^{14}C contamination testing in natural abundance laboratories: A new preparation method using wet chemical oxidation and some experiences.

Cameron P. McIntyre[1,2,3], Franziska Lechleitner[1,4], Susan Q. Lang[5], Negar Haghiour[1,2], Simon Fahrni[2,6,] Lukas Wacker[2], Hans-Arno Synal[2]

[1] Biogeoscience, ETH Zürich, Zürich, Switzerland.
[2] Laboratory of Ion Beam Physics, ETH Zürich, Zürich, Switzerland.
[3] Current address: SUERC AMS Laboratory, SUERC, East Kilbride, UK.
[4] Department of Earth Sciences, University of Durham, Durham, UK.
[5] Earth and Ocean Sciences, University of Southern Carolina, Columbia, USA.
[6] Ionplus AG, Dietikon, Switzerland.

Corresponding author: cameron.mcintyre@glasgow.ac.uk

1. Abstract

Substances enriched with ^{14}C can easily contaminate samples and laboratories used for natural abundance measurements. We have developed a new method using wet chemical oxidation for swabbing laboratories and equipment to test for ^{14}C contamination. Here we report the findings of 18 months work and more than 800 tests covering studies at multiple locations. Evidence of past and current use of enriched ^{14}C was found at all but one location and a program of testing and communication was used to mitigate its effects. Remediation was attempted with mixed success and depended on the complexity and level of the contamination. We describe four cases from different situations.

2. Introduction

The use of radiochemicals enriched with ^{14}C can contaminate work areas used for natural abundance measurements. The concentration of naturally occurring ^{14}C is 10^{-12} and blanks measured by accelerator mass spectrometry (AMS) are $<10^{-15}$. Therefore, commercially available radiochemicals containing 100% atom ^{14}C are >15 orders of magnitude above blank levels and pose a catastrophic danger to AMS laboratories. Several laboratories have reported experiences of contamination and recovery from a single 'hot' sample and we are aware of a number of other unreported examples (Jull et al. 1990; Vogel et al. 1990; Zhou et al. 2012). Practices for preventing contamination, evaluating and monitoring potential workspaces and cleaning contaminated workspaces and laboratory equipment have been described for both natural abundance and bio-AMS preparation laboratories (Buchholz et al. 2000; Zermeno et al. 2004).

In the case of a graphitization of a hot sample, it was found that the majority of the contamination occurred in the graphitization system where cross talk between samples occurs due to extended manipulation of the gases during sample preparation. It was possible to recover from a contamination event from a single hot sample in the 10^{-6} – 10^{-9} range by extensive cleaning of the apparatus and replacement of parts that have been in direct contact with the hot sample. The ion source of the AMS system recovered quickly where it had been in

operated with a hot sample for seconds but required extended cleaning when that time was longer. A general conclusion drawn from in all studies was that whilst an AMS laboratory could accommodate a wide range of ^{14}C concentrations up to 10^{-11} and that recovery from a single severe contamination event was possible, it was not suitable to undertake natural abundance and enriched studies in the same area. It was also noted that refurbishment of contaminated laboratory space in one case had been unsuccessful and was abandoned.

It is known by those skilled in the art, that effective communication and a rigorous monitoring program is invaluable to ensure the isotopic fidelity of measurements. It is often difficult to know whether equipment, samples or facilities have been affected by isotopically enriched materials and therefore it is critical to have the capability to identify legacy contamination, screen for potentially hot samples and monitor the status quo. Major AMS laboratories providing routine analysis ask clients if they are aware of the use of enriched ^{14}C the vicinity of their work and can refer them to further testing services if necessary.

Testing for contamination can be done by liquid scintillation counting (LSC) or AMS and at this time the SWAB program at the University of Miami can check for gross contamination using LSC (see supplementary information). An area is washed down with soapy water and an aliquot of the residue is mixed with scintillator and analysed for ^{14}C. AMS laboratories typically use sealed tube combustion and graphitization of samples taken by swabbing an area with a combusted quartz filter moistened with solvent. Dilution with ^{14}C free CO_2 can be used for small or hot samples before graphitization. Additionally, solid graphite powder can be used to detect airborne contamination by sorption (Zermeno et al. 2004). These procedures are risky in themselves as they can contaminate equipment used for natural abundance measurements so dedicated equipment is often used. In the case of AMS this can be relatively costly as a separate vacuum line and graphitisation system may be required.

In this study we detail a new method for contamination detection using wet chemical oxidation preparation and AMS for analysis. The procedure is convenient, cost and time effective, and minimises cross contamination of equipment. We report on four contamination situations based on real events and the results of more than 800 analyses.

3. Experimental

A printable preparation protocol is given in supplementary information.

3.1 Sampling Apparatus:

A sampling kit was prepared and kept on-standby for use at any time by anyone. In a carry basket, was freshly combusted Exetainer vials (12 ml, Labco, PN-9RK8W), metal tweezers and 25mm quartz fibre filters. Combustion was performed at 450 °C for 2-3 hrs. Also included were fresh Isopropanol (IPA) in

100ml Schott bottle, Kimtech Science Purple nitrile gloves (PN-90627), a roll of aluminium foil, metal vial rack, pen, notebook and a copy of the protocol.

3.2 Swabbing procedure:

The work area was covered with aluminium foil and labelled vials were placed in the rack. A combusted quartz fibre filter was moistened by dipping it in the IPA with tweezers. Then an area of 5-20 cm^2 was wiped by hand with fresh gloves. Three blanks were prepared by placing a moistened filter into a vial without wiping anything. Sample details were noted using fresh gloves and when finished, the rack of vials was covered loosely with a single piece of foil.

3.3 Processing of the samples:

A dedicated area with dedicated equipment was used for drying of the samples and processing prior to AMS analysis to avoid possible contamination of the natural abundance laboratories. All preparatory work was conducted in a ventilated basement room in a separate building with restricted access. Care was taken to maintain the order of the vials and caps for clear sample tracking and to prevent cross contamination. Fresh nitrile gloves were used for each sample to prevent cross contamination.

The wet chemical oxidation method used for sample preparation was adapted from a method developed for analysing dissolved organic carbon (Lang et al. 2016). The rack of vials and quartz filters were first dried in a dedicated oven overnight at 60 °C to remove the IPA. The oxidant was prepared fresh by dissolving 1.5g sodium persulfate (AR grade) in 50 mL of Milli-Q water and adding five drops of 85% H_3PO_4. The solution was stored in a combusted amber glass container and 1 mL of solution was added to each sample vial. The vials were capped and a fresh disposable 50mm 23 gauge syringe tip was put in each vial to allow gas to escape. The vials were purged one at a time for 1 min with 100 mL/min of high purity helium. The helium supply was a cylinder and regulator fitted with a metering valve, flexible tubing and a disposable syringe tip. The helium supply needle was inserted minimally (5-10mm) through the septa and both needles withdrawn when purging was complete. The samples were immediately heated on a heater block at 100 °C for one hour to complete the oxidation of organic carbon to CO_2 and then cooled to room temperature. At this stage, the samples could be stored for months and shipped to an AMS facility for analysis.

3.4 AMS analysis:

The gas samples were analysed for ^{14}C using an AMS system fitted with a gas ion source, a gas interface system and a carbonate handling system (MICADAS-GIS-CHS, Ionplus) (Wacker et al. 2013). The autosampler can accommodate 42 Exetainer vials for the automated analysis of headspace CO_2. The GIS was operated with parameters to maximise the throughput of samples. Samples were flushed onto the zeolite trap with helium for 30 s and the CO_2 trapped at 100 °C and desorbed at 400 °C. Trap cleaning between samples was 30 s and overall this

allowed 12 samples/hour to be analysed. The AMS was calibrated against ^{14}C-free CO_2 and CO_2 from combusted standard oxalic acid (NIST SRM 4990C). Vials showing no contamination were acid washed and reused used while contaminated vials were discarded.

3.5 Dilution of samples:

When necessary or if suspect, samples were diluted with ^{14}C-free CO_2 prior to analysis. Additional empty exetainer vials were flushed with helium for 30 s at 100 mL/min using the CHS autosampler of the AMS system. Then, 0.1 mL of ^{14}C-free CO_2 was injected using a 1 mL insulin syringe and needle (Terumo U100), which was equivalent to 50 μg C. Samples could be diluted 1:12 or 1:120 v/v by injecting aliquots of 1 or 0.1 mL respectively into a vial containing blank CO_2. Additional vials of blank CO_2 were prepared to flush the system after hot samples if required.

4. Results and discussion

4.1 Samples and blanks

The method described here was used to give a semi quantitative 'hot' or not result for screening for and monitoring of ^{14}C contamination. Between January 2014 and May 2015 twenty-seven batches of samples from eight institutions were analysed comprising 750 samples and 80 blanks. This encompassed samples for screening of unknown areas, monitoring of work areas and retesting of cleaned areas. Signs of use of enriched ^{14}C were found at 7 of the 8 institutions checked while the 8th institution was using enriched ^{13}C. Each location was either being used for, or had plans to be used for natural abundance ^{14}C measurements.

The results showed that 14% of the samples (106) had an $F^{14}C > 2$ and 29% (216) had an $F^{14}C > 1$. The number of positive results is artificially high as the tests were largely targeting contaminated areas or were retesting cleaned areas. The highest value recorded was an $F^{14}C > 2000$ and the median value was $F^{14}C = 0.9$. The mean mass of carbon measured was 100 ± 70 μg and the highest mass was 500 μg C. Based on a mean value of 100 μg C, the 1:12 and 1:120 v/v sample dilutions had a dilution of 1:6 and 1:60 w/w carbon respectively. Typically 20s of data acquisition was enough to determine if a sample was hot or not. For samples that contained more than 100 μg C, the GIS would automatically reduce the size of the sample.

The 80 processing blanks had a measured mass of 13 ± 10 μg with a $F^{14}C = 0.55 \pm 0.21$ excluding 4 outliers with high mass and one with high $F^{14}C$ (due to exchange of a hot cap). Based on an upper limit at the 3 sigma error level this is equivalent to detection limit in order of femtocuries (fCi) which was highly sensitive. Performing a mass balance using the mean blank and sample data, any sample with an $F^{14}C > 1$ was considered to be suspicious. The variation of the blank appeared to be related to the type of glove being used to handle the moistened swab with latex gloves giving higher values than other types of gloves. Different

gloves were tested and it was found that the purple nitrile gloves (Kimtech PN-90627) gave the lowest mass and $F^{14}C$ values for the blanks.

A clear advantage of the method was that it could be comfortably used for small samples down to 5 µg C without having to add additional carrier carbon. The use of exetainer vials and simple equipment for the preparation kept the costs and possibility of cross contamination to a minimum. Based on the AMS throughput of 12 samples per hour it was possible to analyse more than 100 samples in 3 days including collection (day 1), preparation (day 2) and analysis (day 3).

At the time of this study the carry over of the system was estimated to be approximately 3 % due to the shortened analysis method and a blockage in the GIS transfer line. Carry over predominantly occurred in the autosampler and gas interface system. Consistent with the findings of previous studies, the ion source could be brought back to normal within half an hour by running several fresh cathodes with blank gas. However, after the hottest sample with an $F^{14}C > 2000$ it took several days to bring the CHS and GIS system back to normal blanks levels requiring repeated of cycles of heating, flushing with blank gas and evacuation to remove excess ^{14}C. After this event, risky samples were diluted before analysis.

4.2 Swabbing strategies

Over the course of our swabbing program we were able to refine our strategy for taking swabs. Additional advice and strategies can be found in the reference and supplementary information. One key initiative that was implemented was a 'swab before you start' program. Here, before a students or colleague commenced a new project, they were asked to swab test their equipment and facilities. This served several purposes including familiarisation with the issues of contamination and ensuring that their equipment and facilities were clean. It was also possible to have them include additional samples from existing and new locations to extend our database of results. A quarantine area was set aside so that items and samples could be tested before being transferred into a ^{14}C work area. Through this program, five new locations with evidence of previous tracer work were uncovered.

Consistent with previous studies, contamination could be found in commonly used areas and on commonly used equipment. Initially, a broad screening run of door handles, fume hoods, freeze dryers and balance areas were targeted. Door handles were particularly useful as it was not necessary to enter a workspace to tell if something hot was inside. Multiple door handles could be swabbed with a single filter paper allowing whole buildings, floors or corridors to be checked. Any suspect areas could be investigated further and 20- 50 specific swabs of furniture, instrumentation and apparatus could be taken. We would focus on switches, fume hoods, computer keyboards, bench tops, drawer handles, ovens, balances, fridges and freezers. Using this procedure we located point sources at most contaminated locations.

4.3 Scenarios

Here, four situations encountered are described without providing specific names or locations. These were the four most significant cases out of ten that were studied.

4.3.1. Hot gas chromatograph in a stable isotope laboratory

During the course of a compound specific radiocarbon analysis (CSRA) project, a number of hot samples with $F^{14}C < 10$ were measured on the AMS resulting in the loss of weeks of compound preparation work and unpublishable data. The contamination appeared to occur randomly and the laboratories used for sample preparation were refurbished just 5 years previously complete with new furniture and instrumentation. Broad screening of the laboratories located a point source at a gas chromatograph (GC) in a stable isotope laboratory used for CO_2 quantification. Further swab tests narrowed this down to set a drawers and a box of syringes which showed levels of $F^{14}C > 100$. One syringe had been previously used in an overseas laboratory that had conducted CSRA with labelled compounds more than 20 years ago. This syringe had cross-contaminated the GC, samples and the surrounding furniture. Clean up was attempted but only reduced the contamination to $F^{14}C < 10$ which was still too high for the very low levels considered necessary for CSRA analysis. The area was abandoned for ^{14}C work and access restricted by signage.

4.3.2 Tracer experiment near a sample preparation laboratory

During routine ^{14}C sample preparation in a dedicated natural abundance laboratory, graphite blanks measured on the AMS increased overnight to $F^{14}C > 0.2$. It was found that a biology group in the same building had undertaken a tracer experiment the previous day using $Na^{14}CN$. The $Na^{14}CN$ was prepared in a secure basement isotope laboratory before samples were transported to scintillation counters in laboratories above. Although the tracer experiment was performed carefully in a fume hood vented directly to the outside, it was speculated that dissolution of the $Na^{14}CN$ in aqueous solvent produced gaseous $H^{14}CN$ which dispersed throughout the building, contaminating the natural abundance samples and blanks.

Operations in the AMS sample preparation laboratory were stopped and around 1 month of prepared graphite samples were contaminated and lost. The AMS preparation laboratory was moved to another building while cleaning and testing was undertaken. ^{14}C levels in the building quickly decreased over several days after the event and after one month blank levels had mostly returned to workable levels. After three months the preparation apparatus could be returned to its original building. In this case, it was fortunate that the contaminant was gaseous and was cleared by ventilation. Nonetheless, consequences of the event were a significant loss of commercial and research samples and time. In terms of revenue and labour the monetary loss was estimated at more than USD 100K. Prevention of future contamination relied on an agreement to cease further tracer experiments in the building and instigation of a ^{14}C monitoring program.

4.3.3 Hot gas chromatograph in a compound specific ^{14}C laboratory

Two students about to commence radiocarbon projects were asked to swab the laboratories and equipment they planned to use. Initial results showed that several places such as the fume cupboards had low level ^{14}C contamination with $F^{14}C < 20$. It was also discovered that ^{13}C tracer work was on-going in the laboratories that could potentially interfere with ^{14}C analysis. The laboratories were wiped down and rearranged to isolate ^{14}C operations into a dedicated section. A final swab of the laboratory found a previously untested preparative GC in the laboratory that had an $F^{14}C > 2000$. Although it was stated that the GC had never been used for tracer work, it had come from another institution that had used enriched ^{14}C materials.

The preparative GC was removed and the laboratory was emptied and totally refurbished. The fume hood was professionally cleaned and the furniture was replaced and the laboratory was re-swabbed. The results showed that the area was more contaminated than before and it was revealed that furniture was second hand from the central repository. Despite extensive cleaning before putting it in the refurbished laboratory, it had not been tested first and had obviously previously been used near enriched materials. The furniture was removed and the laboratory professional cleaned a second time. Brand new furniture was installed in the laboratory and a final batch of swabs found only 2 samples with $F^{14}C$ of 1-2. The whole process of setting up this lab took more than 1 year and it was significant delay to students and cost to the department. Local tracer work in the building was on going however an awareness lecture took place and restricted access and signage were implemented.

4.3.4 Laboratory relocation to a contaminated area

Two students were about to start projects at a new institution. Upon discussion with the institution it was revealed that a number of tracer experiments had been undertaken over the past 20-30 years at that location and that enriched samples had been processed in several laboratories. Renovations were about to be undertaken during which time the laboratories would to be temporarily interchanged within a building. Screening of several laboratories showed areas of contamination with $F^{14}C > 100$ including a TOC analyser, peristaltic pump and freeze dryer. High $F^{14}C$ levels were also found in fume hoods and balance areas. Discovery of these areas was fortunate, as it had been planned to relocate a clean preparation laboratory into one of these areas. As a result of these findings it was recommended to abandon the contaminated areas permanently for ^{14}C work. A newer and recently refurbished building was tested and a clean dedicated laboratory was reserved for natural abundance ^{14}C work. One fortunate consequence of these tests was that samples destined for the calibration curve were saved from certain contamination.

4.4 Cleaning strategies

The main question that was always asked was ‘How do we clean up the contamination?’ How clean is clean enough strongly depends on the size and age of the samples to be prepared. Larger modern samples such are less sensitive to

contamination than smaller older samples and therefore in some situations a higher level of residual contamination could be considered tolerable. Laboratories for natural abundance CSRA require the cleanest conditions i.e. ultra-trace levels. Other studies have reported cleaning strategies that have been successful ((Jull et al. 1990; Vogel et al. 1990; Zermeno et al. 2004; Zhou et al. 2012).

In general it was found that for items with $F^{14}C < 10$, cleaning might be possible to acceptable levels of $F^{14}C < 2$. Above an $F^{14}C$ of 10 we had mixed success and for an $F^{14}C > 100$ we generally recommended abandoning the item for natural abundance ^{14}C work. Typically, the first step of cleaning was washing with detergent and solvent such as IPA and the second step was a baking the items in a muffle furnace if possible. It was more difficult to clean complex apparatus such as a freeze drier. This occurred in 4.3.4 where the freeze drier was initially tested with $F^{14}C > 300$ and after very extensive washing, it was only reduced to $F^{14}C > 100$. In general it can be recommended that anything disposable should be discarded and items to be kept should be cleaned and retested. When setting up a lab, we recommended that all areas and items should be new or proven clean. As in 4.3.1, a single hot item could contaminate an entire laboratory.

5. Conclusions

We have developed a convenient and cost effective method that proved satisfactory for testing laboratories and equipment for enriched levels of ^{14}C. We found that on-going use of and residues from ^{14}C tracer work were a problem and periodically impacted routine natural abundance operations. Transfer of tracer compounds was primarily by contact and could be mostly contained within a single laboratory. It was possible to clean contaminated areas, however this depended the extent and levels of ^{14}C contamination and on the complexity of the item to be cleaned. Consistent with previous studies, mixing of tracer and natural abundance work was found to be not advisable. Good communication about prior use, thorough testing and a monitoring program where essential to ensuring the isotopic fidelity of ^{14}C measurements by AMS. Proven clean areas with restricted access and cessation of nearby tracer work were necessary for ^{14}C AMS laboratories.

6. Acknowledgements

Thanks to our colleagues at the various institutions who allowed us to perform testing and listened to our recommendations when given.

7. Supplementary Information

Attached is copy of the preparation protocol with illustrative photos.

Currently, additional protocols and advice are available from the NOSAMS AMS facility at WHOI, the W.M. Keck Carbon Cycle AMS facility at UCI and the SWAB program at the University of Miami.

(http://www.whoi.edu/nosams/Submitting_Guidelines),
(http://www.ess.uci.edu/researchgrp/ams/protocols)
(http://www.rsmas.miami.edu/groups/tritium/swab/monitoring-of-shipboard-contamination/)

8. References:

Buchholz BA, Freeman SPHT, Haack KW, Vogel JS. 2000. Tips and traps in the C-14 bio-AMS preparation laboratory. Nuclear Instruments & Methods in Physics Research Section B-Beam Interactions with Materials and Atoms 172:404-8.

Jull AJT, Donahue DJ, Toolin LJ. 1990. Recovery from Tracer Contamination in Ams Sample Preparation - Discussion. Radiocarbon 32(1):84-5.

Lang SQ, McIntyre CP, Bernasconi SM, Früh-Green G, Voss BM, Eglinton TI, Wacker L. 2016. Rapid 14C analysis of dissolved organic carbon in non-saline waters. Radiocarbon, Accepted, Proofs returned.

Vogel JS, Southon JR, Nelson DE. 1990. Memory Effects in an Ams System - Catastrophe and Recovery. Radiocarbon 32(1):81-3.

Wacker L, Fahrni SM, Hajdas I, Molnar M, Synal HA, Szidat S, Zhang YL. 2013. A versatile gas interface for routine radiocarbon analysis with a gas ion source. Nuclear Instruments & Methods in Physics Research Section B-Beam Interactions with Materials and Atoms 294:315-9.

Zermeno P, Kurdyla DK, Buchholz BA, Heller SJ, Kashgarian M, Frantz BR. 2004. Prevention and removal of elevated radiocarbon contamination in the LLNL/CAMS natural radiocarbon sample preparation laboratory. Nuclear Instruments & Methods in Physics Research Section B-Beam Interactions with Materials and Atoms 223:293-7.

Zhou WJ, Wu SG, Lange TE, Lu XF, Cheng P, Xiong XH, Cruz RJ, Liu Q, Fu YC, Zhao WN. 2012. High-Level C-14 Contamination and Recovery at Xi'an Ams Center. Radiocarbon 54(2):187-93.

Protocol for preparation of swab samples for ^{14}C contamination testing and AMS analysis using a gas ion source MICADAS fitted with CHS-GIS system.

Based on the method of Lang et.al (2016) Radiocarbon, accepted.

Important notes:

- Freshly combust vials, filters, tweezers at 450 °C for 2-3 hours
- Use fresh gloves for each sample
- Process samples in order so you can track any cross contamination
- Do any suspicious samples last
- Accompany samples with a list and indicate suspicious samples
- Use a separate lab for drying, purging and oxidation to avoid contamination of your ^{14}C natural abundance work areas

Background:

Natural abundance radiocarbon measurements by accelerator mass spectrometry (AMS) are easily affected by contamination derived from the use of radiochemicals enriched in ^{14}C in the same lab, building or area. Start by asking around to see if people know if ^{14}C has been or is being used locally. Communication and information is the best tool to establish the history of ^{14}C radiochemical usage in your area. Swabbing should target areas and equipment to be used for the preparation of samples for natural abundance measurements. Common work areas or equipment are a good place to start before considering specific items. Door handles, shelves, benches, fridges, ovens, fume hoods, freeze dryers and balances have tested positive in the past. Door handles seem to be a good way of picking up if contamination is being transferred around locally.

Materials:

Sampling kit:

- Basket, Al foil, rack, tweezers, pen, notebook
- Kimtech Science Purple nitrile gloves (PN-90627)
- Freshly combusted 25mm quartz fiber filters
- Fresh Isopropanol (IPA) in 100ml Schott bottle
- Freshly combusted Exetainer vials (12 ml, Labco, PN-9RK8W)
- Clean Exetainer caps

Lab Materials:

- AR grade Sodium Persulfate
- Milli-Q water
- 85% H_3PO_4
- Clean amber vial for persulfate solution
- Heater block for vials (or a hot plate with additional block on top)
- HP helium purge line (Cylinder, regulator, valve, tubing, syringe tip)

- Dedicated drying oven (or the heater block for vials)
- 32G, 50mm, syringe tips

Swiping procedure:

- Using tweezers, moisten a combusted quartz filter with IPA
- By hand, wipe an area of 5-20 cm^2 or equipment
- Insert the swiped filter into an Exetainer vial, label and loosely cover with aluminum foil
- Remember to change your gloves after every swipe to avoid cross-contamination
- Prepare 3 blanks by placing a moistened filter in an Exetainer without wiping anything
- Dry the quartz filters in an oven overnight (ca. 60°C), loosely covered by aluminum foil
- Note: You can dry vials in a heater block and avoid the need for a separate oven

Making a new batch of oxidant:

- Weigh out sodium persulfate onto a boat (~1.5 g in 50 mL).
- Transfer into combusted glass amber vial
- Add Milli-Q water gravimetrically
- Add ~5 drops of 85% H_3PO_4 and shake to dissolve
- Store in fridge. Oxidant is good for 2 days so long as care is taken to not contaminate it

Preparing samples:

- Transfer 1 mL of oxidant to the Exetainer vials containing the dried quartz filters.
- Cap the vials with clean caps.

Purging Exetainer vial with He:

- Turn on He gas at least 5 minutes before use
- Insert a syringe tip into each vial for venting
- Insert the helium purge needle and purge for 1 min at 100 mL/min
- After, remove both tips at the same time and dispose of the venting tip
- Repeat until all vials are purged

React samples:

- Heat samples at 100°C for 1 hour on a heater block
- Allow to cool and equilibrate overnight
- Once equilibrated, the samples are stable for a long time (months)
- Samples can be analyzed by CHS-GIS-AMS directly from the Exetainer vials.

Clean up:

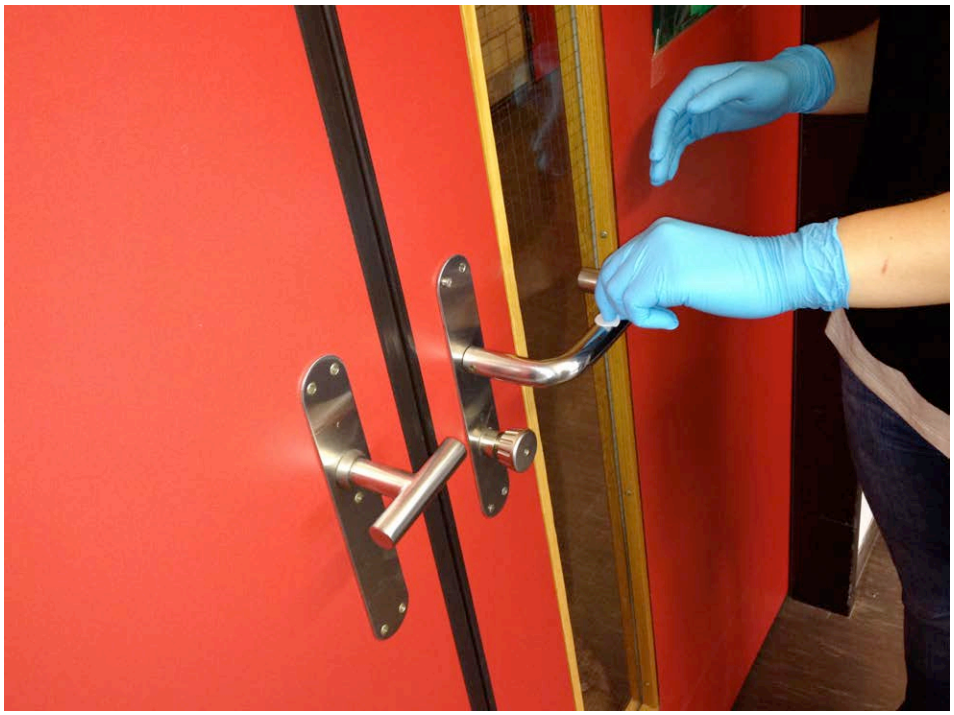
- Dispose of oxidant in a special waste container
- Vials that are not contaminated can be reused
- Acid wash all vials and caps, combust at 450°C for 2-3h

Photos:

1. A Sampling kit:

2. Moistening a filter in IPA

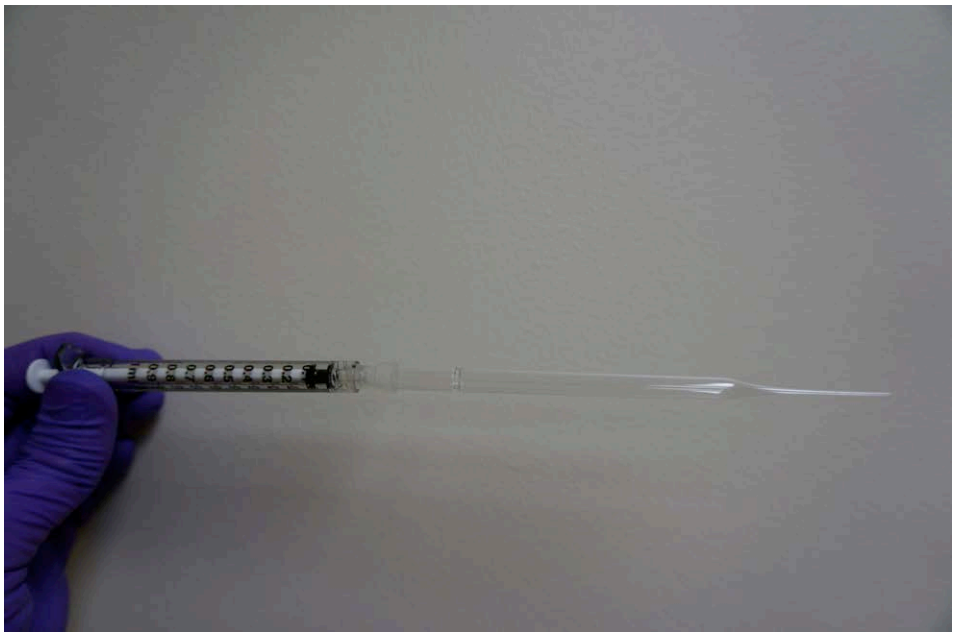
3. Swabbing a door handle:



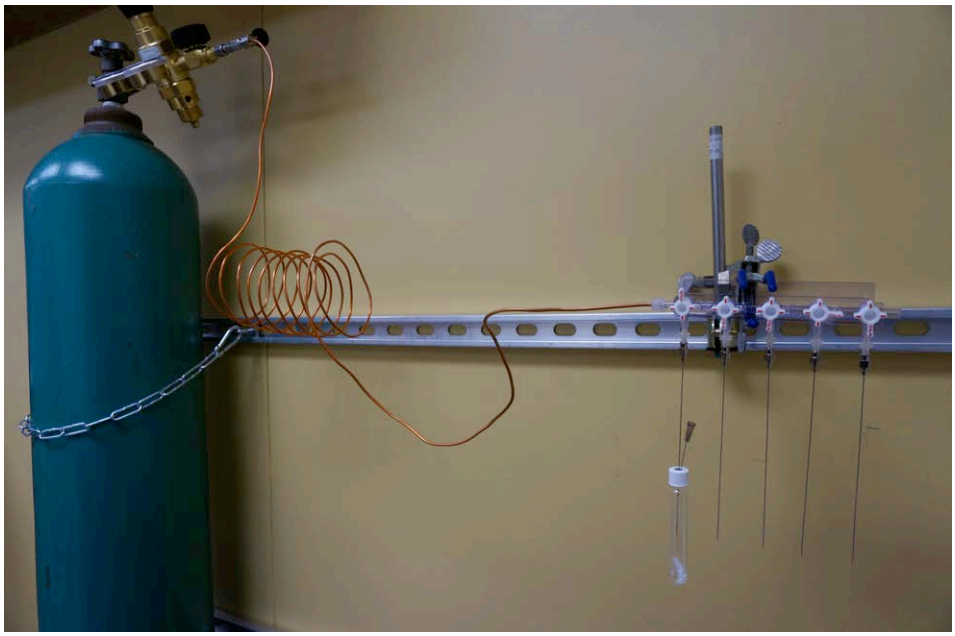
4. A dedicated preparation area away from natural abundance laboratories

5. Drying the swabs and vials in an oven

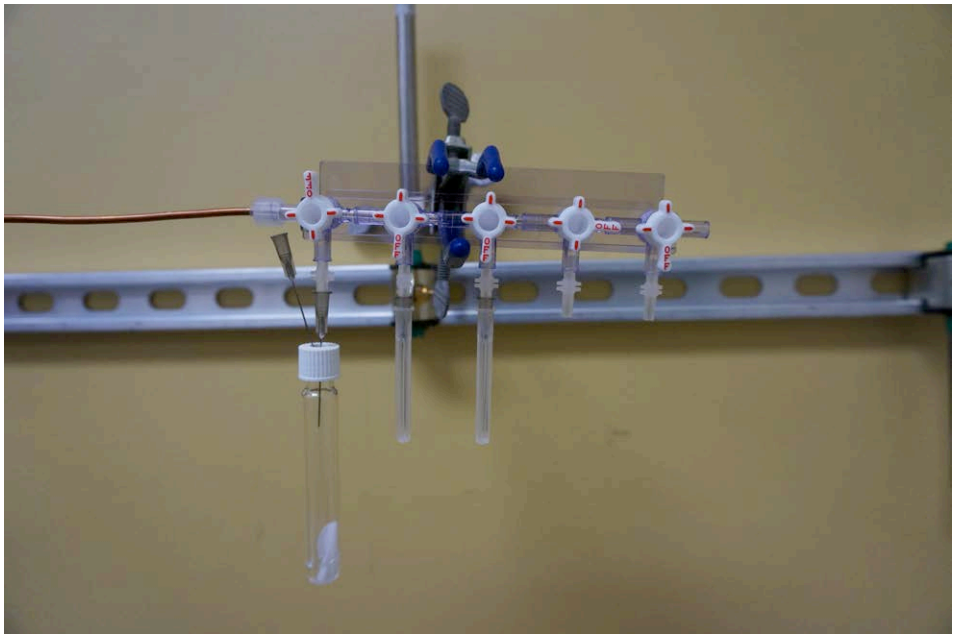
6. Syringe for dispensing 1 mL of persulfate solution



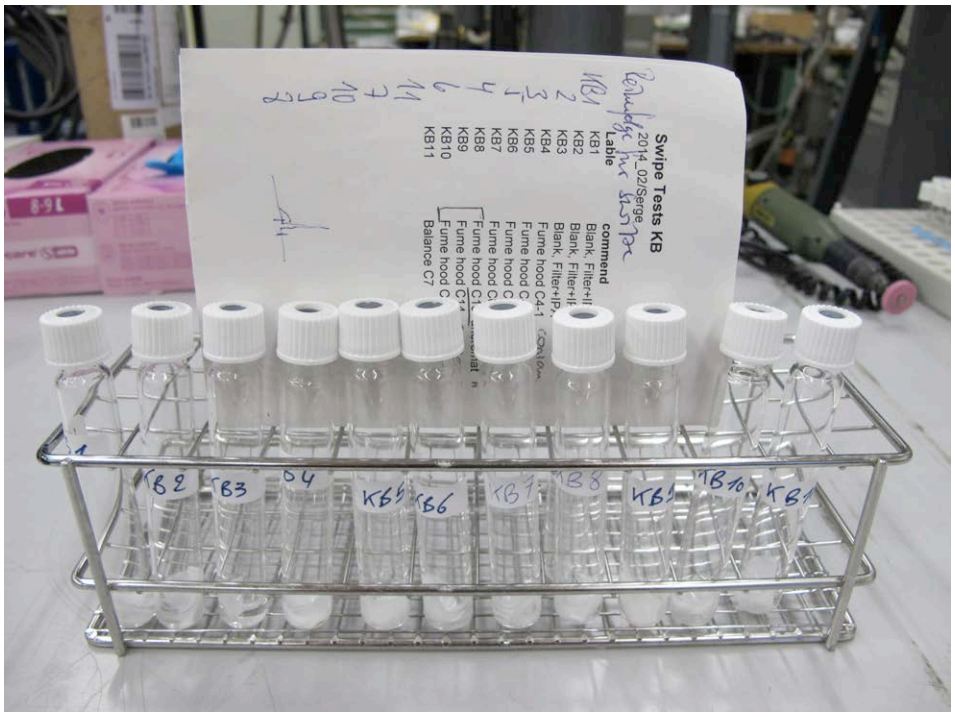
7. A helium purge station



8. Purging vials with Helium using disposable tips



9. Prepared sample waiting for analysis



10. Analysis using the CHS-GIS-MICADAS