

Supporting Document 1: Risk Assessment compiled for particle size analysis. Had this method been carried out in our own laboratories, this risk assessment would have been approved by the lab manager and signed off by every lab user undertaking this process.

Task/Activity Risk Assessment

Description of Task or Activity: <i>(to include enough information to establish the foreseeable hazards)</i>	Location(s): <i>(where will the activity or task take place?)</i>	Date:
Digestion of organic materials in soil/sediment samples prior to particle size analysis, using a 20% solution of 100Vol Hydrogen Peroxide. Flocculation is prevented by the addition of Sodium Hexametaphosphate	D112A and D132	7/12/22
Most significant risk(s):	Equipment required <i>(including PPE, tools, chemicals etc.)</i>	
Exposure to Hydrogen Peroxide Burns and electrocution	Equipment: • Disposable centrifuge tubes (50ml) • Centrifuge • Water bath • Aluminium foil • Glass beakers • Glass rods • Dropper pipettes • Distilled water • Balance Chemicals: • 20% solution of 100Vol Hydrogen Peroxide (H₂O₂) • Sodium Hexametaphosphate solution (Sodium Hexametaphosphate – NaPO₃ and Sodium Carbonate Na₂CO₃ dissolved in distilled water) PPE:	

				• Lab coats and eye protection to be worn in laboratory at all times • Safety gloves to be worn when handling Hydrogen Peroxide		
Work Step/ Tasks <i>(Describe the tasks/steps involved in the order of work)</i>	Hazards Identified for each task/step	Those at risk <i>(people who could be harmed)</i>	How could they be harmed? <i>(nature of injuries, damage that could result)</i>	Uncontrolled risk level <i>(level of risk without control)</i>	Required controls <i>(how the risk can be removed or reduced by for example engineered methods, safe systems of work, training and/ or personal protective equipment)</i>	Controlled risk level <i>(level of risk remaining when controls are in place)</i>
1) Weigh out approx. 0.5g of soil/sediment into 50ml centrifuge tube.	Soil handling/Bacteria in soil	Staff	Risk of contamination from bacteria/organic matter leading to infection and illness Risk of infection if soil makes contact with any cuts/wounds on skin. Risk of cross-contamination from handling to e.g. foods, drink, etc. Potential for respiratory irritation/aggravation of respiratory conditions if	4 x 3 Medium Risk	All required PPE to be worn in lab (lab coat and eye protection). Nitrile gloves can be worn if necessary/preferred. Prior assessment of soil samples by trained personnel to ensure contaminated samples are not sent to the lab. Thoroughly wash hands with soap and water after handling soils. If necessary/preferred, soil sample weighing can be performed under extraction in dust cabinet (D112A or D131).	2 x 2 Low Risk

			soil/sediment is fine and produces a "dust".			
2) Add approx. 20ml of 20% Hydrogen Peroxide to each tube (H ₂ O ₂ is added in excess and breaks down organic matter in sample). Undertaken within fume cabinet.	Exposure to Hydrogen Peroxide from chemical spillages/splashes	Staff	Hydrogen Peroxide can cause serious eye damage and eye/skin irritation, may be harmful if swallowed, and may have pressure build-up within storage bottles. Injuries from slips and trips. Cuts from glass breakages and risk of glass shards remaining in cuts. Risk of chemicals making contact with skin and both new/existing cuts from spillages/splashes.	4 x 4 High Risk (Known to occur, Major)	Staff to demonstrate and observe correct use of chemicals and transfer protocols. All lab users to wear safety glasses at all times. All lab users to wear disposable nitrile gloves at all times. All lab users to read COSHH form for Hydrogen Peroxide before handling. All work with Hydrogen Peroxide to be undertaken within fume cabinet.	2 x 3 Low Risk (Not likely, Moderate)
	Cuts from broken glass	Staff	Cuts from glass breakages and risk of glass shards remaining in cuts. Risk of chemicals		Broken glass to be removed by technical staff for appropriate disposal. Seek first aid immediately if cuts occur and tell first aider which	

			making contact with skin and both new/existing cuts from glass breakages.		chemicals have been handled.	
	Glove allergy	Staff	Allergic reaction to glove material when contact made with skin.	3 x 2 Low Risk (Could occur, Minor)	Wear appropriate gloves and inform colleagues/staff of any allergies.	2 x 2 Low Risk (Not likely, Minor)
3) Place rack of samples into boiling water bath to aid oxidation of matter (digestion).	Burns and electrocution from water bath	Staff	Burns from contact with hot water. Electrocution of user due to electrical malfunction or using equipment with wet hands. Possible fire hazard from overheating. Physical injury from manual handling and lifting equipment. Mechanical injury to hands and injuries from catching loose hair/clothing in equipment.	3 x 3 Medium Risk (Could occur, Moderate)	Work with care to prevent splashes. Do not handle/lift when filled with water. Ensure equipment is not faulty before use (in-date PAT testing). Do not operate with wet hands. Water bath should be safety checked as appropriate. Manual handling training carried out before lifting. Keep loose clothing/hair away from equipment, especially whilst circulation impeller and/or shaker mechanism in use (cover where appropriate).	2 x 2 Low Risk (Not likely, Minor)

					Ensure all equipment placed within water bath is safe to be heated to high temperatures.	
4) Monitor each tube for excessive reaction during digestion – carefully remove rack from heat if sample reacts violently, allow reaction to subside, then return to heat (a small amount of ethanol may be added to reduce frothing at this stage). This may need to be repeated several times until sample reacts steadily but not aggressively ('cold' digestion overnight prior to water bath heating may benefit some samples).	Burns and electrocution from water bath	Staff	Burns from contact with hot water. Electrocution of user due to electrical malfunction or using equipment with wet hands. Possible fire hazard from overheating. Physical injury from manual handling and lifting equipment. Mechanical injury to hands and injuries from catching loose hair/clothing in equipment.	3 x 3 Medium Risk (Could occur, Moderate)	Work with care to prevent splashes. Do not handle/lift when filled with water. Ensure equipment is not faulty before use (in-date PAT testing). Do not operate with wet hands. Water bath should be safety checked as appropriate. Manual handling training carried out before lifting. Keep loose clothing/hair away from equipment, especially whilst circulation impeller and/or shaker mechanism in use (cover where appropriate). Ensure all equipment placed within water bath is safe to be heated to high temperatures.	2 x 2 Low Risk (Not likely, Minor)

	Exposure to Hydrogen Peroxide from chemical spillages/splashes	Staff	Hydrogen Peroxide can cause serious eye damage and eye/skin irritation, may be harmful if swallowed, and may have pressure build-up within storage bottles. Injuries from slips and trips. Risk of chemicals making contact with skin and both new/existing cuts from spillages/splashes.	4 x 4 High Risk (Known to occur, Major)	Staff to demonstrate and observe correct use of chemicals and transfer protocols. All lab users to wear safety glasses at all times. All lab users to wear disposable nitrile gloves at all times. All lab users to read COSHH form for Hydrogen Peroxide before handling. All work with Hydrogen Peroxide to be undertaken within fume cabinet.	2 x 3 Low Risk (Not likely, Moderate)
	Exposure to ethanol from chemical spillages/splashes	Staff	Ethanol is highly flammable as both a liquid and vapour, can cause serious eye irritation, and may cause skin/respiratory irritation. Injuries from slips and trips. Risk of chemicals making contact with	4 x 4 High Risk (Known to occur, Major)	Staff to demonstrate and observe correct use of chemicals and transfer protocols. All lab users to wear safety glasses at all times. All lab users to wear disposable nitrile gloves at all times. All lab users to read COSHH form for Ethanol before handling.	2 x 3 Low Risk (Not likely, Moderate)

			skin and both new/existing cuts from spillages/splashes.			
5) Once sample reaction has stabilized, cover rack of samples with aluminium foil to avoid contamination and maintain oxidizing atmosphere, then return to water bath for 2 hours.	Burns and electrocution from water bath	Staff	Burns from contact with hot water. Electrocution of user due to electrical malfunction or using equipment with wet hands. Possible fire hazard from overheating. Physical injury from manual handling and lifting equipment. Mechanical injury to hands and injuries from catching loose hair/clothing in equipment.	3 x 3 Medium Risk (Could occur, Moderate)	Work with care to prevent splashes. Do not handle/lift when filled with water. Ensure equipment is not faulty before use (in-date PAT testing). Do not operate with wet hands. Water bath should be safety checked as appropriate. Manual handling training carried out before lifting. Keep loose clothing/hair away from equipment, especially whilst circulation impeller and/or shaker mechanism in use (cover where appropriate). Ensure all equipment placed within water bath is safe to be heated to high temperatures.	2 x 2 Low Risk (Not likely, Minor)
6) After 2 hours, check samples for complete digestion of	Burns and electrocution from water bath	Staff	See above.	3 x 3 Medium Risk	See above.	2 x 2 Low Risk (Not likely,

organic material (supernatant liquid appears transparent and is a green or yellowy/brown colour).				(Could occur, Moderate)		Minor)
7) If digestion is incomplete and organic matter is still present (supernatant liquid still cloudy or matter can be seen floating in liquid), centrifuge samples at 4000rpm for 4 minutes, decant supernatant liquid and repeat process from Step 2 until all organic matter is digested.	Burns and electrocution from water bath	Staff	See above.	3 x 3 Medium Risk (Could occur, Moderate)	See above.	2 x 2 Low Risk (Not likely, Minor)
	Exposure to Hydrogen Peroxide from chemical spillages/splashes	Staff	See Step 2.	4 x 4 High Risk (Known to occur, Major)	See Step 2.	2 x 3 Low Risk (Not likely, Moderate)
	Improper use of centrifuge leading to chemical exposure or equipment malfunction	Staff	If the centrifuge lid is not fitted properly, or if the centrifuge is not balanced/loaded correctly, reagents may spill. Improper loading of the centrifuge may cause explosion, potentially leading to chemical exposure and burns. Electrocution from	3 x 4 Medium Risk (Could occur, Major)	Ensure centrifuge is used on stable level surface. Ensure test tube lids are securely fastened prior to loading. Ensure all test tubes are balanced correctly and use blanks to balance if necessary. Long hair to be tied up. Centrifuge should be safety checked as appropriate and checked before use (PAT	2 x 2 Low Risk (Not likely, Minor)

			faulty equipment or contact between electrical equipment and liquid. Mechanical injuries such as entrapment within rotating parts of equipment. Possible injuries from chemical exposure due to splashed from improperly sealed vessels.		testing). Do not operate with wet hands.	
8) When all organic matter has been digested, centrifuge samples at 4000rpm for 4 minutes and decant supernatant liquid. Top up samples with distilled water, centrifuge and decant off supernatant liquid again.	Improper use of centrifuge leading to chemical exposure or equipment malfunction	Staff	See above.	3 x 4 Medium Risk (Could occur, Major)	See above.	2 x 2 Low Risk (Not likely, Minor)
	Cuts from broken glass (beakers)	Staff	Cuts from glass breakages and risk of glass shards remaining in cuts. Risk of chemicals making contact with skin and both new/existing cuts from glass breakages.	4 x 3 Medium Risk (Known to occur, Moderate)	Broken glass to be removed by technical staff for appropriate disposal. Seek first aid immediately if cuts occur and tell first aider which chemicals have been handled.	2 x 2 Low Risk (Not likely, Minor)

9) Add 20ml of distilled water to tube and then 2ml of Sodium Hexametaphosphate solution (wetting agent to aid deflocculation). Sample is now ready to be analysed on Coulter laser granulometer (Geography).	Exposure to Sodium Hexametaphosphate solution from chemical spillages/splashes	Staff	Sodium Hexametaphosphate can cause mild skin irritation at site of contact, irritation and redness to the eyes, throat irritation when ingested, as well as throat irritation and tightness in the chest with inhalation.	4 x 3 Medium Risk (Known to occur, Moderate)	Sodium Hexametaphosphate solution to be provided by Geography. Staff to demonstrate and observe correct use of chemicals and transfer protocols. All lab users to wear safety glasses at all times. All lab users to wear disposable nitrile gloves at all times. All lab users to read COSHH form for Sodium Hexametaphosphate before handling.	2 x 2 Low Risk (Not likely, Minor)
	Cuts from broken glass (beakers)	Staff	Cuts from glass breakages and risk of glass shards remaining in cuts. Risk of chemicals making contact with skin and both new/existing cuts from glass breakages.	4 x 3 Medium Risk (Known to occur, Moderate)	Broken glass to be removed by technical staff for appropriate disposal. Seek first aid immediately if cuts occur and tell first aider which chemicals have been handled.	2 x 2 Low Risk (Not likely, Minor)

Internal Guidance/Linked Documents:		MSDS and COSHH forms for Hydrogen Peroxide, Ethanol, and Sodium Hexametaphosphate solution.	
Competence Requirements:		Training provided to all users prior to sampling.	
Supervisor/Manager Review and Comment:			
Assessment Prepared by		Supervisor/Manager acceptance	
Name:	Aimee Parker	Name:	
Signature:	<i>AB Parker</i>	Signature:	
Date:	7/12/22	Date:	
This risk assessment should be read by those performing and supervising the work in conjunction with all relevant documentation including method statements and safe systems of work			

Health and Safety Risk Matrix

			Probability/ likelihood of risk realisation				
			Almost Impossible (1)	Not Likely to occur (2)	Could occur (3)	Known to occur (4)	Common occurrence (5)
			A freak combination of factors would be required for risk to be realised	A rare combination of factors would be required for risk to be realised	Could happen when additional factors are present otherwise unlikely to occur	Not certain to happen but an additional factor may result in risk being realised	Almost inevitable that risk will be realised
Potential Consequences	Severe (5)	One or more fatalities. Irreversible health problems	5	10	15	20	25
	Major (4)	Partial or medium-term, disabilities or major health problems	4	8	12	16	20
	Moderate (3)	Lost-time injuries or potential medium-term health problems	3	6	9	12	15
	Minor (2)	Minor, very short-term health concerns on recordable injury cases.	2	4	6	8	10
	Insignificant (1)	Inherently safe, unlikely to cause health problems or injuries	1	2	3	4	5

Extreme risk	High risk	Medium risk	Low risk

Supporting Document 2: The protocol for loss on ignition supplied by the Geography Department's technical team.

Department of Geography Laboratory Procedures

Preparation and analysis of samples for moisture content and loss on ignition

Prerequisites

Before carrying out the following procedure ensure you have completed the following paperwork/training.

1. Read through and understood the standard laboratory risk assessment for the determination of moisture content and loss on ignition.
2. Completed the safe laboratory working induction.
3. Have been instructed on the following procedure by a member of the lab staff.

If you run into any difficulties, are unsure what to do or have any accidents inform a member of the lab staff immediately.

Principle

The moisture content (MC) of a sediment sample is determined by drying a known mass of sediment at 105°C for 24 hours giving us our sample's dry mass. These values are required to calculate moisture content.

Loss on ignition (LOI) is a procedure used to determine the approximate percentage of organic carbon within a sediment/soil sample. The process involves igniting a known mass of sample for a specified duration of time and using the subsequent mass difference to calculate the result.

PPE requirements

The following PPE must be worn during this procedure:

- Lab coat
- Gloves
- Safety glasses
- Heat resistant gloves and tongs when handling hot crucibles

Equipment

- | | |
|-----------------------|---|
| • Porcelain crucibles | • Analytical balance (4 decimal places) |
| • Drying oven | • Muffle furnace |
| • Tongs | • Desiccator |

Method

1. MC and LOI require specialized crucibles capable of withstanding high temperatures. These are all marked with unique identifiers which have been burnt on using heat resistant markers. **Do not** add other markings to the crucibles. Dry your crucibles overnight in the drying oven at 105°C. Once cooled, place your crucibles in a desiccator to keep them dry. Remove your crucibles individually and record their mass to 4 decimal places. **This is your crucible mass (A).**
2. Add 3-5 g of sample to each pre-weighed crucible and record the mass to 4 decimal places. Work as efficiently as possible as you want to minimize the amount of time your crucibles and samples are open to the atmosphere. **This step provides you with your crucible plus wet sample mass (B).**
3. Dry your crucibles and samples in a drying oven at 105°C for 24 hours. Place in a desiccator to cool prior to weighing. Once cool, record each crucible's mass to 4 decimal places, removing them from the desiccator one at a time. **This is your crucible plus oven dry sediment mass (C).**
4. Place your crucibles and samples into a **cold** muffle furnace and heat your samples to 550°C for 4 hours. It is essential that samples are placed into the furnace whilst it is cold as a sudden temperature shock can cause samples to ignite, spitting material which will contaminate other samples and invalidate your results. It is also far safer to place items into a cold furnace.
5. After 4 hours turn the furnace off and very carefully remove your samples from the furnace using long-reach tongs. Place the samples carefully inside a glass desiccator and allow to cool.
6. Once your samples have cooled sufficiently (usually after around 45 minutes) record the mass of the crucibles one by one to 4 decimal places. Please take care when opening the desiccator for the first time after cooling as negative pressure will have developed inside the desiccator. To avoid a sudden in-rush of air, the desiccator should be opened slowly. **This step provides you with your crucible plus ash sediment mass (D).**
7. To calculate your results, use the values you have obtained above in the following calculations.

$$MC \% = \left(\frac{\text{wet sample mass} - \text{dry sample mass}}{\text{wet sample mass}} \right) \times \frac{100}{1}$$

$$LOI \% = \left(\frac{\text{dry sample mass} - \text{ash sample mass}}{\text{dry sample mass}} \right) \times \frac{100}{1}$$

Where:

- wet sample mass = B – A
 - dry sample mass = C – A
 - ash sample mass = D – A
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Supporting Document 3: The protocol for particle size analysis sample preparation supplied by the Geography Department's technical team.

Preparation of Samples for Laser Diffraction Particle Size Analyser

Prerequisites

Before carrying out the following procedure ensure you have completed the following:

4. Read and understood the "Preparation of Samples for Laser Diffraction Particle Size Analyser" risk assessment.
5. Read and understood the COSHH form for hydrogen peroxide (H₂O₂) and sodium hexametaphosphate solution.
6. Have been instructed on the following procedure by a member of the lab staff.

PPE requirements

Laboratory coat, nitrile gloves, safety glasses

Chemicals

Hydrogen peroxide (20%)

Strong oxidizing agent and cause explosive reactions with several chemicals. Is an irritant and skin contact can cause burns.

Sodium hexametaphosphate solution 3% w/v (containing sodium carbonate)

Causes eye irritation

Consumables

- 50 ml centrifuge tubes (1 per sample)
- Foil
- Plastic pipettes

Equipment

- Fume cupboard
- Water bath and cover
- Centrifuge
- Balance
- Sediment sampling tools eg. spatula
- Permanent marker pen
- Wash bottle of deionized water
- Glass beaker
- Centrifuge tube rack
- Glass rods

Method

1. Weigh out the sediment sample into a 50 ml centrifuge tube and place tubes into a rack (dry sediment samples must be passed through a 2 mm sieve first). Make sure the weight of the sample is recorded. Do not place lids on the tubes.

The amount of sediment required will depend on the grain size and how organic the material is. Typically, ~ 0.5g of sediment is processed (if mainly clay/silt only ~0.2g; if mainly coarse sand between 1-3g; if high in organic material roughly estimate the percentage of organic material and adjust the weight accordingly to leave approximately 0.5g of sample after peroxide digestion).

2. Move the rack of samples into a fume cupboard. Add approximately 20ml of 20% hydrogen peroxide solution (20 vol) to each of the samples. **Do not** place lids on the tubes (gas is evolved as part of the oxidation reaction and would cause a capped tube to burst). The hydrogen peroxide is added in excess and breaks down the organic matter in the sample; generally, only mineral matter is considered for particle size analyses.
 3. If the hydrogen peroxide does not react violently with the sediment, the rack of centrifuge tubes can be covered with aluminium foil and placed into the water bath (set to 80°C). Covering the samples with foil not only prevents any cross contamination due to vigorous reaction but also helps to maintain an oxidising atmosphere above each sample, aiding the reaction. The foil must be labelled with the owner's name and date. If the samples react vigorously, leave them overnight in the fume cupboard, placing them in the water bath when the reaction has reduced/stopped. A small amount of ethanol may be added to reduce frothing at this stage.
 4. Heat samples for 2 hours in the water bath until all the organic material has been digested by the hydrogen peroxide. Ensure the water bath does not dry out, the water bath cover will reduce the chance of this happening.
 5. Carefully remove the sample rack from the water bath and check your samples in the fume cupboard. If all organic matter has been digested, then the supernatant liquid will be transparent and a green or yellowy/brown colour. If any organic matter is still present (the liquid is still cloudy or matter can be seen floating in the liquid) centrifuge the samples at 5 minutes at 3300 rpm (if using the Rotanta 460 centrifuge) decant the supernatant liquid and repeat the process from Step 2 until all organic matter is digested.
 6. When all organic matter has been digested, centrifuge the samples at 3300 rpm for 5 minutes (Rotanta 460 centrifuge) and decant the supernatant liquid. Top up the samples with distilled water, centrifuge and decant off the supernatant liquid again.
 7. Add 20ml of distilled water to the tube and then 2ml of sodium hexametaphosphate solution. Sodium hexametaphosphate solution is a wetting agent and aids deflocculation. Stir with a glass rod.
 8. Leave the sample overnight. The sample is now ready to be analysed on the laser diffraction particle size analyser.
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Reagent information

Hydrogen Peroxide (20% v/v)

The hydrogen peroxide is prepared in 2 litre quantities by measuring out 400 ml of 100 volumes hydrogen peroxide (bought in concentration) into a pre-labelled reagent bottle and adding 1600ml of deionized water to make up to the 2-litre mark.

Sodium Hexametaphosphate solution (3.3%w/v)

To prepare the sodium Hexametaphosphate solution weigh out 33g of Sodium Hexametaphosphate ($\text{Na}_6\text{P}_6\text{O}_{18}$) and 7g Sodium Carbonate (Na_2CO_3) and dissolve in approximately 500 ml distilled water in a 1 litre volumetric flask, when dissolved make up to the mark with deionised water.
