Pipette Procedures

Supplies

- 1 Liter cylinders (one per sample)
- 400mL beakers (one per sample)
- Aluminum weigh pans (two per sample)
- 20mL volumetric pipette
- #230 (0.0625 mm) sieve (stainless steel)
- 500mL squeeze bottle
- Large bowl with spout (1285mL evaporation dish is suggested)
- Large stirring rod with aerator end
- Small stirring rods for beakers
- Labeling tape
- Deionized water
- 10% $Na(PO_4)_x$ solution (100g $Na(PO_4)_x$ dissolved in 1000mL DI Water)
- Ultrasonic bath
- Clock that displays hours, minutes, and seconds

Safety Note: Be sure to use ear protection when using the ultrasonic bath.

Note: It is easiest to set up a run of 10 to 20 samples at a time.

Prep

1. Label the beakers and weigh (Since the samples are wet there is not really a need to weigh empty beakers unless you are interested in doing water content in which case the dried sample should NOT be used to pipette.). Know approximately what the beaker weighs and fill it to make a rough weight including ~ 20-40 grams of sediments. Coarser more sandy can be on the higher end and the more muddy samples on the lower end. Balance used can be like a triple beam balance. Label Sample ID's on the labeling tape and place on beakers.
2. With a pencil, label the inside bottom and the tabs of the weigh pans, one with 4Φ and one with 8Φ. Weigh these pans.
3. Set up cylinders in order. Place corresponding pans upside down with cylinders.
4. Label the 10cm and 20cm depths on the volumetric pipette.

Wet Sieving

1. Make sure sample is homogenized. If not, mix it. Do this for all the samples you have set up.Take a sarepresentitive sample of 20-40 grams wet weight. Very muddy samples should be in the 20-30g range and sandy ones in the 30-40 g range.

2. Add 10 or 50mL of the 10% $Na(PO_4)_x$ solution to each beaker and stir, being sure to note the prescribed amount (usually, the 50 ml is for samples with high mud content [>10% mud] to prevent flocculation).

3. Fill each beaker to the 250mL mark with deionized water. Stir with small stirring rod.

4. Place beakers 4 at a time into the ultrasonic bath which has been filled to between ⅓ and ½ with warm water. Process for 5 to 10 minutes stirring frequently (if there are still mud "clods" in the beaker continue ultrasonic and stirring a little longer). While wet sieving, replace the one you take from the ultrasonic bath with one that has not been processed (remember to continue to stir all of them in between the seiving).

5. Using a #230 (0.0625 mm) sieve designated for this purpose, wet sieve one sample at a time. This should be done carefully over a large bowl with a squeeze bottle of deionized water, without exceeding the use of 500mL of water, if possible.

6. After wet sieving, wash the sand to one end of the sieve with tap water. Take the sand portion left on the sieve and rinse it into the initial beaker. Alternatively, wash the sand into the filter paper folded into a funnel, let it drain, and then dry the filters. Place in the oven for drying at 65-75˚C being sure to remove labeling tape before placing in the oven.

7. Transfer the label to one of the cylinders. Take the portion of the sample that passed thru the sieve and pour it into the cylinder. Rinse the dish with deionized water into the cylinder. Fill cylinder to about 980mL with deionized water. Stir with large stirring rod. Rinse stirring rod into cylinder with deionized water and fill to 1000mL.

8. Rinse the empty sieve and dish with tap water.

9. Repeat steps until all samples have been wet sieved. Allow cylinders to set overnight before pipetting and check for flocculation.

10. After sands have dried, weigh and continue to dry sieving if desired.

Pipette Analysis

1. Check temperature of sample in cylinders and refer to the provided chart for proper time interval. Fill out the pipette data sheet for times.

2. Get the stirring rod and a cylinder filled with water and a pipette and another cylinder filled with deionized water.

3. For the first withdrawal, stir at least 20 seconds (more is better). Wait only 20 seconds; then take the withdrawal. You will withdraw 20mL of sample from a depth of 20cm, and place sample in the pans labeled 4Φ. Withdraw small amount of water from DI cylinder to rinse pipette and place this remainder in same pan. For simplification, separate the sample withdrawals by two minutes.

   Example:
   Looking at the minutes and seconds on the clock, start stirring the first cylinder NO LATER than 1:10. Stop stirring at 1:40 and take the first withdrawal at 2:00. For the next sample, stir at 3:10, stop at 3:40, withdraw at 4:00.

4. When the first round of the withdrawals (4Φ) are finished, place the pans in an oven to dry at approximately 65-75 ˚C.

5. Check the data sheet for the time of the second withdrawals. The 8Φ is NOT stirred. Take the 8Φ withdrawal of 20mL at a depth of 10cm and place in appropriate weigh pans. Remember to rinse the pipette as above. Place in the oven to dry.

6. Weigh in a humidity controlled room with a humidity of about 50%. After taking the pans out of the oven and into the controlled room, let them sit for at least 1 hour to come to equilibrium, and weigh.

To calculate amount of fines, subtract the weight of pan from the weight of pan and sample, then subtract the weight of the dispersant and multiply this by 50. This gives you the amount of 4Φ material and the amount of 8Φ material. To get the amount of silt, subtract the 8Φ from 4Φ. The 8Φ amount is the amount of clay.