

# Hybrid Organic-Inorganic Framework Compounds

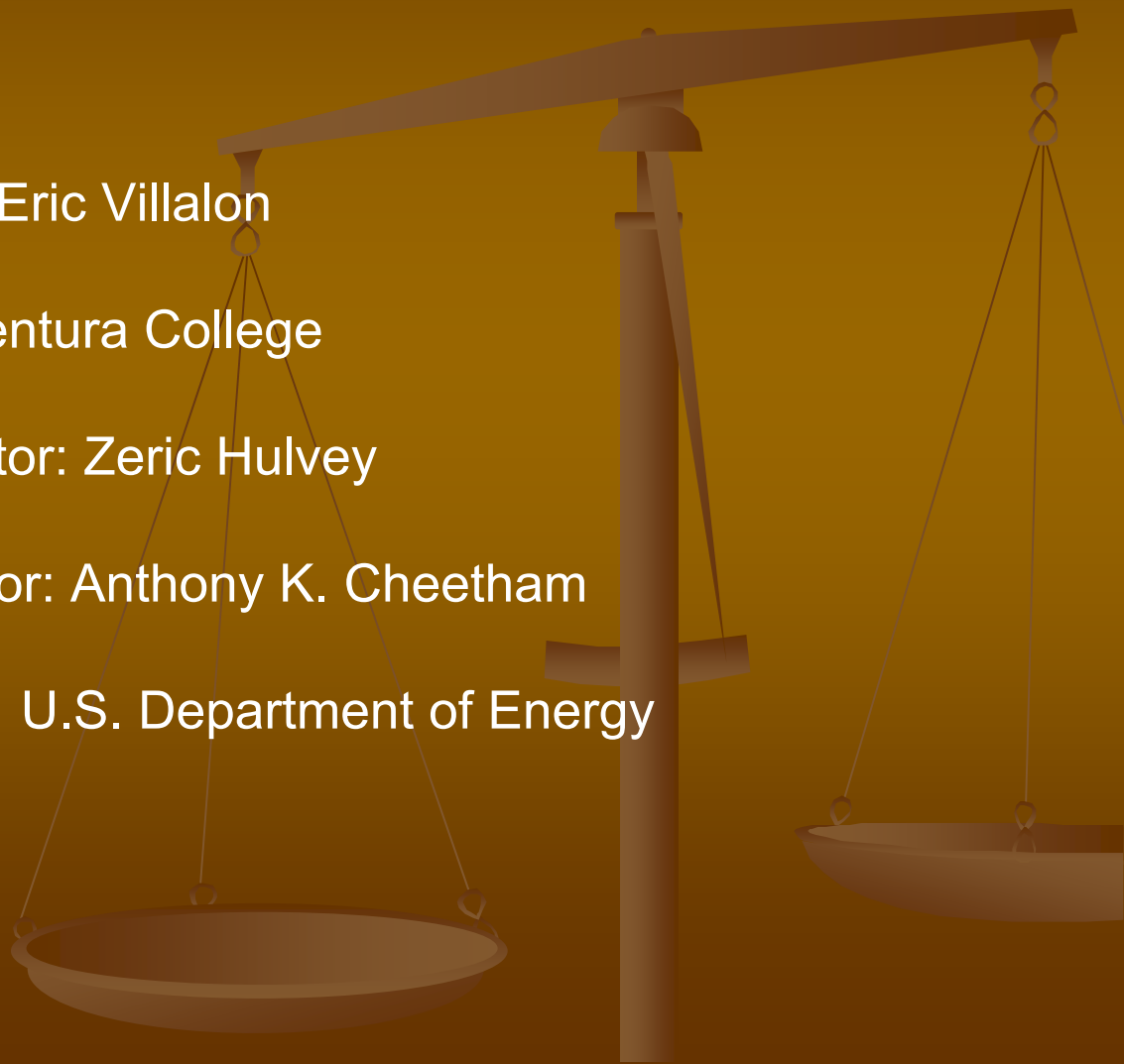
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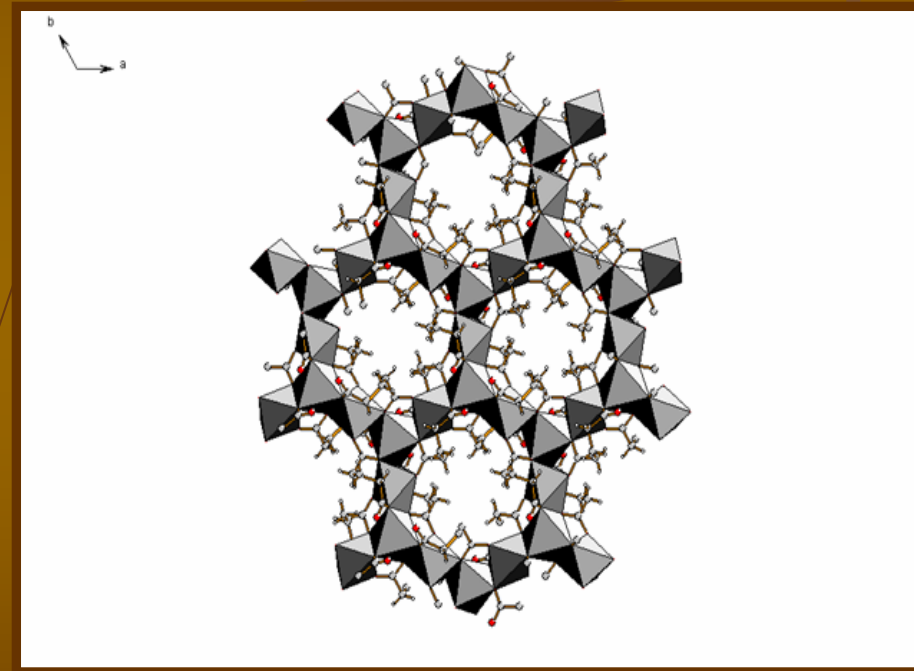
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# What are framework compounds?

- High porosity materials with low structure density
- Metal + organic linker
- Structures often contain 3-D channels or pores



# Applications of framework materials

- Catalysis
- Separations
- Hydrogen storage
  - H<sub>2</sub> adsorbs to framework surface
  - Potentially used in fuel cells



[www.bmwworld.com/hydrogen/h2r\\_racer.htm](http://www.bmwworld.com/hydrogen/h2r_racer.htm)

# Research objectives and approach

- Synthesize new framework structures using ionic liquids as solvents
- Perform as many different reactions as possible
- Vary reaction conditions to get crystals for structure solution
- Product characterization to figure out structure



# Synthesis equipment



Teflon liner



Bomb



Heating oven



# Experimental methods and equipment

Grinding the powder to do X-ray diffraction



Using the X-ray diffractometer



Preparing the sample to be analyzed

# Data

## Typical synthesis:

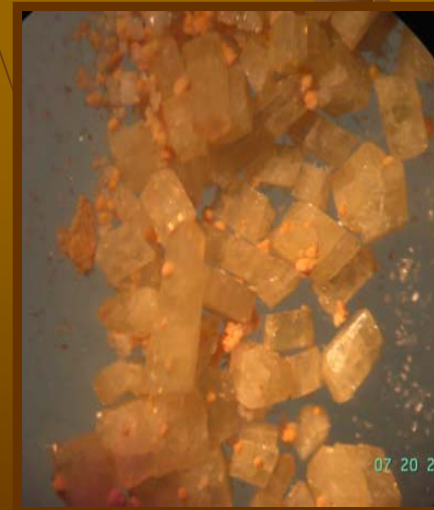
1 gram of solvent  
1:1 ratio of metal to glutaric acid  
Heat for two days



Powder product

## Variables we've changed:

Temperature  
Concentration  
Solvent  
Ratios



Crystals product

# Comparing the reaction results

1-ethyl-3-methyl-imidazolium chloride ( [emim][Cl] )

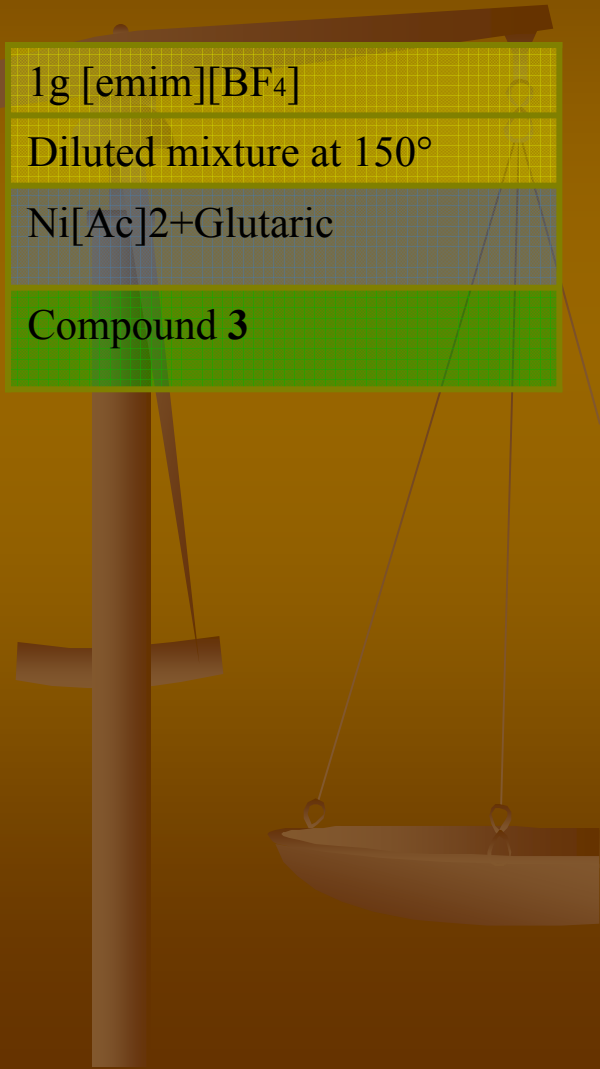
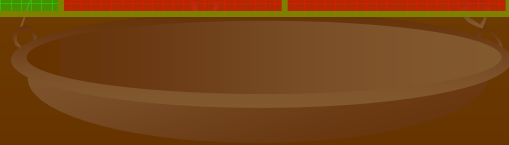
1g of solvent [emim][Cl]	Diluted mixture at 150°	Concentrated mixture at 150°	Diluted mixture at 190°	Diluted mixture at 150° + DABCO
<i>Reactants</i>	Previous reactions	Reaction set #1	Reaction set #2	Reaction set #3
Ni[Ac]2+Glutaric acid	No product	<b>Compound 1</b>	No product	No product
Cu[Ac]2+Glutaric acid	No product	No product	No product	No product
Co[Ac]2+Glutaric acid	No product	No product	No product	No product
Zn[Ac]2+Glutaric acid	No product	No product	No product	No product
Mn[Ac]2+Glutaric acid	No product	No product	No product	No product
Mg[Ac]2+Glutaric acid	No product	<b>Compound 2</b>	No product	No product

1g [emim][BF<sub>4</sub>]

Diluted mixture at 150°

Ni[Ac]2+Glutaric

**Compound 3**





# Several more reactions using different solvents

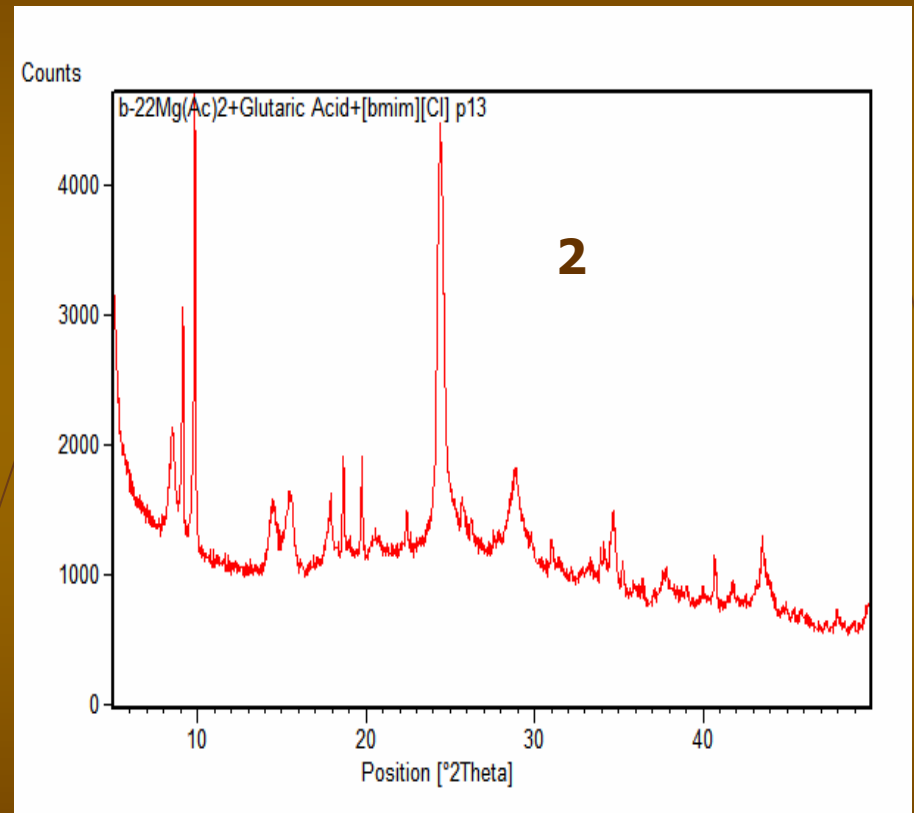
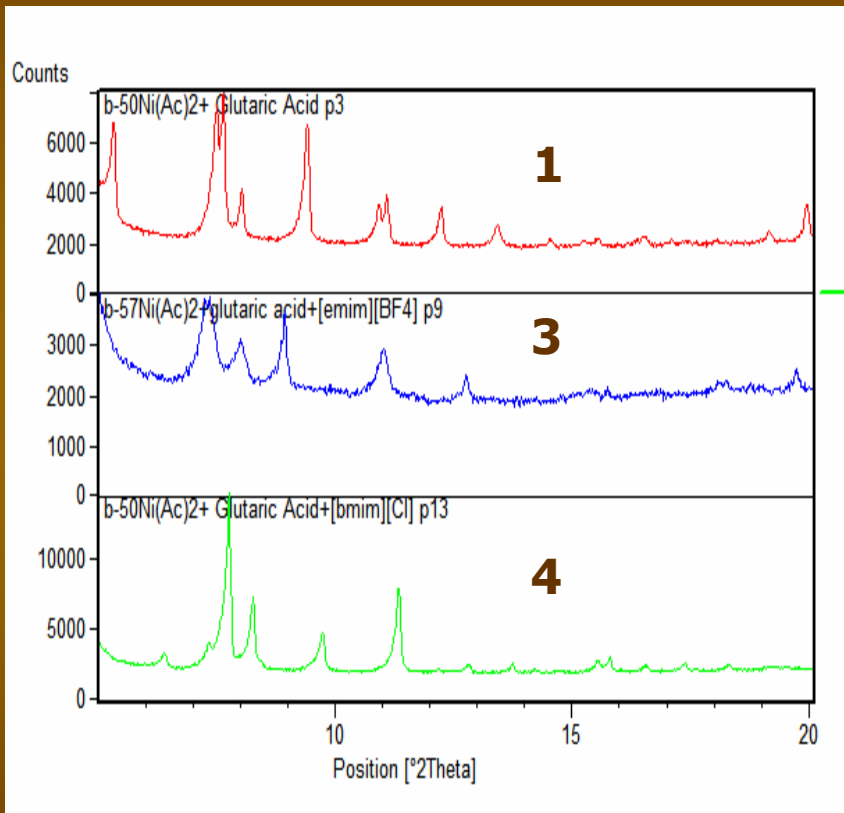
1-butyl-3-methyl-imidazolium chloride ( [bmim][Cl] )

1-ethyl-3-methyl-imidazolium bromide ( [emim][Br] )

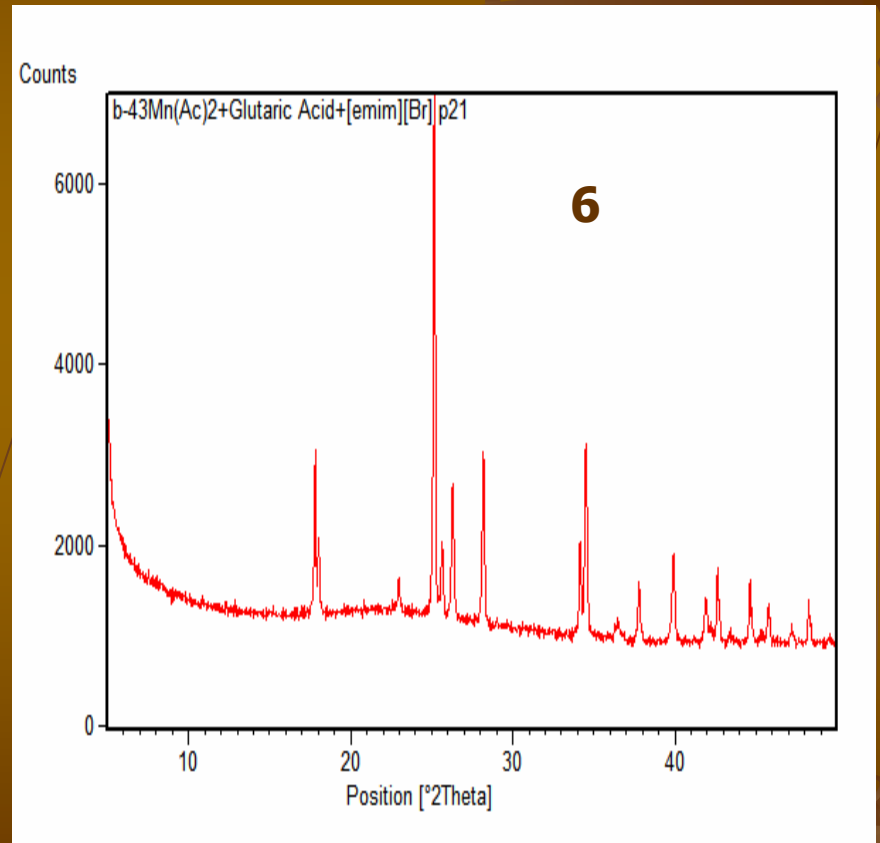
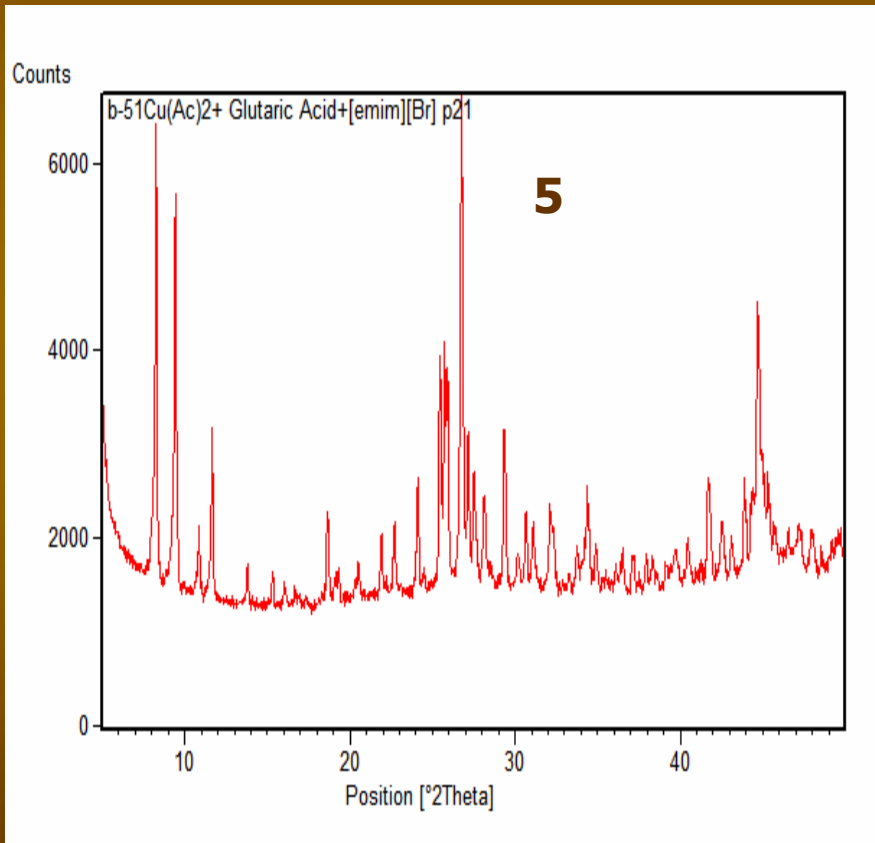
1g of solvent [bmim][Cl] +	Concentrated mixture at 150°	Concentrated mixture at 200°
<i>Reactants</i>	Reaction set # 4	Reaction set #5
Ni[Ac] <sub>2</sub> +Glutaric acid	Compound 4	Compound 1
Cu[Ac] <sub>2</sub> +Glutaric acid	No product	Cu metal
Co[Ac] <sub>2</sub> +Glutaric acid	Amorphous	No product
Zn[Ac] <sub>2</sub> +Glutaric acid	Known zinc glutarate	No product
Mn[Ac] <sub>2</sub> +Glutaric acid	No product	No product
Mg[Ac] <sub>2</sub> +Glutaric acid	Compound 2	Compound 2

1g of solvent [emim][Br] +	Diluted mixture at 150°	Concentrated mixture at 150°
<i>Reactants</i>	Reaction set #6	Reaction set #7
Ni[Ac] <sub>2</sub> +Glutaric acid	No product	Compound 1
Cu[Ac] <sub>2</sub> +Glutaric acid	No product	Compound 5
Co[Ac] <sub>2</sub> +Glutaric acid	No product	Amorphous
Zn[Ac] <sub>2</sub> +Glutaric acid	No product	Known zinc glutarate
Mn[Ac] <sub>2</sub> +Glutaric acid	No product	Compound 6
Mg[Ac] <sub>2</sub> +Glutaric acid	No product	Known Mg-glutarate

# Powder X-ray diffraction patterns

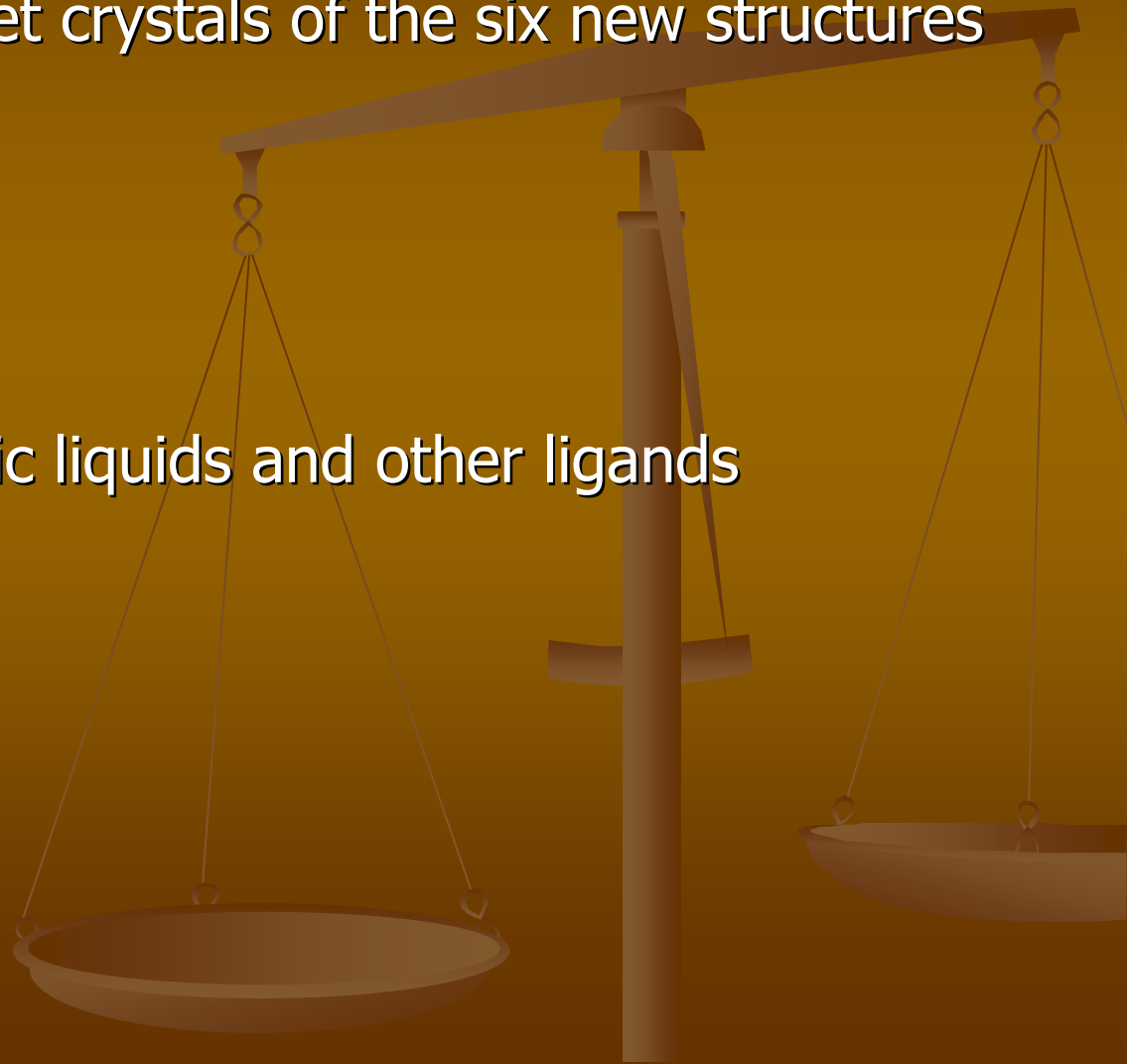


# Powder X-ray diffraction patterns



# Future work

- Continue efforts to get crystals of the six new structures made
- Microwave synthesis
- Investigate other ionic liquids and other ligands



# Acknowledgements



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