Water Sample Pretreatment

Fe Powder Preparation

@706: Prepare Fe powder (each tube 4±0.2mg) beforehand.
*Can make in bulk.
*See video/pdf "鉄粉の秤量"

Prereduction 予備還元 @AMS

Materials required:

- Weighed Fe powder
- PYREX quartz tubes (for new $Mg(ClO_4)_2$)
- Bottle of new $Mg(ClO_4)_2$
- Bottle of old $Mg(ClO_4)_2$
- Spatula
- Box with used PYREX quartz tubes
- Silicone caps

Procedures:

[When there are graphitized samples from previous experiments]

1. Make sure: 1) the end pressure is ~200hpa at the monitor; 2) heater is turned off
2. Mark "End press." Value on the graphitization card
3. Remove the heater. Write "Graphaization Code" & "Sample Name" on the quartz tube with graphitized sample.

Miya's water sample manual ☺
(based on Jian's awesome water sample manual ☺)

4. Take off the quartz tube (with sample), and cover it with silicone cap.
5. Replace the old Fe tube with the new one prepared beforehand
6. Remove PYREX tube with $Mg(ClO_4)_2$, replace with new tube and new $Mg(ClO_4)_2$ (2-3 pieces)
7. Use magnet to open valve (VB) with Fe
8. Heatgun $Mg(ClO_4)_2$ (~5s) to release water soaked inside (*pressure will rise to ~E^{-2} and then
9. Wait until pressure <5.0xE^{-3}
10. Open VA8 (vacuum the tube connected to H_2 can)
11. Close VA6 & VA10 when pressure goes down to <5.0xE^{-2}
12. Add 500hpa H_2 into each port
13. Close all valves (VB) after adding 500hpa H_2 (*VA8 can be either opened or closed)
14. Open VA6 & VA10 (*to vacuum remaining H_2 in the line)
15. Put on heater (*~1cm away from the metal part)
16. Turn on heater. 450° C; 1 hour. (2-3 hours also ok)

----------1 hour later----------

17. Turn off heaters
18. Cool down a bit
19. Use magnet to open VB (*to remove residual H_2)

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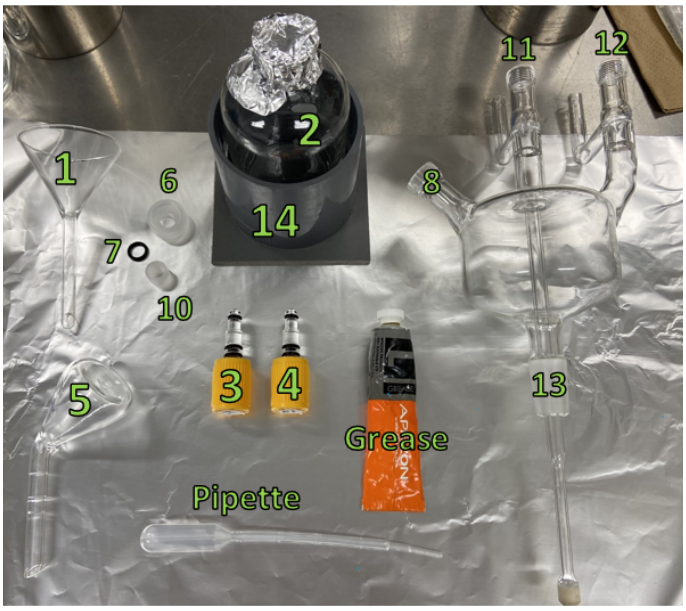
Water Sample Preparation

*Check "水サンプル精製【準備】水銀非含有試料(解説＆PDF あり)"

Materials required:



- 250 mL Sample Bottle (sample) in stand
- 250 mL Sample Bottle (empty)
- small beaker and stand (for excess water sample)
- dropper (to take out excess seawater in sample bottle)
- tweezer (in case O-ring falls off)

Bubbling
- Grease (APIEZON-L)
- Bubbling structure
- Yellow valve ×2
- O-ring set (2 medium + 1 small) ×2 (for yellow valves)

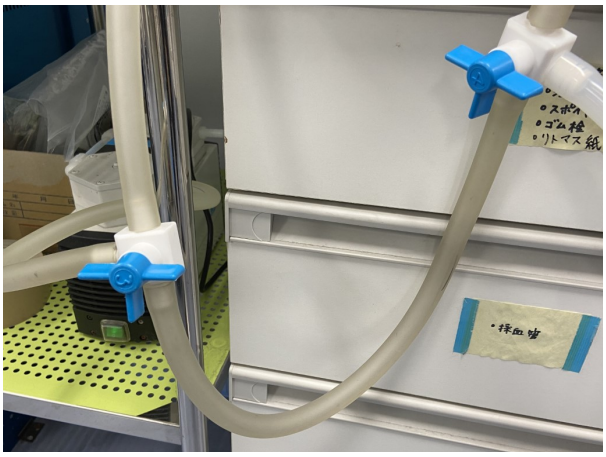
H_3PO
- Flask container for phosphoric acid (H_3PO_4)
- phosphoric acid (H_3PO_4) ($3H^+ + 3HCO^{3-} \leftrightarrow H_2CO_3^- \leftrightarrow CO_2 + H_2O$)
- dropper (for H_3PO_4)
- Big O-ring (for H_3PO_4)
- White ring (for H_3PO_4)
- White cover (for H_3PO_4)

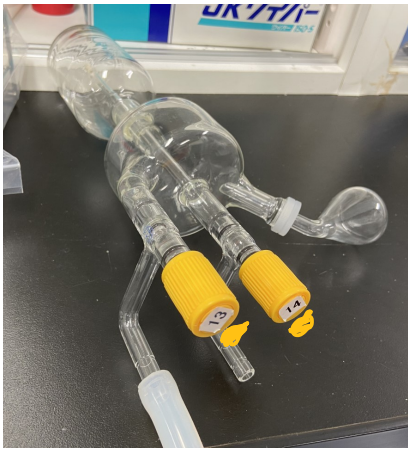
Procedures (adapted from Jian's water manual):

Outside tent

1. Place 7 in 8's opening followed by 10, 6, and 5
2. Grease 3 and 4 (NOT too much) and screw on 11 and 12, respectively

3. Grease 13 generously, placing dollops around the plug
4. Slide 2 onto 13 and twist to evenly apply grease
5. Take the entire structure and attach 12 to the vacuum. (*the way the vacuum dials should be as below)
   Turn the vacuum on.





6. Open 3 on 12 (yellow cap screw part)
7. Allow to vacuum for 30 seconds. The noise of the vacuum should become quieter the more time passes
8. Close 3 on 12 and turn the vacuum off. Remove from vacuum.
9. Place structure (lie flat) in the plastic tent along with water sample, phosphoric acid, a clean plastic pipette, 14 (, and 1
10. Seal the tent and hook up the Ar (g) tube to 15
11. Open Ar (g) tank valve to inflate the tent
12. Once full, close the tank valve
13. Open the seal on the plastic tent and remove the gas completely
14. Repeat steps 10-12 again (this ensures a clean atmosphere in the tent) *always do twice*
15. Slightly open the Ar (g) tank valve so just a bit of gas flows in, enough to keep the tent from deflating

Inside tent

16. Use the gloves attached to the plastic tent to do the following steps: Open yellow lid on 12 (or whichever valve is attached to the OUTER opening, NOT the middle one) and close it to fill it with Ar (g)
17. Remove 5 completely and pipette phosphoric inside (about 2 pipettes worth, ~2 mL). Set aside in a safe place

18. Remove 2 from the structure and place it in 14
19. Put 5 back on, positioned so acid does not enter the sample (pictured below)

20. Re-screw 6 so it is tight
21. Pipette some water sample out (~twice). This is to prevent water overflow when putting bubbling apparatus into the sample
22. Carefully put the bubbling apparatus into the water sample bottle
23. Open the tent and bring the structure to the machine. Position the structure so the yellow caps face towards you (or the opening in the middle is on the left, and the opening on the side is on the right)

Water Sample Line & Graphitization Line

See "14C_WaterPretreatment_Graphitization_20240807_Eng.pdf"