

Sedigraph 5120 Procedure

The Sedigraph method of grain-size analysis consistently determines finer grain-size distribution than the Pipette Method; however, it is a poorer reflection of the actual particle size distribution of the sample. The best results are obtained when organic matter and salts have been removed from the sample.

The type of solutions used with the sample will determine what range of particle analysis can be achieved. If water or 0.05% Sodium Metaphosphate is used then, the maximum clast size that can be measured is approximately 56 microns. As such, the

sedigraph should only be used for samples or the fraction of samples that passes through a 63 micron (very-fine grained sand mesh).

To measure larger clast sizes, you need to use a 70% to 30% de-ionized water mixture, but that will increase the measuring time from 5 minutes to several hours for clay-sized sediment.

- x For sand-dominated sediment (> 95% sand) that are mica-rich, use the Mastersizer (laser particle analyzer) to measure grain size.
- x For sand-dominated sediment (> 95% sand) with high mica-content, sieve the sediment, and determine grain size by weight.
- x For muddy (> 5% mud < 95%) samples must be processed through both the Mastersizer and the Sedigraph and the results combined. For this, process one portion of the sample using the Sedigraph Procedure and a separate portion using the Mastersizer procedure. Combine the two results using 63 microns as the link point.
- x If you prefer, muddy samples can be processed by washing and sieving the sediment and processing the fine fraction through the Sedigraph (this will take longer though). See Shahin Dashtgard for procedure.
- x For mud-dominated sediment (> 95% mud), process the sample using the Sedigraph only.

Dispersing Fluid

- 9) Add 0.5 g of Sodium Metaphosphate (So-Met) to 1 L of de-ionized or distilled water (makes a 0.05% solution). Stir vigorously till all So-Met dissolves and let the solution sit for 24 hours.

Pre-Sieve and Removal of Organics

- 1) Pre-sieve your sample through a 63 micron (very-fine grained sand) mesh
- 2) Put 2 – 6 g of less than 63 micron material in a 100 mL (or bigger) beaker
 - a. More sample is needed the coarser it is. For example, a sandy sample requires 5 to 6 g, a coarse silt-dominated sample requires 3.5 g, and a clay dominated sample may only need 2 g. The amount of sample needed should be determined by visually inspecting the

source sediment. Prep 2 batches of ~~each~~ sample (double the amounts listed above) in case you run into problems ~~ensuring~~ the analysis.

- b. The amount of sample to fluid is key to ~~get~~ an accurate result. You need at least 40 mL to run the Sedigraph, and ~~is~~ easier to dilute a sample ~~later~~ than to increase the concentration *Err on the side of too much rather than too little.*
- 3) Add 10 mL of 30% ~~H₂O₂~~ to the sample. Do it slowly ~~to~~ make sure the bubbling doesn't go over the edge
- 4) Stir with a clean glass rod ~~(careful not to spill any)~~
- 5) Let the samples sit (supervised ~~for~~ half an hour to ensure ~~the~~ reaction won't boil over in your absence) for 24 hours.
- 6) Pipette off fluid (supernatant) ~~without~~ disturbing the sediment.
- 7) Repeat step 3-6 carefully. Record vigor of ~~the~~ reaction. If it is still very bubbly (means there is much organic matter left), ~~repeat~~ the process a third time.
- 8) Once bubbling has slowed to a negligible ~~rate~~, add 40 mL of 0.05% ~~Cap~~ solution to the sample. Stir with a clean glass rod to ~~susp~~ sediment. Store sample for transport.

Sedigraph Set Up

- 11) Remove the cover plate from ~~front~~ of the Sedigraph and ~~ensure~~ that the pumps are engaged and the tubing is not crimped. To do this, move ~~the~~ metal levers all the way to the right, such that the tubing fits tightly into the cavity ~~with~~ the pump. Make sure the metal levers are all the way down. Replace the cover plate and the mixing chamber cover.
- 12) Turn on battery back up (black box). Wait ~~until~~ the plug icon stops flashing and the "buzz" sounds ceases before proceeding.
- 13) Turn on sedigraph. (White power ~~switch~~ on right side of machine).
- 14) Turn on laptop. Log in with password: Summer2008
- 15) Open program on desktop.
- 16) Make sure green light ~~is~~ solid on sedigraph.
- 17) Ensure that the distilled water ~~rinse~~ container is full, and the waste container is empty. The rinse liquid may have to be placed ~~on~~ the bench top to initiate flow.
- 18) Let the Sedigraph sit for 30 minutes for the ~~machine~~ to reach its optimal operating temperature (~35 °C)
- 19) Click: Unit 1/Rinse/Sedigraph Make sure autorinse selected (rinses 3 times). Click Continue. (should take about 5 minutes)
- 20) Turn start key on front of Sedigraph to ~~turn~~ x-ray. Ensure red X-ray light is ~~lit~~ **Turn off machine if it doesn't light up.**
- 21) Collect baseline data.
 - a. Click Unit 1/Baseline Select, "Prepare to load baseline liquid" and make sure "leave mixing chamber empty" is ~~selected~~. While in this window, the kilocounts per second (kCnts/s) should be > 300. If it is "0" then ~~turn~~ the xray key to "standby", and then back to "on". Wait until the xray counts 300 kCnts/s before clicking ~~next~~.

- b. Load clear 0.05% So-Met solution into mixing chamber (about 2/3 full). Ensure that x-ray intensity is set at normal and the mixing chamber speed is 3**. Click Next. (should take 5 minutes or so)
- **You can use a mixing chamber speed up to 6 for coarser sediments, but it will increase the formation of bubbles. For clay, a mixing chamber speed of 2 is adequate; use 3 for silt, and 5 or 6 for sand.
- c. The "baseline" should be relatively straight and should be approxima

- b. Select a suitable lower limit. 0.98 microns can be run in 5 minutes, whereas 0.24 microns takes 20 minutes (in water). It takes much longer in glycerin-water mixtures.
 - c. Double check the sediment density. For sediments 2.65 g cm⁻³ is adequate. For the garnet standard, make sure it says 3.88 g cm⁻³. If you have density measurements for your samples, enter them here.
 - d. Click Save Click Close
 - e. You can either set up all sample files at the start, or you can do them throughout your analyses.
- 31) Click Unit 1/Sample Analysis Click Browse. Select file you just named. Click OK.
- 32) Select your preferred analysis conditions and the same file for the report conditions. Click Next.
- 33) Select "Leave mixing chamber empty" then click Start. Wait for prompt to load sample.
- 34) Once drained, ensure that the mixing chamber speed is set to 3 and load the sample into the Sedigraph mixing chamber. Use the squeeze bottle of 0.05% So-Met to get sediment out of the beaker, but do not add more than 2 ml additional So-Met to the solution.
- 35) After loading the sample, the machine will perform a full-scale scan. The scan line should be relatively straight and approximately 70 kcnts/s. If the counts are too high (the sample concentration is too low), then it will say low, red, in the bottom right hand corner. In this case, you will need to up the sample concentration. A wonky full-scale scan line will also introduce error and will require re-running samples.
- 36) Once samples are run, the machine will automatically save 3 times. After that save and close the graph. Then repeat steps 31-34 for successive samples.

Shutting Down the Sedigraph

- 37) Shutdown of the Sedigraph:
- a. Turn the x-ray key to the "off" position. DO NOT leave the x-ray lamp on when analyses are not being done.
 - b. Do one final rinse cycle. Algae can grow inside the system if water is left in the system.
 - c. Drain the unit of all liquid by clicking Unit 1/Drain and Load. After the system has drained, click cancel.
 - d. Shut down the x-ray and the computer, and make sure the work CLEAN!!
 - e. Remove the cover plate from the front of the Sedigraph and shift the metal levers to the