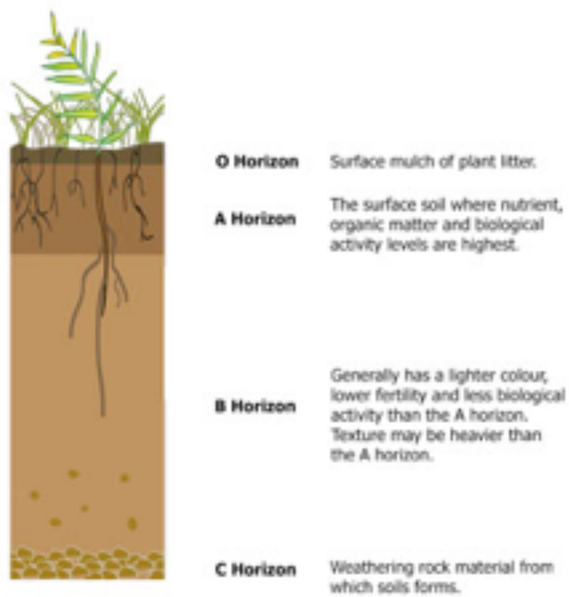
ENVS2001/2014 LABORATORY #4
SOIL ANALYSIS

INTRODUCTION

The chemical and physical properties of soil are vital characteristics which influence the plant and animal communities that grow within it. Many human factors alter the abundance and chemistry of soil, threatening future agriculture and terrestrial biodiversity. From a plant's perspective, soils contain several factors vital for healthy growth, including nitrogen (N), phosphorus (P), and organic carbon (C). We will measure carbon and phosphorus this time, nitrogen later.

Sample	% moisture	pH	$[PO_4^{2-}]$ (mg L^{-1})	% sand	% silt	% clay	% organic	notes
Cheung Chau								
Lung Fu Shan								
Potting				x	x	x		
Compost				x	x	x		



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<u>Field Sampling</u> for soils is varied and depends on the soil composition and the purpose for sampling. Pit cores can be sampled simply by careful digging with a shovel or a motorized post-hole digger. More precise sampling (for analyzing soil layers) can be taken using a variety of soil augers and probes. Depending on the application, care must be taken when removing the core and preparing the sample for transport and storage.

1) measure soil temperature as a function of depth using the **digital temperature probe**
2) practice taking a small soil core using the **soil probe**.
3) observe how a large soil core can be taken using the **auger**.
4) homogenize a subsample of soil from each of the samples provided (see page 1) using a **mortar & pestle**
5) sieve soil samples using the **sieve shaker** in the laboratory.




mortar & pestle (left), soil push probe (left middle), a “russian” auger (right middle), and a soil sieve shaker (right).

Soil Extraction is a fundamental procedure for the study of soil chemistry. Briefly, soil particles are generally charged and attract positive and negative ions (cations and anions). Clays tend to be negatively charged and thus strongly bind cations (like Ca, Mg, K) while organic matter tends to have mixed charges and thus bind all ions depending on it's composition. Plants can secrete ions (like H^+) to "push" other ions off of soil particles. If the pH is slightly acidic, this process is easier for plants. We can analyze ions by forcing ions off using strong ionic solutions. Today you will prepare a soil extraction using 0.5M sodium bicarbonate to measure soil phosphate using the ascorbic acid method as previously conducted on water.

Soil Moisture (this has been done for you) see data on last page and complete the calculations.

1) Dry one 10g subsample (field moist) in oven for 48 hours @ 105°C
2) return to re-weigh the oven-dry sample.
3) calculate % moisture using:

% moisture = (field moist mass - dry mass)/field moist mass

Extracting Soil - $NaHCO_3$

Each team will extract 2 samples. Please refer to the whiteboard for your assignment.

4) Weigh out a 5g subsample of **field moist soil** in a flask. With a graduated cylinder measure 100 mL 0.5M $NaHCO_3$ and add to soil. cover with parafilm.
5) Shake for 30 min at 200 rpm.
6) Allow samples to settle after shaking.
7) filter suspension through a 9cm filter folded into a funnel placed atop of an empty labeled 50mL falcon tube. collect about **10mL** of extract.
8) extracts can be stored frozen.

A filtration system for preparing multiple extracts simultaneously.

Soil Phosphate (PO_4^{2-}) limits terrestrial plant productivity and is vital for essential plant functions including ATP cycling (energy), photosynthesis (reductants like NADP), sugar cycling, and DNA synthesis. Lack of P reduces leaf and root growth and extension. Again, we will measure soluble reactive phosphate (SRP or orthophosphate = dissolved inorganic phosphate), which is directly taken up by plant cells.

Each team will extract 2 samples and measure 4 standards. Please refer to the whiteboard for your assignments.

1) create a combined reagent <u>in the following order</u> with mixing after each addition:

- **i.** 5mL H_2SO_4 (5N)
- **ii.** 0.5mL potassium antimonyl tartrate
- **iii.** 1.5mL ammonium molybdate
- **iv.** 3mL ascorbic acid

2) all solutions should be at room temperature before proceeding.

3) pipette 5mL of each standard and soil extract** into a 15mL “Falcon” tube (see table to the right)

4) add 0.8mL combined reagent to each tube. Screw on cap and vortex.

5) wait 10 minutes but no more than 30 minutes to measure

6) pour 2 mL of each standard and soil extract into the cuvette and record the absorbance at **880 nm**. Make sure the mark on the cuvette is lined up with the mark on the spectrophotometer. Rinse cuvette with DI water between each sample.

** Recall that the soil extract was made by diluting 5g soil in 100mL $NaHCO_3$ solution**

9) calculate the equation of the line of best fit from the standard curve **using the laptop**. (Absorbance = slope * concentration + intercept.

10) using the absorbance values from your extracts, calculate their concentration based on the best fit line.

Concentration ($mg\ L^{-1}$)	**Absorbance (880)**	**Extract**	**Absorbance (880)**	**Concentration ($ug\ L^{-1}$)**
0		Lamma		
10		LFS		
20		Potting		
40		Compost		

Calculate soil [PO_4^{3-}] in mg P g^{-1} soil:

<u>Soil pH</u> is critical for plant growth as the relative acidity/alkalinity of the soil affects the abundance of dissolved free ions in water surrounding soil particles. relatively acidic soils contain more free ions and thus more nutrients available for plant growth. Use of the pH probe is as before (instructions below). This time you will stir the probe in a beaker with the soil solution to improve the precision of our analysis. The probe is calibrated to pH 4 and 7.

<u>You will measure pH of all 4 samples.</u>

1) add 10g of each soil sample to 10mL DI water in a small beaker. stir. (you do not need to sit at the probe to do this, allow other groups to use the probe too!)
2) allow the solution to equilibrate for 15min. stir.
3) carefully remove the pH probe from the storage solution by first unscrewing the cap, removing the bottle, and then sliding the cap off the probe. GENTLY! **DO NOT YANK THE STORAGE BOTTLE OFF THE PROBE OR YOU MAY BREAK IT!**
4) open the blue vent on the side of the probe.
5) rinse the probe with DI water
6) Use the pH probe to measure the pH of two standards pH 4.0 and pH 7.0 **while stirring**. **rinse the probe between each measurement** with DI water.
7) if the standards do not match the pH reading, please inform your demonstrators.
8) if the readings match, Record pH for each soil solution, while stirring and **rinse the probe between each measurement** with DI water.
9) rinse and store the pH probe in the provided storage solution bottle. First, slide on the cap and o-ring with the threads facing down. Then insert the probe into the bottle's solution. Bring the cap down and screw on gently.
10) replace the vent cap

Figure 1. Soil pH and Plant Growth

Soil Reaction	pH	Plant Growth
	>8.3	Too alkaline for most plants
	7.5	Iron availability becomes a problem on alkaline soils.
Alkaline soil	**7.2**	6.8 to 7.2 – "near neutral"
Neutral soil	**7.0**	6.0 to 7.5 – acceptable for most plants
Acid soil	**6.8**	
	6.0	
	5.5	Reduced soil microbial activity
	<4.6	Too acid for most plants

% Organic content is calculated by mass difference. A mass of soil is weighed and then combusted in a **muffle furnace.** Unlike a drying oven which we've used for drying filters and incubating microbes, a muffle furnace is capable of much higher temperatures (>500 °C). As such, the muffle furnace is a suitable tool for removing organic matter via burning. For example, the GF/F filters we used in the water quality laboratory were pre-combusted in the muffle furnace to remove organic matter contamination.

This has been done for you to save time. Please complete the calculations on the last page.

1) pre-weigh a ceramic crucible. to this add 1g oven-dried soil of any type
2) label the crucible on the bottom with pencil (marker will burn off in the furnace).
3) carefully place in the muffle furnace.
4) combust at 500ºC for at least 6 hrs (this will be done for you).
5) move samples from the furnace to a low humidity dessicator to avoid water re-absorption.
6) in 24 hrs, re-weigh the crucible and subtract the initial mass from the final mass to determine the mass of material remaining. the "ashed" sample now contains only the inorganic fraction of the soil sample.
7) calculate % organic matter using the equation below.

A common muffle furnace. Note the thick insulating layer on the inside to prevent overheating of the exterior

% organic matter = (oven dry weight - ash free dry weight) / oven dry weight

Soil texture is an important characteristic that is altered by the proportion of sand, silt, and clay. Clays are the smallest particle size, and although they have a high surface area : volume they can compact to the point that limits water, oxygen, and nutrient availability. On the other end of the spectrum, sand does a poor job of retaining water and is prone to low moisture.

NOTE: Calibrate hydrometer in soil-free solution

1) add 50g of sieved soil to a 1L beaker containing 2g of the detergent sodium hexametaphosphate*, mix
2) add 500mL of DI water to the beaker
3) mix for 5-10 minutes using magnetic stir bar and plate
4) remove stir bar with magnetic wand
5) rinse all the solution and particles into a 1000mL graduated cylinder. top up with DI water to 1000mL
6) cap the cylinder and invert several times to mix.
7) remove the cap and immediately add the soil hydrometer and record the hydrometer value (g soil L^{-1}) at exactly 40 seconds
8) remove and clean the hydrometer, record the water temperature using a digital thermometer
9) after 2hr repeat both measurements (hydro. & temp)
10) correct the hydrometer readings for temperature by adding (or subtracting) 0.36 g L^{-1} per degree above (or below) 20ºC.

40 sec reading __________ **corrected:**_____________

2 hr reading __________ **corrected:**_____________

*** this is a common detergent and acts to separate the soil particles and prevent clumping in solution. It has been added to your beaker already.**

A soil texture triangle assists with classifying soil type.

CALCULATE THE SOIL TEXTURE USING THE EQUATIONS BELOW:

Oven-dry wt. = Air-dry wt./(1 + decimal of % H_2O*) = _______________ (*ex: if soil is 2.2% H_2O use 1.022)
(OPTIONAL, NOT REQUIRED IF SOIL IS PRE-DRIED)

Grams of sand = oven-dry wt. - corrected 40 sec. reading = ________________

Grams of silt + clay = corrected 40 sec. reading. = _______________

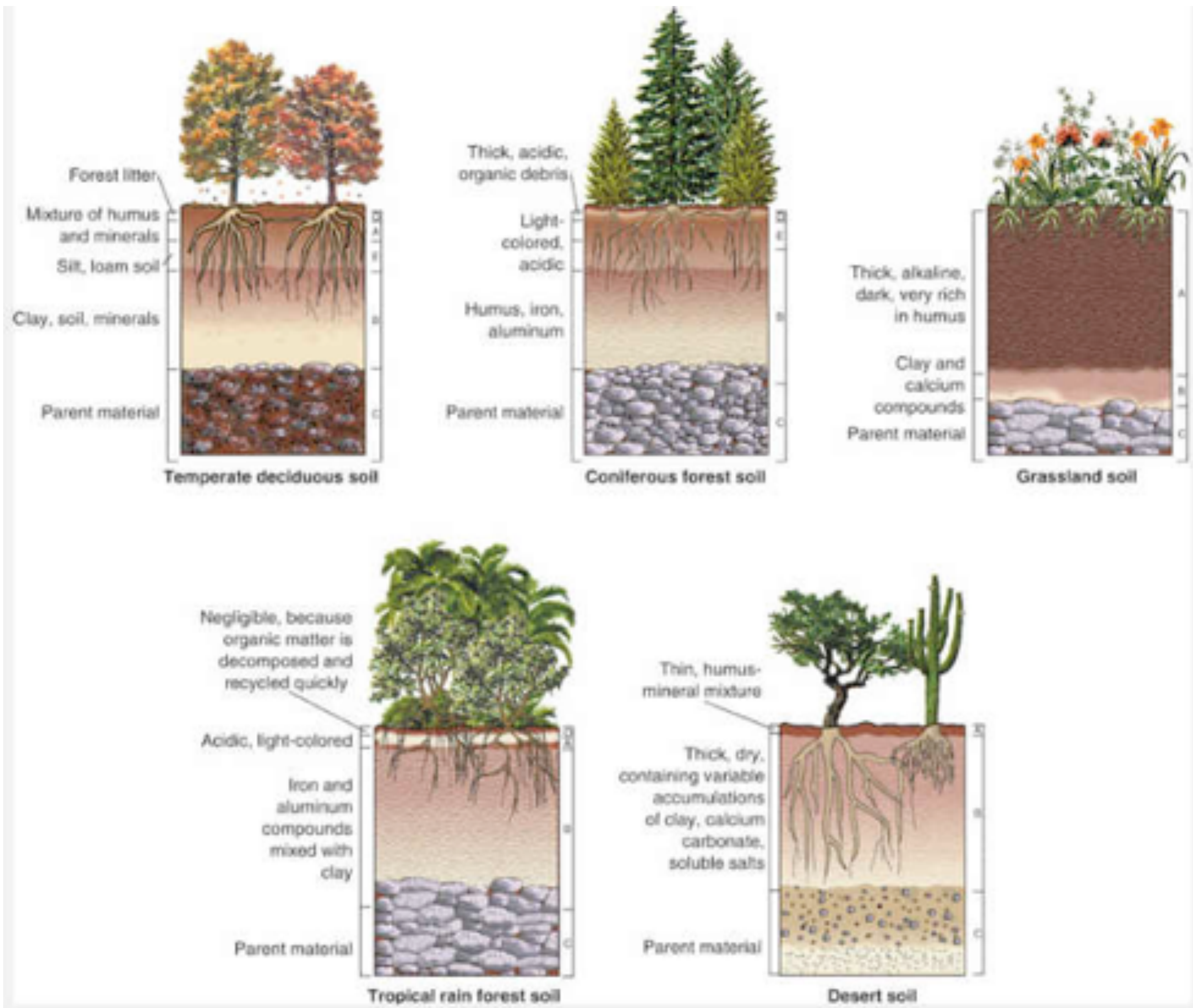
Grams of clay = corrected 2 hr. reading. = _______________

Grams of silt = corrected 40 sec. reading - corrected 2 hr. reading. = _______________

% sand = (grams sand/oven-dry wt.) x 100 % silt = (grams, silt/oven-dry wt.) x 100 % clay = (grams clay/oven-dry wt. x 100 = _______________

Use the texture triangle to determine the texture of your soil sample. = ______________________________

Although we cannot demonstrate it effectively in Hong Kong, soil profiles are an important aspect of terrestrial ecology. The depth of each layer can influence the abundance and biodiversity of plant and animal species inhabiting surface and sub-surface environments. See below for some examples of how soil horizons vary across different biomes. **Which one do you think most closely represents Hong Kong? Why?**



ENVS2001: SOIL ANALYSIS POST LAB REVIEW (8 pts)

1) Why didn't we analyze the soil texture of potting soil and compost?

2) Describe the texture and composition of Hong Kong soil. Based on this information, is the soil good for agriculture? What properties of compost and potting soil make it more suitable for growing plants?

3) Following the previous page, based on what you know about Hong Kong climate and topography, what type of soil do you think is most common in Hong Kong?

4) What was the average phosphate concentration of each soil sample? Why do you think some soil types have more phosphate than others?

NAME:______________________________ **DATE:____________________**

% MOISTURE CALCULATION WORKSHEET

Table 1. ~100g of field moist soil was weighed and dried overnight at 104ºC. The initial and final mass are given.

sample	initial mass (g)	final dry mass (g)	difference (g)	% moisture
compost	10.03	5.54		
lung fu shan	10.36	9.01		
cheung chau	10.04	9.50		
potting soil	7.14	2.87		

% ORGANIC WORKSHEET

Table 2. A few grams of **field moist** soil were placed in a crucible and combusted at 500ºC. The remaining mass of soil represents the inorganic fraction. Calculate the %organic material by determining the amount of soil mass lost during combustion. **You must also factor in the %moisture content!**

sample	initial (g)	final (g)	% organic
compost	1.175	0.073	
lung fu shan	1.330	1.325	
cheung chau	1.123	1.025	
potting soil	1.267	0.102	