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##### Lab manual for UCL clean lab

**ILLOGIC 3B**

**(Isotope Lab, London Geochemistry and Isotope Centre 3B)**

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##### Introduction

This handbook is intended to provide an introduction to geochemical clean labs, the basic principles underlying the maintenance of a safe and clean working environment and an outline of basic protocols in the metal free clean lab at UCL Earth and Planetary Sciences. It is very important for both i) the personal safety and ii) the clean and successful processing of samples, that all users understand and follow the guidelines laid out here. To this end, new users must be 100% supervised by an experienced user until such time as they have demonstrated that they can work safely and cleanly, after which they are permitted to work independently. Reading this manual is part of that training.

This manual will not tell you how to process your samples since the specific procedures used vary depending on the sample material being analysed and the element(s) being purified; for this, you will be individually instructed by an experienced user.

##### What is a clean lab?

The main purpose of a clean lab is to minimize the contamination of samples so as to increase the accuracy of results. The lower the concentration of the element of interest in the sample, the cleaner the preparation must be (Table 1.1). The first step in reducing contamination is clean particle free (or at least particle reduced) air.

**Table 2.1:** Potential contaminants.

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Element ppm** | **House dust** | **Street dust** | **Garden soil** | **Saliva (ppb)** |
| Aluminum | 25948 | 47540 | 55841 |  |
| Antimony | 7.28 | 0.89 | 0.36 |  |
| Arsenic | 7.3 | 1.3 | 3.0 |  |
| Barium | 492 | 576 | 766 | 12.76 |
| Beryllium | 0.56 | 0.97 | 1.40 |  |
| Bismuth | 1.67 | 0.05 | 0.08 |  |
| Cadmium | 6.46 | 0.37 | 0.30 | 0.674 |
| Calcium | 48760 | 96787 | 26978 |  |
| Chromium | 86.7 | 43.3 | 44.8 |  |
| Cobalt | 8.92 | 8.31 | 8.36 |  |
| Copper | 206.08 | 65.84 | 13.19 | 18.44 |
| Iron | 14135 | 18948 | 21481 |  |
| Lead | 405.56 | 39.05 | 64.69 | 5.08 |
| Lithium | 6.3 | 7.4 | 11.3 |  |
| Magnesium | 9826 | 15780 | 8937 |  |
| Manganese | 269.3 | 431.5 | 525.3 |  |
| Mercury | 3.633 | 0.029 | 0.107 |  |
| Molybdenum | 3.16 | 1.39 | 0.64 |  |
| Nickel | 62.9 | 15.2 | 16.3 |  |
| Phosphorus | 1380 | 616 | 1240 |  |
| Potassium | 10305 | 14963 | 18035 |  |
| Rubidium | 25.3 | 37.2 | 52.0 |  |
| Selenium | 1.2 | 0.5 | 0.7 |  |
| Silver | 2.05 | 0.22 | 0.31 |  |
| Sodium | 23224 | 18273 | 22042 |  |
| Strontium | 255 | 459 | 360 |  |
| Tellurian | 0.08 | 0.08 | 0.04 |  |
| Thallium | 0.14 | 0.21 | 0.29 |  |
| Tin | 54.84 | 3.02 | 1.65 |  |
| Uranium | 0.58 | 0.82 | 1.17 |  |
| Vanadium | 24.8 | 34.0 | 46.8 |  |
| Zinc | 716.9 | 112.5 | 113.7 | 127.1 |

###### Design

In order to achieve a sufficiently high level of cleanliness all air entering the Clean Lab passes through a series of filters before it enters the Metal Free Lab. After initial filtration it is forced through high efficiency particulate air (HEPA) filters by fans. The initial bag filters and primary HEPA filters are on the roof. The air then enters dedicated ducting serving the Clean Lab and passes through secondary HEPA filters located above the roof, and enters the lab. Extraction of air occurs in the lab occurs in two ways: 1) though the laminar flow hoods, via the holes above the slides. That air passes through another set of HEPA filters, through the working space, and is extracted through the bottom of the hood back outside; 2) via ducts in the lobby to maintain positive air pressure in the lab. The plant room filters and ducting are the responsibility of Estates whereas we are responsible for the secondary HEPA filters accessed from within the Clean Lab. The air handling in the Clean Lab is controlled by the Building Management System (BMS). This system monitors airflows and flags up errors when airflows are not at their set points.

The specification for air in the lab is approx. ISO Class 6 (equivalent to Class 1,000). The lab is at positive pressure with respect to the lobby, and the lobby is at positive pressure with respect to the outside rooms. Thus airflow in the Clean Lab cascades outwards from the interior towards the exterior of the lab and dust should not blow in (e.g. when a door is opened). However, it is still best practice not to open two doors at the same time as this may disrupt the general outward flow of air by creating eddies.

Particles can also travel into the lab on clothing and shoes, for this reason everyone entering the Clean Lab must wear specially provided lab coats/coveralls and shoes/shoe covers. Particles transferred in this way include dust on you or your clothes, lint from your clothing (especially your socks) and dead skin (Table 1.1).

Within the lab, an additional level of cleanliness is provided by the hoods. These are also the primary means of reducing our exposure to harmful chemicals. Our LAF hood specification is ISO Class 5 (equivalent to Class 100) in the rear 75% of the work space, even with the sash fully up.

There are six multi-use Class 100 laminar flow hoods and one fume hood in the clean lab:

1. The two smaller laminar flow hoods (one red, one green) are nominally for acid evaporation. The red hood contains a perchloric scrubber. The green hood is Boron-free.
2. The other four larger laminar flow hoods (three blue, one green) are working hoods nominally for column chemistry. The green hood is Boron-free (green).
3. The fume hood (Blue) is nominally for acid distillation and/or beaker cleaning.

The laminar air-flow hoods are designed to protect both the *operator* from the chemicals inside the hood, and the *process* from external contamination. The design of these hoods also offers containment in the event of an acid spill.

The perchloric scrubber is designed to avoid condensation of perchloric acid vapour in the exhaust ducting. This is necessary due to the possibility of explosive reactions between perchloric acid vapour and organic materials (see Section 3.7.3).

###### Sources of contamination

Contamination is surprisingly easy, especially for isotope ratio measurements and low level trace element analyses (Table 1.1). In addition, with (MC-)ICP-MS it is not necessarily the analyte that is the contaminant. In many cases the contaminants are other elements which have isobaric interferences on analyte isotopes or polyatomic species having the same mass as analyte isotopes.

Examples:

Isobaric interferences – 180W2+ = 180Hf2+

Doubly charged species - 186W2+ = 93Nb+; 184W2+ = 92Zr+

Polyatomic species – 40Ar40Ar = 80Se; 40Ar16O = 56Fe, 23Na40Ar = 63Cu

###### Cleaning

In order to maintain a high level of cleanliness a clean lab must of course be cleaned regularly. All surfaces are washed and/or dusted regularly so that particles do not accumulate. Even so, air and human borne particles are not the only source of contamination.

Before newly delivered materials are brought into the Clean Lab, all unnecessary packaging, and most particularly cardboard boxes and other materials used as protection during delivery, needs to be removed since we have had no control over where this packaging has been. Materials brought into the lab from outside (new or old from elsewhere) also need to be cleaned, not only to remove any obvious dust adhering to them but also to remove any invisible residue from their manufacture. Furthermore all plastics need to be leached in acid for extended periods before use. This is because they contain trace levels of many elements that can be leached out over time once the plastic is in contact with an acidic (or any other) solution. Coloured plastics need to be avoided as far as possible because colours are produced by trace level addition of metals to the plastic. For example, yellow and red plastics may be produced by adding cadmium, iron or mercury based pigments, blue and green plastics by adding copper, cobalt and chromium based pigments, violet plastics could be Mn based, while white plastics may contain Pb, Zn, Ti and Ba based mineral pigments. With time the colour and the metals leach out of the plastic! Anything that is used and/or re-used in the lab must of course be cleaned before use. Similarly outdoor clothing and bags cannot be brought into the Clean Lab since they are much more likely to have been in contact with dirt and dust than indoor clothing.

##### Lab safety

It is very important for your personal safety that you follow the guidelines laid out here, are aware of the potential hazards of working in a clean lab and know the correct procedures for dealing with them. For this reason your initial work in the Clean Lab will be 100% supervised until your lab work supervisor is satisfied both that it is safe for you to work alone on specific tasks and that your work will not endanger other people or other people’s work.

###### Training

Your training should cover all cleaning and chemistry tasks necessary for your planned work in the clean lab. If you are not confident in any of these tasks after the initial training period, it is perfectly OK to request continued supervision until you are comfortable with the task(s).

1. **Induction and Authorisation to work declaration:** This is a formal declaration that you have read and understood this manual, the separate ‘local lab rules’ document, and all relevant Risk Assessments (RAs). Risk Assessments are stored on the RiskNET server.
2. **Authorised HF Users:** Anyone who intends to work with hydrofluoric acid must receive additional training from the responsible PI, as well as reading the relevant sections of this manual and the RA available on the RiskNET server. All HF users will be given a tube of HF antidote gel to take home, in case burn symptoms develop after work. Grab sheets to take to hospital are available in the lab.
3. **Authorised Users 8am – 8pm:** all users must aim to finish their work in the lab within UCL working hours (8am – 8pm). ***HF use is permitted only during core lab hours (9 am – 5pm)***. No new tasks should be undertaken without a supervised introduction and/or discussion of the proposed protocol. If approved work continues out of hours, no concentrated acids (>50%), and no HF may be used during this time. A “buddy” safety system must be employed, where the user informs a friend or colleague where they are going, and how long it will take.

###### The Law: Risk Assessment & COSHH

The government Health and Safety Executive requires employers to follow regulations for the control of substances hazardous to health (COSHH). Under these regulations both employers and employees have duties aimed at ensuring safety in the workplace. There are regular safety inspections to ensure that we are complying with these regulations (e.g., that our risk assessments and linked COSHH assessments are complete and up to date) and to check that there is no evidence of poor lab practice (e.g. unlabelled or inadequately labelled containers).

* + 1. Employer duties under COSHH

- Assess risk to workers.

Document the risks and safety measures associated with the various tasks carried out in the Clean Lab. These documents (risk assessments and linked COSHH assessments) are stored on the riskNET server, to which all lab users will be given access.

- Prevent or control exposure.

The primary methods used in the Clean Lab are engineering controls (i.e. our hoods), your personal protective equipment (PPE), this manual and your training.

- Ensure control measures are used and maintained properly.

This includes safety inspections, annual maintenance of the air handling plant by UCL Estates, annual testing of the individual hoods (LEV testing).

- Arrange health surveillance, where necessary.

For example, in the event of hydrofluoric acid or acid vapour exposure (see Sections 3.5).

- Inform, instruct and train workers.

Lab manual, initial supervision and training, annual lab safety-talk.

**The Managers** are responsible for:

* Risk assessments identifying the major risks associated with working in the clean lab, who might be harmed and what measures have been taken to control these risks.
* COSHH assessments (linked to RAs on riskNET) for tasks carried out in the lab, e.g., plastic cleaning, sample dissolution, ion exchange chromatography. These identify the specific hazards associated with individual tasks and should be consulted before tackling any unfamiliar job.
* Additional safety information on hydrofluoric acid.
* An up to date list of PAT tested electrical equipment.
  + 1. Employee duties under COSHH

Employees also have duties under COSHH regulations. You are expected to take control of your own safety. This includes:

-Reporting defects and problems.

Community spirit is needed when it comes to reporting defects promptly, watching out for your colleagues’ safety was well as your own, and opening discussions on safety issues if you have any concerns.

-Operating the hoods correctly.

From a safety perspective our major protection measures against inhalation of acid vapour and protection from acid spills are the hoods and the vented storage beneath them. You therefore need to know how to operate the hoods correctly for the various tasks you do. See Section 2 of this manual.

-Using your PPE.

The everyday PPE in the Clean Lab are your lab coat, gloves and safety glasses. These should be worn at all times. You should be particularly aware that there is no ideal glove for our work. See Section 3.6.

-Knowing the Clean Lab first aid procedures.

You should know where to find and how to use the HF antidote gel, eye-washes and first aid kits. Your ability to respond to an accident quickly and appropriately is particularly important for hydrofluoric acid accidents and chemical splashes in the eyes.

-Knowing your chemical safety.

You should ensure that all your reagents are properly labelled and appropriately handled and stored (Section 3.7 and 3.7.5).

-Following lab protocols.

Refer to risk assessments and this manual. If we do something differently here and you don’t understand why or disagree with our protocol, please discuss this within the geochemistry community. If your old methods are an improvement on our current practice then we will happily incorporate them, if not, you must adapt to our protocols. Do not introduce new protocols or new equipment into the Clean Lab without discussion with your PI and other experienced lab users.

* + 1. Pregnancy

Should you become pregnant, you will need to inform the lab manager, health and safety office or line manager as soon as possible, so that safety measured (e.g. respirator) can be organized.

###### Major Safety Concerns

Two safety concerns are highlighted here: 1) the safe operation of the air handling system in the Clean Lab and the appropriate response to the warnings and alarms issued by the BMS; and 2) the dangers of hydrofluoric acid.

* + 1. Hood Safety & Air Handling Problems

Air handling in the Clean Lab is controlled by a BMS. This system monitors airflows and flags up errors when airflows for the system as a whole are not at their set points. Nevertheless, user observations are critical in supplementing this.

There are a number of observations that you should make habitually:

* Firstly, check the pressure gauge in the Gowning lobby. If the air handling is functioning normally, this will usually read ~200. Values much higher or lower than this indicate a problem, as does audible alarm should be going off inside the lab.
* Secondly, use your nose. If you smell anything unexpected (e.g., acid vapour) when you enter the Clean Lab don’t ignore it. If you can determine the source take action to eliminate the problem.

**System Fault (Shutdown)**

The System Fault button will light when the BMS registers either a fire alarm or an air handling failure resulting from a hardware fault. When this happens, the hotplate(s) should turn off automatically, but check this has occurred. Seal any open acid containers in order to minimise the generation of acid vapour and completely close all sashes. **Report the problem to Gary or your PI.** Do not return to work until an all clear is posted on the Clean Lab door.

**Other Faults**

Not all faults with the air handling system generate warnings on the BMS. If you smell acid, even if there is no warning displayed on a hood, report it immediately to your PI or to Gary.

* + 1. Hydrofluoric acid safety

The major chemical safety issue in geochemistry clean labs is in the use of hydrofluoric acid (HF). HF presents a special hazard as both the gas and liquid form are highly toxic and able to penetrate deeply into the tissues and bone. Symptoms (pain) of contact with hydrogen fluoride solutions may be delayed with serious burns resulting. The danger with HF is not only in the H+ ion causing chemical burns, as is the case for most other acids, but also in the F- ion which bonds with Ca (and Mg) in your body. In doing this it destroys skin and bone and lowers the concentration of Ca in body fluids. The resulting low Ca (in blood - hypocalcemia) is what kills you. Unfortunately, because HF is not a particularly strong acid it does not cause “normal” chemical burns except at very high concentration. You are therefore unlikely to realize that you have come into contact with it until sometime after the initial contact. It is therefore necessary to pay great attention to every aspect of any procedure involving HF.

The risks associated with HF cannot be understated. **Skin contact with even small volumes of concentrated HF can result in death.** To ensure that you are fully aware of just how serious the risks are you should familiarise yourself with the HF safety information available in this handbook and online on the riskNET server.

**All lab users, whether they will be using HF or not, need to know what action should be taken in the event of suspected HF contact** – you may be called upon to help someone in the event of an HF accident (see Sections 3.7.2 and 3.5.2).

If you will be working with hydrofluoric acid you are required to attend an introduction to the dangers of HF and be formally signed off by your PI before you are allowed to handle this chemical. After the induction you will be issued with a personal tube of HF antidote gel for off-site use.

HF use is not allowed out of normal working hours.

###### What to do and who to call in an emergency

**Clean Lab manager: Susan Little 020 3108 6575**

**Geochemistry Lab manager: Gary Tarbuck 020 7679 2260 / 2373**

**Departmental Health and Safety Officer: John Bowles 020 3108 6316 / ext 56316**

**Departmental Manager: Leisa Clemente 020 3108 6338**

|  |  |  |
| --- | --- | --- |
|  | **Action** | **Contact Number** |
| **Emergencies:** |  |  |
| Fire | If Fire Alarm not already ringing, activate Fire Alarm Call point (in corridor outside lab) on way out. Evacuate building. | **Phone 222 (or 020 7679 2222 from external line)** |
| Personal injury/sickness  (not lab accidents) | In extreme emergency i.e. deemed life threatening, call 222. Otherwise contact Facilities Staff or Reception in order to contact a first aider for medical attention. |  |
| Flood (water) | Report to Gary Tarbuck or John Bowles. |  |
| **HF accident:** | Follow instructions in lab manual and Risk Assessment. Remove contaminated clothing, rinse affected area with copious amounts of high flow water for 1 min & then, wearing gloves, start massaging in HF antidote gel (C-gel). Anyone assisting must wear full PPE and take care to avoid contact with HF contaminated materials. For suspected contact with concentrated HF go to hospital emergency (with someone). If an ambulance is required call 222 or 999 directly. | **Phone 222 (or 020 7679 2222 from external line)** |
| **First Aid:** |  |  |
| Chemical burns (non-HF) | Follow instructions in lab manual and relevant Risk Assessment Forms. Remove contaminated clothing and rinse effected area with copious amounts of water. |  |
| General | Contact Facilities staff or Reception in order to find a first aider for medical attention. |  |
|  |  |  |
|  | **Action** | **Contact Number** |
| **Chemical spill/ hazard:** | Follow instructions in lab manual and relevant Risk Assessment Forms. Acid/Chemical Spill Kit is in the lobby. Normally, dilute with copious amounts of water, and clean up. |  |

###### All accidents and “near miss” incidents must be reported by clicking 'report an incident' on riskNET Tools, which can be found on the UCL online SafetyNET Page: <http://www.ucl.ac.uk/estates/safetynet/>

**Serious accidents must be reported immediately to UCL Safety Services by telephone ext. 57246, and the DSO (John Bowles, ext 56316)**

###### First aid

The following first aid equipment is available in the Clean Lab:

Hydrofluoric Acid Antidote Gel (C-gel) – The lab has tubes of antidote gel, together with a green Medical Information Card. These are generally located close to the sink. Additional supplies are in the cupboard in the Gowning Lobby.

Eye Washes – The lab has eye wash bottles in the lobby. These are for minor splashes. Additional supplies are in the cupboard in the rear Gowning Lobby.

Emergency water spray - Located in the evaporation hood. This is for major skin contact with acids or other dangerous chemicals.

Standard First Aid Kit - Located in the Gowning Lobby.

**Make sure that you know where to find and how to use this equipment.**

* + 1. Hydrofluoric acid antidote gel

- Even if you will not be working with HF yourself, ALWAYS check that the HF Antidote Gel is where you expect them to be BEFORE you start work. Do NOT start work until they have been found and/or replaced. You do not want to waste time running around looking for HF Antidote Gel after you or a colleague has been burned!

- It is recommended that you keep your personal tube of HF Antidote Gel at home. Symptoms of HF “burns” can be delayed and could occur after you have left work.

* + 1. **Treatment of hydrofluoric acid “burns”**

If at any time you suspect that you have HF anywhere on your person, go immediately to the nearest sink and rinse off the effected area for 1 minute under fast flowing water. Then (for skin contact) start massaging HF Antidote Gel into the skin. This should be done wearing lab gloves to protect your unaffected hand(s) from transfer of HF from the contact area. For HF in the eyes, flush with water or eye wash solution until an ambulance arrives continue irrigation en route to the hospital. Additional bottles of eye wash solution can be found in the lobby. If only one eye is affected take measures avoid contaminating the unaffected eye. For contact lens wearers, do not try to remove contact lenses if they do not flush out. Non-professional removal may damage the surface of the eye. **All eye splashes and inhalation require immediate medical attention.** Anyone assisting the victim of an HF accident must wear full PPE and take care to avoid contact with HF contaminated materials.

In the case of major skin or eye contact (i.e. conc. HF or large skin area) have someone (**remember you should never be working alone with HF without having informed a “buddy” or colleague**) arrange for medical treatment as quickly as possible while you are treating yourself. They should call 222 (or 999) in order to get immediate ambulance transport to a hospital emergency room. They should tell the dispatcher that you have a hydrofluoric acid burn. Take the HF Antidote Gel or additional eye wash solution with you (so that you can keep treating yourself) and take a grab sheet from the lab containing information on HF burns.

For minor skin contact (i.e. ≤ 2.5% HF – approx.1.5M) first aid should be as for a major contact. It is not necessary to go to hospital immediately, however if any symptoms develop later (pain or discolouration of skin), you should seek medical help immediately.

Once first aid for a suspected HF “burn” has commenced it must be reported immediately by a third person (your PI for preference) who will arrange for the victim to be accompanied to the doctor or hospital should medical treatment be necessary. This is to assist and support the victim who, under the stress of the situation, may be unable or unwilling to question medical personnel about their treatment. If you accompany a victim to hospital, you should be prepared to make a nuisance of yourself in order to make it clear to the medical personnel at the hospital, how serious an HF burn could be. The unfortunate reality is that the person admitting your colleague may NEVER have had to deal with an HF burn before and may NOT know how urgent immediate treatment is. The Medical Information Card will carry more weight than your say so and will ensure that the person treating you knows what to do.

Lastly, it is quite possible to have come into contact with HF inadvertently during the day and be unaware of this until much later. For this reason your personal tube of HF Antidote Gel should be kept with you, either in your bag or at home so that you can commence First Aid immediately if you suspect an HF burn after work.

As soon as is practical AFTER the HF burn has been treated, fill out an Accident/Incident Report Form (via riskNET).

* + 1. Treatment of other acid (or chemical) contact with skin or eyes

- The first step in all cases is to decontaminate. Immediately, and as far as possible simultaneously remove contaminated clothing and rinse the effected area under running water: 15 minutes in general (1 min for HF - see Section 3.5.2). Small areas (hand, wrist, forearm) can be rinsed off in the sinks in the clean lab. If you have to rinse off larger or harder to reach body areas use the emergency spray in the evaporation hood and strip off under the running water! Do not put your clothes on again afterwards!

- Chemical splashes to the eyes should be treated by thorough flushing with the eye wash solution found in bottles by the sink in the clean lab. There are instructions on the bottles which you should read before you start work in the lab. You may not be able to read them once you have acid in your eye! If you wear contact lenses and they don’t come out during flushing, **leave removal to a medical professional**. Non-professional contact lens removal might end up removing some of the surface of your eye as well! **All chemical splashes to the eye require professional medical attention after thorough flushing with eye wash solution.**

- If further help is needed contact Reception in order to seek assistance from a First Aider or emergency services (e.g. for a serious chemical burn).

- Fill out an Accident/Incident Report Form (as soon as possible afterwards.

|  |
| --- |
| **AFTER First Aid, it is mandatory to seek medical attention at A & E for:** |
| 1. **Any concentrated acid on the face, hands or feet.** |
| 1. **Acid ingestion.** |
| 1. **Acid in the eye.** |

###### Personal protective equipment (PPE)

Personal protective equipment (PPE) is an individual’s means of protecting themselves from hazards in the laboratory. The PPE used in the Clean Lab are:

* Lab coats/coveralls
* Lab shoes/shoe covers
* Gloves
* Glasses
  + 1. Gloves

There is no perfect choice of glove material to suit all circumstances so you must be aware at all times of the condition of your gloves. Like lab coats, gloves serve two purposes; to protect your hands from contact with dangerous chemicals and to protect your samples from contamination (e.g. by oil from your skin). All gloves used in the Clean Lab are powder-free due the risk of sample contamination by the powder, which is usually talc.

Gloves used in the lab are made of very thin materials designed to be splash resistant. They are not designed to be worn for any great length of time. Therefore even if you have been wearing gloves throughout your lab work, you should wash your hands before leaving the lab, just in case there was a hole in your gloves. This is especially important after working with HF (see Section 3.3.2). Gloves available in the clean lab are normally nitrile. They should not be considered as an invincible barrier between your hands and chemical reagents. **As a rule of thumb double gloving will increase penetration time by a factor of four**.

NB. Neither nitrile (nor other glove types like vinyl) is ideal for hydrofluoric acid, double gloving is therefore mandatory when working with HF.

Natural rubber/latex gloves are discouraged due to the possibility of skin allergies.

If you use reagents other than mineral acids, you should check the relevant COSHH assessment and SDS for guidance on choice of glove material (if given) and/or the chemical resistance guides for gloves available. You should note when referring to these guides that the gloves we use are less than 5 mil thick (~0.13mm) and the tested gloves are generally 11 mil thick. We can therefore expect that the “breakthrough” times for our gloves will be significantly faster than those in the guides, even for the same material.

**Bearing the above information in mind the following protocols should be observed for glove use:**

1. Always wear gloves when working with acids or other dangerous chemicals.
2. When putting gloves on, pick them up by the cuffs and avoid touching the remaining gloves in the packet/box – your fingers may contaminate them.
3. Even when wearing gloves, do not pass hands over open samples or touch the clean parts of bottles, test tubes, pipette tips etc.

**Be aware of what you are doing when wearing gloves.**

When you are wearing gloves, it is important not to touch your face or hair with your gloved hand. Not only may you have touched acid without realizing it and then transfer it to your skin, but you will transfer oil from your skin and hair to your gloves and contaminate any surfaces you touch subsequently. If you must scratch your face or eye or something, the best thing to do is to remove your gloves, wash your hands, and then touch your face/skin with your cleaned hands.

There is no point wearing gloves to keep things clean if you use them to do dirty jobs as well as clean jobs. For example, if something is dropped onto a dirty surface (e.g. the floor) gloves should be removed before picking it up. If for safety reasons you need to pick something up from the floor wearing gloves, throw the gloves away immediately afterwards. If the item is disposable (e.g. a pipette tip) it should be thrown away, if not it needs to be cleaned before being used again or being placed on any clean surface. This includes pens. There is a tendency not to treat pens in the same way as other lab equipment. Pens should not be left where they are likely to be knocked onto the floor (e.g. on the sash bar of a hood) nor should they be put down inside hoods, they may be dirtier than you think or may come in contact with acid which could be transferred to an ungloved hand at a later date.

* + 1. Glasses

Safety glasses must be worn at all times in the Clean Lab; it might not be your own accident that causes acid to splash in your face. Acid in the eye is not a trivial injury and may result in loss of sight. All safety glasses in the Clean Lab meet European Standard EN166. NB. This standard does not cover protection against chemical splashes.

**Special Note for Contact Lens and Glasses wearers**

If you are a contact lens wearer it is your responsibility to make sure that everyone in the lab is aware of this. If you wear glasses, you must also wear safety glasses or goggles over you glasses.

###### Chemical safety

Anyone intending to work in the Clean Lab is required to be familiar with the risks associated with the work they plan to do. This involves being familiar with the contents of this manual, with the contents of the relevant Safety documents on the Sevron server, and with the dangers of working with HF. Familiarity with the dangers of HF and appropriate first aid for HF “burns” is MANDATORY. Even if you never use this chemical yourself, you may be called upon to administer first aid to someone who has been exposed to it.

* + 1. The Acids

The major chemical hazard in geochemistry laboratories is hydrofluoric acid (HF). For the treatment of HF burns every second counts. However, other acids are used in greater quantities. All are very corrosive and must be handled with extreme care in a fume or LAF hood. All acid burns need to be treated very seriously, most especially any acid splash in the eyes.

Below is a brief summary of the characteristics of the most commonly used acids. **This should not be regarded as a substitute for reading the COSHH Forms written for tasks in which the acid is used**.

**Hydrochloric Acid (c. 12M):** Corrosive and Harmful. A volatile fuming liquid. Both liquid and vapour cause severe burns to body tissue. Inhalation causes lung damage.

**Nitric Acid (c. 16M):** Corrosive and Oxidising. A fuming, sometimes pale yellow, liquid. Both liquid and vapour cause severe burns to body tissue (yellowing of skin). Inhalation causes lung damage.

**Hydrofluoric Acid (c. 28M):** Extremely toxic and corrosive. A fuming, volatile liquid. Burns to skin delayed until acid reaches underlying tissue and bone. Rapid treatment essential to prevent further damage.

Further details on Hydrofluoric Acid Safety can be found in other sections of this document.

**Perchloric Acid (c. 12M):** Corrosive and Oxidising. A solution of approx.. 68-70% is used in laboratories because the solid is too unstable. Must only be dried down in the LAF hoods designated for this chemical i.e. the ones fitted with scrubbers. Contact with combustible materials must be avoided. Causes severe burns on human tissue. See Section 3.7.3 on “Perchloric Acid Safety” for further details.

The appropriate safely/hazard labels should be present of all chemical bottles in the clean lab. Rolls of safety/hazard labels can be found in the cupboards in the rear gowning lobby (fridge room). Please do not remove the rolls from this location, instead cut off as many as you need. If in doubt over which labels to use, refer to the poster in the Ancillary Lab.

The globally harmonised system (GHS) of safety labels are now mandatory. These are the white labels with the red border. Old orange/yellow labels must be replaced.

* + 1. Safety guidelines for working with HF.

Unfortunately, because HF is not a particularly strong acid it does not cause “normal” chemical burns except at high concentration. It is possible therefore to be unaware that you have come into contact with it until sometime after the event. It is therefore necessary to pay great attention to every aspect of any procedure involving HF. The guidelines below are aimed at reducing both (a) the chances accidental contact with HF and (b) the severity of any accidental spill.

1. If you use HF regularly, make sure you have a supply of HF Antidote Gel at home to administer first aid if symptoms develop after work.

2. Never work alone. If necessary ask a colleague to sit in with you briefly while you are handling HF. This is especially important between 5 and 7pm when there are fewer people in the building. In the event of an accident the other person can arrange transport to the hospital while you are administering first aid.

3. Only work with HF between 8am and 7pm and never on weekends.

4. Every time you work with HF check beforehand that the HF Antidote Gel and green Medical Information Card are where you expect, just in case it has been moved. Every second counts in the treatment of an HF burn.

5. **PPE.** Always wear two pairs of gloves and safety glasses when working with HF. Wear a face shield as well when handling large volumes (>50mL) of concentrated HF or mixtures containing concentrated HF. Sleeve protectors are also a good idea. Never pick up even a closed bottle of HF without wearing gloves and safety glasses, there may be dried drops of HF on the outside of the bottle that will re-dissolve on contact with skin moisture.

6. Before use, wipe HF bottles with a damp Kimwipe to remove any HF residue from previous use (e.g. a dried drip). It’s very easy to get wet or dry HF onto your gloves and then transfer it to your face with an unconscious gesture. If you see a drop of HF on your gloves wash your gloved hands and then remove and dispose of the glove(s).

7. Do not put HF in squeeze bottles. Squeeze bottles drip!

8. As with all concentrated acids, never pour HF (of any concentration) into a container that you are holding in your hand.

9. The lid of any large volume (greater than 10ml) container of HF should be closed tightly immediately after decanting to or from it.

10. If you need to use HF repeatedly, e.g. add 2ml to 10 samples, do not be tempted to use the reagent bottle directly. Instead transfer an aliquot of HF to a clean Savillex beaker and use a pipette. Cap each beaker immediately after HF has been added.

11. Never leave a beaker of HF open, no matter how small the volume inside.

12. If you leave a closed beaker of HF unattended, the contents must be clearly identified with a label or note in order to alert other lab users.

13. It is a good idea to work on a Kimwipe when dealing with HF; it’s easier to see a wet patch on a Kimwipe than a drop on a shiny bench surface.

14. Any tips or Kimwipes that have come in contact with HF should be rinsed under running water and disposed of inside a glove or plastic bag.

15. When drying down HF bearing solutions you are generating HF vapour so make doubly sure that the extraction in the hotplate hood is working properly and clearly indicate on the frame of the sash that HF is being dried so that fellow lab users are warned.

16. At the end of your task, rinse off your gloved hands under running lab water before removing your gloves. Then wash your hands thoroughly just in case there was an undetected hole in your gloves. Remember, HF burns are not necessarily immediately painful, this final wash may save you from a bad burn.

Having said all this, if you wear gloves, a lab coat, and safety glasses and follow the guidelines, it is unlikely that you will come into contact with HF.

* + 1. Perchloric acid

Beyond causing acid burns on contact with skin, there are two major problems with perchloric acid. Firstly, when it is evaporated the vapour can react explosively with organic materials such as wood and paper (Kimwipes) and secondly if it is heated to dryness at elevated temperatures (above our normal hotplate temperatures)) it can explode. Perchloric acid should therefore only be dried down in specially designed perchloric hoods which incorporate a “scrubber” for washing down the hood ducting with water to dilute and wash away any perchloric acid that condenses on the surfaces of the ducting. **N.B. Never dry down organics in a perchloric hood, even if perchloric is not currently being used in the clean lab. Organic residues may linger and react explosively with perchloric acid vapour if it is used at a later date.**

The clean lab has a single LAF hood that incorporates a scrubber system for use when drying down perchloric acid. These hoods are the only ones that can be used for drying down solutions containing perchloric acid. You should ensure that you know how to set them up for safe handling of perchloric acid and know how the nozzles should perform **(see Section 3.7.3)**.

Perchloric acid should never be stored with organic chemicals.

For additional information on the hazards of perchloric acid you can refer to the University of Glasgow Guidance Note on perchloric acid and as an illustration of just how explosive perchloric acid accidents can be, check out the Henderson Explosion on You Tube:

<https://www.youtube.com/watch?v=2K0cEX9ex3U>

* + 1. Chemical Reactions

Acid dilution results in a chemical reaction which depending on the acid may be extremely exothermic. Therefore **a basic rule for all acid dilutions is that the acid should be added to the water and NEVER the reverse**.

Dangerous by products may be produced as a result of reactions between the chemicals used in your chemical procedure. Some of these by products can be avoided (e.g. by not mixing waste nitric and hydrochloric acids), but others may be unavoidable. Make sure that you are aware of these by reading the relevant Risk Assessments.

* + 1. Acid spills

**Most acid spills in the clean lab should occur inside the fume or LAF-exhaust hoods.** These hoods have built in drains in the plenum under the perforated work surface. Acid spills in hoods can therefore be dealt with by pouring copious amounts of 18M water through the work surface to ensure that the acid is:

1. rinsed away
2. thoroughly diluted.

Spills while working should be diluted with 18M water and the area dried with a Kimwipe. However at the end of the day, the work surface should be lifted to make sure that all the liquid has drained away. Sometimes accumulated debris or an incorrectly inserted drain plug will mean that the plenum does not drain properly. If this happens, the plenum and drain will need to be cleared and then cleaned. If more than a few mL of acid were spilled, additional copious amounts of water should be used to rinse out the plenum and further dilute the acid. Test any remaining liquid with pH indicator sticks to be sure that any remaining liquid is not acidic. N.B. 18M water has a pH ~6 because it adsorbs CO2 from the atmosphere.

**Acid spills outside the hoods are tackled differently.**

Never tackle an acid spill if you are the least uncomfortable with the procedure.

**Concentrated HF spills.** Only those of a few drops should be tackled alone. Wear double gloves for the job. The spill should be wiped up with Kimwipes and the Kimwipes thoroughly rinsed in the sink. Once the acid is wiped up, cover the spill area with 18M water using a clean general-purpose beaker or squeeze bottle and then wipe this up as well. After squeezing out the Kimwipes pick them up in one hand and pull your outer glove over the tissues, repeat with your other outer glove and throw away. For larger spills report immediately to your PI or to Gary Tarbuck. They will take the decision on whether to tackle the spill in house or call the emergency services.

**Perchloric acid spills.** All spills containing perchloric acid must be diluted with large volumes of water before wiping up. The diluted perchloric acid should be wiped up with yellow chemical spill towels that are thoroughly rinsed before being stored in plastic bags for disposal. Perchloric impregnated organic material (e.g. paper) may catch fire when it dries.

**Large volume high concentration spills.** Any large volume (>2L) spill involving ≥50% of our concentrated nitric acid, ≥50% of our concentrated hydrochloric acid or more than 10mL of ≥5% of our concentrated HF may need to be dealt with by the emergency services. Contact your PI or Gary Tarbuck.

**Large (≤2.5L) volume low concentration spills.** These can be tackled in house, but only by those comfortable with the procedure. Before doing so seek advice and assistance from another experienced lab user. **Never try to tackle a large spill alone** and always change into the Emergency PPE located under the sink in the Gowning Room (yellow coveralls, gloves, safety glasses and shoe covers). This is so that you don’t contaminate your lab coat/coveralls during the clean-up.

Collect the Acid Spill Kit from the water plant room. The following instructions are on a laminated sheet in the box:

1. Empty the contents of the kit out on the floor.
2. ¼ fill the plastic box with lab tap water.
3. Carefully place adsorbent pads on the spill starting slightly outside the spill area and slowly releasing the pad down onto the spill. If more than one mat is required, observe how the spill moves as the mats are placed and place each additional mat in such a way as to curtail the spread of the spill as much as possible.
4. Once all the acid has been adsorbed, transfer the wet pads to the plastic spill kit box (the water in the box is to dilute the adsorbed acid).
5. Rinse the spill area with 18M water and use additional yellow pads to wipe this up as well. Transfer these to the plastic box as well.
6. Place the lid on the box and clearly label it as acid waste.
7. Leave the sealed box in the Gowning Room while you fetch a trolley from Reception.
8. Do not remove your Emergency PPE when you exit.
9. Remove the Emergency PPE coveralls outside the Clean Lab and throw away. Thoroughly rinse the safety glasses with lab tap water to remove any splashes. Return to the clean lab with the shoe covers still on so that you can return your lab shoes and the rinsed glasses to the Gowning Room.
10. Report the spill to your PI or Gary Tarbuck.

**Small hydrochloric or nitric acid spills with no more than ~2.5% HF (approx 1.5M HF or approx. 5% of our concentrated HF).** For small spills of these acids (i.e. an area no larger than your hand) locate the adsorbent pads (yellow) used for chemical spills. There should be a small supply in a heavy-duty plastic bag under the sink in the clean lab. If not, go to the water plant room and collect the acid spill kit. Set the RO water running in the sink, and then wearing 2 pairs of lab gloves, cover the spill with as many of the adsorbent pads as necessary to adsorb it. A single pad should be sufficient. The contaminated pad(s) should be placed in the sink under the running water to rinse and dilute the acid. Once the acid is wiped up, cover the spill area with 18M water using a clean general-purpose beaker or squeeze bottle and then wipe this up as well. Thoroughly rinse out the yellow pads in the sink, testing them periodically with pH indicator sticks. Once the pH is ~6, squeeze out as much liquid as possible, and seal them inside the heavy duty plastic bag(s).

##### Clean lab basics

Your personal cleanliness and tidiness are a very important component of your success or otherwise as a clean lab geochemist. A little paranoia is useful!

###### Before you enter

There are a number of things you should think about before entering the clean lab:

1. Do I smoke?
2. What am I wearing?
3. Do I have to touch my shoes in order to take them off?
4. Have I done any dirty jobs today?
5. Are my hands clean?

**Why?**

**Question 1:** If you use an old fashioned cigarette lighter with a “flint”, then you are in frequent contact with one of the only everyday things that can contaminate your samples with rare earth elements (REE). Every time you use it, tiny particles spall off into your face, hair and clothing. These flints are in fact made of ferrocerium. In additions to huge amounts of REE (>70%) they also contain percent levels of Fe and Mg and presumably an assortment of interesting trace elements. If you must smoke use a quartz lighter!

**Question 2:** Never work in the clean lab wearing clothes that leave your legs uncovered i.e. shorts or skirts. Always wear socks. Never wear potentially dusty clothes in the clean lab (see Question 4). Never wear outdoor coats, hats, gloves, scarves or bring outdoor bags into the lab. It is likely that these are dirtier than the rest of your clothing e.g. they may have been in contact with the ground or exterior surfaces of buildings.

**Question 3:** Think about where your shoes have been. Are they your ordinary out door shoes ... outdoors is full of contaminants. Wear slip on shoes that you don’t need to touch in order to remove or, for best practice, change into a pair of slip-on indoor shoes when you arrive at work.

**Question 4:** If you have done any dusty jobs (e.g. moving furniture/boxes; crushing rocks etc...), your hair and clothes are likely to be full of dust which can easily be transferred to samples. If you have dirty jobs to do, don't do them on lab days unless you wash afterwards and bring in a clean change of clothing.

**Question 5:** It's a good idea to wash you hands before or immediately after you enter the lab. There're a lot of nasty things you could have touched since the last time you washed them!

Do not bring anything metallic into the lab, this includes keys, jewellery (if you cannot remove a ring, gloves are mandatory at all times), notebooks or folders with metal bindings. No non-plastic/non-glass containers, bags, etc… should be taken into the clean lab. If you feel that bringing something metallic into the clean lab is unavoidable in any particular case, discuss this with experienced lab users ahead of time in order to determine whether alternatives exist. Some metallic objects are unavoidable (e.g. balances, ultrasonic baths) but these need to be carefully monitored for corrosion and removed as soon as this develops. Once corrosion starts particles of rust or other metal oxides start to spread around the lab causing contamination.

Any equipment (including plastic boxes) being taken into the lab should be wiped piecewise with a Kimwipe and 18M water before being taken beyond the Gowning Room and then more thoroughly cleaned once in the CLEAN LAB. The appropriate cleaning procedure will depend on the item and its intended use. If in doubt, ask.

If you have long hair, keep it tied back or use a hairnet/bouffant. If you have a beard you should wear a beard cover for work in the clear lab.

###### At the door

Think about where you put your feet when you enter or leave the clean lab. When you take your shoes off to enter the clean lab, step as directly as possible into the clean area. Do not walk around without shoes anywhere if you intend entering the clean lab later in the day. For example do not walk in your socks between the clean lab and other labs along the corridor. Any dirt that is on your socks will be transferred into your lab shoes and from there into the lab. Always wear socks in the clean lab. They provide a little extra protection for your feet in the event that you spill some acid.

**Shoe changing**

There are three rules for shoe changing:

Rule 1: Don’t touch any outdoor shoes with your hands.

Rule 2: Put your lab shoes on as soon as possible after you enter the Gowning Room so that fibres from your socks do not contaminate the sticky mat.

Rule 3: Once you have your lab shoes on, take at least three steps per foot before you go into the Clean Lab.

**Why?**

**Your shoes**, especially if they are outdoor shoes, are the dirtiest part of your clothing. Touching them is the same as touching the floor. If you then touch anything clean, e.g. your lab coat, your sample beaker, you might as well have dropped them on the floor outside the lab. The same goes for your lab shoes, however if you have touched your lab shoes, wash your hand once you have put them on the floor. If you want to avoid lots of hand washing then don’t replace them in the cubbyholes until the end of the day!

**Your socks** are made of fibres. Every time you step on the sticky mat fibres are pulled off. This results in the introduction of a lot of lint into the lab (not everything stays stuck to the sticky mat). Most lab fluff is probably sock fluff!

**At least three steps per foot** are necessary to remove a significant proportion of particles from your lab shoes. If you can see your footprints on the mat, then inspect and clean or if necessary, replace your lab shoes.

**Do not pile lab shoes on top of each other** when they are not in use. Doing this transfers accumulated dirt from the soles of the shoes (where it can be removed on the sticky mat) onto the uppers of other shoes, which do not get cleaned off by the sticky mat.

**Doors**

The clean labs are over pressured with respect to the lobby and this in turn is over pressured with respect to the outside room. However this does not mean that we can leave doors open!

Only one door should be open at a time so that drafts/eddies caused by opening doors do not blow dust through into cleaner areas. So check through the windows before opening any door in a clean lab facility. This is particularly important when entering or leaving the Gowning Room.

**Lab coats/coveralls**

The purpose of your lab coat or coveralls is to protect you and your clothes in the event of a chemical accident and to reduce the chances of particles and lint falling from your hair or clothes onto clean working surfaces or into your samples. Lab coats should therefore be done up at all times.

Lab shoes and lab coats should never leave the clean lab except to be cleaned. If you do accidentally leave the lab wearing your lab shoes, wash them before returning them to the Clean Lab.

###### General Conduct

The key factor in running a safe and clean lab is honesty.

Even if you only think you did something wrong, take action as if you had made the mistake. Overkill is much better than underkill.

If the problem/accident needs immediate attention and you don’t know what to do, ask for help.

If you will be seeing to it later, leave a timed and dated note to warn people of the problem and indicating when you expect to have corrected the problem. If you see such a note after it was supposed to have been fixed ... remind the person!

In order to encourage folks to ‘own up’, please remember that everyone does something stupid every now and again, so don’t make other people feel stupid if they come to you with a lab problem!

Be considerate of other lab users, organize the occupation of hoods, hotplates and bench space and cupboards with your colleagues. It avoids cramped working areas and will limit contamination problems. Do not touch other people’s work and do not share hotplates without permission.

**DO NOT** remove items from the clean lab without permission from the Lab Manager (Susan Little). Items in other labs may be needed there and your “borrowing” of them may at the very least cause inconvenience or at worst result in contamination of someone else’s work.

**NEVER** move other user’s beakers, or handle them, without discussing it with that user first. This is because you don’t know what’s in their beakers (it could be dangerous chemicals), they might be in a specific order, and it might risk contamination of precious samples.

Some general points to bear in mind:

**A: LAB NOTEBOOKS ARE MANDATORY**

**Do not rely on your memory!**

Write down everything you do in your lab notebook, label anything you use. Date your notes and labels. This is not just in case you forget details in months or years do come, but to track down mistakes that come to light later in a procedure e.g. an unexpectedly high blank, low yield, or a suspected contamination.

Templates are a good idea for repetitive procedures, e.g. multiple column chemistries. It’s very easy to lose your place otherwise! If you continue in research, your lab notebooks will become useful references for setting up procedures you may not have used for several years. Or if you quit, they will be useful to the people who follow you in the lab.

Last but not least, if you are lucky enough to become embroiled in a case where your results or honesty are being investigated, your lab notes are your only records. Occasionally scientific disputes end up being covered not only in scientific journals but also in the international press. In such cases, investigators have even resorted to forensic testing of lab notebooks in order to determine whether notebook entries had been written in the order given by the dates!

**Keep a detailed professional notebook!**

**B: Clean up BEFORE you start and AFTER you finish**

**Nothing is clean until you clean it yourself!**

Always clean and tidy your work area with 18M water before you start. Do not assume that it is clean just because you cleaned it last night!

Do not leave new or used gloves, tissues, pipette tips, pipettors etc lying on benches, stools or in hoods. If you drop something, pick it up and throw it away (or clean it) as soon as is reasonable. If someone has to clean up after you they won’t know what is on/in the glove, tissue or tip and may unknowingly touch something dangerous e.g. HF.

When you are finished for the day, put everything away in your drawer or the appropriate cupboard. Unlabelled items (e.g. sample beakers) left lying around are fair game for cleaning and/or disposal. Remove all acids from hoods and place in the appropriate ventilated cupboard under the hoods. Wipe all work areas you used (on benches and in hoods) with 18M water and a Kimwipe.

**C: Replace and refill.**

If you use the last of anything (eg. pipette tips etc.) in the clean lab, replace or refill it. If you don’t know where to find it, ask.

###### Our hoods

All the air in the clean lab has been forced through HEPA filters to remove particles. The specification for clean room air is ISO Class 6–7 (equivalent to Class 1,000–10,000). Some jobs can therefore be done on the open benches. However, anything involving acid (e.g. most sample manipulations, cleaning procedures, etc.) and anything requiring extra cleanliness, should be done inside a hood. The LAF hood specification is ISO Class 5 (equivalent to Class 100) in the rear 75% of the workspace, even with the sash fully up.

The clean lab is fitted with 1 fume hood and 6 multi-functional hoods. There are 7 possible combinations of air pressure and laminar flow in these multi-functional hoods:

###### Cleaning

A major activity in all clean labs is (obviously) cleaning. As a community we have to maintain the communal areas to the standards required by the most demanding procedures carried out in the lab. All plastics used in the lab have to be cleaned depending on their intended use and the material of which they are made. The ultimate test of whether our cleaning is successful is THE BLANK.

* + 1. **Cleaning**

All clean lab users are expected to participate in the cleaning of the communal areas of the Clean Lab. A rota is schedules and new users will be added. Please ensure that you do your cleaning duty when scheduled.

There are several standard tasks when cleaning:

**1) Empty rubbish bins.** Fairly obvious! Remember to wear gloves to protect your hands from the possibility of contaminated waste. Don’t let the bins get so full that you can’t tie the plastic bags shut. Double bag full bags if necessary and place in the corridor or dispose of in large bins downstairs (under the bridge to the Housman room).

**2) Wash floor.** Ideally, just spill RO water on the floor and wipe up with mop, squeegee or Wypall. Work towards the door, the final wipe up can be done in the lobby. Remember, when you have done this job your hands/gloves will be dirty. Wash/change them!

**3) Wipe open benches.** The aim of this job is to prevent fine dust/lint accumulating in the lab. If everyone is cleaning up after themselves, as they should be, this should be easy. If you do it on the same day that you clean the floor, the benches should be wiped first. This job is also a chance to do a little tidying. As always clean from cleanest to dirtiest: All you need to do is squirt some 18Mohm water onto the bench/shelf and wipe dry with Kimwipes.

**4) Check MQ water quality.** Check that the Milli-Q water being delivered in the clean lab is 18M. If it’s not, report to the person responsible for the clean lab. Do NOT use the water until it has returned to 18M.