

**School of Geography**

FACULTY OF ENVIRONMENT



**UNIVERSITY OF LEEDS**

# **GEOG3600**

## **Laboratory & Field Work Guide**

### **(For Dissertations 2009/2010)**



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## 1 Introduction

This document is intended to help you plan your dissertation and also to answer some of the frequently asked questions. Please remember that the equipment and reagents you used in laboratory classes were prepared for you by the technicians so allow plenty of time to prepare and analyse your samples. If you find any errors or omissions in this document please e-mail the labs to enable us to improve and update the guide. At the end of your project we would ask you to provide feedback on all aspects of your laboratory and fieldwork, whether good, bad or indifferent: please e-mail [r.l.gasior@leeds.ac.uk](mailto:r.l.gasior@leeds.ac.uk) to leave your comments. **The deadline for the completion of lab work is Friday 15<sup>th</sup> January 2010.**

## 2 Contacts

Technical staff:

The technicians can be contacted: by telephone on 0113 343 3314, by e-mail using [geo-labtechs@leeds.ac.uk](mailto:geo-labtechs@leeds.ac.uk) or in person in room 2.56. To ensure you receive a reply please use the geo-labtechs e-mail for field and lab/fieldwork requests rather than e-mail individuals.

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# LABORATORY GUIDE

### **3 Laboratory Health and Safety**

#### **3.1 General**

- If you are planning to collect samples from outside the EU please see Rachel Gasior before you commence your fieldwork. You will need to take a copy of the Plant Health Licence issued by DEFRA to import samples and a copy of the laboratory protocols.
- Before starting any lab work you must assess the risks:
  1. Contact a member of staff and arrange a laboratory induction
  2. Obtain a COSHH assessment for the procedure. COSHH stands for Control of Substances Hazardous to Health. The assessment explains what safety precautions to take to prevent exposure to harmful chemicals and what you should do in the event of an accident.
  3. Use the COSHH assessment to assess the risks – complete a ‘Student COSHH Risk Assessment Form’
  4. Return the completed Risk Assessment form to your mentor for checking and signing.
  5. The risk assessment MUST be displayed on your work bench when working. This can be stored in the folder in lab 2.60 when not in use.
  6. A copy of a risk assessment can be found at the end of this document.
- Coats and bags can be kept on the hooks in lab 2.60 or 2.58 – do not leave bags in the aisles or on the benches.
- Wear a laboratory coat at all times.
- DO NOT bring food or drink into the laboratory.
- Wear the appropriate personal protection when handling chemicals.
- Dispose of waste chemicals following COSHH procedure.
- Clear any spillages immediately, ask the technicians how to dispose of chemicals if you are unsure. Be considerate of other lab users, do not leave chemical residues on the balances or work benches.
- Contact a member if you break or find any broken glassware, do not use broken glassware and do not put any items of glassware in the general waste bins
- Clearly label all receptacles containing chemicals with the chemical name and concentration.
- Wash your hands before leaving the laboratory.
- Return acids, flammable liquids and chemicals to their proper storage locations.
- Consider the hazard your samples pose to others. You will be aware that your samples maybe: toxic, hazardous or are biohazards, but consider how will other lab users know if you don't tell them. Specify on the sample container/ bag if your samples contain chemicals.
- If you need to leave an experiment in progress leave a note identifying the hazards and specify the date on when you intend to return.

#### **3.2 Fire Safety**

##### **3.2.1 Action to be taken on the discovery of a fire**

- Raise the alarm. Fire Alarm call points are located in Laboratories 2.50 and 2.60, as well as on the 2<sup>nd</sup> floor stairwell.
- Call security on 32222 (0113 343 2222 from mobile phones). Provide the following information: the location of the fire, the location of any trapped persons, the type of fire i.e. the type of fuel involved, report any hazardous substances involved.
- Evacuate the building using the shortest possible route closing the door to the affected room behind you, do not use the lift.
- Report to the Car Park at 41 University Road (Cream coloured building opposite the entrance to Geography). Await further instructions.

##### **3.2.2 On hearing the fire alarm**

- Evacuate the building immediately by the shortest possible route and then report to the assembly point. DO NOT stop to collect valuables as this will hamper your escape, and that of other people.

### **3.3 Healthcare Waste**

All laboratory waste must be placed in yellow healthcare waste bags for incineration, do not put laboratory waste in the black bin bags.

All sharps: pipette tips, small items of broken glassware, needles, cocktail sticks etc must be placed in sharps bins.

## **4 Booking Laboratory Sessions**

### **4.1 Booking**

Sessions can be booked in the lab diary which is located in the pigeon hole on the lab technicians' office door, room 2.56. If you can't attend your lab session please notify [geo-labtechs@leeds.ac.uk](mailto:geo-labtechs@leeds.ac.uk). At certain times of the year the lab staff are busy demonstrating at lab sessions or preparing for them – during these times you are permitted to use the labs (with the exception of the teaching lab, lab 2.50) but please be aware that technical support is limited. The labs are open daily from 9-4.30pm. If you have samples that have to be preserved (invertebrate, samples for metals) please allow plenty of time to be able to complete your work before 4.30pm.

**ALL LAB WORK MUST BE COMPLETED BEFORE FRIDAY 15<sup>TH</sup> JANUARY 2010**

### **4.2 Space**

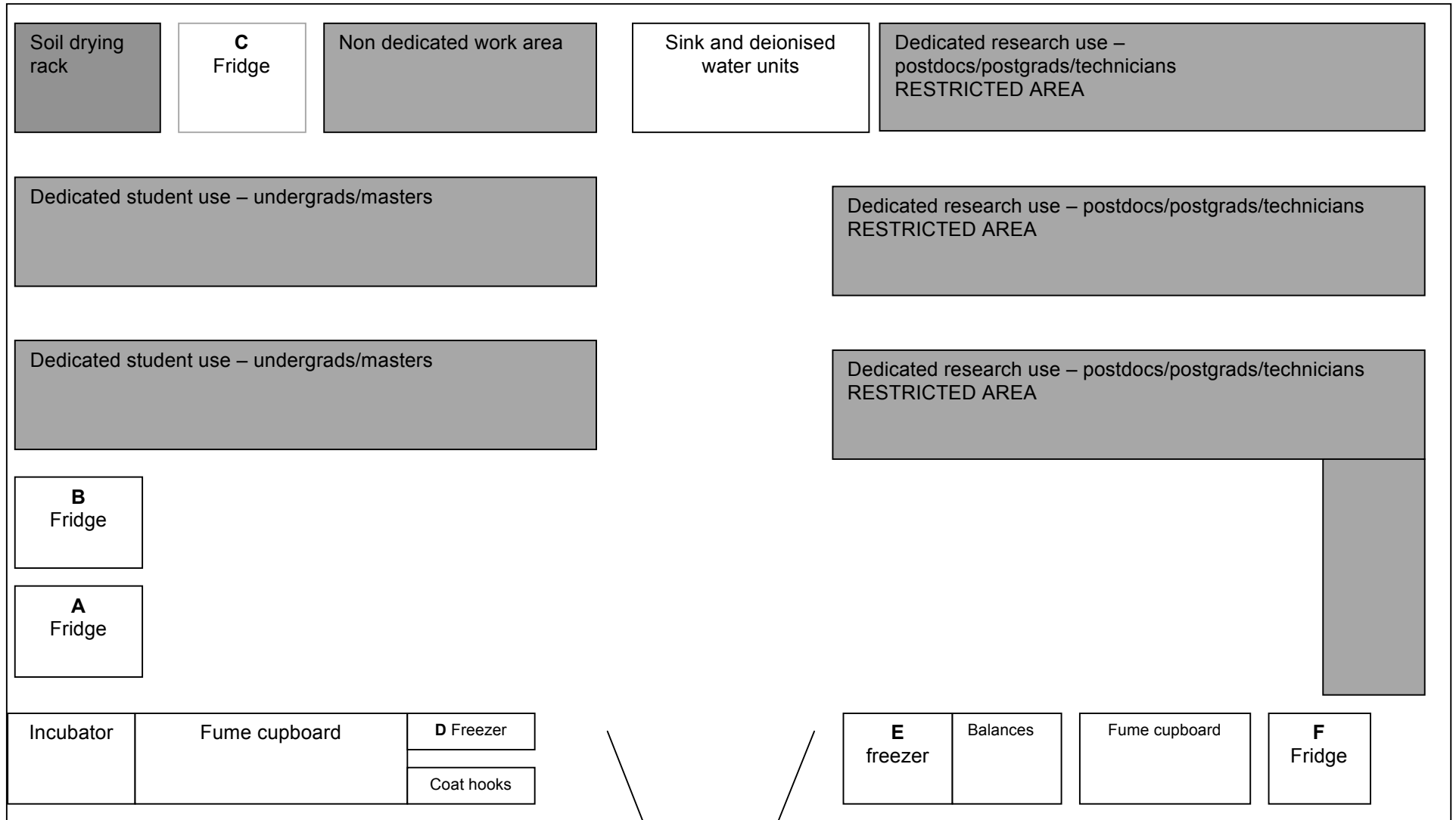
The space for project work is limited to the left hand side of laboratory 2.60 as you enter the room. As you are sharing the Research Laboratory with researchers it is important that you follow Good Laboratory Practice (GLP) to prevent contamination of samples and disruption to other lab users. The diagram below shows the dedicated areas in lab 2.60. Please do not enter the research area of the lab. The majority of equipment you will need is kept in the cupboards in lab 2.60.

Soil sample preparation (grinding and sieving) must be carried out in the sample prep room lab 2.57.

### **4.3 Security**

As secure storage for bags and coats is not available do not bring valuables to the laboratories. Keep your bag with you at all times, if this is not possible leave it in lab 2.60 as this room is occupied more often than other laboratories.

**Laboratory 2.60 Layout**



## 5 Laboratory Opening Hours

The labs are open all year from 9 am to 4.30 pm, Monday to Friday. Working out of hours is not permitted.

## 6 What to Bring to the Laboratory

- This document
- Goggles
- Laboratory coat
- Calculator
- Marker pen
- Methods of Analyses

The technicians have a variety of methods for your use, **but cannot advise you which to use**. You must discuss this with your mentor, read the relevant literature and decide which methods are suitable for your project.

- COSHH assessments
- A4 notebook

Where possible record data in tables, including the following information: date and title of experiment, method used, dilution factors and any other information required so the experiment can be repeated.

## 7 Analysis/Equipment Available

The tables below provide a guide to the types of analyses and equipment available at the School of Geography - if the analytes/equipment you are interested in do not appear on the list please discuss with the lab staff before submitting your dissertation proposal.

### 7.1 General Laboratory Equipment Available for use

Equipment	Manufacturer	Location	Comments
Autoclave	Astell	2.58	See lab techs for training
Automated pestle and mortar	Retsch	2.57	Soil preparation
Centrifuge	Fisherbrand	2.58	See technician
Cutting mill	Retsch	B08	Shredding plant material
Desiccator cabinet	n/a	2.50	Suspended solids determination – ensure desiccant is coloured orange before use
Furnace	Carbolite	2.60	Ensure fume cupboard is on
Furnace	Carbolite	2.60	Ensure fume cupboard is on
High-power biological microscopes	Leica/ Olympus	Portable	100-1000x magnification. Applications in Quaternary palaeoecology (pollen, charcoal, diatoms, etc)
Incubator	Jouan	2.60	For biological oxygen demand samples – read COSHH before analysing samples
Low-power (stereo) microscopes	Various	Portable	10-50x magnification. For studying invertebrates, small plants and animal fossils
Mixer mill	Retsch	2.57	Fine grinding of material
Oven (large floor standing)	Unknown	2.50	Soil drying
Oven	Memmert	2.50	Drying filter papers (suspended solids)
Ovens	Memmert	2.60	Soil drying
Refrigerators	Various	2.60	Undergraduate sample storage
Shaking table	Gallenkamp	2.50	Orbital
Shaking table	Gerhardt	2.58	Reciprocal
Soil drying rack	n/a	2.60	Soil drying
Vacuum filtration	Millipore	2.50	Suspended solids determination

## 7.2 Geological/sedimentological analysis

Analysis	Technique	Max no of Samples <sup>1</sup>	Comment
Pollen/charcoal/ fungal spore/testate amoebae analysis	Chemical preparation, microscopy	36	See John Corr
Plant macrofossil/ mollusc analysis	Sieving, microscopy	-	
Particle size analysis	Laser diffraction; prep	-	Labour intensive allow yourself plenty of time
Geochemistry (alkali metals and aluminium)	AAS ICP <sup>2</sup>	- 50	Glassware must be acid washed prior to sample extraction
Magnetic properties	Magnetic susceptibility	-	See John Corr
Dry bulk density, water/organic carbon/ calcite content	Volumetric sampling and loss on ignition	-	
Biogenic silica	Sodium carbonate extraction	30	Samples must be freeze dried prior to extraction

## 7.3 Vegetation Sample Analysis

Analysis	Technique	Max no of samples <sup>1</sup>	Comment
Alkali metals (Ca, Mg, Na and K)	AAS	-	Dry-ashing followed by dissolution in acid

## 7.4 Environmental Sample Analysis

Analysis	Technique	Max no of samples <sup>1</sup>	Comment
Nitrite	Colorimetry	-	Diffusion tubes
Invertebrate		-	
Gases <sup>4</sup>	Drager kit	30	See technician

## 7.5 Water Sample Analysis

Analysis	Technique	Max no of samples <sup>1</sup>	Comment
Alkalinity	Titration	-	Samples must be analysed within 12 hours of collection. Unfiltered sample
Alkali metals (Ca, Mg, Na and K)	Atomic Absorption Spectrophotometry	-	Filter sample prior to analysis
Ammonium	Autoanalyser /colorimetry	50	Students preparation technician analysis
Biological oxygen Demand	Electrometric	-	Measure DO in the field and then again after incubating sample for 5 days @ 20 °C
Chemical oxygen Demand	Digestion/titration	-	
Chloride (by Dionex)	HPLC-IC	50	Filter through 0.45 µm syringe filter
Chlorine	Colorimetry	50	Field test kit
Conductivity	Electrometric	-	Measure in the field
Dissolved oxygen	Electrometric	-	Measure in the field
Fluoride	HPLC-IC	-	Filter through 0.2 µm syringe filter
Metals	ICP-OES <sup>2</sup>	50	Filter and preserve samples prior to analysis. Cd, Cu, Pb, Ni, Cr, Zn, Al, Fe, Mn
Nitrate (by Dionex)	HPLC-IC	50	Filter through 0.45 µm syringe filter
Nitrate	Autoanalyser /colorimetry	50	Students preparation Technician analysis Sub ppm range
Nitrite	Autoanalyser /colorimetry	50	Students preparation Technician analysis
Particulate organic carbon	Gravimetric	50	Gravimetric determination using Whatman GF/F
pH	Electrometric	-	Measure in the field
Phosphorus	Colorimetry	-	
Sulphate (by Dionex)	HPLC-IC	50	Filter through 0.45 µm syringe filter
Suspended solids	Gravimetry	50	
Total carbon <sup>3</sup>	Combustion/IR analysis	50	Student preparation Technician analysis
Total nitrogen <sup>3</sup>	Combustion/chemiluminescence analysis	50	Student preparation Technician analysis
Total phosphorus	Colorimetry	-	Sample digestion prior to analysis
Turbidity	Nephelometry	-	Unfiltered sample

## 7.6 Soil Sample analysis

Analysis	Technique	Max no of samples <sup>1</sup>	Comment
Alkali metals (Ca, Mg, Na and K)	AAS	-	Ammonium acetate soil pH >7 ammonium chloride soil pH <7 <sup>5</sup> . <2 mm dry soil
Available Aluminium	AAS	-	Ammonium acetate extraction, pH 4.8 on dry <2 mm soil
Ammonium (nitrogen)	Autoanalyser/ colorimetry	50	KCl extraction on field moist soil
Carbon (organic)	Digestion/titration	-	Walkley Black method
Conductivity	Electrometric	-	1:2 soil:water extract on dry <2mm soil
LOI	Gravimetric	-	Ignition of soil on oven dry <2mm soil <sup>6</sup>
Metals (total)	ICP-OES <sup>2</sup>	50	<i>Aqua regia</i> digestion on dry <150 µm soil
Moisture content	Gravimetric	-	Oven dry or field moist soil
Nitrate	Autoanalyser/ colorimetry	50	KCl extraction on fresh soil
Nitrogen (Kjeldahl)	Digestion/distillation/ titration	50	Labour intensive procedure on dry <2 mm soil
Particle size analysis	Laser Diffraction	-	Labour intensive procedure on dry <2mm soil
pH	Electrometric	-	1:2 or 1:2.5 soil:water extract on dry <2 mm soil
Phosphorus (available)	Colorimetry	-	Olsen's P for alkali soils on dry <2mm soil
Phosphorus (available)	Colorimetry	-	0.43 M acetic acid extraction for acid soils on dry <2mm
Water soluble anions	HPLC-IC	50	Follow British Standard Method for extraction

<sup>1</sup>Where a maximum number of samples is specified this is the absolute number of samples that can be analysed over the duration of the dissertation, not per sample batch. Including replicate samples.

<sup>2</sup> Metals by ICP-OES are restricted to 50 samples - this can be either 50 soils, 50 water samples or a mixture of soil and water not exceeding 50 sample in total. Alternatively the AAS can be used to measure most metals in soil solution, but is not particularly suited to water samples. Please note that the AAS is labour intensive so you will need to allow yourself plenty of time.

<sup>3</sup>50 samples maximum by Thermalox combustion analyser – this can be either 50 for carbon, 50 for nitrogen or a mixture of carbon/nitrogen not exceeding 50 samples in total.

<sup>4</sup>30 samples maximum - can be a mixture of analytes (purchased in packs of 10) see Fisher catalogue for list of available determinands

<sup>5</sup>Read reference texts or consult you mentor for an appropriate method

<sup>6</sup> Read reference texts for suitable ignition temperature. Temperatures vary according to soil type.

**NB you will need to include a number of replicate samples in your analysis (suggested replication 1 in every 10 samples)**

## 7.7 Instruments Available for Your Analyses (the technical staff analyse your samples)

### Inductively Coupled Plasma – Optical Emission Spectrophotometer

Manufacturer: Perkin Elmer  
Model: 5300DV  
Determinands: Cd, Cr, Cu, Pb, Ni, Zn, Al, Fe, Mn.  
Sample type: Soil, water and sediment  
Analysis restrictions: 50 samples. The instrument is not available all year round; water samples must be preserved and refrigerated/ frozen until analysis can be complete  
Contact: Rachel Gasior

### Total carbon/ Total nitrogen analyser

Manufacturer: Analytical Sciences  
Model: Thermalox  
Determinands: Total carbon, total organic carbon, total nitrogen  
Sample type: Water  
Analysis restrictions: 50 samples  
Contact: Anthony Kepinski

### High Performance Liquid Chromatograph – Ion Chromatograph

Manufacturer: Dionex  
Model: DX-500  
Determinands: Chloride, nitrate, sulphate  
Sample type: Water  
Analysis restrictions: None  
Contact: Rachel Gasior

### Autoanalyser

Manufacturer: Skalar  
Model: SAN ++  
Determinands: Nitrite, nitrate, ammonia  
Sample type: Water and soil  
Analysis restrictions: 50 samples. This instrument is not always available. Samples can be frozen until the instrument is available. Please see the technician responsible for the instrument for advice.  
Contact: Miles Ratcliffe

## 7.8 Instruments Available For You to Use

### Atomic Absorption Spectrophotometer

Manufacturer: Hitachi  
Model: Z-5300  
Determinands: Ca, K, Na and Mg  
Sample type: Soil water and sediment  
Analysis restrictions: None. Ask the technician to book the instrument and arrange a suitable time for training  
Contact: Miles Ratcliffe

### Kjeldahl nitrogen

Manufacturer: Gerhardt  
Model: Turbotherm TT Digestion block, Vapodest 30 Distillation Unit  
Determinands: Kjeldahl nitrogen  
Sample type: Soil  
Analysis restrictions: 50 samples. Arrange a convenient time with Miles for training  
Contact: Miles Ratcliffe

### Laser Diffraction Particle Size Analyser

Manufacturer: Beckman Coulter  
Model: LS 230  
Determinands: Particle size 0.04 – 2000 microns  
Sample type: Soil, sediment (dry sample or suspended in water). Various synthetic materials.  
Analysis restrictions: None. Ask the technician to book the instrument and arrange a suitable time for training  
Contact: David Ashley or Miles Ratcliffe

### UV/Vis spectrophotometer

Manufacturer: Jasco  
Model: V 630  
Determinands: Wavelength range from 190-1100nm  
Sample type: Aqueous  
Analysis restrictions: None  
Contact: Anthony Kepinski. Ask a technician to book the instrument and arrange a suitable time for training.

## 7.9 Preparation of samples for analysis by technicians

Your samples IDs must be sequentially numbered and transferred to the correct container. Give each sample, including replicates, a unique ID. Samples without clear labels will not be analysed.

Record your details in the analysis logbook in the pigeon hole on the door to room 2.56. The lab technicians will either e-mail the results to you or leave the data in the folder in lab 2.62.

Place the samples in the specified fridge or freezer with a note with: name, date, e-mail, analysis requirements and details of any preparative work you have carried out on the samples. Notes can be found in front of the drying cabinet in lab 2.60.

### 7.9.1 HPLC-IC (chloride, nitrate, sulphate)

Filter the sample through a 0.45 µm syringe filter into a 2 ml glass vial (fill the vial ¾ full) cap the vial. Label the vial with a permanent marker pen. **Fridge G.**

### 7.9.2 ICP-OES (metals)

Do not begin this procedure until you have read the COSHH assessment and assessed the risks. Decant 15 ml of filtered sample into a 15 ml centrifuge tube. Preserve the sample by adding 22.5 µl concentrated nitric acid to each sample. Include a blank sample with each batch of samples; this is done by measuring 15 ml of filtered deionised water into a 15 ml centrifuge tube and adding 22.5 µl concentrated nitric acid. Maximum of 40 samples. **Fridge A**

### 7.9.3 Autoanalyser (nutrients)

Filter the sample into a 30 ml universal container. Check with Miles Ratcliffe where to store the samples – if the analysis cannot be completed for a while the samples may be frozen. Maximum of 50 samples. **Freezer E**

### 7.9.4 Carbon/nitrogen

#### 7.9.4.1 Dissolved

Transfer ~ 3 mL of sample which has been filtered through a 0.45 µm syringe filter (or use sample already filtered from suspended solids determination) into a small capped vial. Place the samples in a test tube rack along with a note providing the following information: name, date, e-mail and list the analytes required. Maximum of 50 samples. **Fridge G.**

## **7.10 Water sample handling [1]**

### **'Rationale**

From the moment water samples are gathered they begin to deteriorate as a result of chemical and microbiological processes. Three methods of slowing this deterioration are to be used in ECN – filtration, cold storage, and (for Al and Fe determinations only) acidification.

### **7.10.1 Initial storage**

Samples must be placed in cold storage, at a temperature between 1 and 4 °C, if the interval between the collection and the measurement of conductivity and pH is more than seven hours. They should be returned to cold storage if filtering is not completed on the same day as these measurements. Samples in transit must be placed in a cool box with pre-frozen cool blocks.

### **7.10.2 Conductivity measurement**

Conductivity must be measured within 36 hours of collection on an unfiltered subsample sample at a temperature of 25 °C according to the method given by the HMSO (1978). Results should be expressed to one decimal place (0.1 uS cm<sup>-1</sup>).

### **7.10.3 pH measurement**

pH must be measured within 36 hours of collection on an unfiltered subsample. The same subsample can be used for conductivity and for pH measurement, but conductivity should be measured first and the subsample not returned to the main sample after measurement. Results should be expressed to two decimal places (0.01 pH unit).

### **7.10.4 Filtering**

Filtering must take place within 60 hours of collection. All parts of the filtration equipment assembly must be thoroughly rinsed with deionised water before the first sample and between subsequent samples. The filters and the surfaces of the filter assembly must not be touched by hand. Filters should be moved using tweezers and each filter should be used only for one sample.

### **7.10.5 Storage prior to chemical analysis**

Storage temperature should be between 1 and 4 °C. Analysis should be completed preferably within 16 days of collection but definitely within 28 days.

### **7.10.6 Bottle and vial washing**

Containers must be washed in a laboratory cleaning agent before being used for the first time, and subsequently at approximately six-monthly intervals, or if subjected to high levels of soiling. The cleaning agent should be free of phosphate and hypochlorite. If a laboratory washing machine is available, Decomatic (not Dri-Decon) is suitable, and, if no machine is available, overnight soaking in Decon 90 is suitable. Subsequently, containers will be rinsed four times in tap water and three times in deionised water. At other times, after use, containers will be rinsed three times in deionised water and retained for use with the same sampler on subsequent occasions. After washing, containers must be shaken to remove drops of deionised water, dried in warm air in a dust free environment, and re-capped immediately.'

### 7.10.7 Analysis guide for water samples

#### **FIELD WORK**

- Take field measurements: pH, conductivity, flow, DO etc.
- Collect samples for water chemistry, invertebrate etc.
- Take separate sample, in a 50ml flip top container for BOD



#### **RETURN TO THE LAB ASAP**

- Incubate sample for BOD
- Analyse samples requiring unfiltered samples
- Preserve samples: acidify metals, add ethanol to invertebrates
- Freeze samples for nutrients – see lab techs



#### **LAB WORK**

- Carry out any preparative work and analyse samples
- Measure DO on BOD sample on day 5
- Clean work area
- Check data and dispose of samples

### **7.11 Soil sample handling**

The majority of the determinations carried out in the laboratories are on dry soil ground to less than 2mm, if you are unsure check before you dry your samples as there are a few determinations that must be carried out on field moist soil.

#### **7.11.1 Air drying**

Leave your samples on the soil drying rack in lab 2.60; open or split the bags to expose the soil to the air. Check your samples periodically to see if they are dry. When the samples are dry they can be transferred to the tidy boxes for storage until you are ready to process the samples further. Leave a note with your name, date and e-mail address with your samples. Use a spatula to reduce the size of the larger clods to reduce drying time.

#### **7.11.2 Oven drying [2]**

'A drying temperature of 40 °C in an oven is preferable to air drying at room temperature because the increased speed of drying limits changes due to microbial activity.

It should be noted that every type of sample pre-treatment will have an influence on several soil properties.

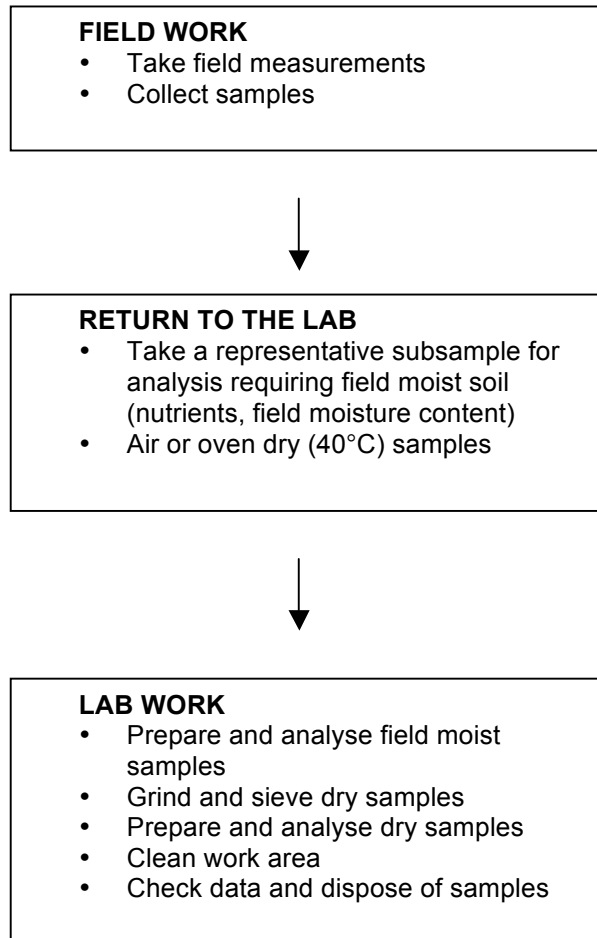
Storing soil samples, including samples that are as received, air dried, refrigerated, or stored in the absence of light, for a long time may have an influence on a number of soil parameters, especially solubilities of both inorganic and organic fractions [3].

Special measures should usually be taken for samples from contaminated soils. It is important to avoid contact with skin and special measures should be taken when drying such samples (ventilation, air removal, etc). Samples may be hazardous because of the presence of chemical contaminants etc, fungal spores, or pathogens such as leptospirosis, and appropriate safety precautions should be taken.'

#### **7.11.3 Preparation**

When samples are dry crush them lightly by hand using a mortar and pestle. Before crushing remove stones, fragments of glass and rubbish etc. Pass the soil through an appropriately sized sieve.

#### 7.11.4 Analysis guide for soil samples



## 7.12 Converting chemical species

To convert from	To...	Multiply by...
mg L <sup>-1</sup> Al	mg L <sup>-1</sup> Al <sub>2</sub> O <sub>3</sub>	1.8895
mg L <sup>-1</sup> B	mg L <sup>-1</sup> H <sub>3</sub> BO <sub>3</sub>	5.7
mg L <sup>-1</sup> Ca-CaCO <sub>3</sub>	mg L <sup>-1</sup> Ca <sup>2+</sup>	0.4004
mg L <sup>-1</sup> CaCO <sub>3</sub>	mg L <sup>-1</sup> Ca <sup>2+</sup>	0.4004
mg L <sup>-1</sup> CaCO <sub>3</sub>	mg L <sup>-1</sup> Mg <sup>2+</sup>	0.2428
mg L <sup>-1</sup> Cr <sup>6+</sup>	mg L <sup>-1</sup> CrO <sub>4</sub> <sup>2-</sup>	2.231
mg L <sup>-1</sup> Cr <sup>6+</sup>	mg L <sup>-1</sup> Na <sub>2</sub> CrO <sub>4</sub>	3.115
mg L <sup>-1</sup> Cr <sup>6+</sup>	mg L <sup>-1</sup> Cr <sub>2</sub> O <sub>7</sub> <sup>2-</sup>	2.077
mg L <sup>-1</sup> Mg-CaCO <sub>3</sub>	mg L <sup>-1</sup> Mg <sup>2+</sup>	0.2428
mg L <sup>-1</sup> Mn	mg L <sup>-1</sup> KMnO <sub>4</sub>	2.876
mg L <sup>-1</sup> Mn	mg L <sup>-1</sup> MnO <sub>4</sub> <sup>-</sup>	2.165
mg L <sup>-1</sup> Mo <sup>6+</sup>	mg L <sup>-1</sup> MoO <sub>4</sub> <sup>2-</sup>	1.667
mg L <sup>-1</sup> Mo <sup>6+</sup>	mg L <sup>-1</sup> Na <sub>2</sub> MoO <sub>4</sub>	2.146
mg L <sup>-1</sup> N	mg L <sup>-1</sup> NH <sub>3</sub>	1.216
mg L <sup>-1</sup> N	mg L <sup>-1</sup> NO <sub>3</sub> <sup>-</sup>	4.427
mg L <sup>-1</sup> Cl <sub>2</sub>	mg L <sup>-1</sup> NH <sub>2</sub> Cl	0.726
mg L <sup>-1</sup> Cl <sub>2</sub>	mg L <sup>-1</sup> N	0.197
mg L <sup>-1</sup> NH <sub>3</sub> -N	mg L <sup>-1</sup> NH <sub>3</sub>	1.216
mg L <sup>-1</sup> NH <sub>3</sub> -N	mg L <sup>-1</sup> NH <sub>4</sub> <sup>+</sup>	1.288
mg L <sup>-1</sup> NO <sub>2</sub> <sup>-</sup>	mg L <sup>-1</sup> NaNO <sub>2</sub>	1.5
mg L <sup>-1</sup> NO <sub>2</sub> <sup>-</sup>	mg L <sup>-1</sup> NO <sub>2</sub> <sup>-</sup> -N	0.3045
mg L <sup>-1</sup> NO <sub>2</sub> <sup>-</sup> -N	mg L <sup>-1</sup> NaNO <sub>2</sub>	4.926
μg L <sup>-1</sup> NO <sub>2</sub> <sup>-</sup> -N	μg L <sup>-1</sup> NaNO <sub>2</sub>	4.926
mg L <sup>-1</sup> NO <sub>2</sub> <sup>-</sup> -N	mg L <sup>-1</sup> NO <sub>2</sub> <sup>-</sup>	3.284
μg L <sup>-1</sup> NO <sub>2</sub> <sup>-</sup> -N	μg L <sup>-1</sup> NO <sub>2</sub> <sup>-</sup>	3.284
mg L <sup>-1</sup> NO <sub>3</sub> <sup>-</sup> -N	mg L <sup>-1</sup> NO <sub>3</sub> <sup>-</sup>	4.427
mg L <sup>-1</sup> PO <sub>4</sub> <sup>3-</sup>	mg L <sup>-1</sup> P	0.3261
μg L <sup>-1</sup> PO <sub>4</sub> <sup>3-</sup>	μg L <sup>-1</sup> P	0.3261
mg L <sup>-1</sup> PO <sub>4</sub> <sup>3-</sup>	mg L <sup>-1</sup> P <sub>2</sub> O <sub>5</sub> <sup>-</sup>	0.7473
μ L <sup>-1</sup> PO <sub>4</sub> <sup>3-</sup>	μg L <sup>-1</sup> P <sub>2</sub> O <sub>5</sub> <sup>-</sup>	0.7473
mg L <sup>-1</sup> SiO <sub>2</sub>	mg L <sup>-1</sup> Si	0.4674
μg L <sup>-1</sup> SiO <sub>2</sub>	μg L <sup>-1</sup> Si	0.4674

Check source document prior to performing calculations

<http://www.hach.com/fmmimgach?/CODE%3AWAHINTRO5890%7C1>

## 8 Minor Laboratory Equipment in lab 2.60

Beaker (100 mL plastic use for pH)  
Beaker (100 mL) glass  
Beaker (1000 mL glass)  
Beaker (1000 mL plastic)  
Beaker (150 mL)  
Beaker (250 mL)  
Beaker (50 mL)  
Conical flask (125 mL)  
Conical flask (250 mL)  
Conical flask (500 mL)  
Crucibles  
Evaporating basin  
Filtration stands  
Funnel (small)  
Magnetic stirrer  
Measuring cylinder (10 mL)  
Measuring cylinder (100 mL)  
Measuring cylinder (1000 mL)  
Measuring cylinder (25 mL)  
Measuring cylinder (250 mL)  
Measuring cylinder (50 mL)  
Measuring cylinder (500 mL)  
Metal preparation equipment (sieve holders)  
Mortar (ceramic)  
Pestle (ceramic)  
Pipette (1 – 25 mL)  
Riffle box  
Shaker (end-over-end)  
Shaking bottle  
Sieve  
Sieve  
Spectrophotometer  
Stirrer  
Turbidimeter  
Universal containers (30 mL plastic)  
Volumetric flask (10 mL)  
Volumetric flask (100 mL)  
Volumetric flask (1000 mL)  
Volumetric flask (200 mL)  
Volumetric flask (25 mL)  
Volumetric flask (250 mL)  
Volumetric flask (50 mL)  
Volumetric flask (500 mL)  
Watch glasses

**NB All glassware must be cleaned before use**

## 9 Housekeeping

Any samples left in the laboratories (especially in fridges) without your details (name, date and e-mail) will be disposed of. To reduce contamination and exposure of harmful substances to others clean your work area before commencing lab work and again after you have completed. If you have to leave an experiment in progress leave a note with the equipment providing the information as above and also specify any hazards that are associated with the samples or reagents.

NB space is at a premium in the laboratories please be considerate of others.

### 9.1 Cleaning Equipment

All equipment must be thoroughly cleaned before use.

1. Clean plastic and glassware with detergent, hot water and a brush.
2. Rinse thoroughly with tap water.
3. Rinse three times with deionised water.
4. Allow to oven to dry if necessary at 50 – 100 °C (do not put any plastics in the oven at temperatures above 50 °C, including plastic trays!)

All equipment used in the collection, preparation and analysis of samples for metals should be acid washed overnight prior to use.

1. Clean plastic and glassware using detergent, hot water and a brush.
2. Submerge equipment in a nitric acid bath in lab 2.58. Ensure the insides of containers/flasks are filled. Warning – 0.5 M nitric is corrosive, wear lab coat, goggles and nitrile gloves.
3. Remove equipment from acid bath and rinse three times with deionised water.

### 9.2 Sample Storage

Water samples may be stored in the fridges in lab 2.60 for a maximum of 2 months. Leave a note on the box in the plastic wallet on the front of each grey container with the samples with your name, date and e-mail. When you have checked your data or after two months dispose of your samples. Unlabelled samples will be disposed of. See Miles or Kelvin if the trays aren't labelled.

Soil samples may be stored in the green plastic storage boxes, under the benches in lab 2.60, until the dissertation deadline. Leave a note in the plastic pouch on the front of the box with your name, date, e-mail and sample disposal date. When you have completed your analysis, dispose of your samples via the Healthcare waste disposal route.

Invertebrate samples can be stored in the green storage boxes in lab 2.60. Label the samples to indicate that they contain ethanol. When you have completed your lab work dispose of the samples in the ethanol waste container provided.

### 9.3 At the End of the Lab Session

Ensure that all the equipment you have used has been cleaned and returned to its original location. Clean the work surfaces and return samples to storage. All equipment should be thoroughly cleaned after use, but please pay particular attention to cleaning balances and spectrophotometers. Balances should be gently brushed to remove particulates and spectrophotometers be wiped with a damp cloth. Dispose of chemicals following procedure in COSHH assessment. Dispose of any unwanted samples.

As space is limited it is not permitted to leave equipment/samples out on the bench in between lab sessions. Do not leave equipment in soil storage containers.

Soil samples from outside the EU must be disposed of following EU protocol, please see Rachel Gasior for advice and copies of the laboratory protocols.

## 10 Quality Control

### 10.1 Certified Reference Materials

Wherever possible it is desirable to include a Certified Reference Material CRM with each batch of samples, as a means of verifying the accuracy of a procedure. The CRM is supplied with a certificate of analysis stating the analytes present, the concentration of each analyte and the measurement uncertainty. The disadvantages of CRMs are: the expense and lack of availability. Ask the technician if a CRM is available.

### 10.2 Blank Samples

A blank sample is a sample that is taken through all the steps of a procedure with the reagents only. You will ideally include 3 blank samples in every procedure: for water samples, replace sample with deionised water; for soil/sediment samples use the reagents only e.g. in the extraction of Ca with ammonium acetate, measure 125 mL of extract solution into an empty container take this through the same process as the soil samples. If the analyte of interest is present in the reagents, the amount that has been added to the sample can be deducted i.e.  $\text{mg L}^{-1}$  result obtained for the sample –  $\text{mg L}^{-1}$  result for the blank = true  $\text{mg L}^{-1}$  result in the sample.

### 10.3 Duplicate samples

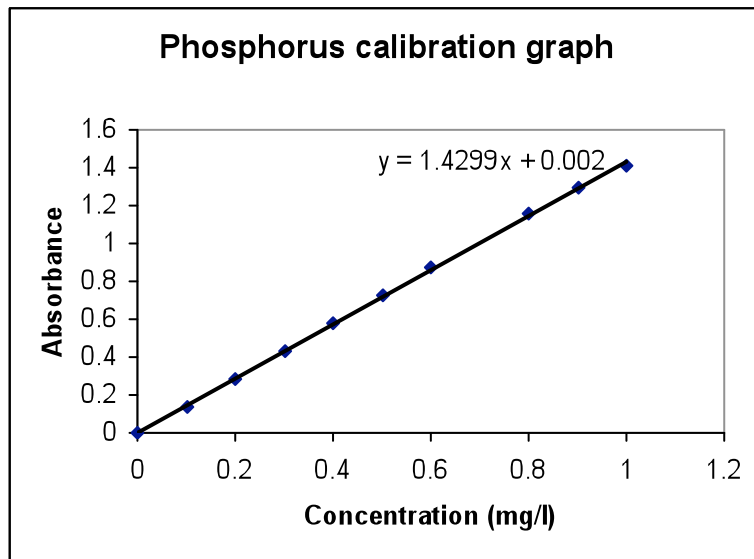
As part of your quality control scheme you should include a number of duplicate samples. The term describes two separate test portions that are subjected to the same test procedure. The number of duplicate samples depends on your project, but 10 % of the total number of samples is a good guide.

### 10.4 Calibration Graphs

For some techniques the concentration of the unknown sample is determined using a calibration graph see fig 1.

- Prepare a series of standards containing the analyte of interest (see the section later on how to prepare calibration standards).
- Measure the response of the samples e.g. by AAS or spectrophotometer.
- Plot a calibration graph of concentration v response, use excel or a calculator in linear regression mode.
- Give the graph a title, date and label axes.
- Draw a line of best fit and determine the correlation coefficient (r). 'A correlation coefficient of >0.999 is generally considered as evidence of an acceptable fit of the data to the regression line' [4].
- If the r value is unacceptable, repeat the process.
- If the r value is acceptable measure the response of the samples.
- Calculate the amount of analyte in the sample using the calibration graph.  
You know the values for y, m and c, so you can rearrange the equation of the straight line to determine x.
  - y = response (absorbance)
  - x = concentration ( $\text{mg L}^{-1}$ )
  - m = gradient
  - c = intercept
- Ensure that the absorbance figures for your samples are within the calibration range. If the absorbance figures for your samples are greater than the absorbance of the top standard, the samples must be diluted.

Fig 1. Example of a calibration graph



## 11 Calculations

### 11.1 Preparing calibration standards

How to prepare a set of five calibration standards in the range 0 – 10  $\mu\text{g ml}^{-1}$  ( $\text{mg L}^{-1}$ )

Assuming that we are starting with a 1000  $\mu\text{g ml}^{-1}$  stock solution, then you will need: six 100 mL volumetric flasks; one 100 mL beaker; and, an automatic pipette with pipette tip (100 – 1000  $\mu\text{L}$ ).

- Ensure that all glassware is clean and beakers are dry.
- Use the equation below to calculate the amount of stock solution needed in each flask:

$$\frac{\text{Concentration required, } \mu\text{g mL}^{-1}}{\text{Concentration of stock solution, } \mu\text{g mL}^{-1}} \times \text{Volumetric flask size, mL}$$

$\mu\text{g mL}^{-1}$  cancel out, leaving you with the amount of stock solution in mL required in 100 mL.

e.g. to prepare a 2  $\mu\text{g mL}^{-1}$  standard from a 1000  $\mu\text{g mL}^{-1}$  stock solution in a 100 mL volumetric flask:

$$\frac{2 \mu\text{g mL}^{-1}}{1000 \mu\text{g mL}^{-1}} \times 100 \text{ mL} = 0.2 \text{ mL}$$

There are 1000  $\mu\text{L}$  in 1 mL  $\therefore 0.2 \text{ mL} \times \frac{1000 \mu\text{L}}{1 \text{ mL}} = 200 \mu\text{L}$

You will need 0, 200, 400, 600, 800 and 1000  $\mu\text{L}$  to prepare 0, 2, 4, 6, 8, 10  $\mu\text{g mL}^{-1}$  in 100 mL volumetric flasks.

- Label a beaker with 1000  $\mu\text{g mL}^{-1}$  stock solution and transfer  $\approx 4 \text{ mL}$  of the stock solution into the beaker.
- Label each volumetric flask.
- Pipette the required amount of stock solution into each flask and dilute to volume with deionised water.
- Stopper the flask and shake thoroughly to mix.

If for instance you required a 0.2  $\mu\text{g mL}^{-1}$  standard, the amount needed 20  $\mu\text{L}$ , cannot be accurately measured using automatic pipettes. In this case, prepare an intermediate standard from the stock solution. Prepare a 100  $\mu\text{g ml}^{-1}$  solution from the 1000  $\mu\text{g ml}^{-1}$  use this intermediate solution to prepare the working standards.

If higher concentrations are required, 20, 40, 60, 80 and 100  $\mu\text{g mL}^{-1}$ , the total amount of stock solution required would be 30 mL: this is too much to use from a stock solution which are typically supplied in 100 mL bottles. In this case use smaller volumetric flasks or try to obtain a more concentrated stock solution.

### 11.2 Converting the concentration of the extract ( $\text{mg L}^{-1}$ ) to the concentration in the original soil sample ( $\text{mg/kg}$ )

$$\text{mg/kg} = \frac{(A - B) \times (C)}{D}$$

Where

A = reading for sample,  $\text{mg L}^{-1}$

B = reading for blank,  $\text{mg L}^{-1}$

C = volume of extract solution added to soil/ sediment sample, ml

D = weight of soil/ sediment, g

### 11.3 Converting $\text{mg L}^{-1}$ to ppm

$$\text{mg L}^{-1} = \text{ppm}$$

### 11.4 Preparing a molar solution from a solid

Molarity is a term used to denote molar concentration, expressed as moles of solute per litre volume of solution ( $\text{mol L}^{-1}$ ). The concentration can be expressed in grams per litre ( $\text{g L}^{-1}$ ) or in moles per litre ( $\text{mol L}^{-1}$ ) of the dissolved substance. The unit  $\text{mol L}^{-1}$  is the same as  $\text{mol dm}^{-3}$  and M, and is indicated by square brackets [ ].

You will need a volumetric flask and stopper, spatula, beaker or weighing boat, COSHH assessment and an accurate balance.

- Ensure that all glassware is clean and beakers are dry.
- Use the equation below to calculate the amount of solute needed:

$$\text{Mass of solute (g)} = \text{Solution volume (L)} \times \text{Molarity (mol L}^{-1}\text{)} \times \text{Molar Mass (g mol}^{-1}\text{)}$$

Dimensionally, L and mol cancel leaving g. You know the solution volume and molarity, the molar mass of a solid can be found on the side of the chemical bottle.

- Accurately weigh the solid into a beaker or plastic weighing boat.
- Transfer the solid to the volumetric flask.
- Use a wash bottle containing deionised water to rinse any solid residue into the flask and dilute to volume with deionised water.
- Stopper the flask and shake thoroughly.
- Clear any spillages, paying particular attention to the balance.

N.B. if the dissolution reaction produces heat, allow the contents of the flask to cool, and then make up to the mark with deionised water.

If the solid doesn't immediately dissolve, place the flask in the sonic bath until it does then dilute to volume.

e.g. to prepare a  $1 \text{ mol L}^{-1}$  solution of ammonium acetate in 2 litres

Solution volume: 2 L  
Molarity required: 1 M  
RMM of ammonium acetate: 77.08 g

$$\text{Mass of solute} = 2 \text{ L} \times 1 \text{ mol L}^{-1} \times 77.08 \text{ g mol}^{-1}$$

$$\text{Mass of solute} = 154.16 \text{ g}$$

### 11.5 Preparing a molar solution from a liquid

You will need a volumetric flask and stopper, measuring cylinder or pipette, beaker and COSHH assessment.

- Ensure that all glassware is clean and that beakers are dry.
- Use the equation below to determine the volume of liquid required to prepare a 1 mol L<sup>-1</sup> solution in 1 L:

$$\frac{100 \%}{\% \text{ of chemical}} \times \frac{\text{Molar mass g mol}^{-1}}{\text{specific gravity}}$$

- Use the value obtained in the equation below to work out how much of the solute needed:

$$\text{Volume of solute, mL} = \frac{\text{Solution volume (L)} \times \text{Molarity (mol L}^{-1}\text{)} \times \text{Molar Mass (g mol}^{-1}\text{)}}{1000}$$

- The liquids most frequently prepared are from concentrated acids. The procedure must therefore be carried out whilst working in a fume cupboard. Obtain the specific COSHH assessment. ALWAYS ADD ACID TO WATER.
- Transfer an amount slightly greater than the amount of the solute needed into a beaker.
- Half fill the volumetric flask with deionised water.
- Use a measuring cylinder or pipette to transfer the solute to the volumetric flask.
- Dilute to volume using deionised water.
- Stopper the flask and shake thoroughly. You may now remove the flask from the fume cupboard.

**NB if the dissolution reaction produces heat, allow the contents of the flask to cool, then make up to the mark with deionised water.**

**NB Do not put volumetric flasks in the ovens or fridges.**



# Fieldwork Guide



## Field Equipment

### 12 Booking field equipment

When you book field equipment you must also ensure that your Risk Assessment has been completed (<http://www.geog.leeds.ac.uk/studentinfo/index.html>). Risk Assessments are a “pass to progress” element of your dissertation and must be submitted towards the end of your second year. You should complete the assessment and discuss this with your nominated mentor before submission to John Corr (further information will be provided in the dissertation guide provided by Louise Waite and Frances Drake). Field equipment will not be provided unless an assessment has been completed. It is your responsibility to gather information to assist you in filling in this form.

- The first point of contact for booking field kit is Miles Ratcliffe.
- Collect a ‘Field Equipment Request’ form from the notice board outside room 2.56 or download a copy from: <http://www.geog.leeds.ac.uk/support/labs/index.html>
- Complete a new form for each booking.
- Enter your details and clearly list the equipment required. Be specific: if you want a tape, what size? plastic bags, how many? level, to what accuracy?
- Hand the form to the technicians in room 2.56 at **least one week before the equipment is needed.**
- Have you requested the right containers for your analysis? see the table below. See the table on page 35.
- The technician will make an appointment for you to collect the equipment. You can request an equipment demonstration if you haven’t used it before.
- Collect the equipment at the time and date given. When the technician demonstrates the equipment to you, please make notes to ensure you can use the equipment correctly and return it in proper working order.
- If you experience any problems in the field please contact the lab staff on 0113 343 3314.
- A £25 deposit will be taken when you collect the equipment please make cheques payable to the University of Leeds, cash is not accepted. Debit card forms are also available.
- When equipment is returned on time and clean, your cheque will be returned. **IF EQUIPMENT ISN'T RETURNED BY 5.00 PM ON THE EQUIPMENT DUE DATE, YOUR CHEQUE WILL BE CASHED. TO PREVENT YOUR MONEY BEING CASHED BY MISTAKE ENSURE THAT THE TECHNICIANS DESTROY YOUR VISA CARD FORM OR CHEQUE.**
- If you need to extend the loan period e-mail [geo-labtechs@leeds.ac.uk](mailto:geo-labtechs@leeds.ac.uk)
- Do not leave wet equipment in closed boxes.
- Ensure all electrode caps are replaced to prevent electrodes drying out.
- Allow yourself sufficient time to return to the labs to preserve samples.
- The Graphics unit (room B01) is able to provide Dictaphones upon request
- Instruction manuals can be found on the labs website in the field equipment section.
- **Equipment will not be issued unless you have a signed risk assessment and deposit.**



## Sample containers

Analysis	Min Vol of sample req <sup>d</sup> , ml, for analysis <sup>1</sup>	Container	Container treatment	Comment
Anions	2	125 ml PP	None	Anions: fluoride, chloride, nitrite, nitrate, sulphate
Cations	10	125 ml PP	None	Cations: calcium, magnesium, sodium and potassium
Biological oxygen demand (BOD)	50	50ml flip top container	Clean in situ before collecting sample	Fill container to the brim, exclude from light, return to the lab asap to incubate the samples for 5 days at 20°C – if you cannot return to the lab keep samples in a box. COSHH required.
Metals	15	125 ml PP	Acid wash	Do not use the same container for collecting samples for nutrients and anions. COSHH required.
Invertebrate	n/a	Twistloc bags	None	When the samples have been preserved label the bag to show it contains ethanol. COSHH required.
Phosphorus	10	125 ml PP	None	COSHH required.
Total Phosphorus	20	125 ml PP	None	COSHH required.
Dissolved organic carbon (DOC)	5	125 ml PP	None	Check sample restrictions
Nutrients	15	125 ml PP	None	Nutrients: ammonia, nitrate, nitrite. Check sample restrictions
Silica	10	125 ml PP	None	
pH	5	125 ml PP	None	Best measured in the field
Conductivity	50	125 ml PP	None	Best measured in the field

### NB

PP - polypropylene

PP 125 ml containers are used once and disposed of to prevent contamination of sample

<sup>1</sup>Double the quantity of sample should be collected for replicate samples for quality control – a duplicate sample should be collected every 10 samples

## 13 Field Equipment Available

### 13.1 Sampling for soil physical analysis

- Auger (Dutch, Screw, Bucket/Posthole, Gouge)  
see <http://www.vanwalt.co.uk/demonstrations1.htm> for auger demonstration
- Bulk density rings
- Infiltration rings  
see  
<http://www.geopacks.com/MapMarketing/geopacks/manuals/Geopacks%20Infiltrometer.pdf#s>  
each=%22mjp%20geopacks%22 for infiltration guide
- Mini disk infiltrometers
- Russian peat corer

### 13.2 Soil physical analysis

- Cone penetrometer
- Moisture meter (Theta probe)  
see <http://www.mea.com.au/products/theta-probe/> for further information
- Munsell colour chart

### 13.3 Sediment sampling

- Grab sampler (Van Veen)  
see <http://www.vanwalt.co.uk/demonstrations7.htm> to see this in action
  - Corer (piston)
  - Russian corer (ideal for peats)
  - Livingstone piston corer (ideal for lake sediments)
  - Gouge and Dutch augers
- Discuss your plans well in advance with a member of staff – special training may be required

### 13.4 Sampling for water analysis

- LaMotte water sampling kit (water colour comparator, Secchi disk, water sampler, algae sampler, Van Veen grab)  
see <http://www.vanwalt.co.uk/demonstrations7.htm> to see this in action

### 13.5 Meteorological equipment

- Anemometers (air-flow meter)
- Atmospheric data centre
- Hygrometer
- Pyranometer
- Net radiometer
- Milton multiband radiometer
- Temperature meter (air/ground)
- Wind vane
- Wind watches

### 13.6 Hydrometry

- Conductivity meter
- Dissolved oxygen meter
- Flow meters (water flow)  
see <http://www.geopacks.com/MapMarketing/geopacks/manuals/Geopacks%20FlowMeter.pdf>  
for instructions
- Handheld fluorometer – Phycocyanin and turbidity  
<http://www.turnerdesigns.com/t2/instruments/aquafluor.html>
- pH/temp meter
- Rain gauge (tipping bucket)

### **13.7 Ground Surveying**

- Echo sounder
- GPS (hand-held)
- Levels (Dumpy, Kern, Abney, Quick set)
- Plane table
- Total station
- Trundle wheel
- Tripod

### **13.8 Other**

- Clinometer
- Compass
- First aid kits
- Gas sampler (Drager)
- Meter rule
- Quadrat
- Tape (100 m)
- Tape (30 m)
- Tape (5 m)
- Tape (diameter 5 m)
- Wellington boots
- Waders

If the equipment you require is not on this list please see the technicians

- Make sure you understand the objectives of the fieldwork, the potential hazards and the appropriate responses to such hazards, before you set out.
- Your work must be designed carefully, to allow for the experience of the participants and the locations visited. Don't overestimate what can be achieved – fieldwork is more demanding than laboratory work.
- Any physical disabilities must be brought to the attention of your dissertation mentor, so that appropriate precautions can be taken.
- Never work under the influence of alcohol or drugs other than prescribed medicines.
- A comprehensive first aid kit **must** be carried. Be alert for hypothermia or heat exhaustion.
- Never work alone without the permission of the organiser or leader.
- Make sure that you can read a map and use a compass: your group should have both.
- Your clothing (see table below) and equipment must be suitable for all of the weather conditions likely to be encountered during the work.
- Check the weather forecast before departure: look for changes in the weather at all times and do not hesitate to turn back if necessary.
- Leave full details of your intended working locations, routes and times. Never change these arrangements without informing someone.
- Make sure that you know the international distress calls.
- Always wear a life jacket when working in or on water.
- Check tide times when working on the coast, and try to work on a falling tide.

List of PPE for fieldwork (if you require anything else, please specify)

Riggers' gloves	Vinyl/nitrile gloves
Ear defenders	Overalls
Safety footwear	Waders (thigh length)
Life jackets	Hard hat
Fluorescent tabard	Rope
Blankets	GPS

Clothing and Climate

<b>Cold weather</b>	
Head	Warm, waterproof wear in winter
Trunk	Thick wool/fibre jumper over normal shirt and underwear. Waterproof, not showerproof, jacket/cagoule/macintosh which is also windproof
Legs	Loose-fitting, heavy-duty trousers containing wool/cotton fibres. <u>Jeans are not advisable</u>
Feet	Thick socks and stout boots giving ankle support. Wellington boots are not suitable for long walks

<b>Hot weather</b>	
Head & Trunk	Keep covered with thin, light-coloured garments to avoid sunstroke
Exposed areas	Use a high-factor sun-block lotion

**15 Recommended Texts**

The following texts may be borrowed from the technicians to photocopy or read in the laboratory:

Allen, S.E., (ed.) (1989) *Chemical Analysis of Ecological Materials*. Blackwell Scientific Publications, Oxford.

Dean, J.R., *et al.* (2002) *Practical Skills in Chemistry*. Prentice Hall, Harlow.

Gupta, P.K. (2000) *Soil, Plant, Water and Fertiliser Analysis*. Agrobios, Jodhpur.

Hesse, P.R. (1971) *A Textbook of Soil Chemical Analysis*. John Murray, London.

Jones, A., *et al.* (2000) *Practical Skills in Environmental Science*. Prentice Hall, Harlow.

Rowell, D.L. (1996) *Soil Science: Methods and Applications*. Longman, Harlow.

Sparks, D.L., (ed) (1996) *Methods of Soil Analysis Part 3 Chemical Methods*. Soil Science Society of America, Madison.

Carter, M.R., Gregorich, E.G. (eds.) (2008) *Soil Sampling and Methods of Analysis*, 2<sup>nd</sup> Ed. Taylor and Francis Group, Florida.

Prichard, E., 1995. *Quality in the Analytical Chemistry Laboratory*. Chichester: John Wiley and Sons Ltd.

Sykes, J.M., Lane, A.M.J. (eds) (1996) *The United Kingdom Environmental Change Network Protocols for Standard Measurements at Terrestrial Sites*. London: The Stationery Office

## **16 Websites**

### **Analysis**

Methods of Phosphorus Analysis for Soils, Sediments, Residuals and Waters

[http://www.sera17.ext.vt.edu/Documents/Methods\\_of\\_P\\_Analysis\\_2000.pdf](http://www.sera17.ext.vt.edu/Documents/Methods_of_P_Analysis_2000.pdf)

Conversions - chemical species

<http://www.hach.com/fmmimghach?/CODE%3AWAHINTRO5890%7C1>

Sea Water – dissolved constituents

[http://www.cleanwaterstore.com/technical/water-sources/body\\_sea\\_water.html](http://www.cleanwaterstore.com/technical/water-sources/body_sea_water.html)

Trace analysis – reliable measurements

<http://www.ivstandards.com/tech/reliability/>

Soil, plant and feed analysis

[http://groups.ucanr.org/danranlab/Methods\\_of\\_Analyses545/](http://groups.ucanr.org/danranlab/Methods_of_Analyses545/)

### **Field equipment**

Van Walt - augers

<http://www.vanwalt.com/demonstrations-environmental-water-level.htm>

MJP Geopacks – Flow meters, infiltration guide

<http://www.geopacks.com/manuals.aspx>

Garmin – GPS for beginners

[http://www8.garmin.com/manuals/GPSGuideforBeginners\\_Manual.pdf](http://www8.garmin.com/manuals/GPSGuideforBeginners_Manual.pdf)

Garmin – Instruction manuals

<http://www8.garmin.com/support/userManual.jsp?market=3&subcategory=37&product=All>

## 17 Bibliography

[1] The UK Environmental Change Network Protocols for Standard Measurement at Terrestrial Sites

[2] International Standard, (1994). ISO11464:1994(E). *Soil Quality – Pretreatment of samples for physico-chemical analysis*.

[3] Bartlett, R.J. (1981) Oxidation-reduction status of aerobic soils (Chapter 5), in: *Chemistry of the soil environment*. American Society of Agronomy. Soil Science Society of America. ASA Special publication No. 40, Madison Wisconsin. pp. 77-103

[4] Green, M.G. (1996) A Practical Guide to Analytical Method Validation in *Analytical Chemistry* 68, p. 305A-309A

[5] Jones, A. (2000) Duck, R., Reed, R. and Weyers, J. *Practical skills in environmental science*. Prentice Hall. p. 7



**SCHOOL OF GEOGRAPHY**  
**STUDENT COSHH RISK ASSESSMENT FORM**



**NOTE: Before completing this assessment you must read all method sheets and COSHH information available. Use this information to complete this assessment form.**

<b>Location of work:</b>	<b>Dates: From</b> _____ <b>to</b> _____
<b>Student Name:</b>	

<b>Summary of experiment (s)</b>
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	Hazardous substances (if mixing chemicals creates a dangerous mixture please note and complete a separate line for this mixture)	Quantity used	Frequency	Duration of contact	Chemical hazard classification <sup>1</sup>	Personal protection/control measures (tick all that apply)				Exposure/contamination <sup>2</sup>	Is health surveillance required?  Yes/No
		g/ml	Hours/day	Hrs		Fume cupboard	Vinyl Gloves	Goggles	Other (specify)		
<b>E.G</b>	acetic acid 99%	200ml	daily	<1 (30min)	Harmful	√	√	√		Standard procedures	No
1											
2											
3											
4											

<sup>1</sup> Chemical hazard classification : Very Toxic, Toxic, Flammable, Corrosive, Harmful, Irritant (these can be found on the chemical container)

<sup>2</sup> Standard procedures are listed below. If special procedures are required these must be stated above.

- a. **Mouth, Eyes, Skin exposure** – flush area of contamination with plenty of water. Contact a member of the lab staff.
- b. **Inhalation** – notify lab staff. Move casualty to a well ventilated area.
- c. **Ingestion** – notify lab staff, medical attention will be immediately sought.

	Hazardous substances	Hazard potential	Likelihood of occurrence	Risk Ranking	Spillages <sup>3</sup>	Disposal procedure (for liquid/spillage kits)	
						General disposal <sup>4</sup>	Special waste <sup>5</sup>
E.G	acetic acid 99% (glacial)	2	1	2	Use absorbent granules	√	
1							
2							
3							
4							

Hazard Potential		Likelihood of Occurrence	
3	High - Loss of life/ Permanent disability/ Major injury.	3	High - once or several times per day/ or per activity
2	Medium - Serious Injury or illness requiring first aid and medical treatment.	2	Medium - once or several times per month/ or per activity
1	Low - Minor/ non-disabling injury or illness requiring immediate control action then return to work.	1	Low - once or few times per year / or over lifetime of project

**Risk Ranking = Hazard Potential x Likelihood of Occurrence :**

1 and 2	= <b>LOW Risk</b>
3 and 4	= <b>MEDIUM Risk</b>
6 and 9	= <b>HIGH Risk</b>

<sup>3</sup> Use absorbent granules for neat chemical spillages. Weak chemicals can be absorbed using paper towels. Report ALL spillages immediately to a laboratory technician.

<sup>4</sup> solutions can be washed down laboratory sink with copious amounts of water

<sup>5</sup> solutions must be collected in clearly labelled chemical waste drums for special disposal. DO NOT PUT SOLUTIONS DOWN THE SINK.

Declaration	
I, _____ have read and understood all the method sheets and standard COSHH information sheets provided by the laboratory staff relating to the experiment(s) outlined in this COSHH form. Using the information available I have assessed the risks involved with my experiment(s). I agree to abide by the laboratory rules and regulations and will wear all Personal Protective Equipment at all times when using the listed chemicals. I understand that failure to do so will result in expulsion from the laboratories.	
Signed (student)	Date
<b>This assessment has been reviewed by a laboratory technical and assessed as safe</b>	
Signed (technician)	Date

