2.2. Expert Experimentalist Rating: *"The Single-Crystal Shakedown"*

Overview:

X-Ray diffraction is an important and powerful tool for determining the solid state structure of compounds. Modern advances have made data collection and structure solution almost routine for many small molecules. To use this technique, however, good quality single crystals are still needed. In this exercise, you will experiment with the art of growing single crystals.

Techniques Checklist:

- Manipulation of milligram quantities of material ❑
- Syringe use ❑
- Crystallization techniques for growing good quality single crystals ❑

Pre-Lab Discussion:

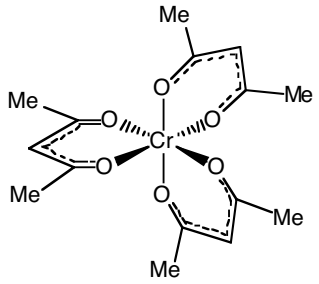
- Use of different recrystallization techniques: vapor diffusion, solvent layering, temperature variation

Goal:

Synthesize $Cr(acac)_3$,[2] then perform several slow recrystallizations to obtain a single crystal of satisfactory quality.

Equipment:

- Magnetic stir plate
- Heating mantle and Variac
- 50-mL Round-bottomed flask
- 2-mL Glass syringe
- Condenser
- Stir bar
- Glass frit (D)
- 250-mL Filter flask and rubber filter adaptor
- 3 Large vials (white cap)
- 4 Small vials (black cap)
- 2 Glass jars

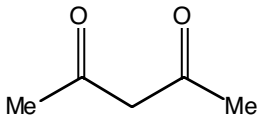


Chromium Acetylacetonate
$Cr(acac)_3$

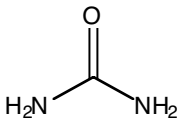
[2]Adapted from Szafran, Z.; Pike, R. M.; Sing, M. M. *Microscale Inorganic Chemistry: A Comprehensive Laboratory Experience*; Wiley: New York, 1991; "Synthesis of Metal Acetylacetonates" p. 224-229.

Experiment Outline:

• ***Before coming to the lab***, perform the necessary calculations to fill in the following table.



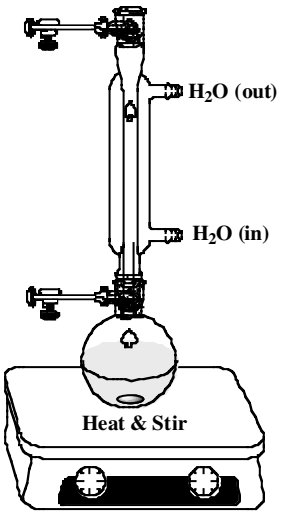
2,4-Pentanedione



Urea

Reagent	Source	F.W.	Density	Mass or Volume	mmols	Equiv
$CrCl_3 \cdot 6H_2O$					1.00 mmol	1
Urea						17
2,4-Pentanedione						8
$Cr(acac)_3$	Product					

• Dissolve $CrCl_3 \cdot 6H_2O$ in 2 mL of distilled water in a 50-mL round-bottomed flask, equipped with a stir bar.
• Add the urea in one portion to the flask, and stir until completely dissolved.
• Add the 2,4-pentanedione dropwise via syringe.
• Attach the condenser to the flask, and heat the mixture to vigorous reflux (*this is important!*), with stirring, for about 1 hour.
• Cool the reaction flask to room temperature, and collect the product by vacuum filtration on a size D glass frit funnel, washing the product with cold water.
• Dry the product in your desiccator overnight, and obtain a yield.
• Set up multiple recrystallizations to grow X-ray qualitysingle crystals - *see Growing a Single Crystal Guide*.

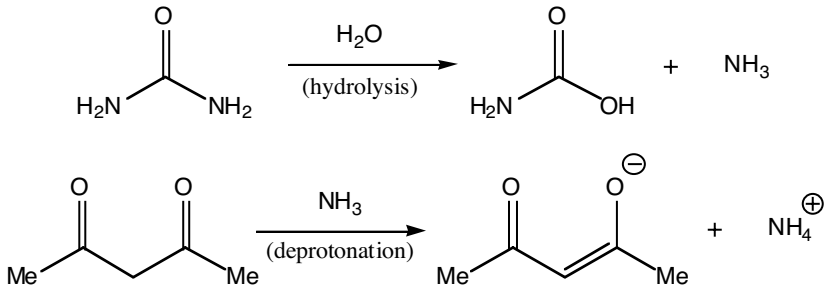


Reaction Setup

Note:

• Urea slowly hydrolyzes under the reaction conditions, liberating ammonia (NH_3), which controls the pH of the reaction. As more NH_3 is generated, the solution becomes more basic, making it easier to remove the proton from the acetylacetonate (acac), also known as 2,4-pentanedione. It is the anion of the acetylacetonate that coordinates to the

metal to form the desired $Cr(acac)_3$ complex. What is the limiting reagent? Calculate your percent yield.



Helpful Hints:

• When using a saturated solution to grow crystals, it is important that you filter the solution through a plug of glass wool in a pipet before setting up the crystallization.

Results:

• To obtain your "EE Rating," you must obtain ≥ 50% yield of $Cr(acac)_3$, and you must produce at least one single crystal that is suitable for X-ray analysis.