

Rules for the use of the ISTerre Geochemistry Clean Room

- **General presentation**
- **Rules for the use of the rooms**
- **Moral commitment (to be signed in paper or electronic form)**

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PREAMBLE

This charter includes a description of the essential principles of the laboratory's operation necessary to work in optimal conditions. The provisions, aimed at ensuring the safety of all and those essential for the performance of coherent scientific studies (cleanliness, cohabitation), are described more specifically.

After reading this document, permanent staff, students, trainees and post-doctoral fellows must sign the "moral contract" and hand it in to a responsible. This commitment is a prerequisite for using the facilities (laboratories and measuring equipment). Managers and tutors must ensure that the provisions contained in this charter are respected.

In the event of failure to comply with the health and safety rules detailed in this charter, the user may be banned from the laboratory after consultation with the managers.

Warning!!! A service order form must be completed and returned to Sylvain Campillo before the start of the analyses.

1. General presentation of the Clean Room and the Annex

Room

Laboratories are clean environments (clean rooms). Make sure you are as clean as possible so as not to "pollute" these spaces.

1.1. Annex Micro-wave Room

This room is adjacent to the cleanroom. Specific Lab coats (of the clean room), clogs and gloves should not be used in the cleanroom.

It is dedicated to:

1. Initially at the weighing of the natural samples analysed in powder form, for the time being relocated to the ICP-MS room. When weighing the powder in the ICP-MS room, it will be necessary to add a few drops of ultrapure water to agglomerate the powder before returning the agglomerate to the clean room for the addition of reagents. Powders are not allowed in the clean room. **Ensure cleanliness during container transfers.**

2. To the Parr bomb attacks:

The rocks are attacked in a bomb in the oven, allowing an attack under high pressure and temperature conditions (T = 150°C, not more!!!).

The procedure for solution bombing is as follows:

-Samples in powder form are weighed and agglomerated with a few drops of UP water in the bomb buckets (white Teflon pots) in the annex room before transfer to the clean room

-The reagents are added to the buckets of bombs and the bombs are closed in a fume-hood in the clean room. It will be necessary to use a plastic tray under the bombs to avoid scratching and polluting the benches or fume-hood.

- transfer and heating in the oven in the annex room

-cooling on a wooden tray in the annex room (beware of acid vapour emissions)

-when cold, open the bombs and buckets of bombs in the clean room under the fume-hood !

Do not leave the metal casings of the bombs lying around in the clean room (in a plastic box in the SAS).

The protocol will be the same for washing the buckets.

Caution, never put the Teflon buckets of Parr bombs on a hot plate. They will deform.

The metal parts of the Parr bombs must be cleaned with the sandblaster in the garage.

3. To the UltraWave microwave oven attacks:

The solution procedure is the same as for Parr bomb attacks:

-Weighing the powders in the containers and agglomeration with UP water in the annex room

-Adding the reagents and closing in a fume_hood in the clean room. It will also be necessary to use a plastic tray under containers to avoid scratching and polluting the benches

-heating in the annex room

-cooling in the annex room (beware of acid vapour emissions)

-Opening the containers under the fume-hood in the clean room.

The protocol will be the same for washing the containers

1.2. Trace area

This area is used for the preparation and processing of samples for trace and major element analyses, but also for isotope preparations. It is equipped with evaporation boxes containing hot plates for the etching (acid) of natural samples in Teflon (Savillex) containers.

The acids are distilled in this room under the fume-hood on the left. The distillation of the acids is reserved for the laboratory managers. The HF hydrofluoric acid dispenser is also stored under this fume cupboard. **This acid can only be handled by permanent staff. Do not use the left fume-hood.**

This room also contains the jerricans for the temporary storage of waste generated by the various experiments. These jerricans must be stored below the fume-hood to avoid any acid vapours in the work area. Finally, the right-hand fume-hood in this room, is used for handling acids. No acid will be handled outside the fume-hood.

Any wipes that have been wiped with acid will be disposed of in the waste trash below this fume-hood and not in any other trash.

In the corner of the trace area is the safe weighing hood. No powder will be weighed under this hood. The weighing area should be cleaned with water/ethanol and wipes before and after each use.

1.3. Isotopy Area

This clean room is reserved for elemental separations for isotope analysis.

The UP water dispenser is located in this area, **so you will need to request access if any isotope separation is in progress.**

The laminar flow hoods are exclusively dedicated to isotope separations on columns and to drying of containers after washing..

2. Rules for the operation of rooms

2.1 Cleanliness and good manners

The two areas of the clean room are spaces where the presence of dust is controlled to the maximum of our means.

The vast majority of the preparations that we carry out in the laboratory are extremely sensitive to particles, hence the need to work in a clean environment. **For the success of everyone's preparations, it is essential to maintain a maximum state of cleanliness.**

The primary pollution factor in a clean room is the people in it! They bring in dust from outside; the movements they make cause turbulence that raises particles; and finally, skin flaking generates abundant particles that can pollute your samples. **Wear a specific lab coat, gloves, glasses and a hairnet. For safety reasons, you are asked to wear**

clothing that covers your legs and socks. Scarves, hats and woollen jumpers are not allowed in the room.

For these reasons the clean room is limited to 3 people at a time and the annex room to 2 people.

It is imperative that everyone respects the simple rules for using the clean room to keep it clean and to ensure everyone's success.

Do not undertake an experiment if you do not have the time to complete it (including washing the equipment and bench and tidying up).

Many of you work in the laboratories and on different subjects, with different materials and different compositions. In order for everyone to be able to carry out their experiments in good conditions, it is essential to respect the cleanliness of the workbenches and the equipment shared by all.

Clean and tidy up everything you use to leave the space clean for the next person and to avoid any accidents.

Clean room entry/exit:

- Leave your shoes at the entrance of the SAS in front of the separation bench (no "dirty" shoes on the bench!)
- Do not store clothes from outside on the lab coat hooks.
- Dress/unclothe in the SAS and not in the room.
- Put on a hairnet (in the SAS)
- Put on a pair of gloves (two for HF handling) (in the room).
- protective glasses are in the room. If needed, protective masks are in the SAS cabinet.
- If someone is already in the cleanroom, ask if you can come and work alongside them.
 - Do not leave the SAS in socks or bare feet as you will bring dust from the corridor into the chemistry lab. Put your shoes back on when you leave the SAS!
 - Remove the lab coat before exiting the SAS. Do not use the same lab coat for the annex room.
 - Clogs do not leave the SAS. Take your shoes for the annex room

In the Clean room :

- Avoid fidgeting for no reason.

- Avoid storing materials on benches that you are not using.
- Store equipment and products in their proper place.
- All containers (reagents, etc.) must be labelled
- All preparations (samples, etc.) must be labelled with the name of the manipulator, the date and the storage reagent
- After use, or before in case of doubt, systematically clean all equipment used.

Clean benches with alcohol after use.

- Avoid storing old samples.
- Remember that you are not the only user of the cleanroom and that the common work area should always be accessible.

A golden rule for maintaining cleanliness and the success of your preparations
"THINK CLEAN"

In practice, you will find that you will save time and considerably increase the chances of success of your preparations.

2.2 Health and safety rules

- lab coat required throughout the chemistry laboratory.
- protective glasses and gloves are mandatory when handling concentrated acid or when someone is handling closed to you.
- Respect the safety signs and the protocols for using the equipment, which are displayed near each piece of equipment or will be explained to you by one of the laboratory managers.

Chemical products: Before starting your experiments with a chemical product (not usually used in the laboratory), you must carry out a study on the toxicity of the product in order to know the risks linked to the handling. This study will be submitted to the person in charge of the study (internship or thesis) and to one of the geochemistry laboratory managers, in order to assess the feasibility of the experiments. Experiments involving toxic products must not be started before validation

by one of the laboratory managers. For this purpose, the toxicological data sheets for the products are available on the INRS website. It is also possible to supplement the existing documentation by requesting additional toxicological data sheets from the laboratory's prevention assistant (Mr Carcaillet - isterre-acmos@univ-grenoble-alpes.fr) or from suppliers.



- Jerricans are available in the "Traces" room to dispose of your waste (acids).
Follow the filling instructions posted next to the containers.

- Wastes management : <https://isterre-intranet.osug.fr/spip.php?rubrique370>

- In the event of an incident, please consult the instruction panels on the doors of the geochemistry laboratory. In the event of an accident, call 15 or 112 from a mobile phone. In case of fire, dial 18.

The CleanRoom is not yet equipped with a fixed telephone. You are asked to carry a mobile phone with you.

- In case of an accident (acid splashes,...), a safety shower with eyewash is located in the immediate proximity of the entrance to the SAS.

- A water spray extinguisher is available near the safety shower. Other fire extinguishers (water spray and CO₂) are located in the corridor and adjacent corridors. It is preferable not to use a dry chemical extinguisher (powder) in the clean room or the annex room.

For a fire involving waste paper, paper baskets, etc., water spray or CO₂ extinguishers should be used. If the fire is a chemical one and/or is located on live equipment, a CO₂ extinguisher should be used..

2.3 Storage of equipment

2.3.1 Samples

- Labelling: To facilitate the storage of samples in the fridge, **all vials, tubes, tube racks, boxes, etc. must be legibly labelled with your name** (or initials), **date, contents** (or sample name and concentration for prepared solutions). Any unlabelled/mislabelled samples may be discarded without notice.

- During your stay you will be provided with drawers and cupboard shelves in the room to store your vials and other samples (without acid inside). **Nothing is left on the benches.**

-You will be given a set of savillex. **You will be responsible for the use and washing of these containers throughout your stay.** You will be responsible for the cleaning of your consumables (bottles, micropipette cones, centrifuge tubes...).

- At the **end of your stay, your scientific supervisor must ensure that you have emptied all the spaces at your disposal.** To do this, you must empty and wash (or throw away) all your preparation bottles and sort your samples with your scientific manager so that only what is essential is stored in the laboratory.

This sorting is a prerequisite for the validation of your internship by your scientific supervisor. The final responsibility for sorting lies with the scientific supervisor.

2.3.2 Chemicals reagents :

Chemicals should be stored where you found them. Don't hesitate to ask if you have any doubts.

2.3.3 Fume hoods :

The fume-hood available in the "Traces" area should be used for any experiments requiring extraction and for handling toxic products. **Do not leave containers filled with acid solution, opened without any cap, under the fume-hood.** It is for storage and washing only, for reasons of hygiene (acid vapours) and safety (risk of burns, injuries).

The distiller's fume-hood should not be used for sample storage. It is reserved for technical staff.

2.3.4 Evaporation boxes :

The evaporation boxes also need to be completely cleaned after each experiment. **The hot plate on which you are working should be cleaned with UP water and paper w wipes (no sponges).** The glove boxes should be cleaned with paper wipes regularly.

2.4 Orders

For management convenience, chemicals and small equipment should be ordered as soon as possible. Be aware that delivery times can be long. **It is essential for the smooth running of the laboratory to inform the person in charge of the laboratory of any**

depletion of chemicals (alcohol, acids, etc.) **and other materials** (bottles, gloves, etc.) **and to note this on the whiteboard in the laboratory entrance SAS before the depletion is effective !**

Report to one of the laboratory managers when the stock is running low (not when there are no more left).

The catalogues can be consulted in office No. 036.

Before ordering a product, make sure that it is not already in the clean room or ICPMS room (part of the storage for Clean room). **Please note that the reagents used do not have the same purity as those in the geochemistry laboratory on the first floor.**

For reasons of pollution risk, the reagents and consumables used in the clean room are exclusively reserved for this room and must not leave it (and vice versa). It's the same for standards. **If you receive a package in the absence of the laboratory managers, deposit it in office N°036 (no reagents), in the ICPMS room or in the SAS entrance of the laboratory.**

3. Moral commitment: Geochemistry laboratory safety

guidelines

Managers of the lab : Alexandra Gurlan / Sylvain Campillo / SimSabine Sentenac

Général

- When entering the cleanroom, wear gowns, gloves, headgear and protective glasses. Socks are mandatory and legs must be covered (no shorts or skirts)
- A glove is an imperfect protection that gives a false impression of safety as it is often pierced by fingernails. Rinse your hands at regular intervals and also when leaving the laboratory.
- Be respectful of highly corrosive acids.
- Have respect for highly corrosive acids.

HF hydrofluoric acid is particularly sneaky because it does not "sting". Its attacks are very painful after a few hours and usually VERY dangerous. It attacks glass rapidly, also bones and nerves. **When handling it, it is compulsory to double the gloves!**

Less dangerous, hydrochloric acid HCl irritates the skin and lungs, nitric acid HNO₃ turns the skin yellow, normally without any after-effects, and destroys clothing. aqua regia (HCl + HNO₃) and perchloric acid (HClO₄) and to a lesser extent sulphuric acid (H₂SO₄) eat away at the skin very quickly.

- **The aqua regia (HCl + HNO₃)** gives off free radical gases which are very carcinogenic.
- When you enter a clean room, put on **special lab coat, gloves, cap and protective glasses.**
- A glove is an imperfect protection which gives a false impression of safety because it is often pierced by fingernails and in fact porous to acids. **Rinse your hands at regular intervals** and also when leaving the laboratory.
- If you have the slightest doubt about liquid contact, rinse immediately with plenty of water.
- Familiarise yourself, without waiting for the accident, with the location and operation of safety products: eyewash, HF neutralizing gel, shower.
- Don't pick up acid bottles by the cap, someone may have forgotten to screw them back on. A loose cap on a bottle can splash the contents onto colleagues or yourself.
- Don't fill the pissing bottles above the line, they become incontinent.
- **Any liquid, acid or water, spilled on a bench should be wiped up immediately.** The person working next to you, or after you, may not see it or may mistake the liquid for water.
- Check the condition of electrical connections (rust) and report any faults.

- If you make any kind of handling error (pollution of standard, acid suction in a micro-pipette, pollution of the balance, breakage of any kind, etc.), **do not hide it**, as the consequences may be significant. Inform a competent person

Washing - cleaning

- Recycle washing acids in the bottles provided (labelled). You can pour only solutions (HCl and HNO₃ only) with a normality of less than 0.5N into the sink (under running water).
- Pour the washing acids only under the fume-hood to avoid steam in the room.
- Savillex and Teflon beakers: All markings should be removed with alcohol beforehand. The inside of the containers should be washed once with ultrapure water and dried, ensuring that no solid deposits remain. Follow the washing procedure detailed in the binder in the room or on the intranet.

Evaporation

- The softening temperatures on the plate are very different for glass (>600°C), Teflon (200°C) and polyethylene (80°C). Therefore, **never put plastic containers on the hot plate**.
- **Perchloric acid** is a potential explosive, especially at high temperatures. Keep this in mind when evaporating it on a very hot plate in a Teflon beaker (T<200°C). Tap the bottom on the plate often. This will be faster and less dangerous than raising the temperature. Minimise the amount of this acid. **Never use it directly on a sample**, but dilute it with another acid at the beginning of the attack. Contact a permanent staff member for further explanation.

Acid titration

- **Never pipette (acids and bases) with your mouth!** Use a micro-pipette.

Working hours

Trainees (masters and short stays) are not allowed to work outside the laboratory's opening hours (**8.30 a.m. to 6 p.m.**) or at **weekends**, unless a permanent staff member is present to supervise them.

4. Specific rules concerning Hydrofluoric Acid

4.1 Training/Authorisation

- All staff handling HF, or likely to accompany them when handling HF, must (1) undergo reinforced safety training (chemical risk) organised by the university's Health & Safety department. (2) This training must be supplemented by specific training for the workstation (safety instructions, wearing of PPE, waste management, emergency procedures, etc.)
- (3) A medical examination with the occupational physician must be carried out before any handling of HF
- (4) For non-permanent staff (excluding trainees), an exemption request must be made to the management for any handling of hydrofluoric acid (article D4154-1 of the Labour Code)
- Only trained permanent staff and PhD students (presenting their certificate) are authorised to handle HF.
- PhD students will also be authorised to handle HF. They must attend the annual (bi-) training course organised by the university's Health & Safety department in the same way as permanent staff. To do this, they must contact their tutor and the lab's PAs to be included on the list of applicants. They will be authorised to handle after validation and signature by the unit director of appendix 2 (Nominative authorisation form for each substance handled by temporary employees on fixed-term contracts listed in article D4154-1 of the labour code).

- All users of the clean room will be informed of the risks involved, in particular the risks linked to HF (training at the workplace (2) but only permanent staff and PhDs who have fulfilled all the conditions will be authorised to handle HF.
- In the event of an HF spill, persons not authorised to handle this reagent must not intervene, but must evacuate the laboratory, block the entrance to the room and quickly inform those in charge who will intervene.
- A HF spill kit (white cardboard) and 1 kit for any other reagent (NB! no vermiculite on HF, Calcite powder authorised)

4.2 Personal protective equipment.

- Use a labcoat with a side opening, double gloves (nitrile+neoprene) as well as sleeves + an integral face mask (standard EN 166)
- No handling with contact lenses
- A half-mask with a specific B2 E2 P3 cartridge is available in the SAS cupboard to intervene in the event of a spill or accidental projections. Check the expiry dates of the cartridges. The mask must be stored away from dirt (+ dangerous or contaminating substances) and the cartridges must be stored in an airtight bag and re-sealed with the lids provided by the manufacturer

4.3 Good handling practices

- Add the reagents slowly, wait for the reaction to stabilise, open the containers towards the bottom of the fume cupboard.
 - Work in the presence of another person. This second person must not handle HF but must be present to intervene quickly and effectively in the event of an accident.
- Work under the fume cupboard specifically dedicated to the handling of HF (right-hand fume cupboard).

4.4 Storage

- The HF bottles will be stored in the ventilated cupboard under the left-hand fumehood.
- The bottle connected to the vacuum pump is stored under a bell in the left-hand fumehood. The acid will be used in the right-hand fumehood.

4.5 Waste management

- All solids soiled with HF (paper towels, gloves, empty bottles, etc.) must be disposed of in a specific way.
- The soiled products must be dried at the bottom of the fumehood with a specific HF identification clearly visible and then immediately afterwards placed in a specific waste bucket (white Impact environnement bucket) and not in the standard waste garbage. This bucket must remain under the fumehood.

4.6 First aid

- The safety showers are located outside the clean room, in the immediate vicinity. Check regularly that they are working properly.
- Calcium gluconate can be found in the SAS medicine cabinet. Also check the expiry dates.
- People who regularly handle HF are advised to keep a calcium gluconate kit available at home.
- An absorption kit is available in the SAS cupboard in case of a spill.

WHATEVER YOUR EXPERIENCE, DO NOT PUT YOURSELF ABOVE THE SAFETY RULES: THIS DECISION ALSO CONCERNS THE SAFETY OF OTHERS. TRUST AND HABIT ARE THE FIRST ENEMIES OF SAFETY;
ANTICIPATE, ANTICIPATE, ANTICIPATE!

Emergency numbers for the Isère department :

N° European emergency call	112
SAMU Emergency	15
Police	17
Firefighters	18
Emergency number for the deaf and hard of hearing	114
Poison Control Centre	0-04 72 11 69 11
SOS Doctor	0-04 38 70 17 01
On-call pharmacy	0-04 76 63 42 55



5. Valuation of results

- **Publications**

- If **major participation** of the technical staff (important number of samples taken in charge: syntheses, preparations, analyses and/or field missions and/or writing/reading of the valorization): you must associate the technical staff as co-authors.

- If **minor participation** of technical staff: you must thank them by name in the acknowledgements.

- **Without participation** of technical staff: you must thank the platform as follows:

« *Sample preparation and /or Chemical analysis and/or Sample Characterization and/or Sample synthesis (cite the type of analysis or other: ICP-AES, MEB etc...) have been performed at the geochemistry-mineralogy platform of ISTerre (Grenoble, France)* ».

The Acknowledgements section must contain in all cases the sentence:

« *The geochemistry-mineralogy platform of ISTerre (Grenoble, France) is partially funded by a grant from Labex OSUG@2020 (investissements d'avenir, ANR10-LABX56)* ».

- **Conferences (oral and poster)**

The rule of association of the technical staff and the acknowledgements of the platform is the same as the one described above.

CERTIFICATE

I, the undersigned, present at the ISTERRE laboratory and using the geochemistry clean room for the period from / / to / / in the capacity of, acknowledge that I have read the rules for the use of the ISTERRE laboratory's geochemistry clean room and its safety instructions and undertake to respect them. The keys (pass, laboratory key, badge, etc.), as well as the analyses and handling books, are the property of the lab. Therefore, everything must be returned at the time of final departure from the laboratory (or a copy of the data must be made for outsiders). For safety reasons, isolated work is prohibited. Working at weekends and on public holidays is only allowed with the agreement of the supervisor and the permission of the laboratory director. Working conditions in the laboratory require the wearing of a lab coat, gloves, hairnet, safety glasses and socks..

Phone numbers:

e-mail :

Person to contact in case of accident:.....

Done at, the

Signature :

Note: This certificate (page 19) must be signed and returned to one of the responsible before the start of the experiments in the laboratory's clean rooms. Keep pages 1 to 18 as a reminder