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We use this protocol and it's working

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Particle size analysis by laser diffraction

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ABSTRACT

This method is a relatively rapid protocol for the determination of soil particle size analysis (texture) using laser diffraction. A critical step is to wet sieve the sand fraction prior to laser analysis.

MATERIALS

- Hamilton Beach HMD200 Drink Mixer
- Malvern Mastersizer 2000 laser diffractometer and Hydro 2000S (detection limit is 600um)
- 53-um sieve (3" diameter)
- 500-mL Nalgene bottles
- 125-mL wide-mouth Nalgene bottles
- Balance
- Pans for drying (smooth-walled metal tins)
- Oven
- Dry, well-mixed soil sieved to <2mm
- 10% sodium hexametaphosphate solution
- Deionized water
- Stir plate and stir bars
- Transfer pipettes (7-ml)
- Electric pipettor and 25-ml disposable pipette

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SAFETY WARNINGS

The mixing spindle spins at a high speed. Failure to wait for the mixing to stop before removing cup should be avoided to minimize the risk of accidental cuts.

Ear protection is recommended during long-term or chronic use of the mixer and/or laser.

Reagents

1 **Sodium hexametaphosphate (NaHMP) 10% solution**

- Chemical formula: $(NaPO_3)_6$
- Molar mass: 611.77 g/mol
- Procedure: Mix 100 g of NaHMP in 1000 mL of deionized water.

Materials

2 **Preparation**

- Dry, well-mixed soil sieved to <2mm
- 125-mL wide-mouth Nalgene bottles ("small bottles")
- 10% sodium hexametaphosphate solution
- Deionized water

Extraction

- 500-mL Nalgene bottles ("large bottles")
- Metal mixing cups and milkshake mixer (Hamilton Beach HMD 200 Drink Mixer)
- Large funnel
- 53um sieves (diameter of sieve = 3")
- Glass stir rod
- DI water (in squeeze bottles and plastic beakers)
- Smooth-walled metal tins
- Stir plate and stir rod
- Empty plastic beaker
- Electric pipettor with a plastic 25-mL pipette

Mixer, mixing cups, sieves, bottles, and funnel used in this analysis.

Laser analysis

- Stir plate and stir bars
- DI water in plastic beaker
- Empty plastic beaker
- 7-mL transfer pipette
- "Mastersizer 2000" laser analysis machine

Sample preparation

3 5m

1. Gather soil samples to be weighed. Samples should be dry and sieved to <2mm. Label the 125-mL wide-mouth Nalgene bottles ("small bottles") with the sample IDs.
2. Weigh 5 g of soil into the small bottle with the corresponding sample ID. Record mass to three decimal places.

> **Note**
>
> Before weighing, mix each sample well to ensure a representative result.

1. Add 50 mL of 10% sodium hexametaphosphate solution.
2. Add 50 mL of deionized water.
3. Cap small bottles and shake vigorously.
4. Allow prepared samples to soak Overnight
5. Label and weigh smooth-walled metal tins. Record mass to at least three decimal places and set aside to be used in extraction.

Sample extraction

4 Prepared samples are ready for extraction after they have soaked overnight.

1. Gather prepared samples ready for extraction. Label the 500-mL Nalgene bottles ("large bottles") with the sample IDs.
2. Transfer the prepared sample from its small bottle into a metal mixing cup.
3. Rinse any remaining soil from the small bottle and cap into the mixing cup. Using a plastic beaker containing DI water, fill the small bottle halfway, then cap it and shake vigorously. Pour the contents into the mixing cup. If needed, rinse again using a squeeze bottle. **Try to use as little water as possible.**

> Note
>
> All of the prepared sample must be transferred into the metal mixing cup. The small bottle should be rinsed into the mixer cup so that it is free of any remaining particles.

5 Place the metal mixing cup into the milkshake mixer on the upper position of the cup rest, which will activate the mixer. Mix the sample for 00:05:00 on low speed. 5m

- While sample is mixing, transfer other samples ready for extraction into remaining metal mixing cups. Keep the labeled small and large bottles close to the unlabeled metal mixing cups that hold the corresponding samples.

1. After 5 minutes, stop the mixing action by moving cup to the lower position of the cup rest. Once the mixer has stopped, remove the newly mixed sample from the milkshake machine. Rinse spindle as needed to remove any adhering soil particles. **Place the next remaining unmixed metal mixing cup into the milkshake machine and begin agitating.**
2. Gather a large funnel and 53um sieve. Place sieve into funnel and arrange above the large bottle labeled with the sample ID that corresponds with the newly mixed sample.

Funnel and sieve set up for separating sand from the silt+clay fraction.

3. Slowly pour mixed sample into sieve. If necessary, gently stir the liquid in the sieve with a glass stir rod to help the solution pass through.
4. Using a DI water squeeze bottle, rinse all remaining soil from the metal mixing cup into the sieve.

Note

All of the mixed sample must be transferred into the sieve. The metal mixing cup should be rinsed into the sieve so that it is free of any remaining particles.

6 Sand particles larger than 53 µm will remain on top of the sieve.

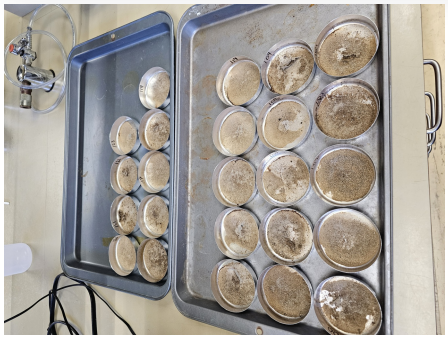
1. Using a squeeze bottle, rinse the sieve with DI water until all fine particles have passed through. Gently tilt the sieve back and forth, moving the particles across the top as you rinse them. Use the glass stir rod or small rubber spatula to ensure no fine particles remain. **All fine particles have passed through the sieve when the water that exits the sieve is clear no matter which way you tilt it.** Be careful to not exceed the large bottle maximum volume.
2. Gather a labeled smooth-walled tin. Arrange the funnel and sieve over the tin. Flip the sieve over to transfer the particles larger than 53um into the tin through the funnel. Rinse the sieve and funnel until all remaining particles are in the tin. **Try to use as little water as possible.**
3. Place tin on an oven tray. When the tray is filled with tins, put it into the oven at 105 °C.

- When all water has evaporated, cool the tins. Weigh and record mass to three decimal places. Tins may require 4-8 hours to fully dry.

Cap the large bottle, gather the corresponding small bottle, and set both aside for the following steps.

Note

If working alone:
Once half of the prepared samples have been mixed, processed through the sieve, and separated into large bottles + tins, pause milkshake mixing and proceed to the following steps.
If working in a group:
The following steps can be done concurrently with the milkshake mixing.



Soil particles > 53µm (i.e., sand particles) in drying tins (following oven drying).

7 Samples inside the large bottles must now be transferred back into the corresponding small bottles.

1. Assemble a stir plate, stir bar, empty beaker, and DI water beaker into a comfortable

arrangement. Gather the labeled pairs of small and large bottles that had been set aside.

2. Drop the stir bar into a large bottle and place on stir plate. Stir on medium speed for at least 20 seconds, ensuring the formation of a visible vortex.
3. Using the 25-mL electric pipette, transfer approximately 100 mL from the labeled large bottle into the correspondingly labeled small bottle. Rinse the pipette tip with DI water between different samples.

- This volume does not need to be exact: fill the small bottle until it is almost full. The tip of the electric pipette should be located approximately halfway into bottle when extracting to ensure a representative sample.

Cap the small bottles and set aside on a plastic tray. Deliver the plastic tray with small bottles upstairs to be analyzed with the laser.

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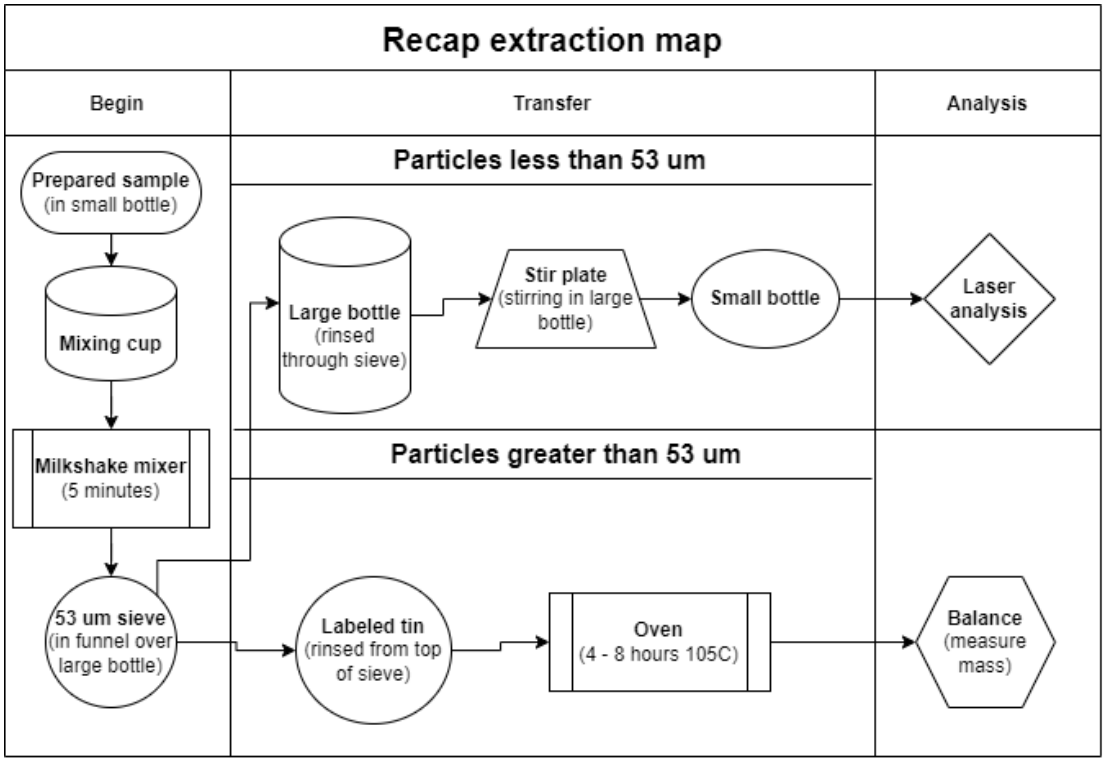

Recap extraction map:
Prepared sample in small bottle-> Unmixed in mixing cup-> Mixed in mixing cup-> 53um sieve

Soil particles **less than 53um**-> rinsed through sieve into large bottle-> stirred and put into small bottle-> laser analysis.
Soil particles **greater than 53um**-> left on top of sieve-> rinsed into tin-> oven dried and weighed.

Laser analysis SOP settings

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Note

These settings should be already contained with the appropriate SOP. Refer to this section if making a new SOP or troubleshooting SOP issues.

The appropriate SOP (currently file "20230825_soil.sop") has the following settings:

- Stirring speed is 3500 rpm.
- Sonication power is 100% and runs for 60 seconds pre-measurement.
- Mode is MIE scattering general purpose model for quartz material with **non-spherical shape**.
- Particle **refractive** index is 1.55
- Particle **absorption** index is 0.1
- **Water** refractive index is 1.33
- Density is 2.65g/cm3
- Laser obscuration is default settings: 10-20%

Note

The sample concentration is controlled by monitoring the obscuration of the laser beam caused by the sample. The obscuration is simply the fraction of light “lost” from the main beam when the sample is introduced. For example, an obscuration of 30% means that 30% of the incident laser beam (recorded during the Measure Background step) has been lost through scattering or absorption.

- **Background** measurement duration is 15 sec
- **Sample** measurement duration is 30 sec
- Level sensor threshold is 64%, which is the default value (for water)
- Degas is checked
- Align is checked
- User defined sizes are selected as follows: 0.01 um, 6.325 um, 56.368 um, 2000 um. These are the particle size cut offs for clay (0.02-6 um), silt (6-56 um), and sand (56-2000 um).

Laser analysis measurement

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Now that the samples have been fully separated and the sand-sized particles have been extracted, the proportion of clay and silt-sized particles in the sample can be analyzed by the laser.

Note

Before beginning the first laser measurements of the day:

- Switch on the optical bench and hydro 2000S units. Leave them powered on for at least 30 minutes before making a measurement to allow the temperature to stabilize.
- Gather a stir plate, seven stir bars, and one 7-mL disposable transfer pipette. Arrange samples neatly near the stir plate.
- Uncap seven samples and drop a stir bar into each uncapped sample. Place one sample on the stir plate.
- Open the "Mastersizer 2000" computer program.
- Clean the laser by clicking **Configure->Accessories->Clean (4 cycles)**
- Start the SOP by clicking **Measure->Start SOP->"20230825_soil.sop"** (or choose the appropriate SOP)

Make sure that a sample containing a stir bar is stirring on the stir plate at medium speed.

1. Once the SOP begins, it will ask for a sample ID. Enter the sample ID that corresponds with the sample that is on the stir plate. The SOP will then clean the tank, align the laser, and measure background light.
2. When the instrument is ready for sample to be added, the computer will indicate "Auto-Dilution is in progress... Please add sample". A soft beeping noise will also sound aloud.
3. Using a 7-mL transfer pipette, dispense the sample into the tank of the machine. Dispense enough sample to reach an obscuration level between 10-20% (approximately 14 mL).
4. The instrument will automatically begin to measure the sample. The process lasts for a duration of approximately 4 minutes.

Note

To ensure a representative sample, place the tip of the pipette **halfway** into the sample while it is **stirring**. Rinse the pipette tip with DI water between different samples.

While the instrument is measuring,

- Remove the sample from the stir plate. Remove the stir bar, then cap and place on a plastic tray to be discarded later.
- Place the next sample containing a stir bar on the stir plate and begin spinning at a medium speed.
- Clean the iron shavings from the used stir bar. When the stir bar is clean, uncap a new sample and drop the stir bar inside.

After the measurement is complete, the computer will ask if the SOP should be run again. Click "Yes". Enter the correct sample ID which corresponds with the next sample that is on the stir plate.

Note

After the measurement is complete, double-click on the cell that says "(sample-ID)-Average". Record the percent volume for clay (0.010um to 6.325um), silt (6.325um to 56.368um), and sand (56.368um to 2000um) in a properly labeled spreadsheet. This spreadsheet should be on a computer connected to internet and BOX.

At the end of each day, print the results which were added to the spreadsheet that day.

Calculations

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1. Sand1 fraction (f_{sa1}) = Total dried soil sample (M_T) - Mass of sand particles following sieving and drying (M_{sa})/M_T.
2. Clay fraction (f_{cl}) = [(M_T - M_{sa}) x $f_{cl\text{-}LD}$]/M_T
3. Silt fraction (f_{si}) = [(M_T - M_{sa}) x $f_{si\text{-}LD}$]/ M_T
4. NOTE: there is some fine sands that can be in the silt + clay fraction. Add the correct proportion from the dried sand fraction to the sand fraction from the laser.
5. Sand2 (laser) fraction (f_{sa2}) = [(M_T - M_{sa}) x $f_{sa\text{-}LD}$]/ M_T
6. Total sand fraction (f_{Tsa}) = f_{sa1} + f_{sa2}

$f_{cl\text{-}LD}$ = Laser fraction cumulative volume of particles less than a clay-silt cutoff of 6.325µm.

$f_{si\text{-}LD}$ = Laser fraction cumulative volume of particles greater than the clay-silt cutoff.

f_{sa2} = Laser fraction cumulative volume of particles greater than a sand-silt cutoff of 56.368µm.

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Website for soil textural classification (calculator)

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Plot_On_Triangle.xls

The attached spreadsheet can be used to generate a soil texture triangle plotting all samples in a given set.

Clean up

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Always leave your workspace clean at the end of the day!
At the end of the day, wipe down benches, balances, and all instrumentation.
Turn off balance.
Log out and close all websites and any data sheets.
The mixer should be wiped down and completely cleaned each day.

The laser and hydro 2000S should be turned off at the end of each run.