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TL Sample Preparation Procedure for Sediments (Fine Grain Centrifugation)

courtesy of Steve Forman, based on procedure used by Ann Wintle

- I. Carbonate Removal
 - A. Place 50-100g of sediment into 1000ml beaker.
 - B. Label beaker with sample number.
 - C. Add approximately 200ml of 10% HCl.
 - D. Let react for at least 4 hours.
 - E. Decant off HCl, careful not to include sediment.
 - F. Transfer sediment to centrifuge tubes and centrifuge for 1-3 minutes; pour off a clear supernatant.
 - G. Wash sediment with distilled water, centrifuge and decant a clear supernatant. Soak in reagent grade H₂O₂ overnight to remove organics.
 - H. Repeat step G two more times. In final washing use dispersant.

- II. Separation of 4-11 micron fraction.
 - A. Wash sample from centrifuge tubes through 63 micron mesh with dispersant, collecting the <63 micron fraction in a 1000ml beaker. Label beaker with sample number.
 - B. >63 micron fraction remaining on mesh should be dried at ca. 40 C and transferred to labeled vial and stored.
 - C. Make certain that the <63 micron fraction in the beaker is well dispersed. Ultrasonication may be necessary, or soak in dispersant for 24 hours.
 - D. Transfer <63 micron fraction from 1000ml beaker to 1000ml graduated cylinder; wash out beaker with dispersant.
 - E. Fill up graduated cylinder to 600ml with dispersant.
 - F. Place stopper on cylinder and turn over twice, to suspend all particle sizes.
 - G. Let stand for 30 minutes to separate the <11 micron fraction.
 - H. Decant the supernatant (<11 microns) into a clean 1000ml beaker, leaving the >11 micron fraction that has settled to the bottom of the cylinder. Wash >11 micron fraction from bottom of cylinder into beaker; dry at ca. 40C and store in labeled vial.
 - I. Repeat steps E, F, G, and H if necessary.
 - J. Transfer <11 micron solution to clean centrifuge tubes. Fill to the level of the black line on the tube.

- K. Load tubes into centrifuge in an opposite, balanced configuration.
- L. Centrifuge for 5 minutes and 30 seconds at 400RPM. Periodically check speed of centrifuge with calibration meter.
- M. Carefully decant the supernatant (the clay fraction) from centrifuge tube into the sink, making certain not to disturb the sediment (4-11 micron fraction) collected at the bottom of the tube.
- N. Rinse 4-11 micron fraction in tubes with methanol. Centrifuge for 1-3 minutes and then decant methanol. Centrifuge for 1-3 minutes and then decant off a clear supernatant. Repeat procedure three times.

III. Deposition of 4-11 micron fraction onto aluminum discs.

- A. Wash with methanol the 4-11 micron fraction from centrifuge tubes into a "medicine" bottle.
- B. Dilute mixture in medicine bottle with methanol until one's finger is barely visible viewed through the bottle, when held up to a red light (Place finger on back of bottle, not in the bottle!).
- C. Place aluminum discs in glass "shell" vials with a forceps, making certain that they are lying flat at the base of the vials. Also be sure that the discs are clean, not bent, or have burrs.
- D. Add a small volume (0.1ml) of methanol to vials containing discs. Tap down discs to bottom of vials with a glass stirring rod, making certain that there is not any methanol or air bubbles between the disc and the vial.
- E. Place clean plastic tip on 1 ml automatic pipettor.
- F. Shake or sonicate medicine bottle containing 4-11 micron mixture to insure suspension of mixture.
- G. Insert pipettor into medicine bottle and draw off 1 ml of 4-11 micron solution. Discharge aliquot into shell vial containing aluminum disc. Repeat this step for subsequent vials. Re-shake or re-sonicate every fifth draw.
- H. Examine vials to make certain that discs are flat lying within vials. If not, tap down with glass rod.
- I. Place vials with disc and mixture into ca. 40C oven overnight or until methanol has completely evaporated and sediment is plated onto discs.
- J. Remove discs from vials by turning over and tapping vial sides. Place in storage box with forceps.