Potassium persulfate recrystallization

Source: Northern Temperate Lakes LTER Website with modifications by R. McHorney July 2014

1. About 30 minutes before you start, place a DI squirt bottle with fresh DI in the refrigerator to cool. After 30 minutes fill a container with ice and place the cold DI into it. Set up two stir plates, one hot, one cold.

2. Dissolve 50 g of potassium persulfate in 300 ml of DI previously heated to 60˚ C. In the process it should cool somewhat. Some heating (with stirring!) may be necessary to get the solution to between 55 and 60°C at which point complete dissolution should occur. The solution should NOT be heated at any time above 60°C.

3. Filter the solution rapidly through a 0.22um membrane or GFF filter (47 mm or larger diameter). Preheat the filter flask to prevent cooling of the solution during filtration.

4. Rinse the 500 mL flask used to pre-heat the potassium persulfate solution and pour the filtrate back into it.

5. Cool solution to about 4°C by placing the flask in ice water with stirring (a 500 mL flask will fit in a 2000mL beaker as an ice bath). The flask and ice bath should be placed on a second, cool stir plate. While this is cooling, heat a second 500 mL flask with 225mL DI to 60°C.

6. Filter the 4°C solution and wash with 1 or 2 squeezes of ice cold DI. Save the white solid and discard the filtrate from the sidearm flask.

7. Place the crystals into a second flask and heat the solution as necessary to between 55 and 60°C to completely dissolve the crystals.

8. Repeat steps 5 and 6. Apply vaccum until no more filtrate comes out then an additional two minutes. The white granules on top of the filter are purified crystals!

9. Place the crystals in a clean wide mouth container and dry in a vacuum dessicator over anhydrous calcium chloride (or Drierite). Rapid drying in a good vacuum and thus at a low temperature is essential as this will minimize the sulfuric acid formation on the crystals. It may be necessary to do the final drying in an oven set to 55°C after vacuum dessication. During this process, record the weight of your container without crystals, with wet crystals and with dry crystals. This will not only give the yield, but can indicate the moisture content immediately after filtration. This may come in handy if you need to use the persulfate without drying.

The yield should be >50%. The procedure *should* yield very low reagent blanks, less than about 10uM for the reagent or 1 uM after dilution with DI.

You will need: *two stir plates (hot and cold), sidearm flask w/tubing, filter holder/funnel (47mm), 47 mm gff or membrane filters, vacuum pump, 500 mL flasks (2), 2L beaker, ice, ice bucket, cold DI in a squirt bottle, spatula, thermometer, clean wide mouth bottle for final crystals.*