

### Total P Protocol using persulfate digestion

- Ellen Esch, May 2019, persulfate reaction adapted from EPA/600/R-93/100, Method 365.1.
  - Method is sensitive to 0.005 mg P/l.
  - The autoclave digestion converts all P to orthophosphate, after which you run the orthophosphate protocol ☺ . Note Total P = OrthoP (dissolved reactive P) + particulate P. You can determine particulate P if you run both the total P digestion, and then the not digestion one too.
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#### Make solutions:

##### 5.6 M H<sub>2</sub>SO<sub>4</sub>

- Add 154.7 ml of concentrated H<sub>2</sub>SO<sub>4</sub> into a 500 ml volumetric with nanopure (fill with ~300 ml nano first).
  - $18.1 \text{ M} * v_1 = 5.6 \text{ M} * 500 \text{ mL} \rightarrow v_1 = 154.7 \text{ ml}$  concentrated H<sub>2</sub>SO<sub>4</sub> into 500 ml
  - $(96.5 \text{ g H}_2\text{SO}_4 / 100 \text{ g acid}) * (1.84 \text{ g acid / mL}) * (1 \text{ mol H}_2\text{SO}_4 / 98.072 \text{ g}) * (1000 \text{ ml / L}) = 18.1 \text{ M}$
  - $(\text{acid purity by weight}) * (\text{density of acid}) * (\text{molecular weight}) * (\text{unit conversion}) = \text{molarity}$
- Toxic!!!! Wear PPE, don't inhale/eat. Store, but if must dispose, H<sub>2</sub>SO<sub>4</sub> is **HAZARDOUS WASTE**.

##### Acid Digestion Solution \*\*make fresh daily!!

- In 50 ml volumetric, add: (this is good for ~200 samples, feel free to scale down as needed)
  - 6.4 g ammonium persulfate (stored in flammable cabinet)
  - 16 ml 5.6M H<sub>2</sub>SO<sub>4</sub>
  - Bring to volume with nanopure
- Dispose of as **HAZARDOUS WASTE**.

#### Digest Samples:

1. Add 4 mL sample to a test tube (make sure to label them well!). (make sure to run a blank (DI water) and your standard curves too!)
2. Add 0.25 ml of Acid Digestion Solution to each tube.
3. Vortex (speed of 2 is good) and cover with caps (loosely capped).
4. Place the test tubes into the plastic autoclave tray, then fill the tray with water to the depth of the solution in the tubes. This will minimize leaks due to rapid changes in temperature and pressure.
5. Autoclave for 30 min at 121C and 15-20 psi (Liquid 20 cycle). Follow all IB autoclave protocols (place our tray inside the aluminum tray, etc).
6. Remove tray from autoclave, cool, securely cap test tubes! After digestion, check water height of samples. If they are inconsistent, there were likely leaks during the digestion, and the digestion should be redone.
7. Run the extracts using the orthophosphate protocol. Be sure to use your digested samples as the standard curve. To be extra efficient, you should probably run the P protocol on you digested and undigested samples on the same day (since the P reagent in the orthoP protocol is only good for ~4hrs).

#### Reporting results:

“Total P was determined using an ammonium persulfate digestion.”