	Ligand / **Complex**	Ligand / **Complex**
A1	-		-		
A2	8.20	7.65	8.088	7.8 / 7.508	8.09 / 5.68
A3	7.59 / 5.89	7.54 / 5.84	7.271 / 5.89	6.9 / 7.406	7.39 / 5.68
A4	7.53 / 6.80	7.50 / 6.54	7.60 / 6.57	7.2 / 7.341	- / -
A5	7.59 / 6.62	7.54 / 6.80	7.271 / 6.80	7.65 / 7.406	7.39 / 6.67
A6	8.20 / 7.57	7.65 / 7.85	8.088 / 7.52	7.8 / 7.508	8.09 / 7.44
B2	- / -	- / -	- / -	- / -	- / -
B3	7.80 / 7.95	- / -	7.861 / 7.75	6.05 / 8.364	7.77 / 7.87
B4	7.74 / 7.88	7.60 / 7.61	- / -	7.59 / 7.597	7.73 / 7.76
B5	7.26 / 6.82	7.20 / 6.70	X.XX / 6.66	- / -	7.24 / 6.79
B6	8.80 / 9.22	8.62 / 9.29	8.764 / 9.06	7.75 / 8.390	8.78 / 9.21
Methyl	- -	2.41 / 2.87	2.554 / 3.29	2.78 / 2.324	2.45 / 1.97

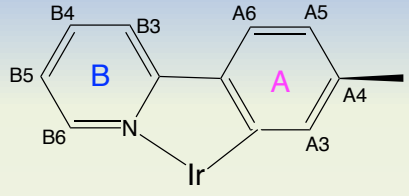
^{13}C NMR Resonance Assignments





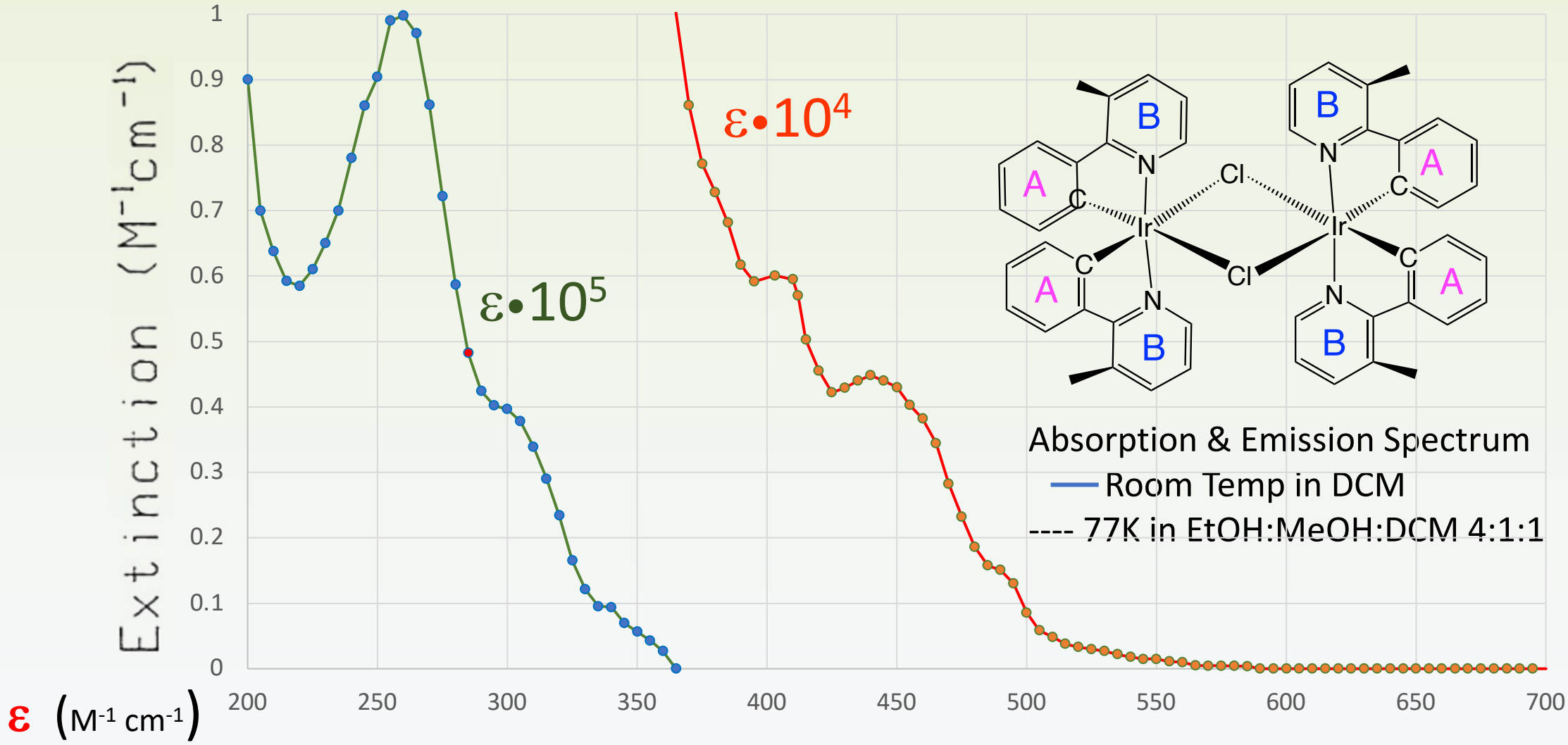






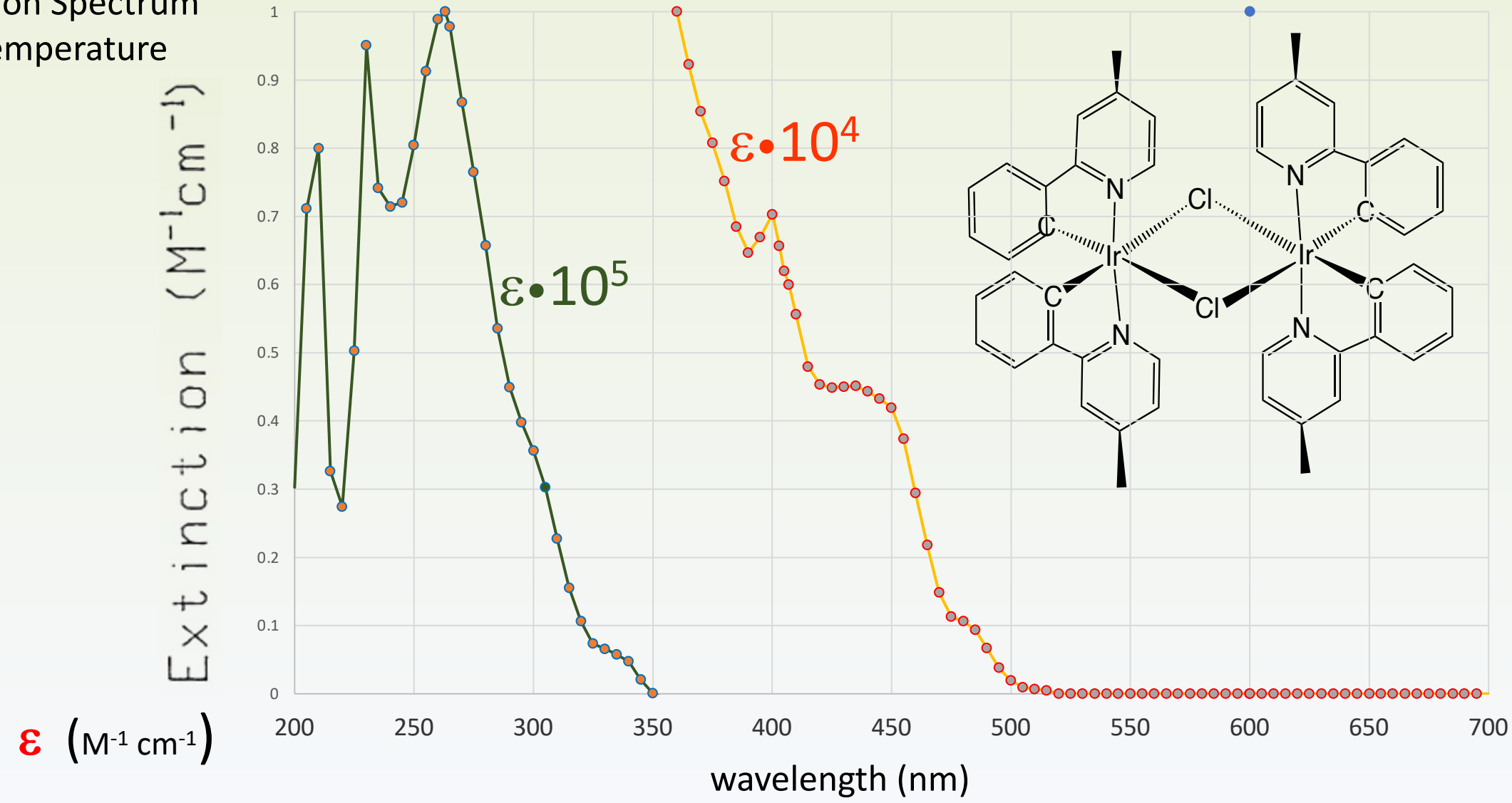
Resonance	2-phenylpyridine	3-me-2-phpyridine	4-me-2-phpyridine	5-me-2-phpyridine	2-(p-tolyl)pyridine
	Ligand / **Complex**	Ligand / **Complex**	Ligand / **Complex**	Ligand / **Complex**	Ligand / **Complex**
A1	139.6 / 144.8	141.2 / 147.4	138.0 / -	137.4 / 137.13	139.4 / 145.4
A2	127.1 / 144.0	129.4 / 146.6	128.3 / -	127.7 / 124.56	127.1 / 141.8
A3	129.0 / 130.3	128.3 / 130.9	128.4 / -	128.4 / 128.73	129.7 / 131.5
A4	129.2 / 129.1	128.1 / 128.4	127.8 / -	127.8 / 123.70	136.9 / 139.6
A5	129.0 / 122.5	128.3 / 121.4	128.4 / -	128.4 / 122.42	129.7 / 122.7
A6	127.1 / 123.6	129.4 / 128.2	128.3 / -	127.7 / 123.70	127.0 / 123.9
B2	157.4 / 168.0	158.6 / 166.4	**162.4** / -	156.2 / 167.08	157.5 / 168.4
B3	120.6 / 118.7	131.1 / 132.1	117.6 / -	122.7 / 133.39	120.6 / 118.6
B4	136.9 / 136.6	138.6 / 141.3	147.7 / -	135.03 / 124.56	136.9 / 136.8
B5	122.5 / 122.5	122.3 / 122.6	124.6 / -	136.22 / 162.69	122.5 / 122.4
B6	150.0 / 151.5	147.2 / 151.0	139.1 / -	148.38 / 138.04	150.0 / 151.8
Methyl	- / -	20.2 / 23.8	21.3 / -	24.4 / 26.53	21.4 / 21.7

Absorption and Emission Spectrum (1)
$[Ir(3\text{-methyl-2-phenylpyridine})_2Cl]_2$



Absorption and Emission Spectrum (2)
$[Ir(4\text{-methyl-2-phenylpyridine})_2Cl]_2$

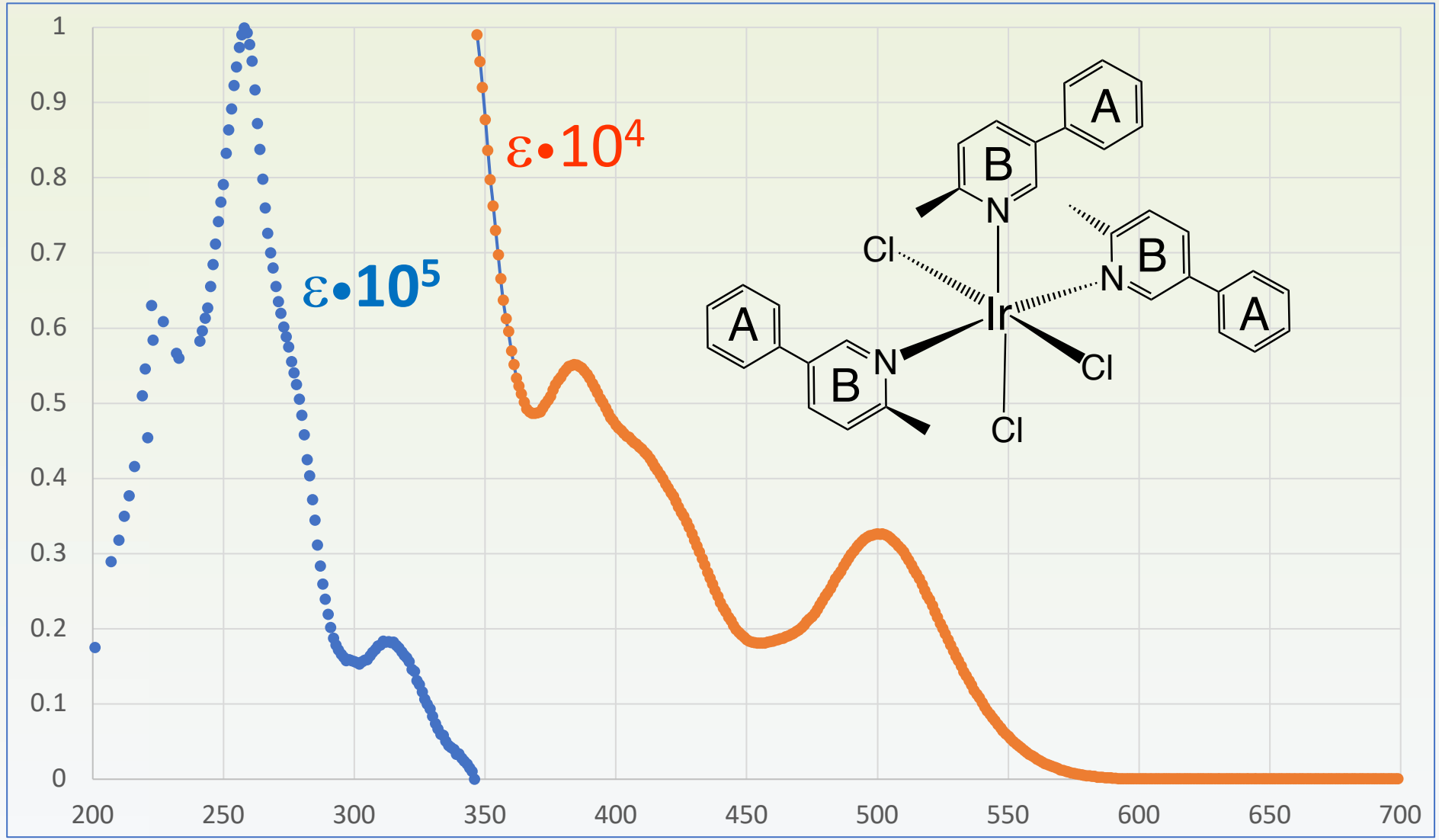
Absorption Spectrum
Room Temperature
in DCM



Absorption and Emission Spectrum (3)

$[Ir(2\text{-methyl-5-phenylpyridine})_2Cl]_2$

Absorption Spectrum
Room Temp
in DCM



ε $(M^{-1}\ cm^{-1})$

Conclusion

Over the summer, our team of budding researchers, initially inexperienced, embarked on an exciting journey into the world of iridium polypyridyl complexes. Through dedication and hands-on learning, we honed our laboratory techniques and analytical skills. Our efforts yielded four distinct iridium orthometallated complexes using 2-phenylpyridine derivative ligands. While one complex posed yield and purification challenges, we successfully synthesized the others, with yields ranging from 30% to 60%, and reaction times varying from one to 24 hours.

Our compounds exhibited vibrant colors, evident in UV-VIS spectra, and surprisingly, all but complex 3 displayed luminescence properties. We diligently assigned proton and carbon resonances and were thrilled to obtain quality crystals for X-ray crystallography analysis for all complexes. This research experience not only expanded our knowledge but also instilled the fundamentals of laboratory research, proving that undergraduate science students at Miramar College can make significant strides in scientific exploration.

Acknowledgement

We extend our heartfelt gratitude to the following individuals and organizations whose unwavering support and contributions were instrumental in the success of our summer research project on synthesizing iridium complexes:

Title V HIS STEM Exito Project We are deeply thankful to the Title V HIS STEM Exito Project for their generous sponsorship of our research. Their commitment to advancing STEM education has provided invaluable opportunities for our community college students and enabled us to pursue this exciting project.

Dr. Linda Woods, Dean of Miramar College and Department Chair, Dr. Namphol Sinkaset: Their leadership and resourceful contributions were instrumental in initiating this research and securing funds for key chemicals.

Lab Technicians: Our heartfelt thanks go to our dedicated lab technicians: Tien Nguyen, Calvin Le, and Bryce Thompson. Their tireless efforts in maintaining our lab facilities and overseeing the procurement of chemicals ensured the smooth operation of our research and the realization of our objectives.

This project would not have been possible without the collective effort and commitment of these individuals and organizations. We are grateful for their support, mentorship, and belief in our research endeavors