



Synthetic Protocol for Pd Nanocubes

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Preparation

- Set up an oil bath by filling a 250 ml beaker about ¾ full with Si oil. Heat and stir oil bath on hot plate to 80° C.
- Put a black capped reaction vial (with a stir bar, 320 rpm) into oil bath with an o-ring as a support on a vial holder.











Making Solutions

- Measure 105 mg poly(vinyl pyrrolidone) (PVP), 300 mg KBr and 60 mg ascorbic acid with end of a disposable glass pipette and mix in 8 mL deionized water. Seal and allow chemicals to dissolve; vortex if necessary.
- Next, measure and dissolve 57 mg Na₂PdCl₄ in 3 mL deionized water.











Adding Chemicals

- Remove cap and add 8 mL of the PVP, KBr and ascorbic acid mixed solution to the reaction vial. (Keep reaction vial clamped in place)
 Replace cap loosely and wait 2-3 minutes until the temperature is back to 80° C.
- After 2-3 minutes, add 3 mL of the Na₂PdCl₄ solution to the reaction vial.









Running Reaction

 The reaction will take approximately 3 hours and the vial should be moved to an ice bath after the reaction.









Isolating Nanocubes from Solution

- Transfer the reaction solution to a 50-mL centrifuge tube, add acetone up to 45 mL, and then spin down the product at 11,000 rpm for 10 min. Remove and discard the supernatant with a disposable pipette.
- Add 10 mL deionized water to the tube, agitate via sonication and vortex to redisperse the products, and then transfer to 10 1.5-mL centrifuge tubes. Spin down the product at 13,000rpm for 10 min. Remove and discard the supernatant. Repeat the water washing step 2-3 times.









