Iridium Rollover isomer of bipyridine Complexes

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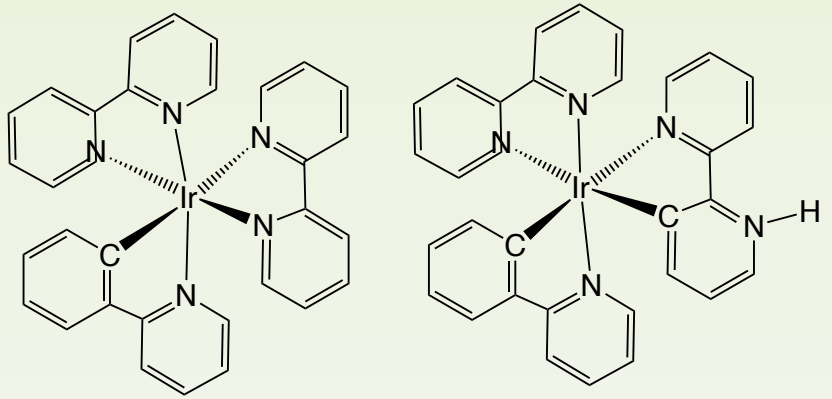
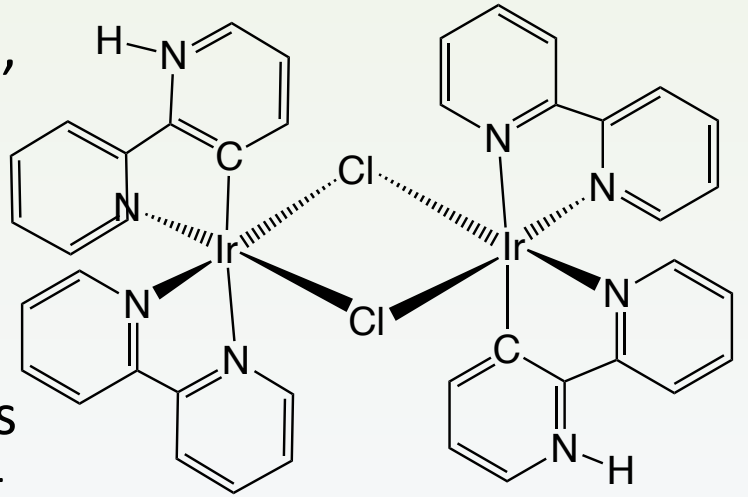
Summer Research

Area of Research:

Rollover cyclometallation of bipyridine to iridium is a rare occurrence, with the first reported instance dating back to the 1970s. Extensive debates regarding its structure ensued until Watts et al. elucidated it through NMR and X-ray crystallography. They confirmed that one of the three 2,2-bipyridine ligands coordinates to iridium(III) in a manner where iridium bonds to the carbon atom.

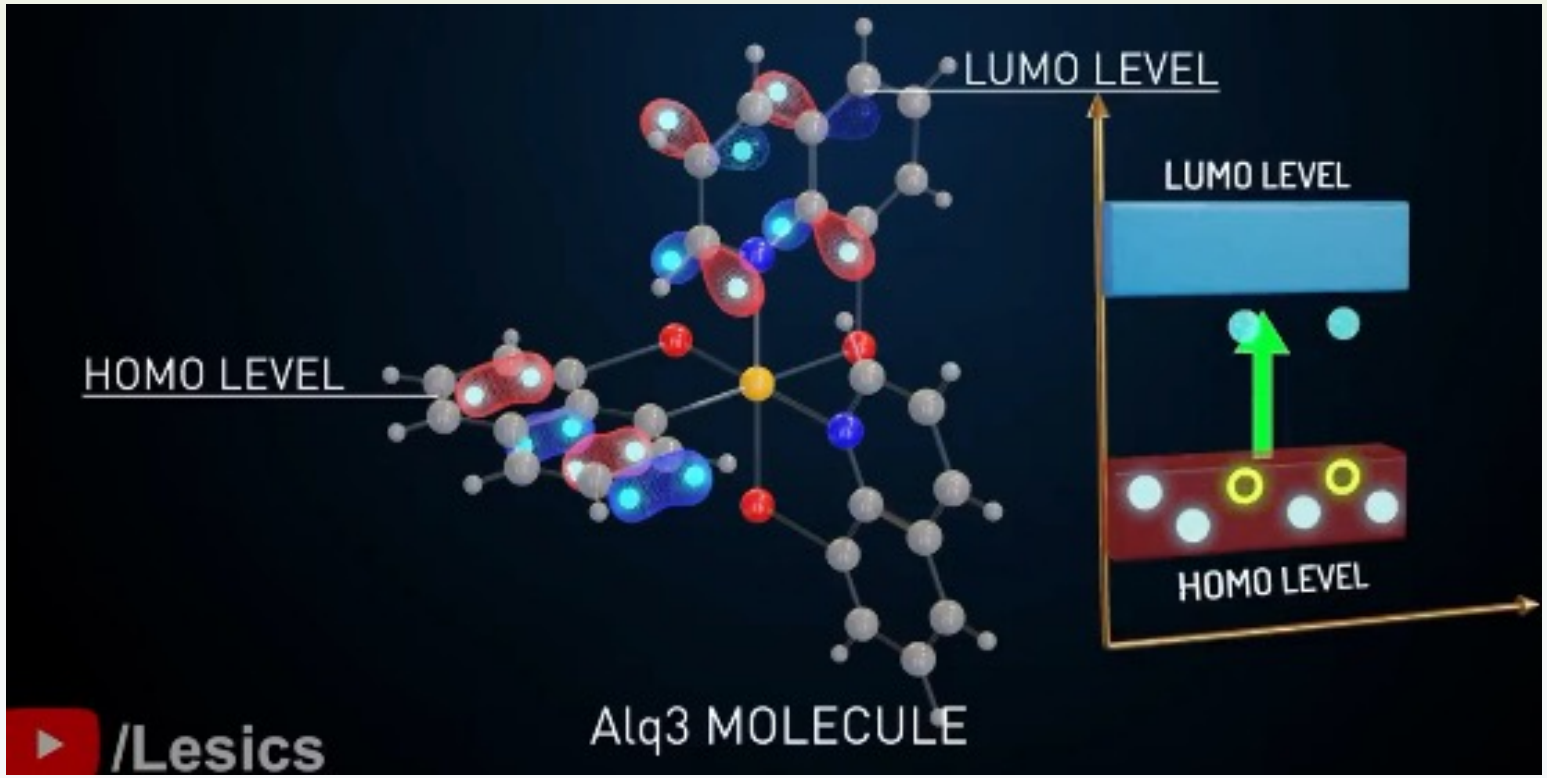


A dimeric relative of the Watt's Complex, [Ir(Hppy-C3,N')(bpy-N,N')Cl_2], can be synthesized using Ir+3 and Ir+4. However, this synthesis is challenging due to the formation of numerous byproducts. In the summer of 2023, we attempted to synthesize a rollover cyclometallation dimer, resulting in the production of several byproducts. This poster documents the synthesis attempts, the various products obtained, and the characterization of these byproducts using NMR spectroscopy.



Applications: OLED

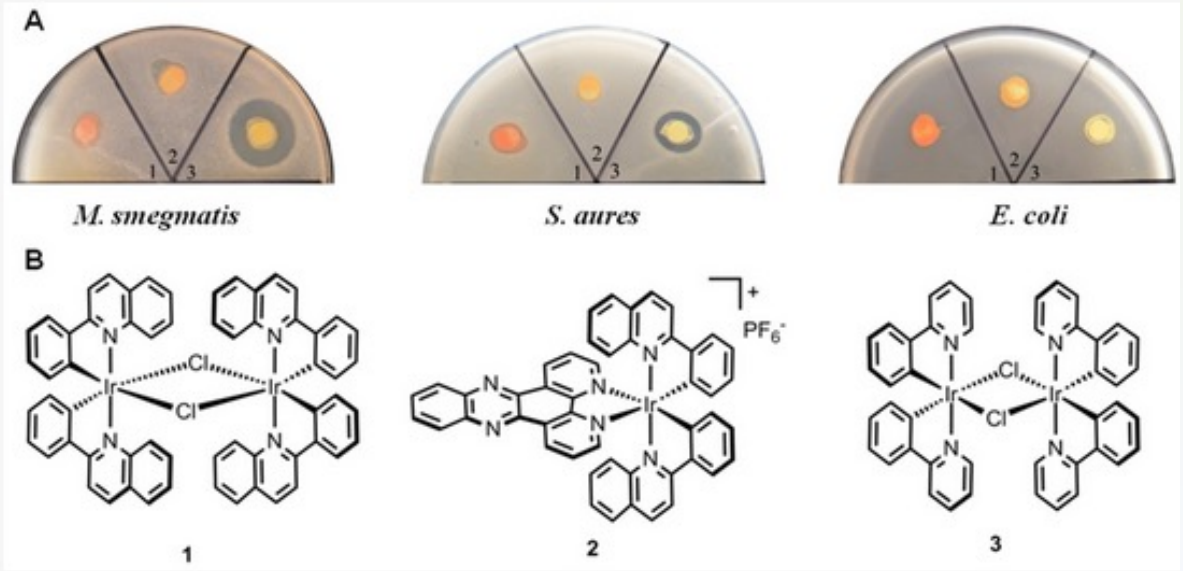
OLED –Organic Light Emitting Diodes is a display technology that uses organic compounds, i.e., $Ir(mppy)_3$ to emit light when an electric current passes through them. It offers vibrant colors, high contrast, and flexible form factors, but may be prone to burn-in and higher production costs compared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



Anti Cancer Bacterial Agent

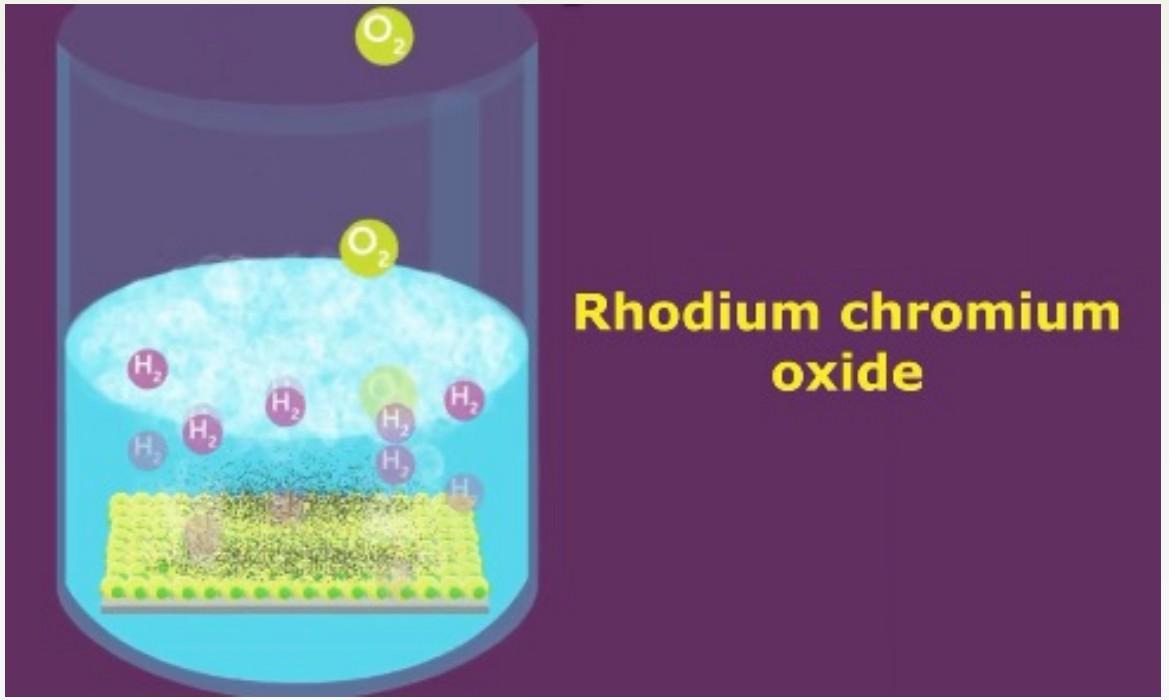
Anti Cancer (Bacterial) Agents –Iridium-based compounds have shown promise as anticancer agents, exhibiting potential for targeted cancer therapy. Additionally, iridium complexes have demonstrated antibacterial activity against drug-resistant bacteria, suggesting their potential application in combating 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**.



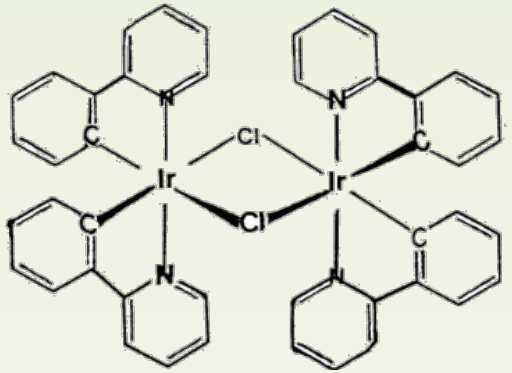
Photocatalysts

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.

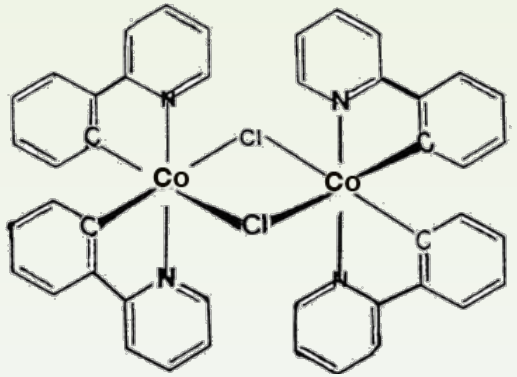


Area of Research, Summer 2023

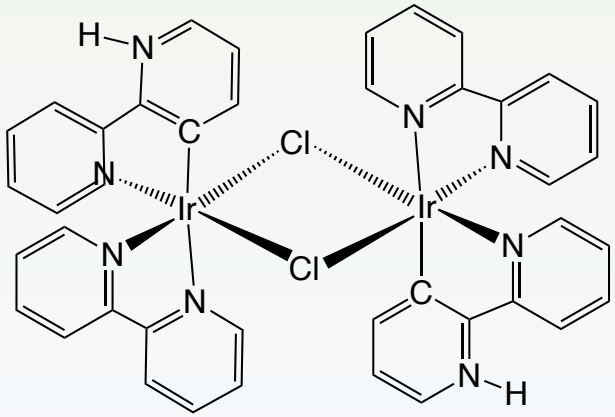
1. **Iridium 2-phenylpyridine derivatives**



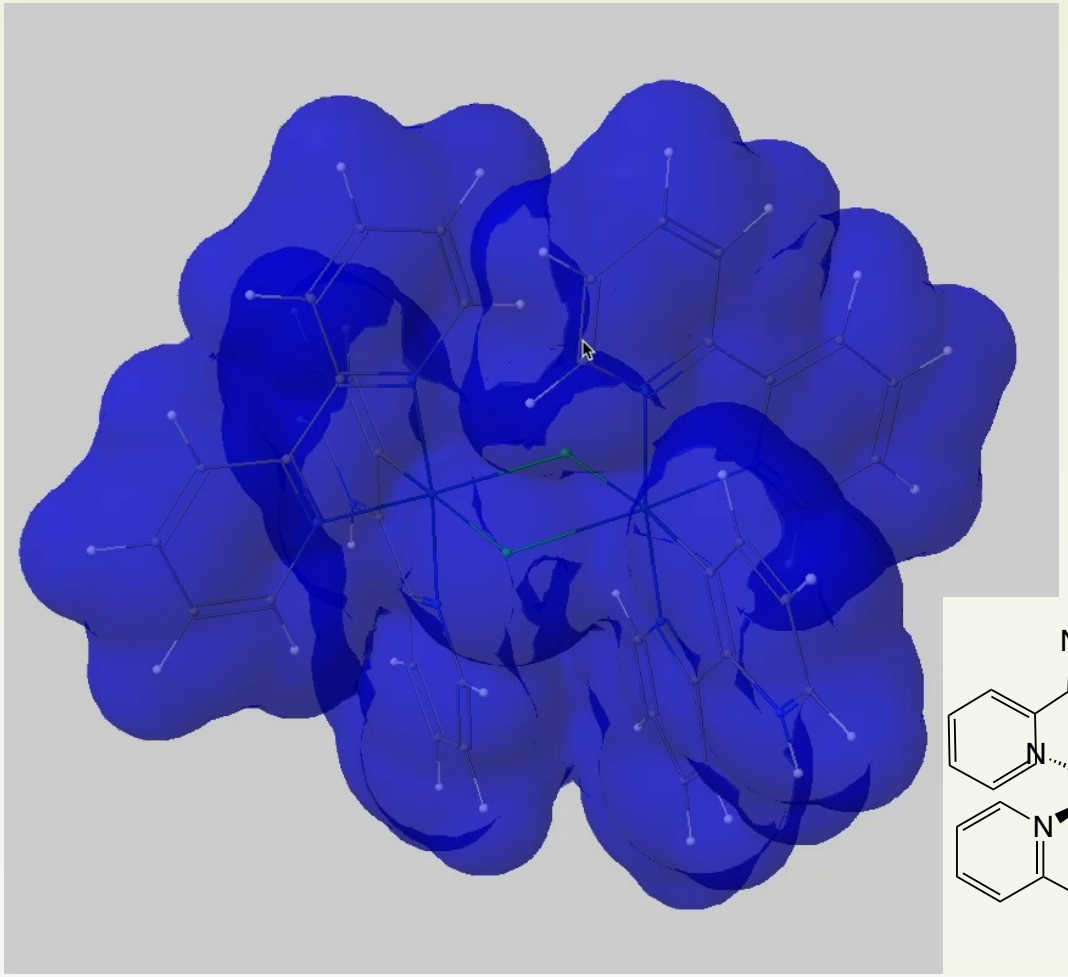
~~2. **Cobalt 2-phenylpyridine derivatives**~~

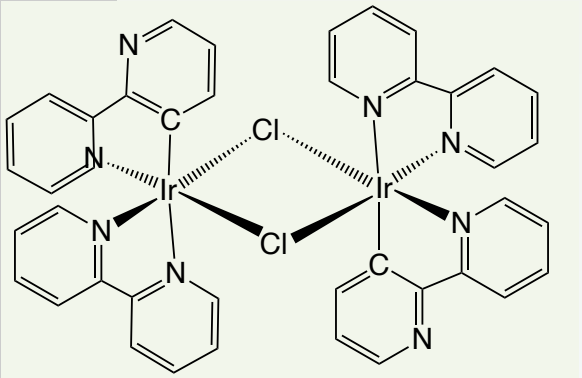


2. **Iridium C-bonded Bipyridine (Watts Dimer)**



MolView





Mol View Link

May not work with smartphone

Synthesis of $[Ir(bpy\text{-}C^3,N')(bpy\text{-}N,N')_2]^{2+}$

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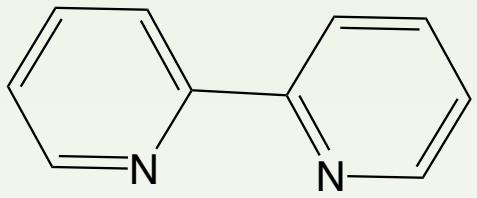
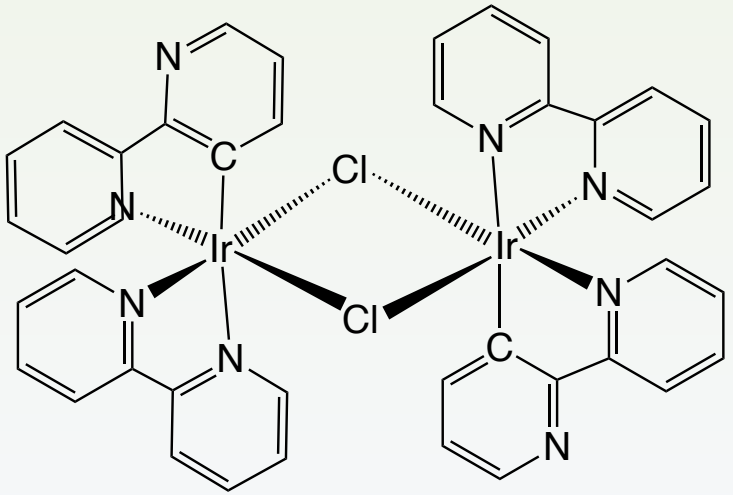
$[Ir(bpy\text{-}C^3,N')(bpy\text{-}N,N')_2]^{2+}$: Synthesis

$$K_3IrCl_6 \quad + \quad K_2IrCl_6$$

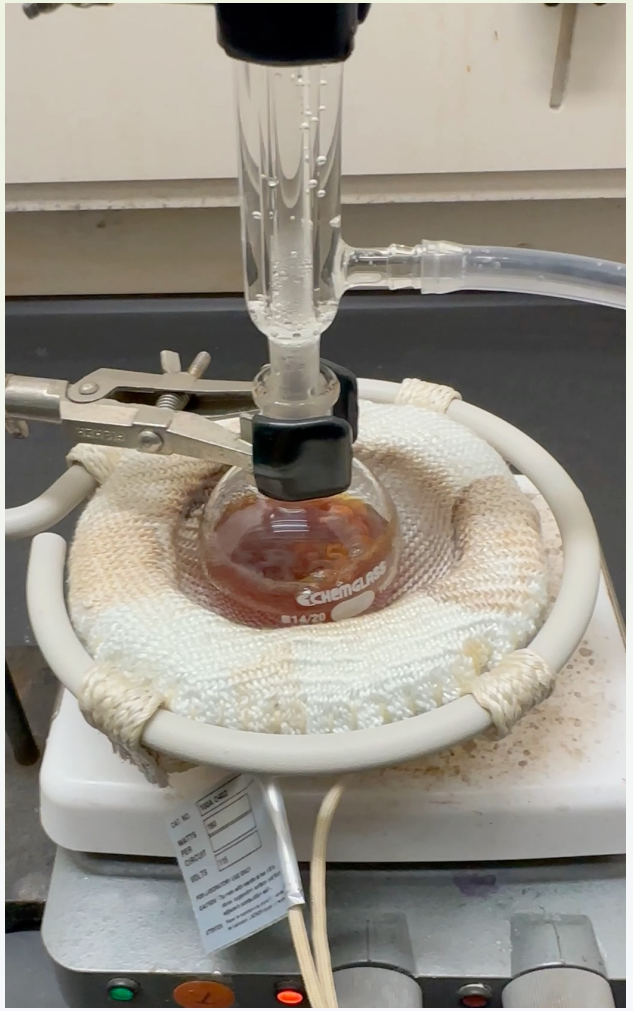
Ethoxyethanol

reflux 24 hr.





Target Molecule



Mix Product of Iridium bipyridine Complexes

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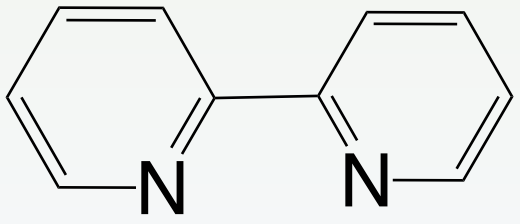
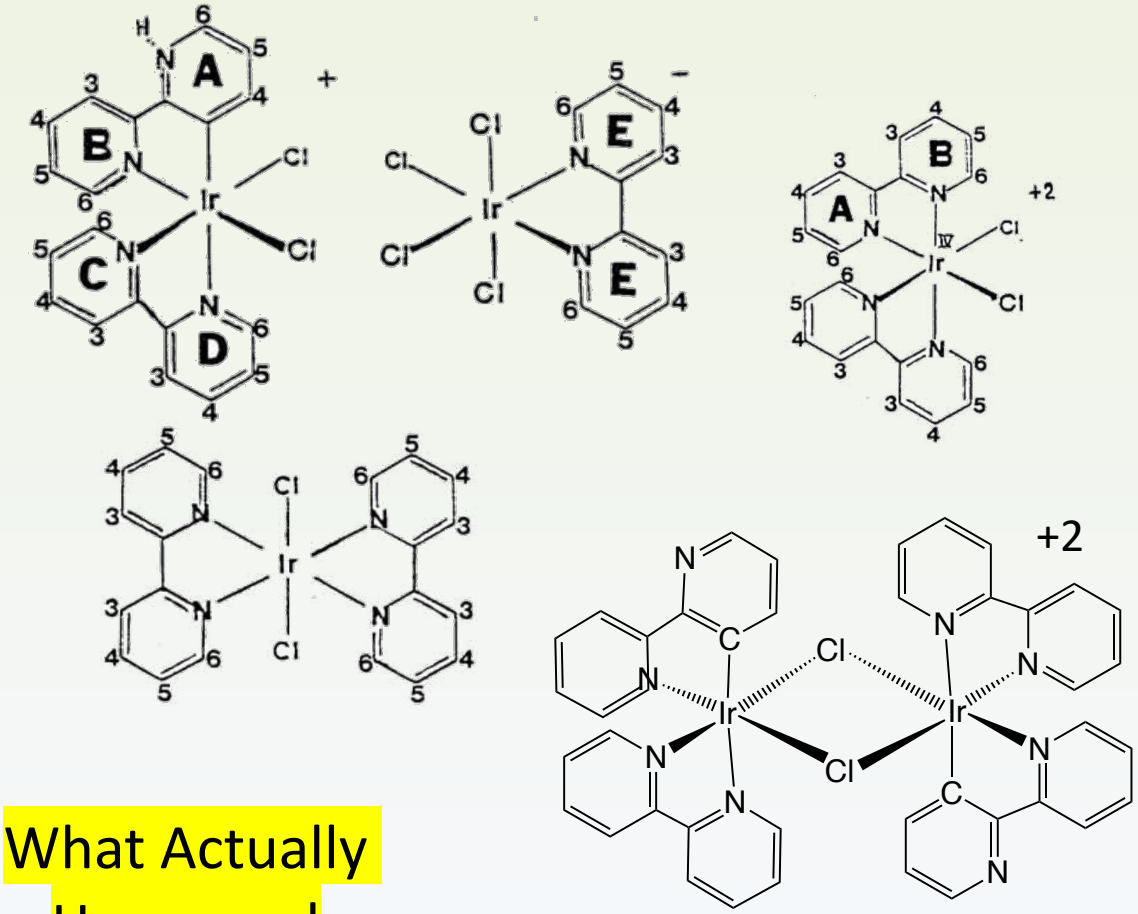
Iridium C-bonded Bipyridine (Watts Dimer)

K_3IrCl_6

K_2IrCl_6 → Ethoxyethanol, reflux 24 hr.





What Actually Happened

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

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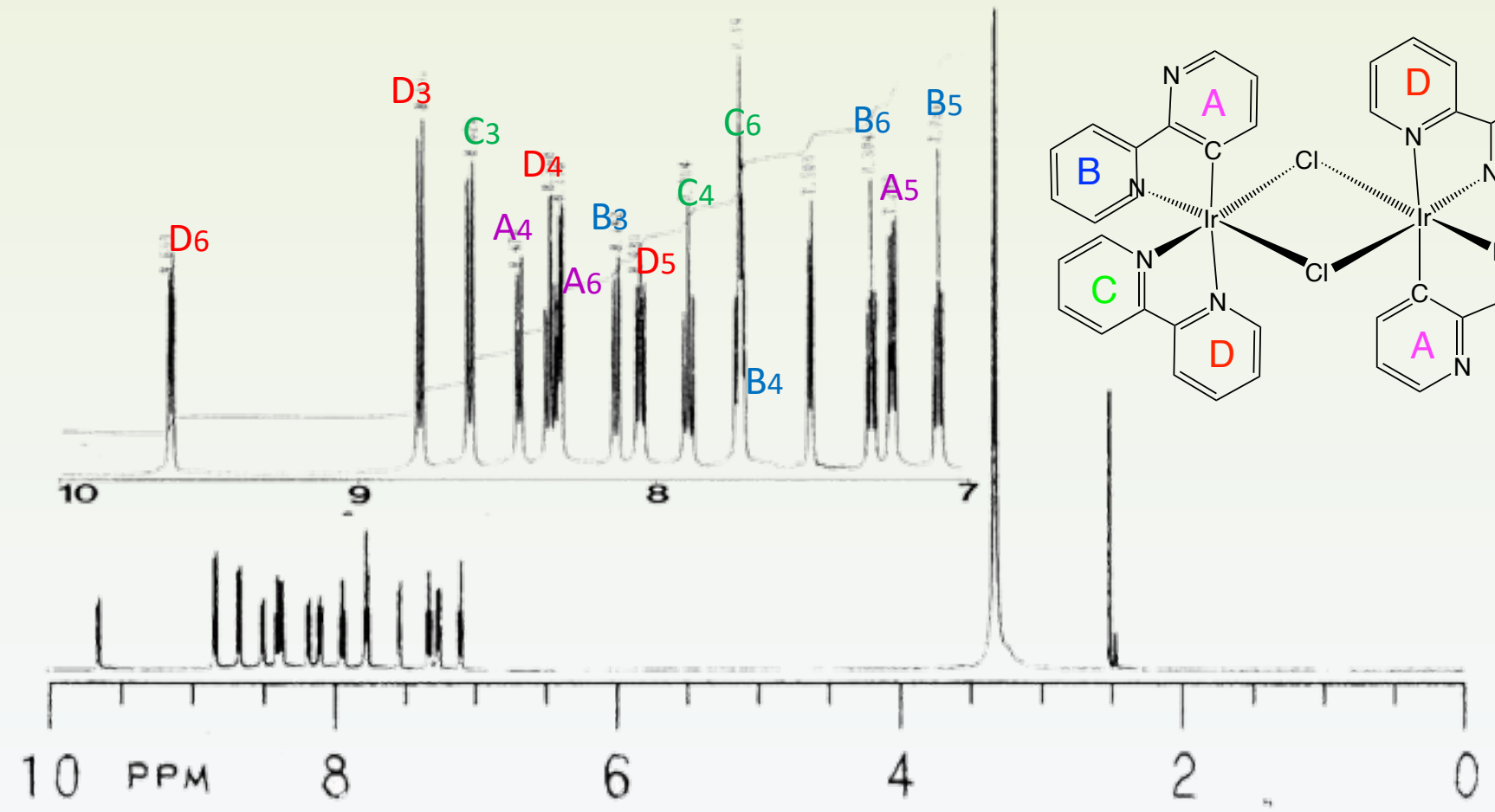
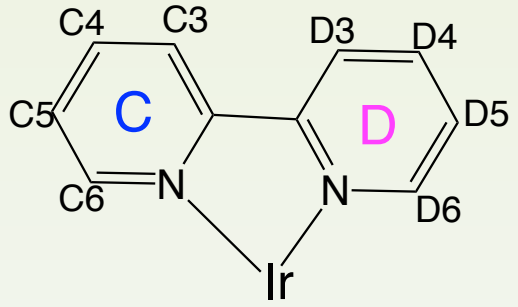
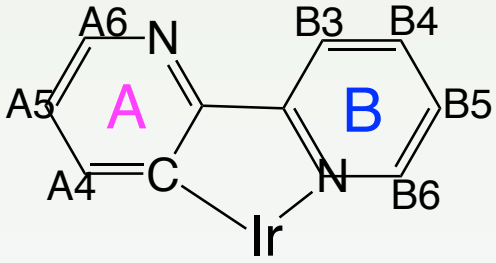
1N NMR Spectrum : DCM



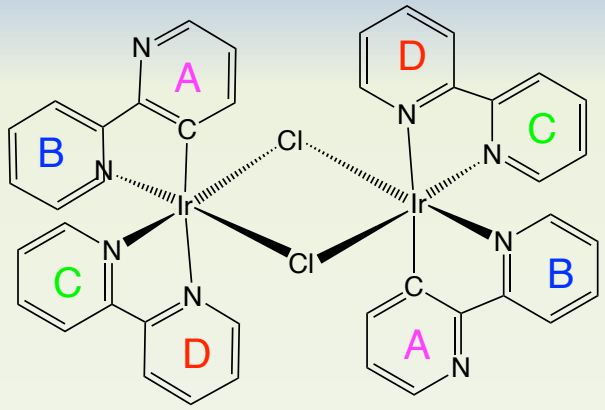
1H resonance assignment Scheme

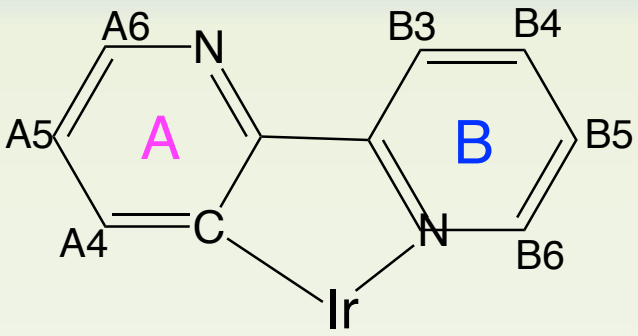


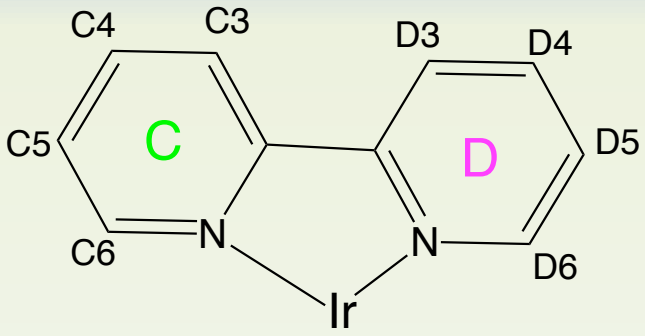


What we want, ^{1}H NMR Spectrum, Evidence of Watt's Dimer

^{1}H NMR Resonance Assignments







Resonance	A-Ring	B-Ring	C-Ring	D-Ring
	Ligand / Complex	Ligand / Complex	Ligand / Complex	Ligand / Complex
1	- / -	- / -	- / -	- / -
2	- / -	- / -	- / -	- / -
3	- / -	8.496 / 8.18	8.496 / 8.66	8.496 / 8.83
4	7.658 / 8.50	7.658 / 7.76	7.658 / 7.95	7.658 / 8.41
5	7.124 / 7.26	7.124 / 7.12	7.124 / 7.34	7.124 / 8.10
6	8.587 / 8.37	8.587 / 7.54	8.587 / 7.78	8.587 / 9.67

$[Ir(bpy\text{-}C^3,N')(bpy\text{-}N,N')_2]^{2+}$

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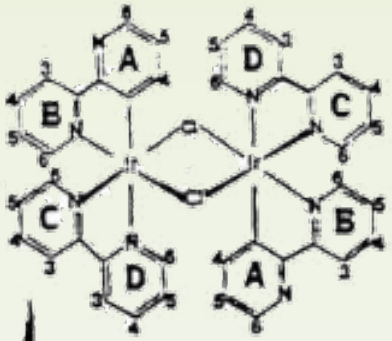
Watts Dimer



What we got·

^{1}H NMR of Product, Evidence of the Watts Dimer

$[Ir(bpy\text{-}C^3,N')(bpy\text{-}N,N')_2]^{2+}$

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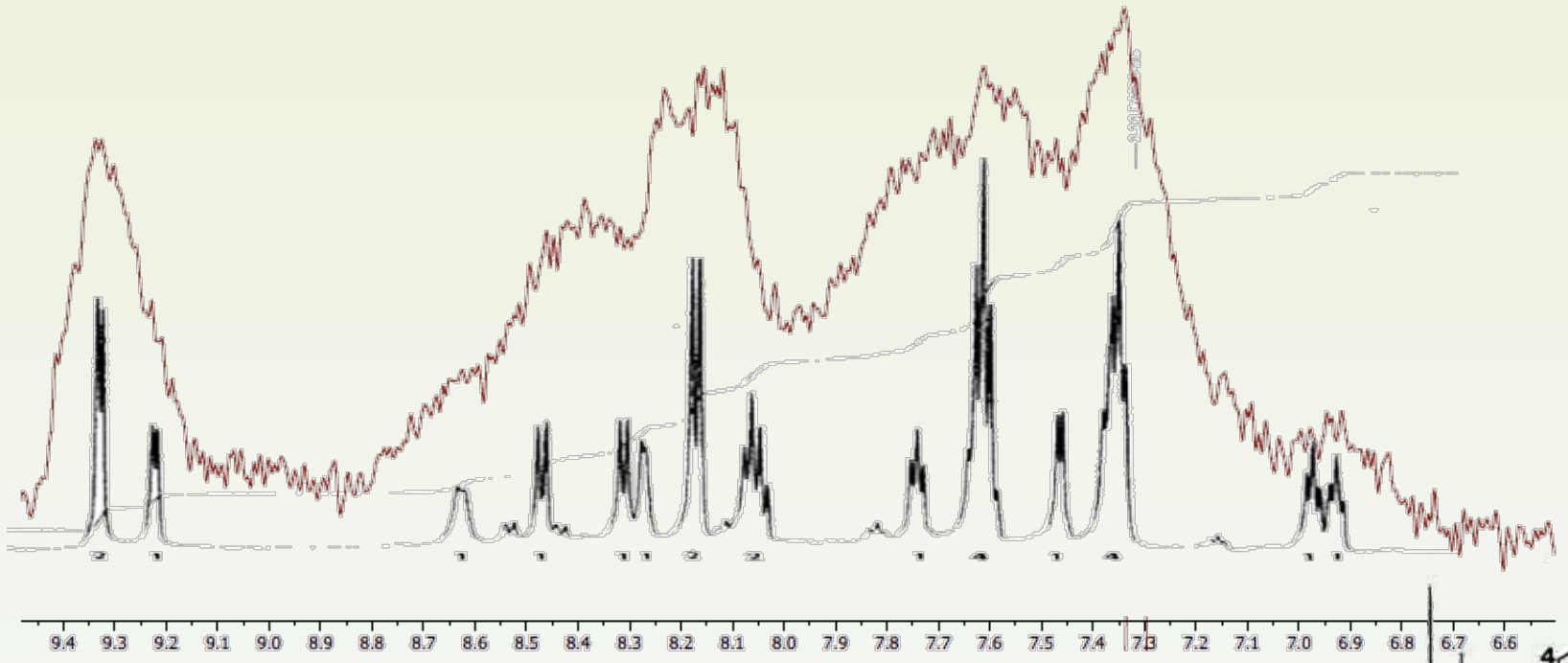
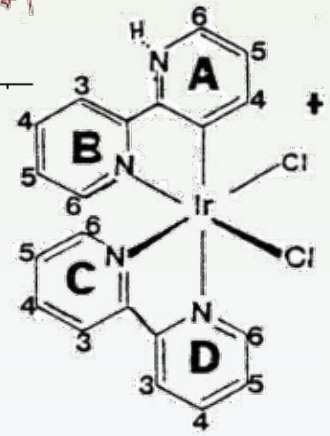
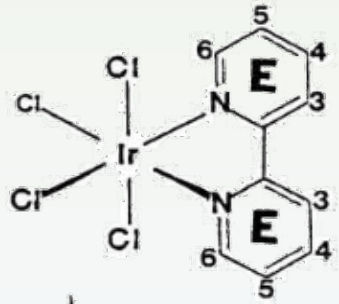
Ion-Pair







What we got·

^{1}H NMR of Product, Evidence of the Ion-Pair

Possible Products

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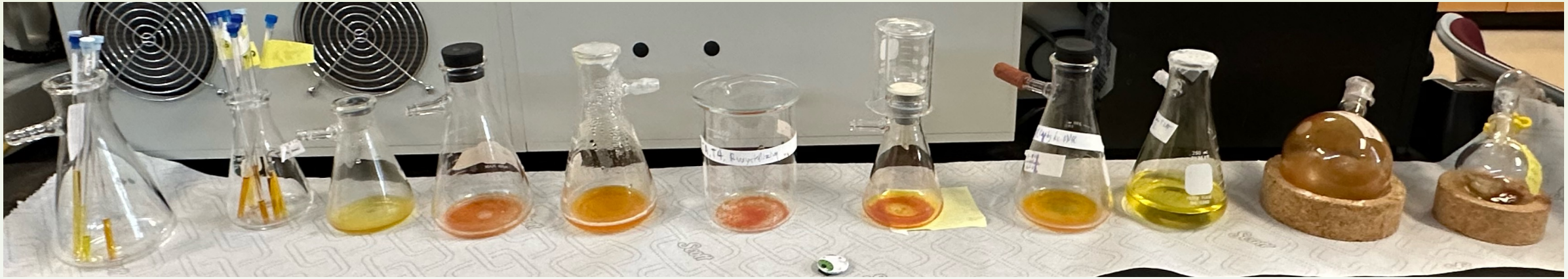
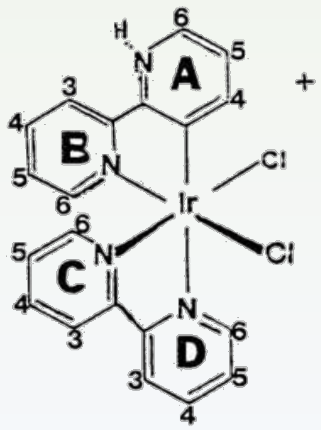
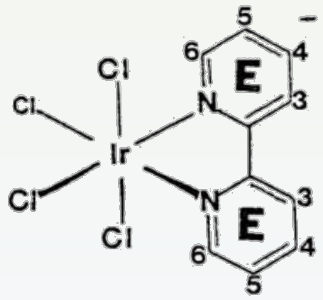
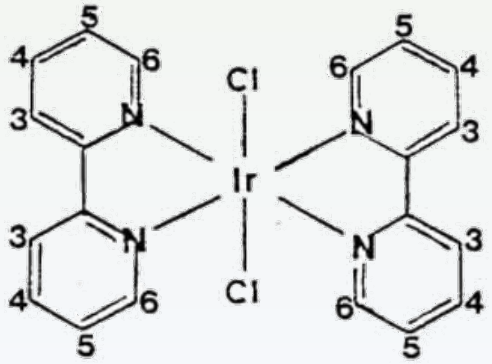
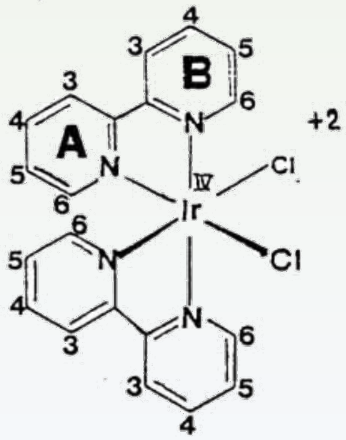
$[Ir(bpy\text{-}C^3,N')(bpy\text{-}N,N')_2]^{2+}$: Possibilities

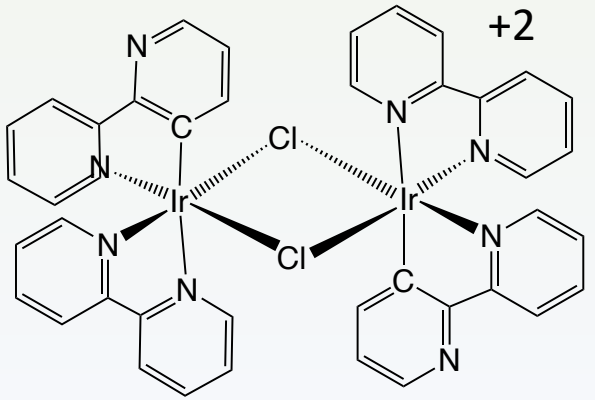












Acknowledgement

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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.

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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.