How to Make Nanocrystals in Several Not-so Easy Steps:

One day in advance:

0. Check for solvents: Dry oxygen free TOPO, pyridine, methanol will be needed.
1. The TOPO can be weighed outside the box if need be, want to be near 12.0±0.1g. Growth, of course, is strongly
related to concentration so the amount of TOPO is very important, especially for the larger sizes. Use a mortar and
pestle to crush thoroughly (helps to get it in the flask). Leave in the ante chamber under vacuum overnight. **DO NOT**
heat over 80°C under vacuum.
2. Pyridine and methanol need to be cannulated from the stills. Only use “fresh” solvents. If there is some left in the
still reservoir, drain and redistill. The methanol still must be refluxed for 24hrs to ensure deoxygenation. The
pyridine is refluxed until blue over sodium benzophenone.
3. When pulling into the dri-box be sure to rinse room temperature glassware with acetone.
4. Check for dimethyl cadmium, 166µl (1.9:1) or 123µl (1.4:1) will be needed. If not make arrangements to have some
stock transferred on the vacuum line. **Note:** The CdMe₂ is expensive and **VERY** reactive, keep capped when not
in use to avoid spilling!
5. Check for selenium/tributyl phosphine (TBP) solution. It can be prepared in 100ml batches from 0.966g Se in 100ml
TBP (to get enough Se to weigh accurately). Shake until dissolved. Use 3.864g Se in 100ml TBP for 4x solution.
**Note:** Phosphines are **VERY** hard (poison) on the dri-train, keep capped when not in use and if spilled clean up
IMMEDITALY! Large volumes (>3ml) of TBP are pyrophoric (especially on paper towels) and stink. Remove any excess material capped directly to the fumehood.
6. Check for needed glassware. All glassware should be put into the antechamber hot (>130°C) from the drying oven
don’t dally) and immediately placed under vacuum. It is much easier to wrap the ground glass joints with Teflon
tape BEFORE they are put in the oven!

**Needed:** A100ml three necked flask, Pear shaped bump trap, Dry ice condenser, Teflon swagelock thermocouple
adapter, Syringes: 10ml (2), 1ml (1), 250µl (note: remove plastic top before heating), Needles: 12 gauge (1) and 18
gauge (2), Two (or more) 25ml Erlenmeyer flasks, Two 11 dram centrifuge vials, threads wrapped with Teflon tape.

Reaction Day:

1. With the heating mantle power cord UNPLUGGED/SWITCHED OFF, set the temperature controller to 360°C by
holding the **up arrow key** until the green setpoint numbers change to 360.
2. Add the ATOPO to the 100ml multi-necked flask. The 14/22 jointed funnel helps.
3. Insert the Teflon swagelock thermocouple adapter into one side joint of the flask and a septum into the other side
joint (for long cooking times the silicone septa are preferred). The bump trap/condenser should occupy the center
joint. Adjust the thermocouple so that it does not touch the bottom of the flask, but still immersed in solvent.
4. Plug in or turn on (press and hold the **up arrow** and **F** keys together) and begin heating the TOPO.
5. Using a 10ml syringe with an 18 gauge needle, withdraw 10ml of Se/TBP solution and add to a 25ml Erlenmeyer.
Withdraw 166µl (1.9:1) or 123µl (1.4:1) of CdMe₂ and add to Se solution, swirling well for the injection solution.
Cap until needed. The first method (step 7) requires an additional reaction solution prepared by adding 664µl (1.9:1)
or 492µl (1.4:1) of CdMe₂ to the 4x Se/TBP solution. The second method (step 8) only requires injection solution.
6. Using the other 10ml syringe with the 12 gauge needle, quickly inject 5-9ml of the reaction solution into the hot
(360°C) TOPO. **Warning:** Due to the low boiling point (100°C) of the TBP the injection is a violent event! **Note:**
A 12 gauge needle is a large needle, it is easy to drip from and therefore advisable to practice and injection with
acetone in an empty flask outside the box first to practice technique.
7. Immediately follow up a 5ml initial injection with 0.5-3ml of 4x solution. Allow the reaction to proceed until the
desired size is reached. Follow the size with 1/2 ml aliquots every 30 min until close to the size desired, then more often (intervals shorter than 10 min are not necessary). The amount of 4x solution added will be determined by what size the nanocrystals are desired to focus at. More solution focuses later in time (size).

**OR**

8. Allow the reaction to proceed for 90 minutes, or until the desired size is reached. Rule of thumb suggests that the best size distribution is achieved at 17 min, for a 5ml injection, 25 min for 6ml, and 35 min for 9ml. All times are approximate. At 90 min a second aliquot of reaction solution (30% initial injection) can be added to focus the size. This process can be repeated at 120 min with 40-45% of the initial injection volume. This works best for 5-6ml initial injection volumes.

9. Remove from the heating mantle when desired size is reached to speed cooling **WARNING: THE HEAT WILL MELT THE DRI-BOX GLOVES BEFORE THE HEAT CAN BE FELT!!!** and allow to cool to room temperature (solid TOPO), wash in anhydrous methanol until the solution becomes flocculent. Add evenly between vials and centrifuge. Decant supernate, add more methanol, shake vigorously. Repeat for three washes.

10. Uncap after last wash (in the box!) and allow to dry. Yield: ~100mg.

**Ligand Exchange:**

1. Dissolve TOPO capped nanocrystals in anhydrous pyridine (1mg/ml).

2. Stir at 80-90°C overnight.

3. Add capping ligand of choice and then precipitate with the appropriate solvent. Centrifuge to collect and wash as necessary.