Hands-on Synthesis, Characterization, & Structural Analysis of Iridium (III) complexes; Introducing Undergraduate Community College Students to Organometallic Research

Summer 23 & Summer 24

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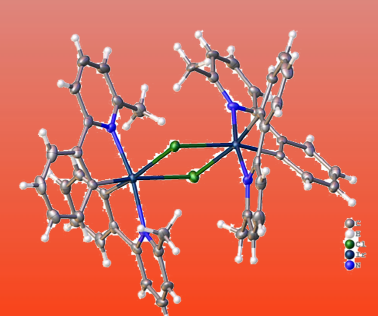
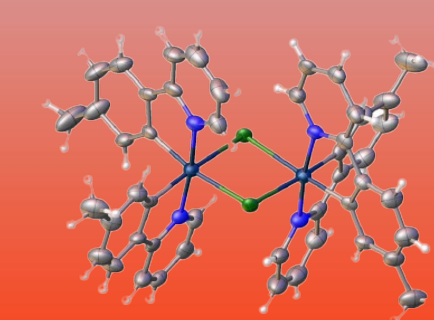
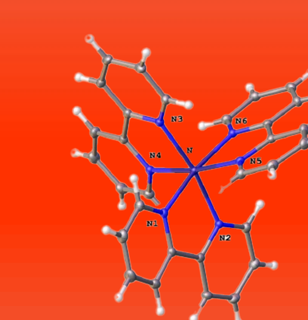




San Diego, Miramar College, Summer Research in Chemistry



Abstract

Fred Garces*, Gary Smith, Milan Gembicky, Lara Cokic, Brian Lam, Cao Tran, Danny Martin, Tyler Bui, Lisa Akai, Tam AboNabout, Joejose Donato, Adrieon Key, Cooper Ambre, Oceana Yu, Ilene Jahanpour

San Diego Miramar College undergraduates engaged in summer research projects centered on the synthesis and characterization of iridium(III) metal complexes. The primary objective was to immerse students in synthetic chemistry while providing them with practical experience using advanced instrumentation within the department. Throughout the project, students successfully synthesized several iridium(III) cyclometallated complexes, utilizing methylated 2-phenylpyridine ligands as the primary building blocks. In addition to these, students investigated coordination isomerism by synthesizing an iridium complex where one pyridine ring in 2,2-bipyridine flipped 180 degrees, resulting in a carbon atom binding to the iridium center rather than the typical nitrogen coordination.

The research required students to optimize reaction conditions, including temperature control and solvent selection, to maximize yields, which ranged from 30% to 60%. The synthesized iridium complexes exhibited distinct UV-Vis absorption characteristics, and many demonstrated luminescence properties. Students also employed 1H and 13C NMR spectroscopy to confirm structural integrity, with some complexes subjected to advanced techniques like COSY and HETCOR NMR for detailed resonance assignments. Electrochemical analysis of selected complexes was carried out using cyclic voltammetry to assess their redox properties.

Furthermore, some of the complexes produced crystals suitable for X-ray crystallographic analysis, allowing for high-resolution structural determination. This aspect of the project provided students with invaluable experience in the process of crystal growth and structure elucidation. The overall research experience fostered students' proficiency in both synthesis and instrumental analysis, exposing them to methodologies commonly used in cutting-edge scientific research. Through this project, students developed a deeper understanding of organometallic chemistry, equipping them with practical skills and knowledge essential for their future careers as chemists.

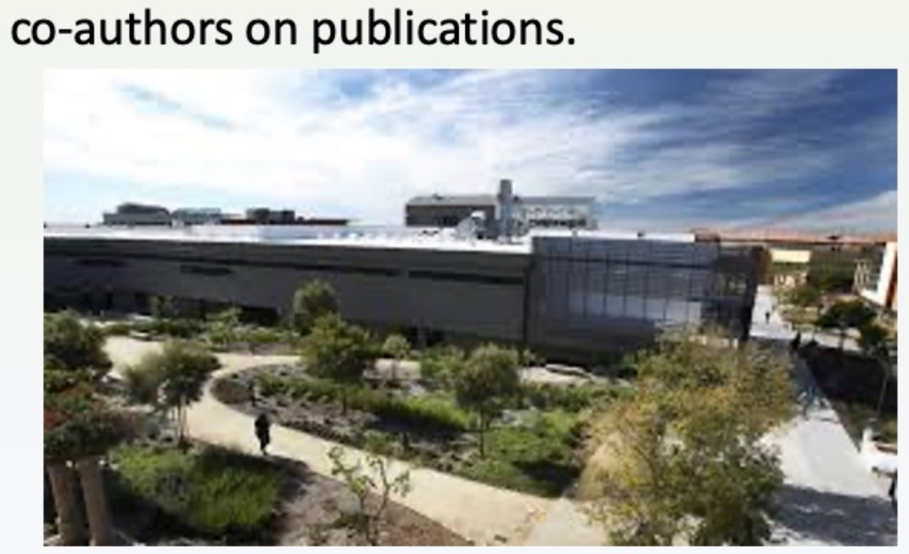
Undergraduate Research at a Community College

Over the past two summers, San Diego Miramar College provided undergraduate students with an exceptional opportunity to engage in advanced research comparable to that at four-year institutions. The program centered on three main areas: synthesizing polypyridyl iridium complexes, developing instrumentation and operation manuals, and attempting the synthesis of an iridium bipyridine dimer.

Participants gained hands-on experience in chemical analysis and laboratory techniques, working with advanced instruments such as Spectroscopy-Electrochemistry (SEC), Inductively Coupled Plasma Optical Emission Spectroscopy (iCAP-OES), Gas Chromatography-Mass Spectrometry (GCMS), Ultra-High-Performance Liquid Chromatography (UHPLC), and Nuclear Magnetic Resonance (NMR). They also conducted original organometallic synthesis involving iridium complexes while refining their skills in literature review and scientific communication.

The program emphasized collaboration through regular group meetings where students discussed progress and explored relevant research. Those who made significant contributions had the chance to be co-authors on publications.

Although unpaid, eligible students could apply for financial support through the Learn and Earn Advantage Program (LEAP) work-study program. Research took place in dedicated facilities, with an orientation held in early June. This opportunity allowed community college students to gain invaluable research experience and develop essential skills for future academic and professional success in chemistry.



Why Iridium(III) 2-phenylpyridine complexes

The pioneering work on Iridium(III) phenylpyridine complexes by Richard Watts and colleagues at UCSB in the 1980s and 1990s laid the foundation for a field with far-reaching implications. Their discovery of tris-phenylpyridine iridium(III) complexes revolutionized organic light-emitting diodes (OLEDs) and beyond (1).

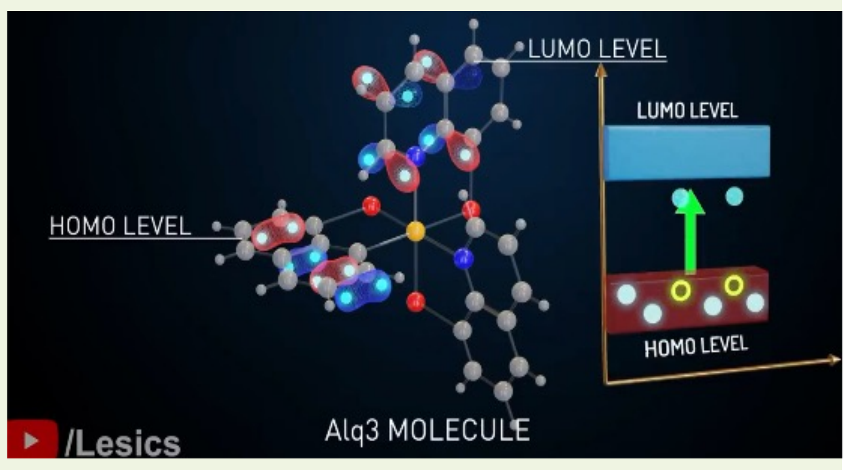
These complexes exhibit exceptional photoluminescence efficiency and tunable emission wavelengths, making them ideal for OLED applications (1). Their photocatalytic properties have opened new avenues in synthetic organic chemistry, enabling challenging transformations under mild conditions (2).

The versatility of these complexes extends to biological applications, showing promise in bioimaging, photodynamic therapy, and antibacterial treatments (2 3). Water-soluble variants have expanded their utility in biological applications and green chemistry (2).

Future applications are vast, including more efficient solar cells and artificial photosynthesis technologies (4). The integration of these complexes with nanomaterials could lead to novel sensors and advanced drug delivery systems. In quantum information science, their long-lived excited states could be exploited for quantum sensing or computing applications (4).

Ongoing research in ligand design continues to unlock novel properties, such as asymmetric tris-heteroleptic iridium(III) complexes for improved OLED performance. The antibacterial properties of certain iridium complexes offer potential solutions to antibiotic resistance (2).

The field of iridium chemistry with phenylpyridine ligands remains dynamic, with applications spanning from materials science to medicine. Watts' pioneering work continues to inspire researchers worldwide, driving progress in diverse fields (1 4).



Applications

Iridium(III) phenylpyridine complexes have a wide range of applications across various fields. Here are at least 10 applications:

1. Organic Light-Emitting Diodes (OLEDs)
2. Photocatalysis
3. Antibacterial agents
4. Antitumor treatments
5. Bioimaging
6. Sensors
7. Photodynamic therapy
8. Solar energy conversion
9. Quantum computing
10. Photoredox catalysis

Top 3 applications with examples and citations:

1. Organic Light-Emitting Diodes (OLEDs): Iridium(III) complexes are widely used in OLEDs due to their high luminescence efficiency and color tunability. They serve as green and red emitters in commercial OLED displays (1).
2. Photocatalysis: These complexes are employed as powerful photoredox catalysts in synthetic organic chemistry. They enable challenging transformations under mild conditions, facilitating single-electron transfer processes (2).
3. Antibacterial / Antitumor agents: Iridium(III) phenylpyridine complexes have shown significant antibacterial activity against various strains, including drug-resistant bacteria. For example, a study reported that these complexes exhibited higher antibacterial activity than ciprofloxacin against several bacterial strains (3).
4. Antitumor treatments: Research has demonstrated the potential of these complexes as antitumor agents. A study showed that iridium(III) complexes exhibited antiproliferative activity against cancer cell lines such as A549 and HeLa, with IC50 values ranging from 2.8 ± 0.8 µM to 39.5 ± 2.7 µM (4)

Top 3 Applications

OLED –Organic Light Emitting Diodes is a display technology that uses organic compounds 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.

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.

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.

Properties

Key Features:

- High quantum yields
- Color tunability
- Long-lived excited states
- Photostability

Iridium(III) cyclometallated phenylpyridine complexes have found numerous applications due to their unique photophysical and chemical properties. The listed applications showcase the versatility of iridium(III) cyclometallated phenylpyridine complexes, spanning from materials science and catalysis to biomedical applications and sensing technologies.

OLED

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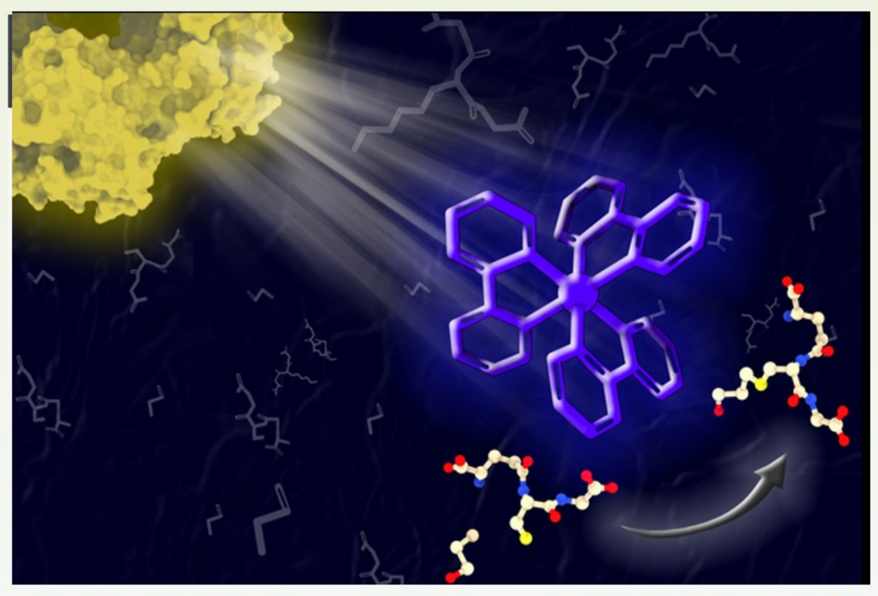
Photocatalyst

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.

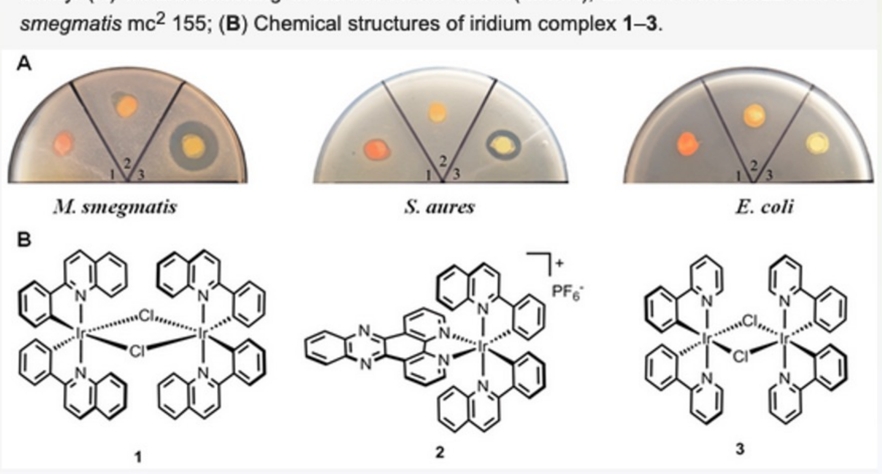


https://chemistry.mit.edu/chemistry-news/this-light-powered-catalyst-mimics-photosynthesis/

Anti-Cancer and Anti-Bacterial

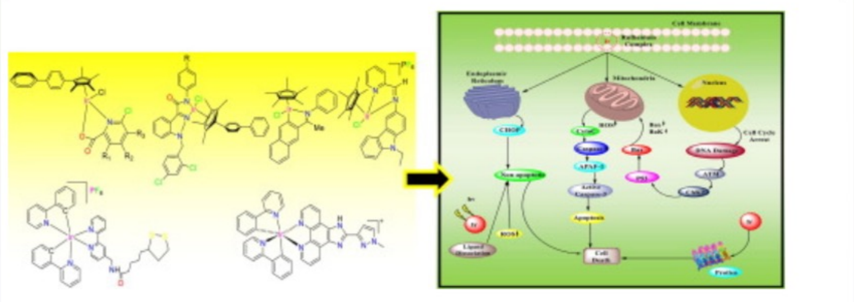
Anti Cancer (Bacterial) Agents –Iridium-based compounds have shown promise as anticancer agents, exhibiting potential for targeted cancer therapy. Additionally, iridium complexes have demonstrated antibacterial activity against drug-resistant bacteria, suggesting their potential application in combating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

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**.



The need to synthesize an effective anticancer drug with minimal side effects has been the aim and concern of the researchers around the world. Cytotoxicity and selectivity are the two important factors of an anticancer drug and the current clinically approved drugs such as cisplatin, oxaloplatin and carboplatin have low cytotoxicity and selectivity towards cancer cells. In the recent times, transition metal based scaffolds other than platinum have gained a lot of attention. In this regard, iridium based complexes have been promising anticancer scaffolds. In this review we summarize the recent advances in iridium complexes as anticancer agents based on their cytotoxicity results.

Graphical abstract



Instruments @ SD Miramar College

Miramar College offers students access to a wide range of state-of-the-art instruments for chemical analysis and research. The chemistry department features advanced spectroscopic tools, including UV-Vis spectrophotometers (Agilent Cary 50, ThermoFisher NanoDrop, Genesys 10), fluorescence spectrophotometers (FluoroLog-3), and FTIR spectrometers (Thermo Scientific Nicolet 380 and iS5).

For elemental analysis, students use the ThermoScientific iCAP OES 7000 for inductively coupled plasma optical emission spectrometry. The department also houses a ThermoScientific ICQ 7000 Quadrupole GC-MS, integrating gas chromatography and mass spectrometry for detailed chemical analysis.

A key instrument is the Anasazi EFT 90 MHz NMR Spectrometer, capable of advanced nuclear magnetic resonance studies, including COSY and HETCOR experiments, with Maestra Nova software for data analysis. Additional resources include electrochemistry tools (eDAQ eCorder) and Vernier Logger Pro sensors.

Through a partnership with UCSD, students also gain access to X-ray crystallography facilities. This comprehensive instrumentation suite provides hands-on experience with professional-grade equipment, preparing students for advanced research and industry careers.

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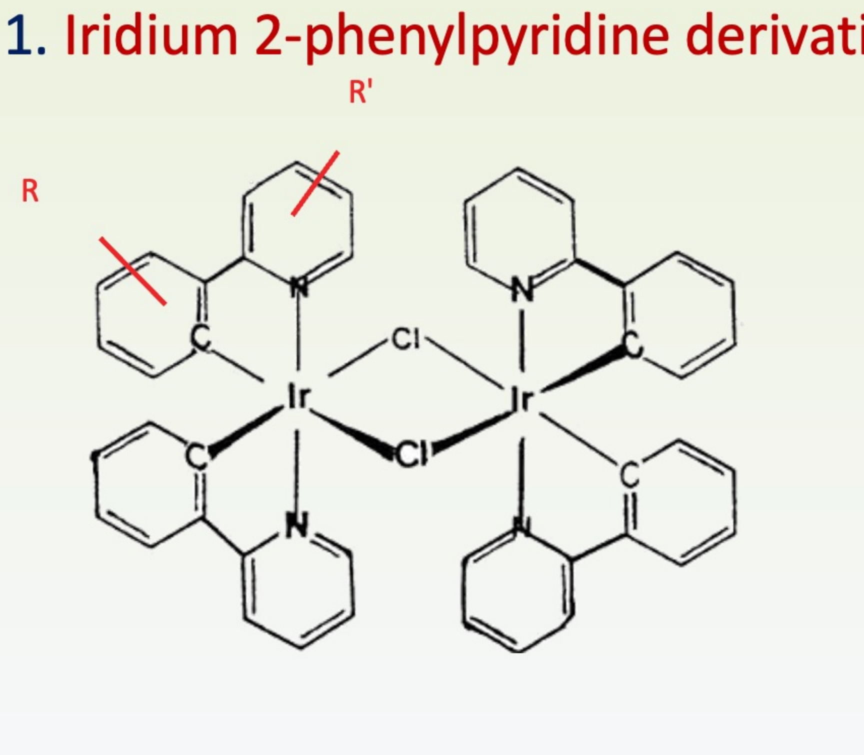
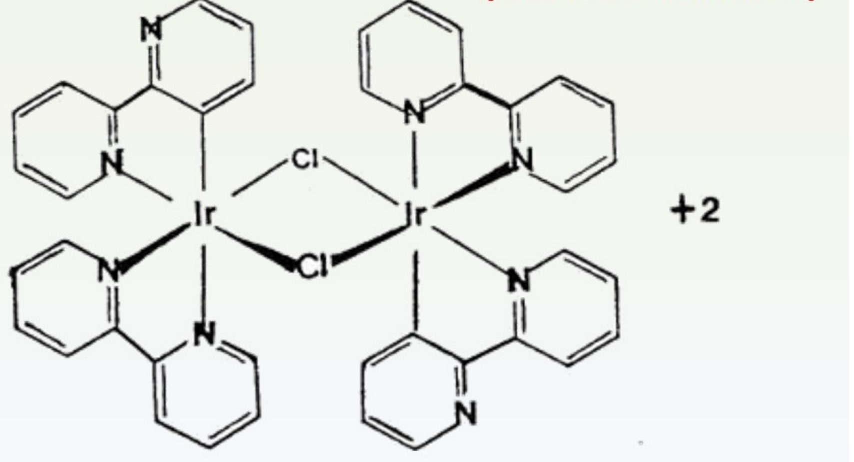
Area of Research, Summer 2023, 2024

1. Iridium 2-phenylpyridine derivatives

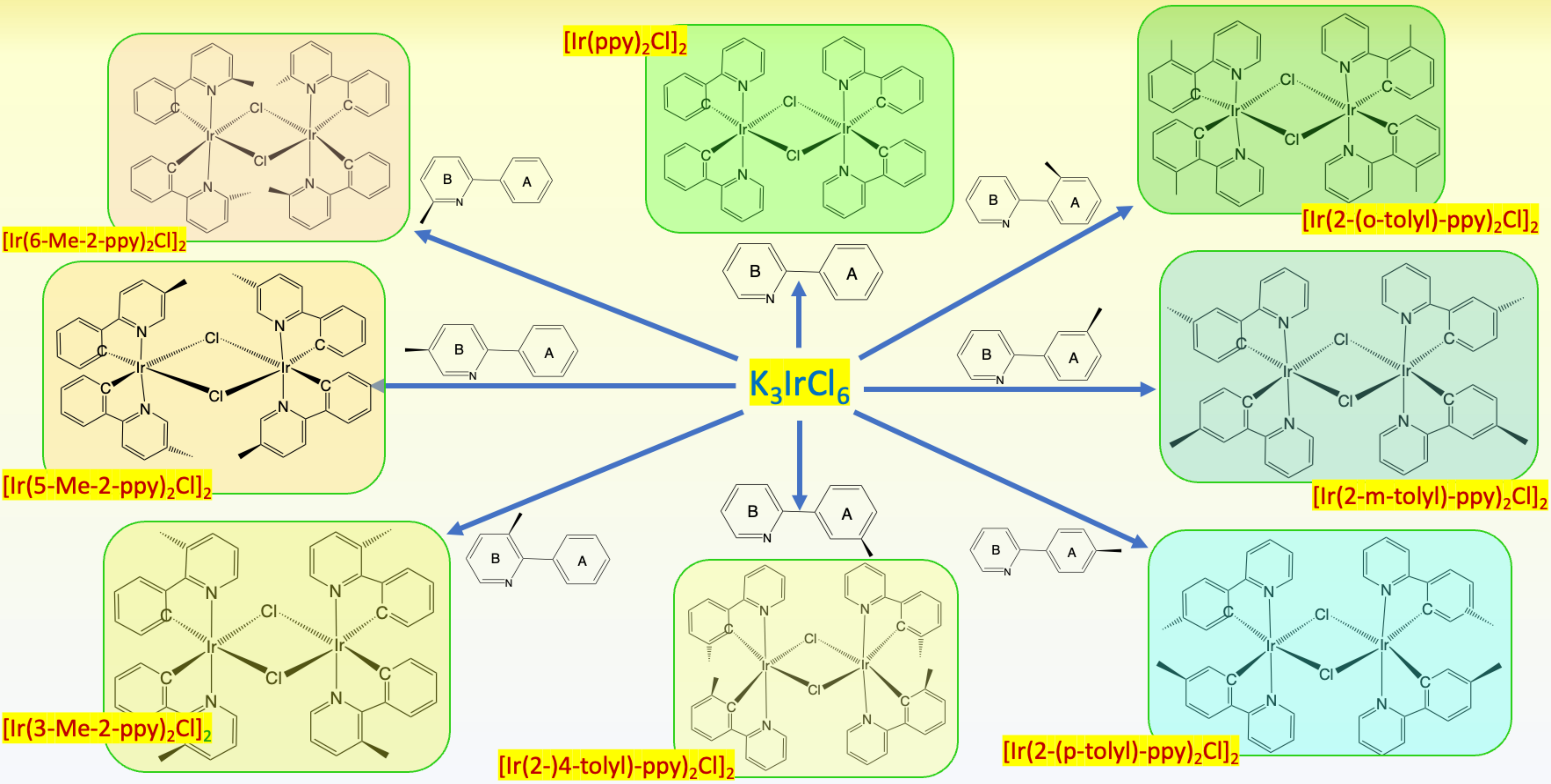


2. Iridium C-bonded 2,2'-ipyridine (Watts Dimer)



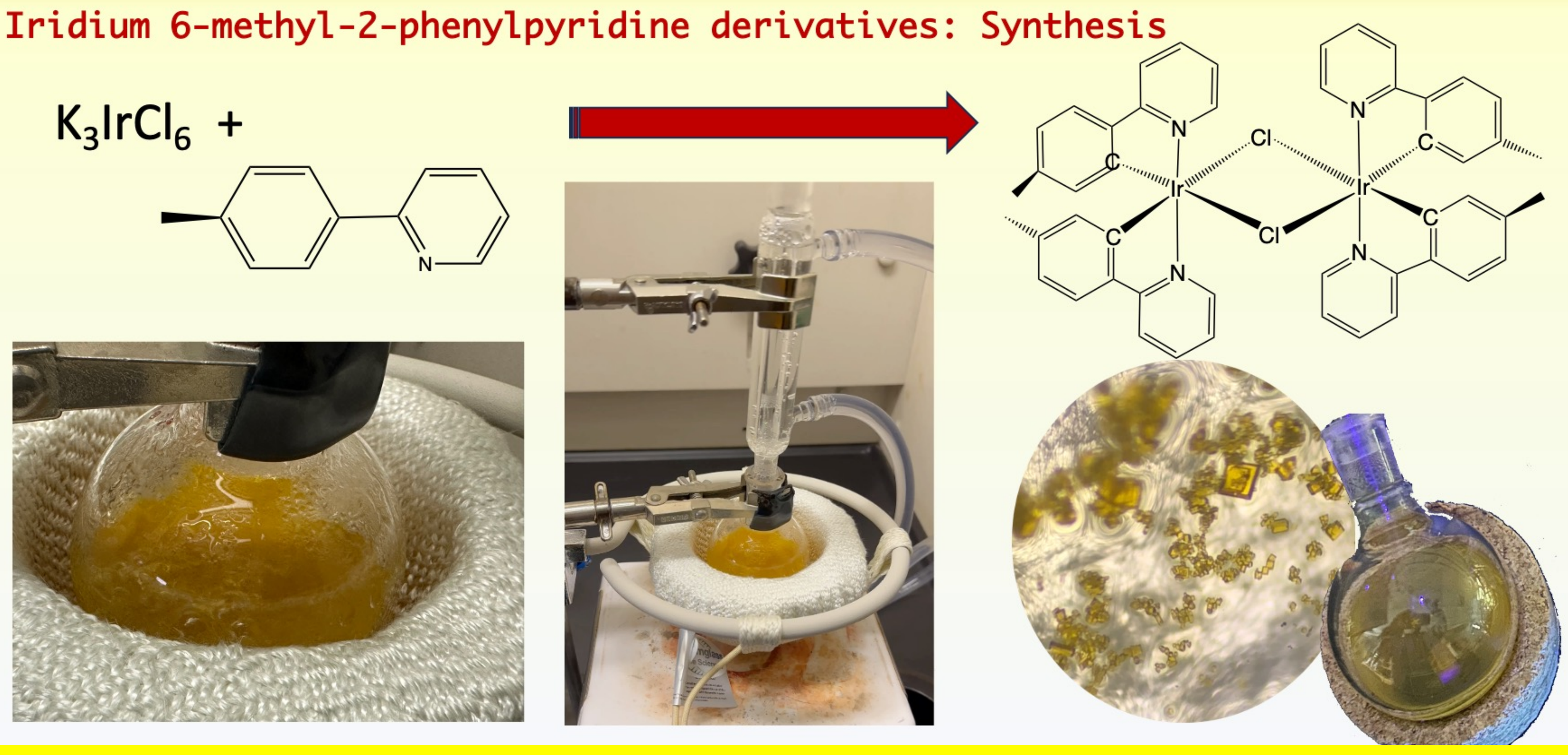
Synthetic Scheme & Crystals

Ir(III) w/2-ppy complexes synthesis



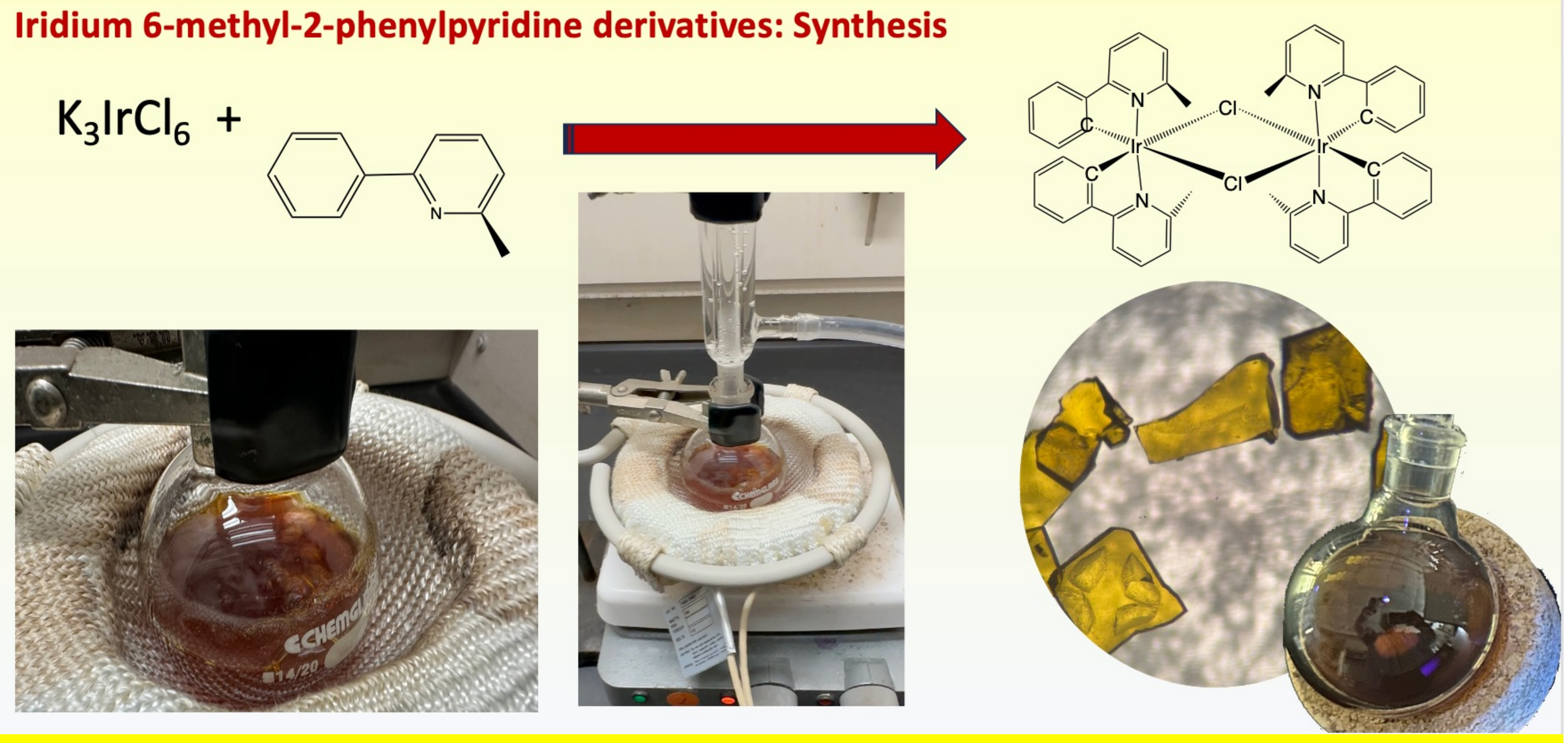
[Ir((2-p-tolyl)pyridine)$_2$Cl]$_2$

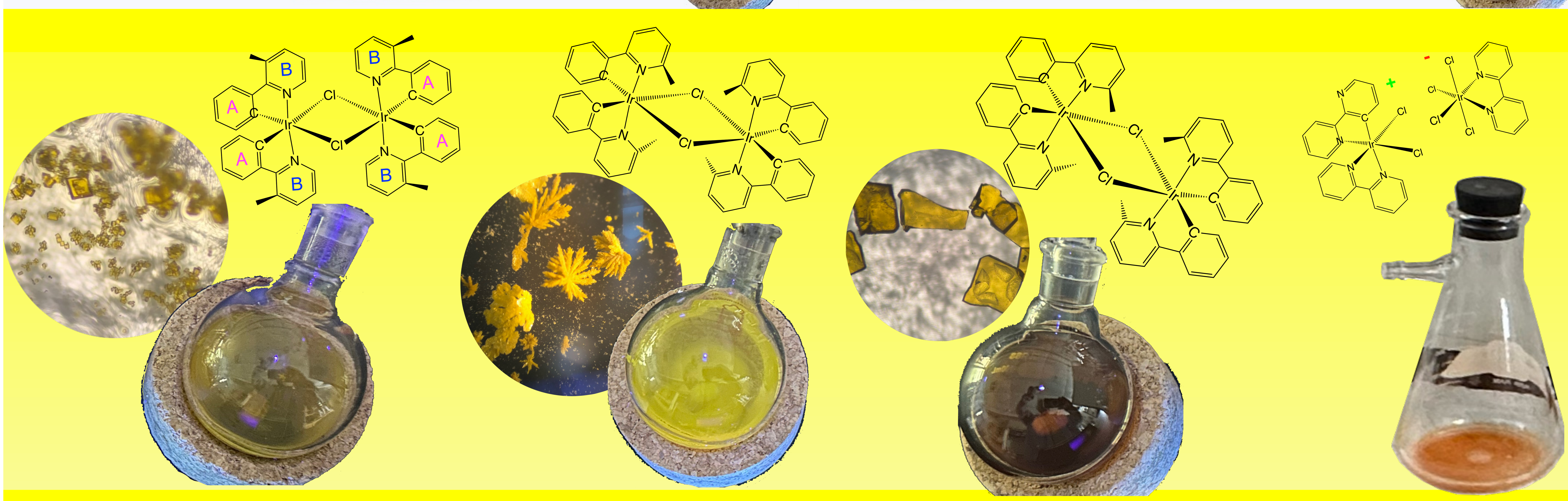
Iridium 6-methyl-2-phenylpyridine derivatives: Synthesis



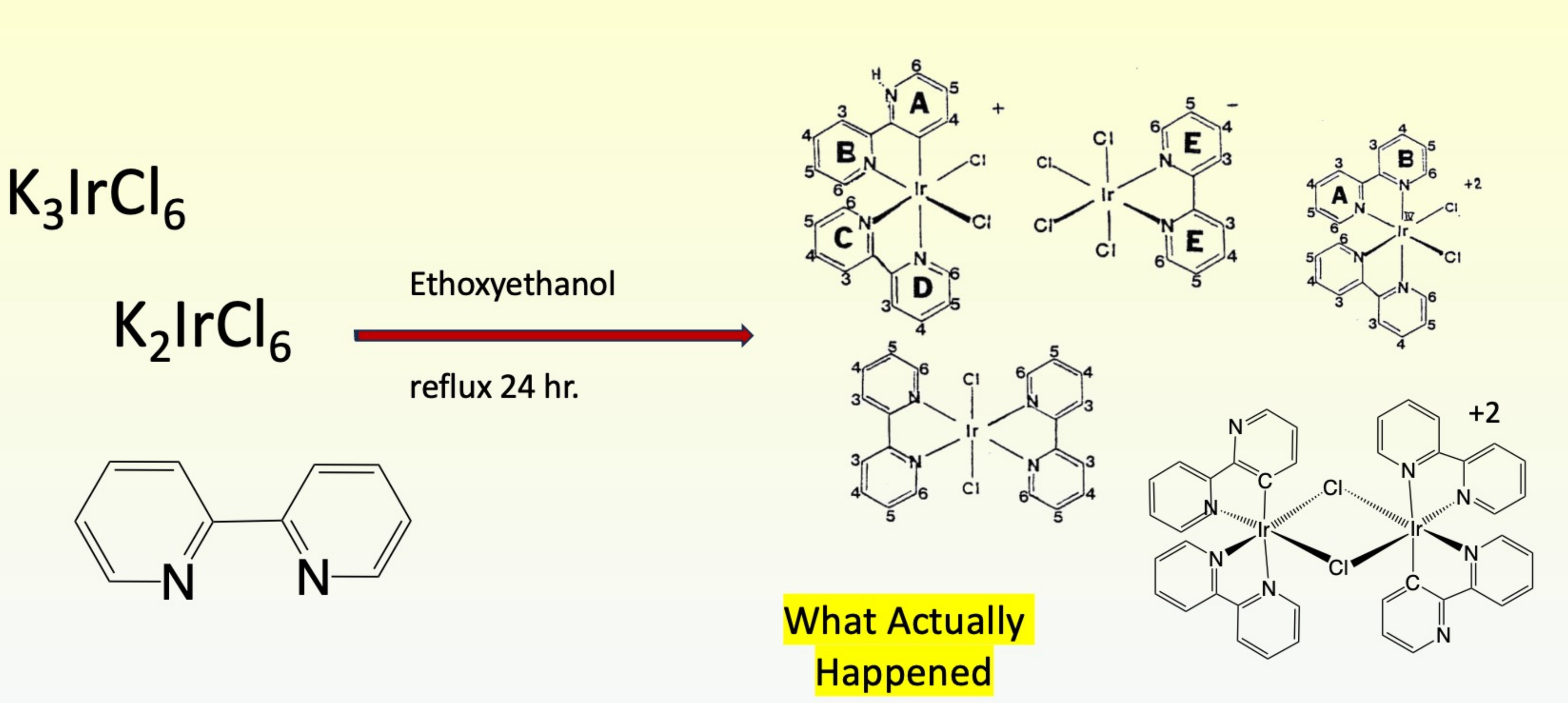
[Ir(6-methyl-2-phenylpyridine)$_2$Cl]$_2$

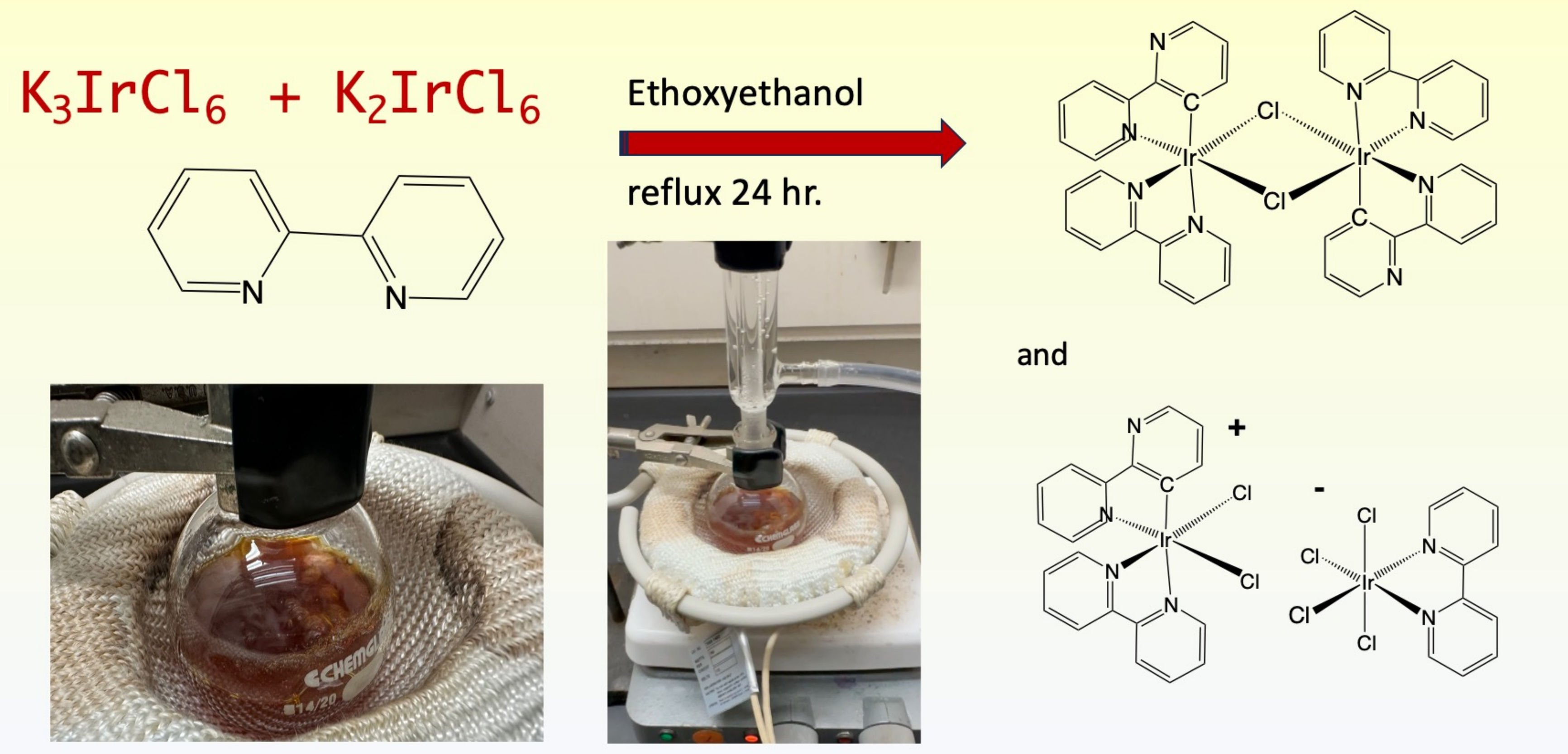
Iridium 6-methyl-2-phenylpyridine derivatives: Synthesis





Synthetic Scheme for the roll-over Iridium2,2'-bipyridine Complex, [Ir(bpy-C^3,N')(bpy-N,N')Cl]$_2$



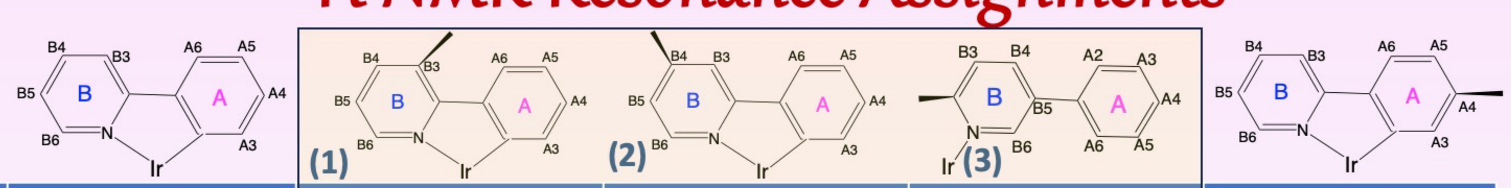


^1H & ^{13}C NMR Analysis

1H and 13C NMR characterization was performed for all synthesized complexes in DCM using the Anasazi EFT 90 MHz NMR Spectrometer. To assign resonances for the metal complexes presented in the table, COSY (Correlation Spectroscopy) and HETCOR (Heteronuclear Correlation) 2D pulse sequences were employed. MNOVA (MestReNova) software was utilized for processing both 1D and 2D NMR free induction decays (FIDs). This software, known for its user-friendly interface and automated data analysis, provided students with an accessible platform for spectral interpretation. In particular, MNOVA simplifies COSY and HETCOR workup by offering intuitive visualization tools, peak picking, and automated cross-peak assignments, making complex 2D spectral analysis more manageable for students.

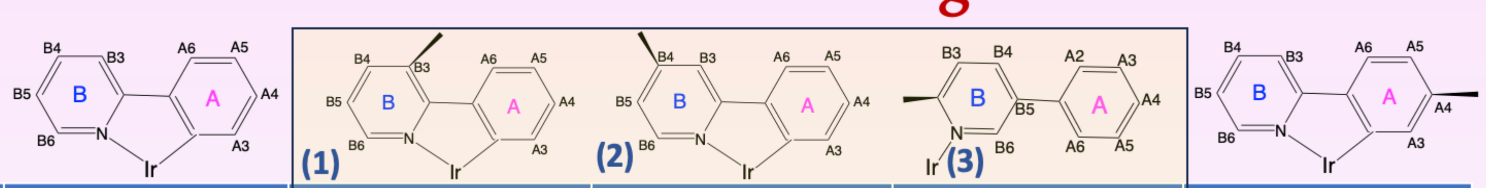
Miramar College provides access to MNOVA in its computer lab, allowing students to independently process and analyze their NMR data. Through hands-on experience, students successfully learned to navigate 2D NMR processing, enhancing their understanding of spectral correlation techniques. The spectra below showcase the students' MNOVA-processed results.

^1H NMR Resonance Assignments



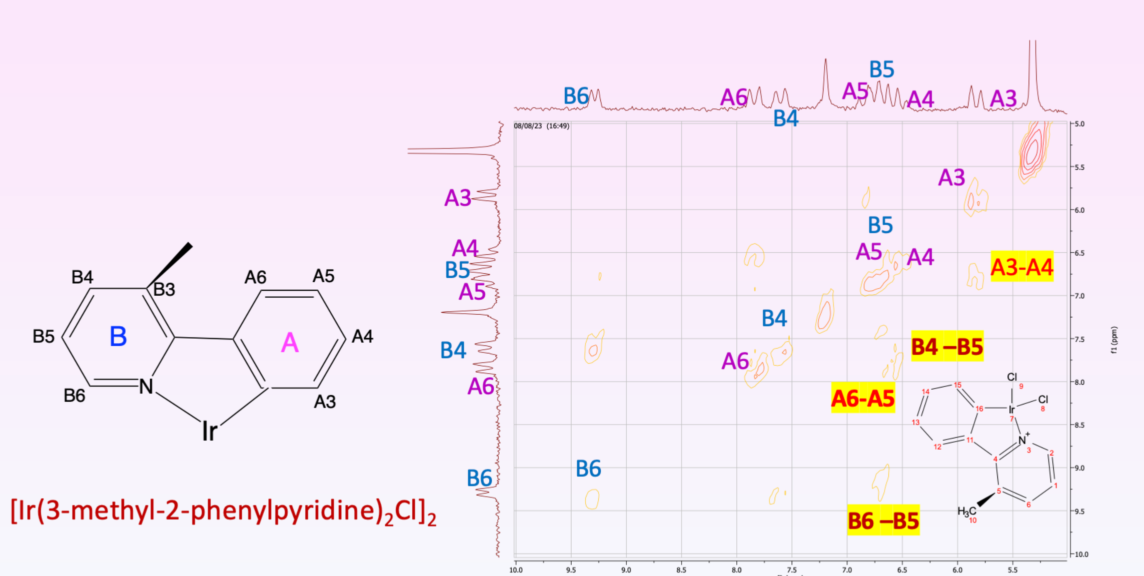
Resonance	2-phenylpyridine	3-me-2-phpyridine	4-me-2-phpyridine	5-me-2-phpyridine	2-(p-tolyl)pyridine
	Ligand / Complex	Ligand / Complex	Ligand / Complex	Ligand / Complex	Ligand / Complex
A1	-		-		
A2	8.20	7.65	8.088	7.8 / 7.508	8.09 / 5.68
A3	7.59 / 5.89	7.54 / 5.84	7.271 / 5.89	6.9 / 7.406	7.39 / 5.68
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

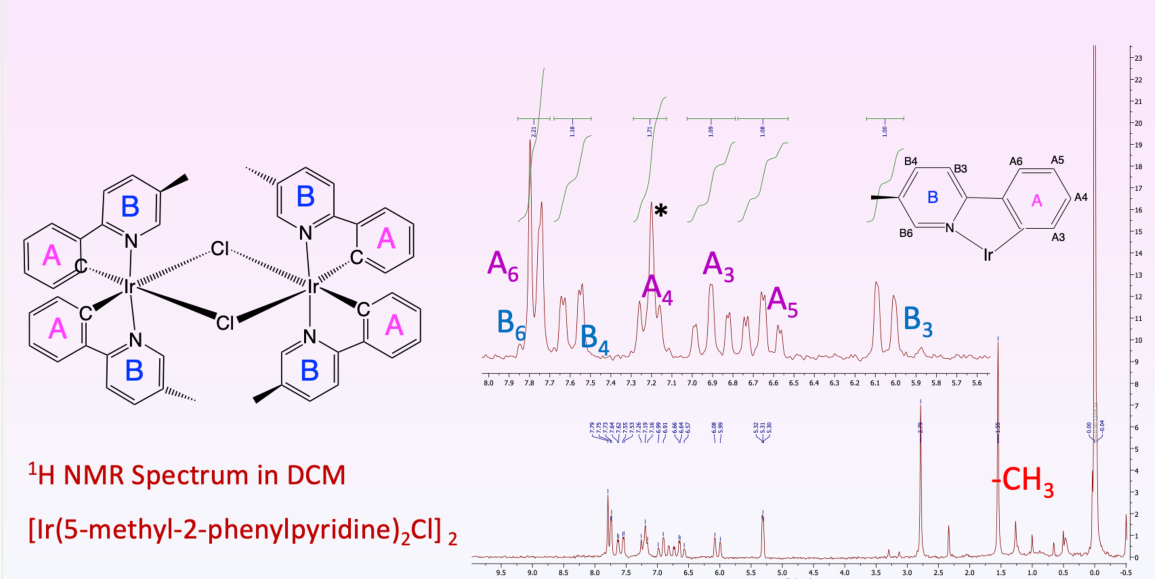


Resonance	2-phenylpyridine	3-me-2-phpyridine	4-me-2-phpyridine	5-me-2-phpyridine	2-(p-tolyl)pyridine
	Ligand / Complex	Ligand / Complex	Ligand / Complex	Ligand / Complex	Ligand / Complex
A1	139.6 / 144.8	141.2 / 147.4	138.0 / -	137.4 / 137.13	139.4 / 145.4
A2	127.1 / 144.0	129.4 / 146.6	128.3 / -	127.7 / 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

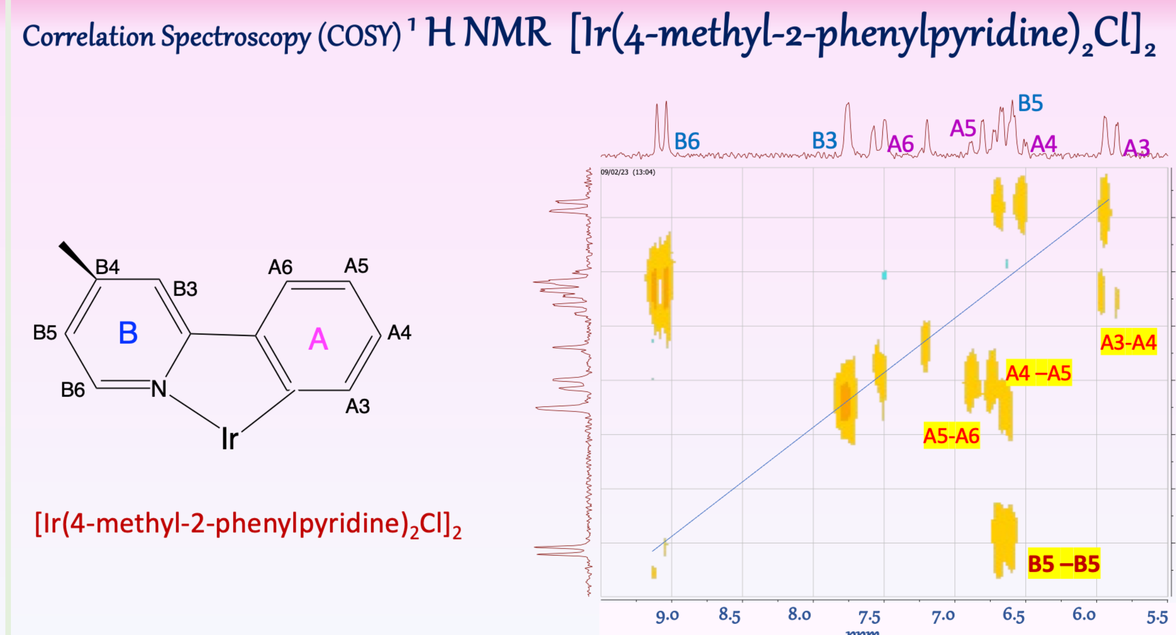
Correlation Spectroscopy (COSY) ^1H NMR $[Ir(3\text{-methyl-2-phenylpyridine})_2Cl]_2$



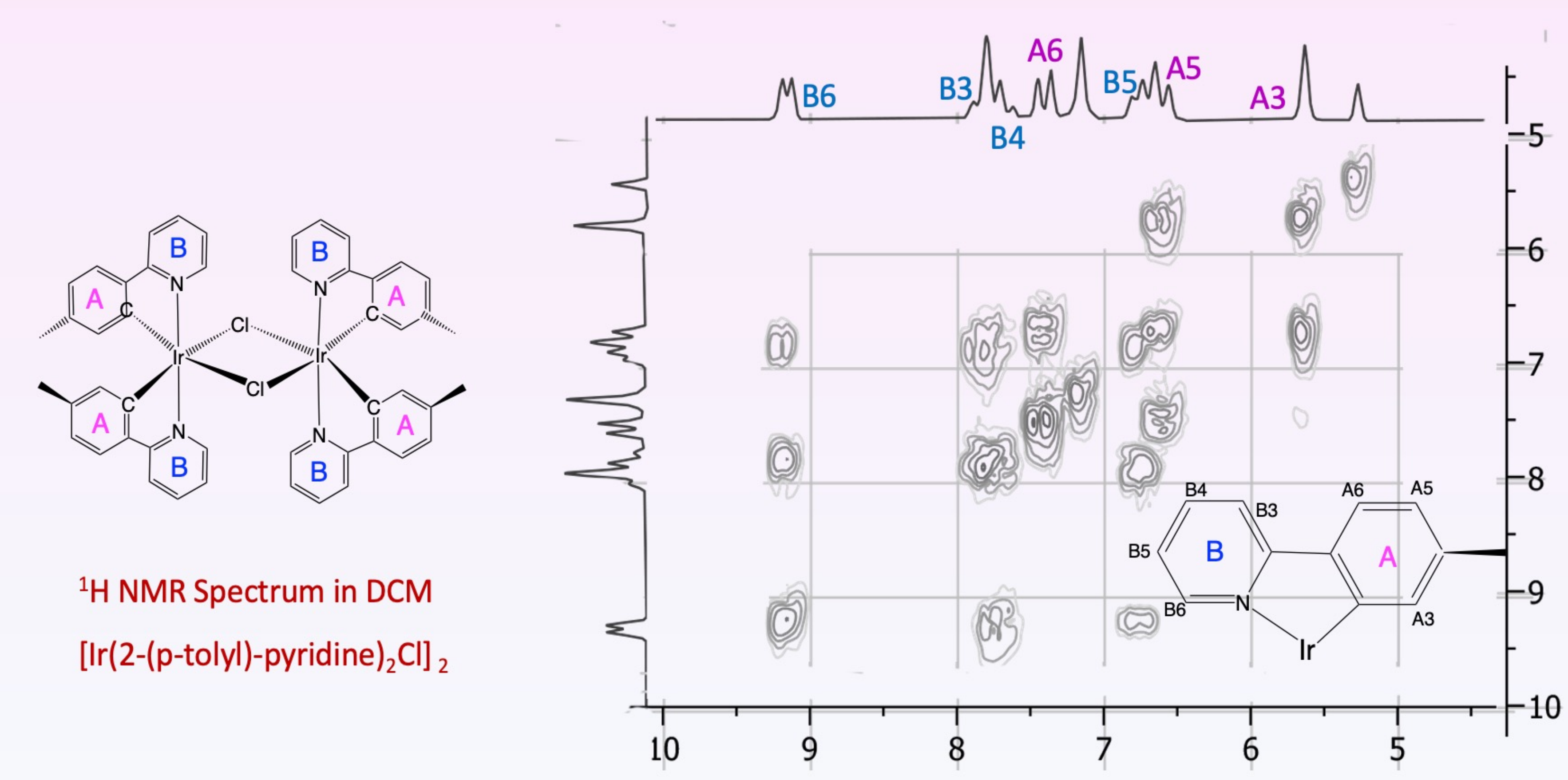
^1H NMR Spectrum for $[Ir(5\text{-methyl-2-phenylpyridine})_2Cl]_2$



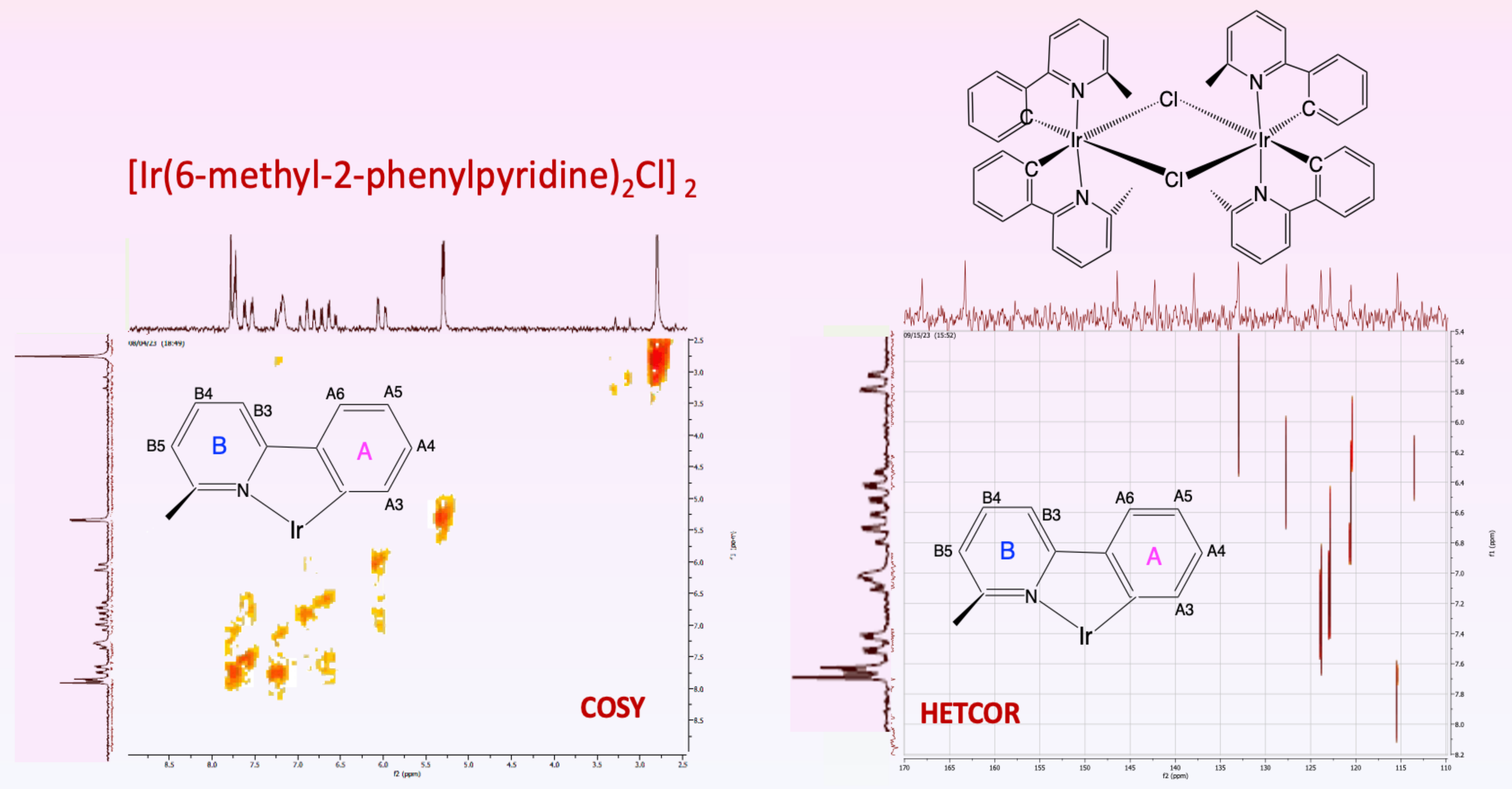
Correlation Spectroscopy (COSY) ^1H NMR $[Ir(4\text{-methyl-2-phenylpyridine})_2Cl]_2$



^1H-COSY Spectrum: $[Ir(2\text{-(p-tolyl)-phenylpyridine})_2Cl]_2$

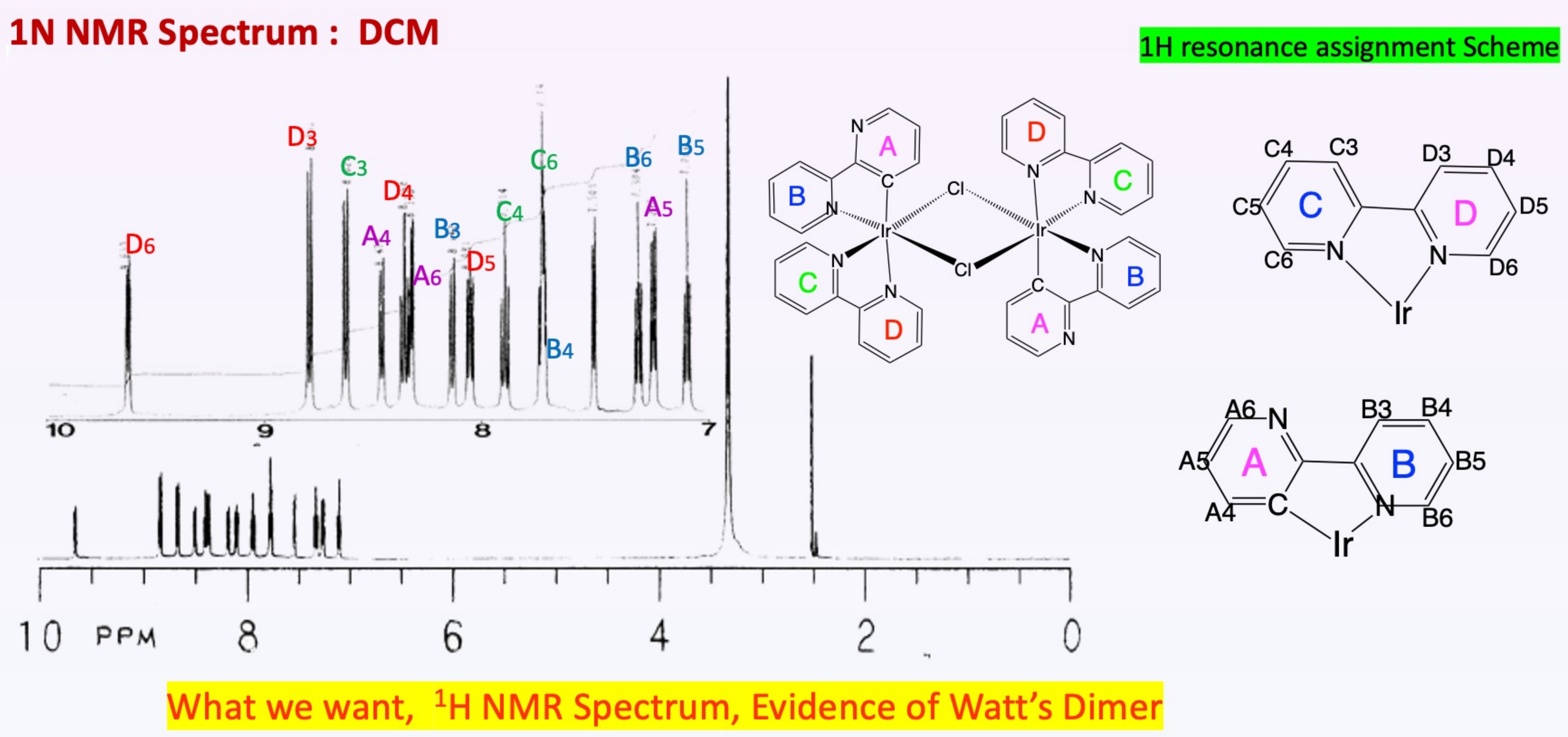


^1H-COSY & ^1H-^{13}C HETCOR Spectrum $[Ir(6\text{-methyl-2-phenylpyridine})_2Cl]_2$



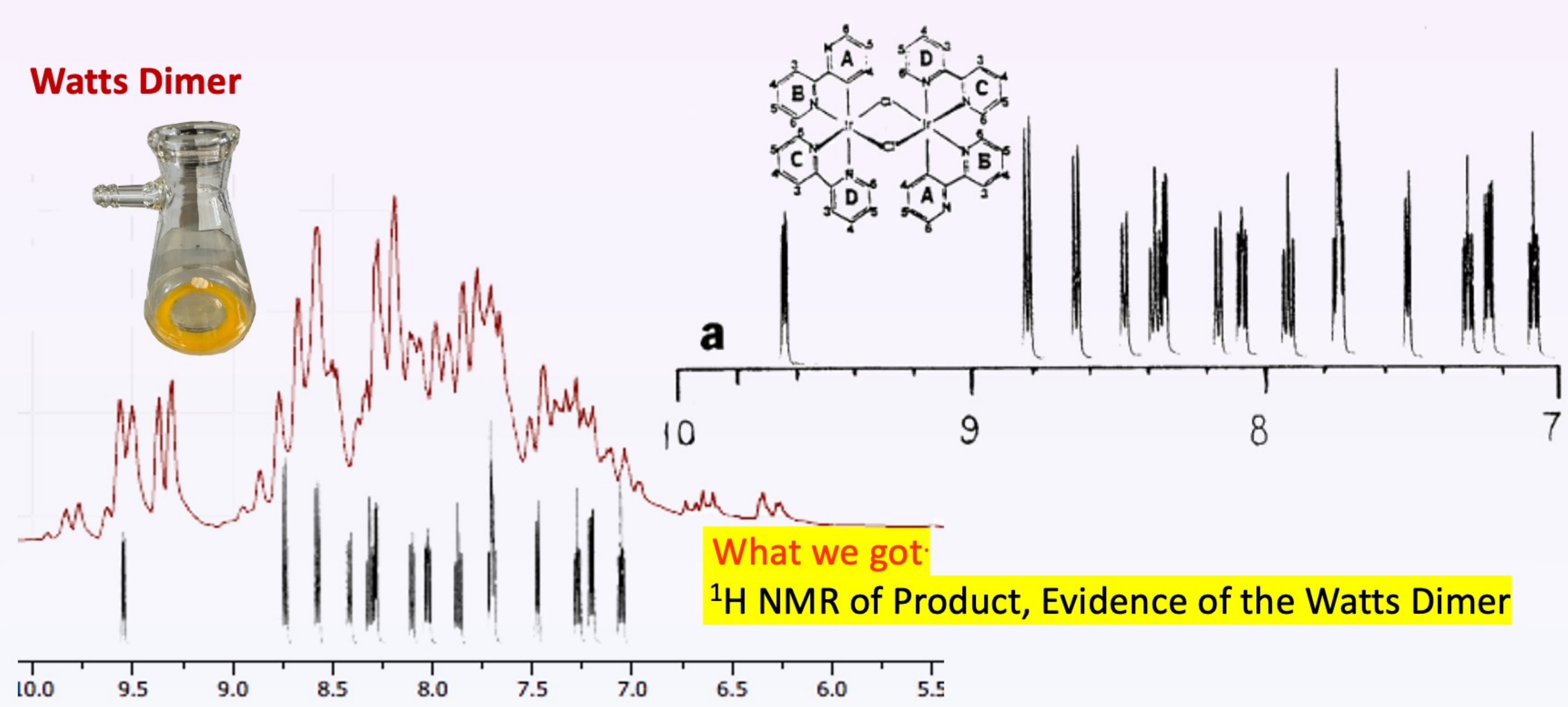
^1H NMR Analysis of the Roll-Over Isomer $[Ir(bpy\text{-}C3,N')(bpy\text{-}N,N')Cl]_2$ The 1H NMR spectrum of the synthesized roll-over isomer, $[Ir(bpy\text{-}C3,N')(bpy\text{-}N,N')Cl]_2$, was obtained to confirm its structural identity. The NMR spectra on the left represent data from the original complex synthesized in 1990 (https://fogarces.com/fgarcesinfo/fogdoc/05_irbpy/5irbpy.htm), serving as a historical benchmark for comparison. The NMR spectrum on the right corresponds to the attempted synthesis of the same complex in the present study. Key spectral differences between the two samples may provide insight into variations in ligand coordination, reaction efficiency, or purity of the final product.

Ideal ^1H NMR Spectrum: $[Ir(bpy\text{-}C^3,N')(bpy\text{-}N,N')_2]^{2+}$



What we want, ^1H NMR Spectrum, Evidence of Watt's Dimer

1H NMR Roll-over Dimer: $[Ir(bpy\text{-}C^3,N')(bpy\text{-}N,N')_2]^{2+}$



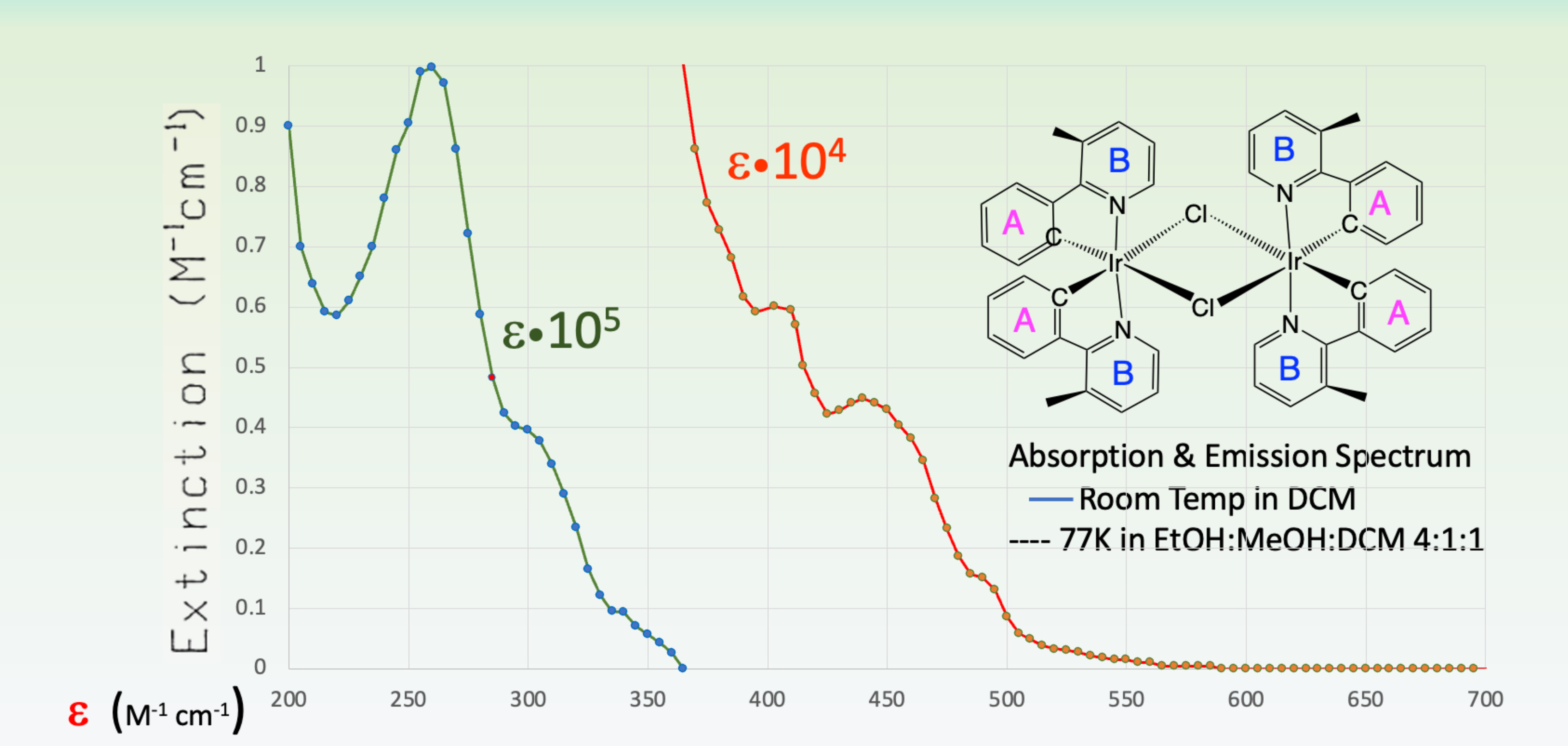
What we got ^1H NMR of Product, Evidence of the Watts Dimer

Photophysical properties and Crystal Structures

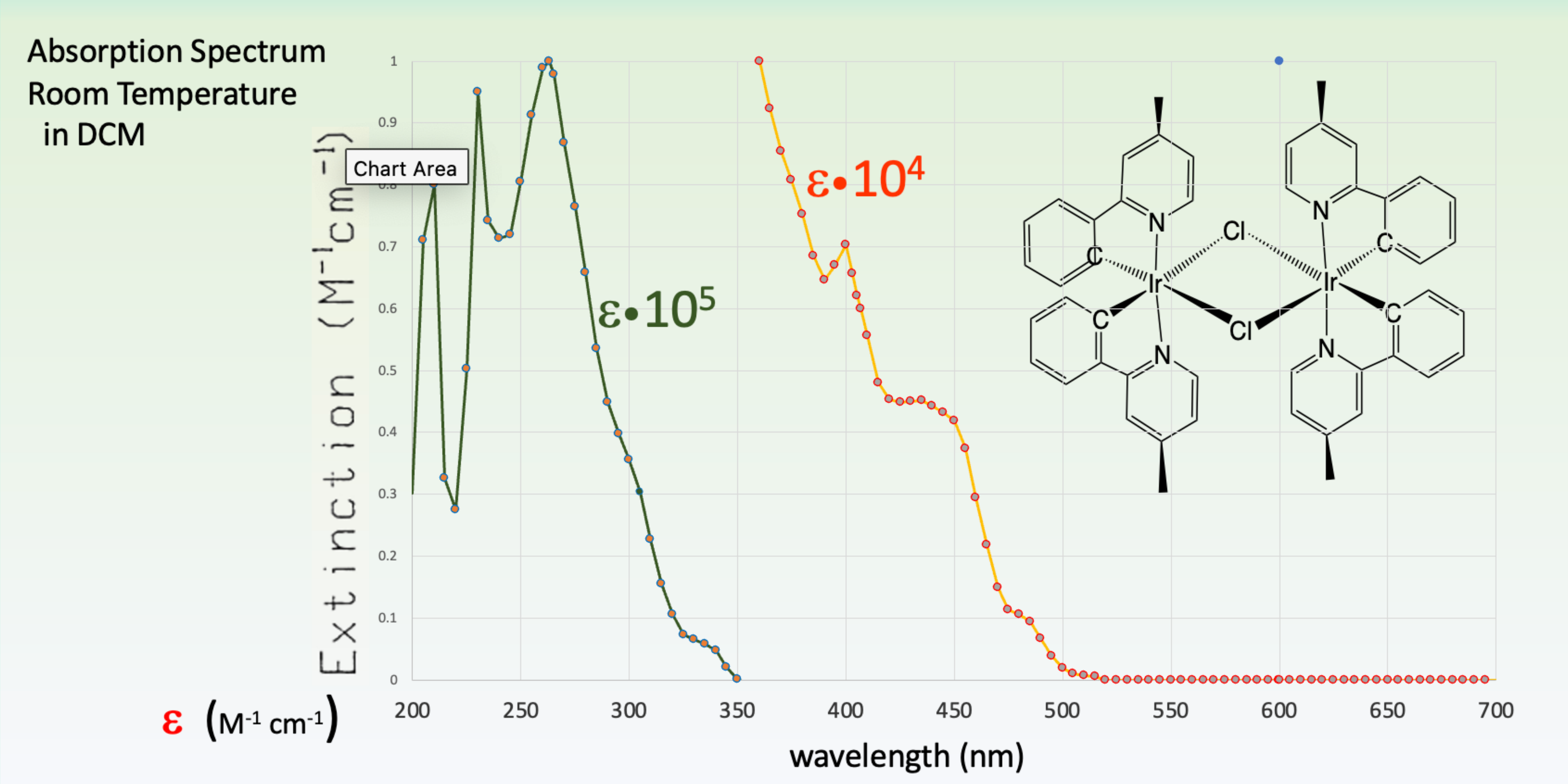
Miramar College undergraduate researchers successfully synthesized and characterized a series of iridium(III) ortho-metallated complex dimers. The project provided community college students with hands-on experience in organometallic synthesis and advanced characterization techniques.

Following the successful synthesis of the complexes, the students conducted UV-Vis absorption spectroscopy to investigate their photophysical properties, including the calculation of extinction coefficients using Beer's law analysis. Luminescence properties were also measured, and cyclic voltammetry was performed on selected complexes to explore their redox behavior. Beyond spectroscopic characterization, the study extended to structural analysis. Crystals obtained from three of the synthesized complexes were successfully analyzed using X-ray crystallography, confirming their molecular structures and coordination environments.

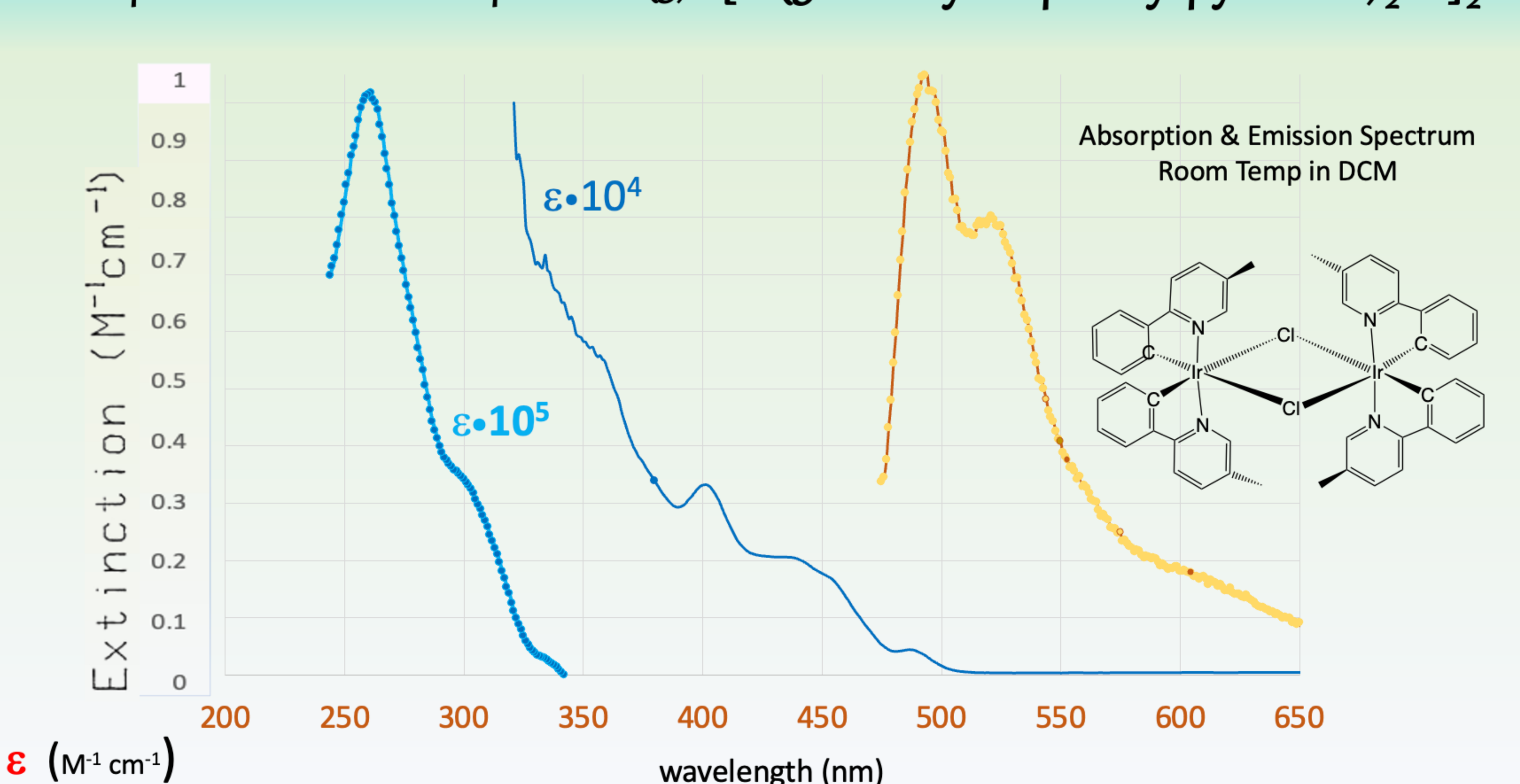
Absorption and Emission Spectrum (1): $[Ir(3\text{-methyl-2-phenylpyridine})_2Cl]_2$



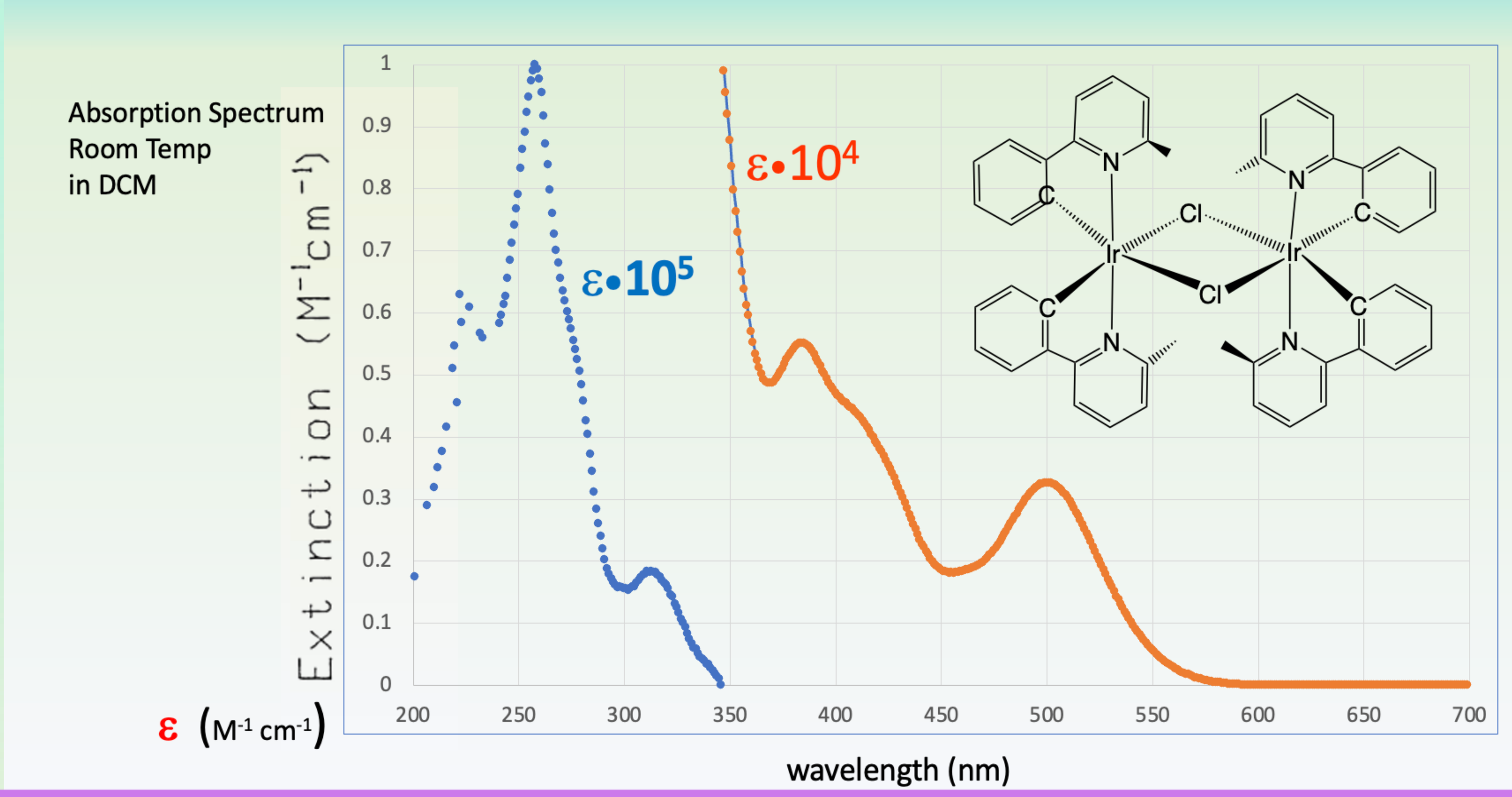
Absorption and Emission Spectrum (2): $[Ir(4\text{-methyl-2-phenylpyridine})_2Cl]_2$



Absorption and Emission Spectrum (3): $[Ir(5\text{-methyl-2-phenylpyridine})_2Cl]_2$



Absorption and Emission Spectrum (4): $[Ir(6\text{-methyl-2-phenylpyridine})_2Cl]_2$



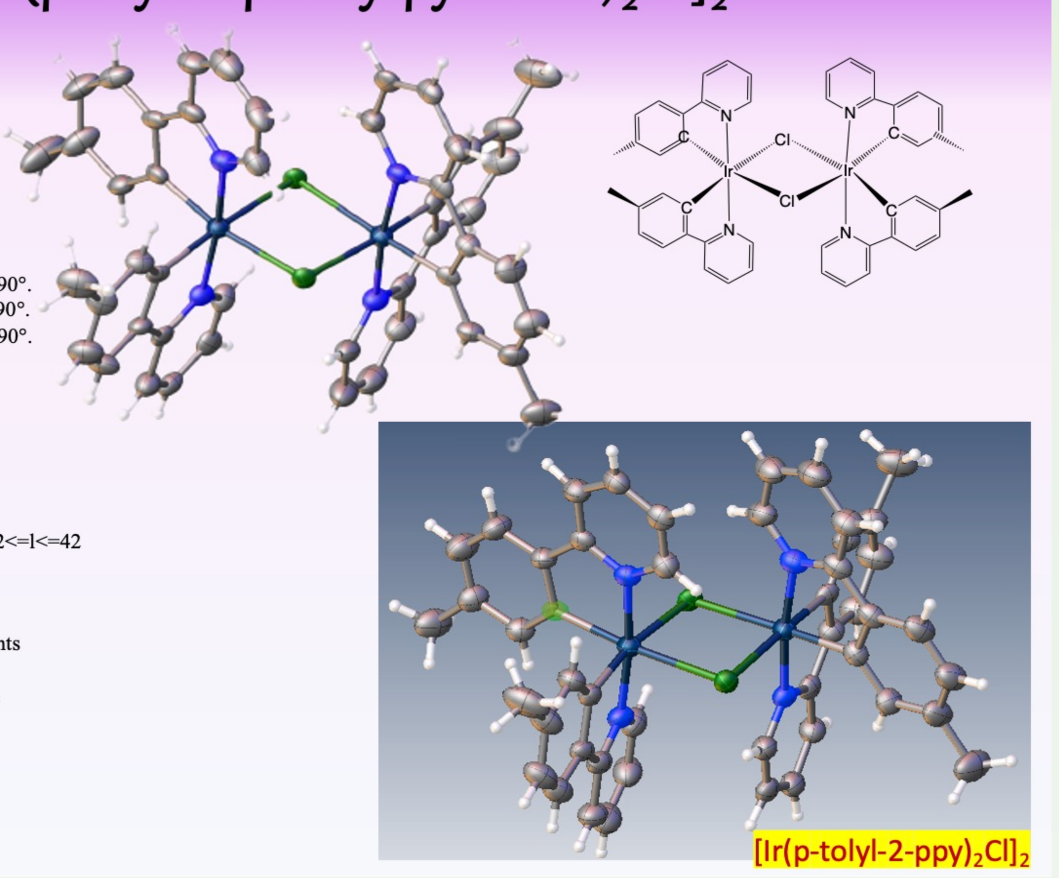
In collaboration with the UCSD X-ray crystallography Facility and with the assistance of Dr. Milan Gembicky, crystal structures were successfully obtained for several of the synthesized iridium(III) complexes. The high-quality diffraction data allowed for precise structural determination, providing critical insights into coordination environments, bond lengths, and overall molecular geometry.

The R-indices for all datasets were approximately 0.035 or lower, indicating high structural quality. The R-factor, a measure of how well the calculated model fits the experimental X-ray diffraction data, ranges from 0 (a perfect fit) to approximately 0.6 (poor structural agreement). An R-factor below 0.05 is generally considered excellent, further confirming the accuracy and reliability of the crystallographic data obtained in this study.

X-Ray Crystal Structure: $[Ir(p\text{-toyl-2-phenylpyridine})_2Cl]_2$

Table 1. Crystal data and structure refinement for Miramar06B.

Identification code	Miramar06B	
Empirical formula	C55 H48 Cl2 Ir2 N4	
Formula weight	1220.358	
Temperature	100.00 K	
Wavelength	0.71073 Å	
Crystal system	Tetragonal	
Space group	$I4_1cd$	
Unit cell dimensions	a = 16.5896(4) Å	α= 90°.
	b = 16.5896(4) Å	β= 90°.
	c = 32.5983(13) Å	γ = 90°.
Volume	8971.5(5) $Å^3$	
Z	8	
Density (calculated)	1.807 Mg/m^3	
Absorption coefficient	6.090 mm^{-1}	
F(000)	4741.967	
Crystal size	0.08 x 0.08 x 0.055 mm^3	
Theta range for data collection	2.14 to 27.91°.	
Index ranges	-21<=h<=21, -21<=k<=21, -42<=l<=42	
Reflections collected	79913	
Independent reflections	5357 [R(int) = 0.0582]	
Completeness to theta = 25.2417°	99.90 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.0473 and 0.0202	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5357 / 292 / 257	
Goodness-of-fit on F^2	1.0384	
Final R indices [I>2sigma(I)]	R1 = 0.0289, wR2 = 0.0708	
R indices (all data)	R1 = 0.0302, wR2 = 0.0717	
Absolute structure parameter	0.012(4)	
Largest diff. peak and hole	0.7436 and -1.1210 e.$Å^{-3}$	

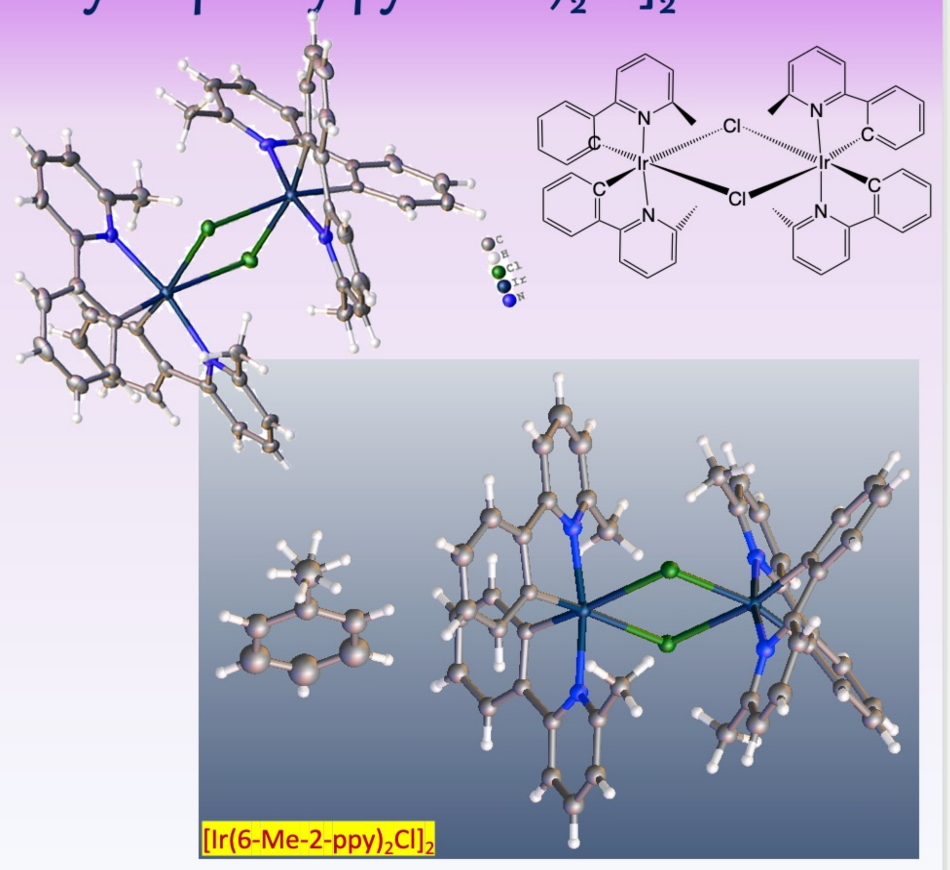


$[Ir(p\text{-tolyl-2-ppy})_2Cl]_2$

X-Ray Crystal Structure: $[Ir(6\text{-methyl-2-phenylpyridine})_2Cl]_2$

Table 1. Crystal data and structure refinement for Miramar05.

Identification code	miramar05	
Empirical formula	C55 H48 Cl2 Ir2 N4	
Formula weight	1220.27	
Temperature	100.00 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C 1 2/c 1	
Unit cell dimensions	a = 15.3143(10) Å	α= 90°.
	b = 32.122(2) Å	β= 130.236(2)°.
	c = 11.6345(8) Å	γ = 90°.
Volume	4369.2(5) $Å^3$	
Z	4	
Density (calculated)	1.855 Mg/m^3	
Absorption coefficient	6.253 mm^{-1}	
F(000)	2376	
Crystal size	0.13 x 0.11 x 0.045 mm^3	
Theta range for data collection	1.268 to 26.795°.	
Index ranges	-19<=h<=14, 0<=k<=40, 0<=l<=14	
Reflections collected	42689	
Independent reflections	4664 [R(int) = 0.0563]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.452 and 0.2107	
Refinement method	Full-matrix least-squares on F^2	
Data/restraints / parameters	4664 / 0 / 290	
Goodness-of-fit on F^2	1.052	
Final R indices [I>2sigma(I)]	R1 = 0.0281, wR2 = 0.0730	
R indices (all data)	R1 = 0.0358, wR2 = 0.0770	
Largest diff. peak and hole	1.412 and -1.397 e.$Å^{-3}$	



$[Ir(6\text{-Me-2-ppy})_2Cl]_2$

Conclusion

This research was conducted over two consecutive 12-week summer projects at Miramar College, focusing on the synthesis and characterization of iridium(III) and nickel(II) metal complexes. Students successfully synthesized four distinct iridium orthometallated complexes using methylated 2-phenylpyridine ligands, achieving yields of 30% to 60%. These reactions were monitored by color changes and purified through recrystallization and filtration. Structural characterization was performed using ^1H and ^{13}C NMR spectroscopy, with some complexes further analyzed by COSY and HETCOR techniques. Three of the complexes crystallized successfully for X-ray crystallographic analysis.

In addition to synthesis, students investigated the photophysical properties of the iridium complexes using UV-Vis spectroscopy, with most compounds exhibiting luminescence. They also explored nickel(II) tris(bipyridine) complexes and identified a novel iridium coordination isomer.

Through these two summer projects, students gained hands-on experience in organometallic research, developing advanced laboratory skills and demonstrating their ability to contribute to scientific exploration.

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We sincerely thank the individuals and organizations whose support and contributions made our summer research project on synthesizing iridium complexes possible.

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