To experimentally determine a single solvent for crystallization, use the following procedure. A flow chart describing this process is shown in Figure 3.11.

- Place \(100 \text{ mg}\) of the solid to be crystallized in a test tube and add \(3 \text{ mL}\) of solvent. (These quantities correspond with the usual guideline of solubility, where a compound is deemed "soluble" if \(3 \text{ g}\) of compound dissolves in \(100 \text{ mL}\) solvent.) Note: quantities may be scaled back depending on compound availability.
- While holding the test tube by the top, flick the tube to vigorously mix the contents.
- If the solid completely dissolves at room temperature, the solvent will not work for crystallization since it needs to be insoluble when cold. If the solid appears to remain insoluble, the solvent may work.
- Bring the solid suspended in the solvent to a boil using a steam bath or hot water bath. If the solid dissolves when the solvent is boiling, it may work for crystallization since it needs to be soluble when hot. If it never dissolves in the hot solvent, the solvent won’t work.
- If the solid dissolves in the hot solvent, allow it to cool to room temperature, and then submerge it in an ice bath for 10-20 minutes. If most of the crystals return, the solvent should work for crystallization. If few or no crystals return, try to scratch the flask with a glass stirring rod to initiate crystallization. If crystals still don’t return, the solvent won’t work for crystallization.

![Flow chart for testing a crystallization solvent by mixing solid and solvent, as described on this page.](image)

This testing procedure is shown for \(\ce{N}\)-bromosuccinimide in Figure 3.12, using water as the solvent.

![Testing the crystallization of \(\ce{N}\)-bromosuccinimide (NBS) using water: a) NBS insoluble in cold water, b+c) Dissolving NBS in hot water, d+e) Cooling and crystallization (ice bath was not needed, as crystals returned).](image)

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