Synthesis of Thermochromic Materials

- **Wash and oven dry:**
  - Two centrifuge tubes
  - Two test tubes
  - Two droppers

- **Collect:**
  - One Styrofoam cup (for ice-water bath)
  - One thermometer
  - Four rubber septa

- **Prepare**
  - Centrifuge (underneath lab bench)
  - Test tube rack and test tubes
  - Glass rod and 100 mL beaker
Objective

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  To synthesize a thermochromic solid from diethylamine hydrochloride and copper(II) chloride

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2\text{(CH}_3\text{CH}_2\text{)}_2\text{NH} \cdot \text{HCl} + \text{CuCl}_2 \rightarrow [(\text{CH}_3\text{CH}_2\text{)}_2\text{NH}_2]_2[\text{CuCl}_4]\]

- **Thermochromic materials**
  - Substance that changes color with temperature
  - Divided into two types: continuous and discontinuous thermochromic
  - According to the crystal field theory, changes in geometry, ligands, or coordination number in the coordination sphere may cause the color change
Introduction

- \([(\text{CH}_3\text{CH}_2)_2\text{NH}_2]_2[\text{CuCl}_4]\)
  - **Bright green** at room temperature
  - **Yellow** color when temperature increases
- The color change is caused by a change in the geometry of the complex ion, \([\text{CuCl}_4]^{2-}\)
Step 1-2. Prepare Reactants and React

Wash & oven dry:
- Centrifuge tubes
- Glass dropper
- Test tubes

*All glassware must be dried

0.001 mol (0.13 g) CuCl₂

0.002 mol (0.22 g) diethylamine hydrochloride

- Placed in a centrifuge tube
- Add 1 mL isopropyl alcohol
- Stir to dissolve and produce product
- Place in an ice-water bath
Step 3-7. Wash and Collect the Product

- **Wash the product**
  - Ice-water bath to crystallize completely
  - Centrifuge and decant the supernatant, iPA
  - Add 1 mL ice-cooled ether
  - Stir and mix well to wash the product
  - Centrifuge and decant the ether layer

- **Rinse with ether several times until the supernatant is colorless**

- **Collect product**
  - Take parts of product to a clean test tube
  - Add 1 mL ether to rinse the product
  - Settle and decant the ether layer
  - Place the tube in a warm water bath ca. 40°C to evaporate residual ether

[(CH$_3$CH$_2$)$_2$NH]$_2$[CuCl$_4$]
Step 8-9. Examine the Thermochromic Temperature

- Fix the sample test tube to the thermometer
- Place the test tube and thermometer in a water bath in a 100 mL beaker
- Heat the water bath slowly to observe the color varied with temperature
- Replace into ice-water bath to check if the color change is reversible

Place the thermometer in the center

Thermochromic temp.? Reversible?
Both reactants are hygroscopic; put the cap back after use.

Cap the test tube with rubber septum to reduce the evaporation of organic solvent.

Cool the centrifuge tube with product after each rinsing to reduce solubility of product.

During centrifuge, centrifuge tubes should be placed in **opposite sides to keep balanced**.

Evaporate the ether solvent in the fume hood.

Collect all the organic solvent in a test tube, then pour into organic waste bin.

Use small portions of water to dissolve the product and discard into the heavy metal recycling bin.