

### Reagents and preparation

- Rinse rule of three times for everything.
- Prepare all solution inside a fume hood and wear gloves and goggles. Read all SDS for each chemical before using.
- Depending on the volume you're making for manganous chloride, sodium iodide/sodium hydroxide, and sulfuric acid, make sure to have adequate size beakers to measure in and stir the solution.

### Supplies:

- 2 L beaker (for homogenizing solution)
- beaker to weigh dry chemical in
- 1 L volumetric flask and cap (to bring to volume)
- Big metal scoop
- Big stir bar
- Stir plate
- DIW squeeze bottle
- Gloves
- Disposable pipet
- Water bath
- Mettler Toledo 3100 g scale (manganous chloride, sodium iodide, sodium hydroxide)
- Mettler Toledo 120 g analytical balance (thio)

Chemical	Name	Chemical Formula	CAS	Amount	Link
Manganous chloride	Manganese Chloride Tetrahydrate (Crystalline/Certified ACS), Fisher Chemical	$\text{Cl}_2\text{H}_8\text{MnO}_4$	13446-34-9	500 g	<a href="https://www.fishersci.com/shop/products/manganese-chloride-tetrahydrate-crystalline-certified-ac-s-fisher-chemical-2/M87500">https://www.fishersci.com/shop/products/manganese-chloride-tetrahydrate-crystalline-certified-ac-s-fisher-chemical-2/M87500</a>
Sodium iodide	Sodium Iodide (Crystalline/Certified), Fisher Chemicals	NaI	7681-82-5	500 g	<a href="https://www.fishersci.com/shop/products/sodium-iodide-crystalline-certified-fisher-chemical-2/S324500#?keyword=sodium%20iodide%207681-82-5">https://www.fishersci.com/shop/products/sodium-iodide-crystalline-certified-fisher-chemical-2/S324500#?keyword=sodium%20iodide%207681-82-5</a>
Sodium hydroxide	Sodium Hydroxide (Pellets/Certified ACS), Fisher Chemical	NaOH	1310-73-2, 497-19-8	500 g	<a href="https://www.fishersci.com/shop/products/sodium-hydroxide-pellets-certified-ac-s-fisher-chemical-7/S3181#?keyword=1310-73-2%20sodium%20hydroxide%20certified%20acs%20pellets">https://www.fishersci.com/shop/products/sodium-hydroxide-pellets-certified-ac-s-fisher-chemical-7/S3181#?keyword=1310-73-2%20sodium%20hydroxide%20certified%20acs%20pellets</a>
Sulfuric acid	Sulfuric acid (ACS Grade), Fisher Chemical	$\text{H}_2\text{SO}_4$	7664-93-9	500 mL	<a href="https://www.fishersci.com/shop/products/sulfuric-acid-certified-ac-s-plus-fisher-chemical-11/A300500#?keyword=7664-93-9%20242206">https://www.fishersci.com/shop/products/sulfuric-acid-certified-ac-s-plus-fisher-chemical-11/A300500#?keyword=7664-93-9%20242206</a>
Sodium thiosulfate	Sodium thiosulfate pentahydrate, 99+%, ACS reagent OR Sodium Thiosulfate Pentahydrate (Crystalline/Certified ACS), Fisher Chemical	$\text{H}_{10}\text{Na}_2\text{O}_8\text{S}_2$	10102-17-7	500 g	<a href="https://www.fishersci.com/shop/products/sodium-thiosulfate-pentahydrate-99-5-ac-s-reagent-thermo-scientific/AC424460051#?keyword=10102-17-7%20sodium%20thiosulfate%20pentahydrate">https://www.fishersci.com/shop/products/sodium-thiosulfate-pentahydrate-99-5-ac-s-reagent-thermo-scientific/AC424460051#?keyword=10102-17-7%20sodium%20thiosulfate%20pentahydrate</a> OR <a href="https://www.fishersci.com/us/en/catalog/search/products?keyword=10102-17-7+sodium+thiosulfate+pentahydrate">https://www.fishersci.com/us/en/catalog/search/products?keyword=10102-17-7+sodium+thiosulfate+pentahydrate</a>
Potassium iodate	Iodate Standards	$\text{KIO}_3$	NA	100 mL	<a href="https://osil.com/product/iodate-standards/">https://osil.com/product/iodate-standards/</a>

A. *Manganous chloride (3M)* (stable indefinitely when filtered and stored in sealed amber glass):

Measure 600 g of  $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$  in a beaker. In a 2 L beaker, slowly add the  $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$  to 500 – 700 mL of DIW (if making a batch of 2 L, then use a little over 1 L of DIW to dissolve 1200 g of  $\text{MnCl}_2$ ). Using a large metal scoop, put large scoops in at a time and let dissolve. Using a stir bar and stir plate, allow solution to become homogeneous. Rinse beaker with DIW to get all remaining chemical out of beaker and into solution (repeat 3 times). A foam hat will probably foam on top. The solution is endothermic. Transfer solution into 1 L volumetric flask. Rinse beaker with DIW three times to ensure no solution remains in beaker. After it reaches ambient temperature, bring to volume of 1L with DIW and stir. Using a coarse glass fiber filter to filter the solution, remove particulate material.

B. *Sodium iodide (4 M)/Sodium hydroxide (8 M)* (stable indefinitely when filtered and stored in sealed amber glass):

Weigh 320 g of NaOH into a beaker. Starting with about 500 mL of DIW in a 2 L beaker, slowly add scoops of NaOH. Using a stir bar and stir plate, allow solution to become homogeneous and clear. Rinse beaker with DIW to get all remaining chemical out of beaker and into solution (repeat 3 times). The solution is exothermic. Place in water bath (with a small amount of ice) to bring to room temperature. Weigh 600 g of NaI into a beaker. Slowly add scoops of NaI. Using a stir bar and stir plate, allow solution to become homogeneous. Rinse beaker with DIW to get all remaining chemical out of beaker and into solution (repeat 3 times). Transfer solution into 1 L volumetric flask. Rinse beaker with DIW three times to ensure no solution remains in beaker. Place in water bath (with small amount of ice) to bring to room temperature. Bring to volume of 1 L with DIW and stir. This may cause solution to get warm again. Meniscus is liquid beneath the foam. Place cap on and mix by hand. Using a coarse glass fiber filter to filter the solution, remove particulate material. The NaI/NaOH dispenser will require regular disassembling and cleaning because the plunger will stick/bind to the glass overtime.

C. *Sulfuric acid (5 M)* (stable indefinitely when sealed):

In a volumetric flask (placed in a tub of cold water), slowly add 280 mL of concentrated reagent grade  $\text{H}_2\text{SO}_4$  to 500 mL of DIW. **NEVER ADD WATER TO ACID. BE CAREFUL: This reaction will produce a lot of heat.** After the solution reaches ambient temperature, bring to volume of 1 L with DIW and stir.

D. *Thiosulfate* (stable for around two weeks – gets weaker over time so ensure standardizing often – keeping it stirring helps):

Mixing:

Weigh 17.5 g of dry thiosulfate pentahydrate in a scintillation vial to the tenth decimal place on Mettler Toledo 120 g analytical balance. Add around 250 mL of DIW to a 500 mL volumetric flask. Add the 17.5 g of thiosulfate to the volumetric flask. Rinse the scintillation vial with DIW to get all remaining chemical out of vial and into solution (repeat 3 times). Dissolve crystals fully mixing by hand. Bring to volume. Can mix by hand or with magnetic stir bar.

Storage:

Thiosulfate solution should be prepared 12-24 hrs before standardizing. Some believe it is more stable and consistent after 2-5 days (stored in proper conditions), but some believe the lifespan is ~ 5-6 days (stored in proper conditions). The solution should be stored in an amber glass bottle in a dark place to avoid degradation to light. It should remain in an amber glass bottle during use. The bottle cap should have a small hole in the top for the intake tube. It is recommended that the thiosulfate solution is consistently stirred using a stir bar and stir plate at a slow speed.

Concentration:

For a concentration of  $\sim 0.14$  M make 17.5 g thiosulfate pentahydrate/0.5 L of DIW.

e. *Potassium iodate* (0.00167 M or 0.0100 N) (stable 18 months – 3 years if stored out of direct sunlight, room temp, dry, well-ventilated – info from Osil email exchange)

### Filtering

Supplies:

- GF/F 47 mm glass microfiber filters (in drawer in O2 lab)
- pump (closet in engineering space or engineering team should have one as well)
- 1 L Erlenmeyer flask with spout on side (O2 lab)
- tube (O2 lab)
- filter cup (O2 lab)
- 500 mL beaker (waste)

Set up filter system. Ensure tubing isn't suctioning in on itself from a too strong vacuum pressure. If it is, adjust gauge. Filter a small amount so you can use that to rinse the flask with the reagent and dispose of that amount of reagent (repeat 3 times). Change filter. Add 100 – 150 mL of reagent into amber filter cup and allow it to filter. Change filters once it clogs up (normally around every 150 mL). Once filter has been used for one reagent, rinse it thoroughly to ensure no remnants are left when filtering the next reagent. Do this by running tap water through amber container and where filter sits and into flask (same set up but without white filter and not attached to vacuum) (refer to video Rachel sent me). Rinse with DIW (repeat 3 times). Reagents do not need to be filtered the day they are made.

