

2.11: Finding a Representative Lithology

From sediment to sample

Sample sediments are typically sent in a large plastic bag inside a brown paper bag labeled with the company or organization name, drill site name and number, and the depth the sediment was taken (in meters).

The first step in determining a lithology is to prepare a sample from your bulk sediment. To do this, you will need to crush some of the bulk rocks of your sediment into finer grains (Figure 2.11.1). You will need a hard surface, a hammer or mallet, and your sediment. An improvised container such as the cardboard one shown in Figure 2.11.2 may be useful in containing fragments that try to escape the hard surface during vigorous hammering. Remove the plastic sediment bag from the brown mailer bag. Empty approximately 10-20 g of bulk sediment onto the hard surface. Repeatedly strike the larger rock sized portions of the sediment until the larger units are broken into grains that are approximately the size of a grain of rice.



Figure 2.11.1 A hammer and hard surface for crushing. The makeshift cardboard shield on the left can be placed around the hard surface to control fragmentation.

Some samples will give off oily or noxious odors when crushed. This is because of trapped hydrocarbons or sulfurous compounds and is normal. The next step in the process, washing, will take care of these impurities and the smell.

Once the sample has been appropriately crushed on the macro scale, a micro uniformity in grain size can be achieved through the use of a pulverizing micro mill machine such as the Planetary Mills Pulverisette 7 in Figure 2.11.2



Figure 2.11.2 A Pulsette micro mill, milling cup removed. The mill is set to 520 rotations per minute and a five minute run time.

To use the mill, load your crushed sample into the milling cup (Figure 2.11.3) along with milling stones of 15 mm diameter. Set your rotational speed and time using the machine interface. A speed of 500-600 rpm and mill time of 3-5 minutes is suggested. Using higher speeds or longer times can result in loss of sample as dust. Load the milling cup into the mill and press start; make

sure to lower the mill hood. Once the mill has completed its cycle, retrieve the sample and dump it into a plastic cup labelled with the drill site name and depth in order to prepare it for washing. Be sure to wash and dry the mill cup and mill stones between samples if multiple samples are being tested.



Figure 2.11.3 A milling cup with mill stones and the crushed sample before milling.

Washing the Sample

If your sample is dirty, as in contaminated with hydrocarbons such as crude oil, it will need to be washed. To wash your sample you will need your sample cup, a washbasin, a spoon, a 150-300 μm sieve, household dish detergent, and a porcelain ramekin if a drying oven is available (Figure 2.11.4).



Figure 2.11.4 A washbasin, with detergent in a squirt bottle and the sample in a cup for washing (sieve not pictured).

Take your sample cup to the wash basin and fill the cup halfway with water, adding a squirt of dish detergent. Vigorously stir the cup with the spoon for 20 seconds, ensuring each grain is coated with the detergent water. Pour your sample into the sieve and turn on the faucet. Run water over the sample to allow the detergent and dust particles to wash through the sieve. Continue to wash the sample this way until all the detergent is washed from the sample. Once clean, empty the sieve onto a surface to leave to dry overnight, or into a ramekin if a drying oven is available. Place ramekin into drying oven set to at least 100 $^{\circ}\text{C}$ for a minimum of 2 hours to allow thorough drying (Figure 2.11.5). Once dry, the sample is ready to be picked.



Figure 2.11.5 A drying oven with temperature set above the temperature of evaporation for water at 105 $^{\circ}\text{C}$

Picking the Sample

Picking the sample is arguably the most important step in determining the lithology (Figure 2.11.6).

During this step you will create a sample uniformity to eliminate random minerals, macro contaminants such as wood, and dropstones that dropped into your sediment depth when the sediment was drilled. You will also be able to get a general judgment as to the lithology after picking, though further analysis is needed if chemical composition is desired. Remove sample from drying oven. Take a piece of weighing paper and weigh out 5-10 g of sample. Use a light microscope to determine whether most of the sample is either silt, clay, silty-clay, or sand.

- Clay grains will have a gray coloration with large flat sub-surfaces and less angulation. Clay will easily deform under pressure from forceps.
- Silt grains will be darker than clay and will have specks that shine when the grain is rotated. Texture is long pieces with jagged edges. Silt is harder in consistency.
- Silty clay is a heterogenous mixture (half and half mixture) of the above.
- Sand is defined as larger grain size, lighter and varied coloration, and many crystalline substructures. Sand is hard to deform with the forceps.



Figure 2.11.6 A light microscope being used to 'pick' the sample. The sample is being separated according to the dominant lithology in preparation for chemical analysis.

Pelleting the Sample

To prepare your sample for X-ray fluorescence (XRF) analysis you will need to prepare a sample pellet. To pellet your sample you will need a mortar and pestle, pellet binder such as Cereox, a scapula to remove binder, a micro scale, a pellet press with housing, and a pellet tin cup. Measure out and pour 2-4 g of sample into your mortar. Measure out and add 50% of your sample weight of pellet binder. For example, if your sample weight was 2 g, add 1 g of binder. Grind the sample into a fine, uniform powder, ensuring that all of the binder is thoroughly mixed with the sample (Figure 2.11.7).



Figure 2.11.7 A mortar and pestle being used to grind the sample to a powder for pelleting in the pellet press. A binding agent (Cereox) is also added using the scapula.

Drop a sample of tin foil into the press housing. Pour sample into the tin foil, and then gently tap the housing against a hard surface two to three times to ensure sample settles into the tin. Place the top press disk into the channel. Place the press housing into the press, oriented directly under the pressing arm. Crank the lever on the press until the pressure gauge reads 15 tons (Figure 2.11.8). Wait for one minute, then twist the pressure release valve and remove the press housing from the press. Reverse the press and apply the removal cap to the bottom of the press. Place the housing into the press bottom side up and manually apply pressure by turning the crank on top of the press until the sample pops out of the housing. Retrieve the pelleted sample (Figure 2.11.9). The pelleted sample is now ready for X-ray fluorescence analysis (XRF).

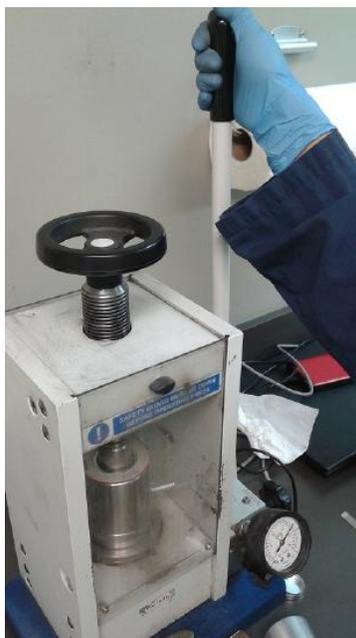


Figure 2.11.8 A pellet press being pressurized to 15 tons.



Figure 2.11.9 A completed pellet after pressing.

XRF Analysis

Place the sample pellet into the XRF (Figure 2.11.10 and Figure 2.11.11) and close the XRF hood. The XRF obtains the spectrum from the associated computer.



Figure 2.11.10 A Spectro XEPOS X-Ray fluorescence spectrometer.



Figure 2.11.11 The inside of the spectrometer where the sample pellets are placed for analysis

The XRF spectrum is a plot of energy and intensity. The software equipped with the XRF will be pre-programmed to recognize the characteristic energies associated with the X-ray emissions of the elements. The XRF functions by shooting a beam of high energy photons that are absorbed by the atoms of the sample. The inner shell electrons of sample atoms are ejected. This leaves the atom in an excited state, with a vacancy in the inner shell. Outer shell electrons then fall into the vacancy, emitting photons with energy equal to the energy difference between these two energy levels. Each element has a unique set of energy levels, therefore each element emits a pattern of X-rays characteristic of that element. The intensity of these characteristic X-rays increases with the concentration of the corresponding element leading to higher counts and higher peaks on the spectrum (Figure 2.11.12).

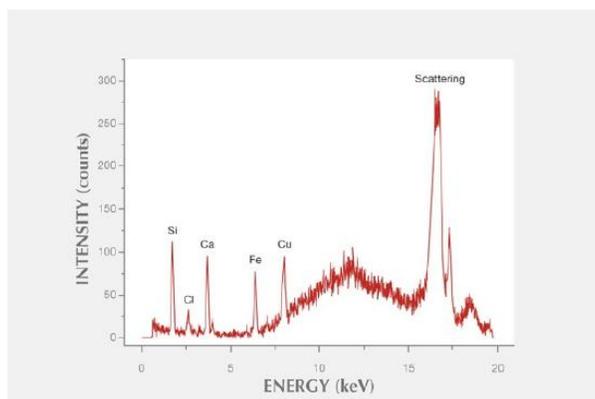


Figure 2.11.12 The XRF spectrum showing the chemical composition of the sample.

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