The crystallization pictured in this section shows purification of a roughly \(1 \text{ g}\) sample of *trans*-cinnamic acid. *Trans*-cinnamic acid is soluble in methanol and insoluble in water, and this crystallization uses a mixed solvent of methanol and water to give a \(74\%\) recovery.

![Figure 3.56: a) Initial sample of trans-cinnamic acid, b) Crystallization using methanol/water, c) Crystallized trans-cinnamic acid.](image)

It is assumed that students performing this technique have previously performed or read about a single-solvent crystallization.

![Figure 3.57: a) Trans-cinnamic acid is insoluble in water and soluble in methanol, b) Adding solid to the flask, c) Adding methanol off the heat source, d) Adding enough methanol to dissolve the solid.](image)

1. Determine two miscible solvents that can be used for the crystallization (Figure 3.57a): the desired compound should be soluble in one solvent (called the "soluble solvent") and insoluble in the other solvent (called the "insoluble solvent").

2. Transfer the impure solid to be crystallized into an appropriately sized Erlenmeyer flask (Figure 3.57b).

3. Place some of the "soluble solvent" into the flask (Figure 3.57c), add a boiling stick (or boiling stones if preferred), then heat atop a steam bath (Figure 3.57d). A hotplate can be used cautiously if using the mixed solvents methanol/water or ethanol/water.

![Figure 3.58: a) Addition of just enough soluble solvent (methanol) to dissolve the solid, b) Addition of the insoluble solvent (water) dropwise, c) Sustained cloudiness after addition of enough insoluble solvent, d) Addition of soluble solvent to clarify.](image)

4. Add more of the "soluble solvent" in portions until the solid just dissolves (Figure 3.58a). Be sure to allow time in between additions, and allow each addition to come completely to a boil before adding more.

5. Add the "insoluble solvent" in portions with heating until the solution becomes just cloudy (Figure 3.58c).

6. Add the "soluble solvent" dropwise with heating until the solution again clarifies (Figure 3.58d).
7. Remove the flask from the heat source, remove the boiling stick and set the flask atop a paper towel folded several times. Cover the mouth of the Erlenmeyer flask with a watch glass, and allow the solution to slowly cool to room temperature (Figure 3.59a).

8. As the solution cools, eventually solid crystals should form (Figure 3.59b). Use a glass stirring rod to scratch the flask and initiate crystallization if necessary. Place the crystals in an ice-water bath for 10-20 minutes and collect the solid by suction filtration.

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