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:

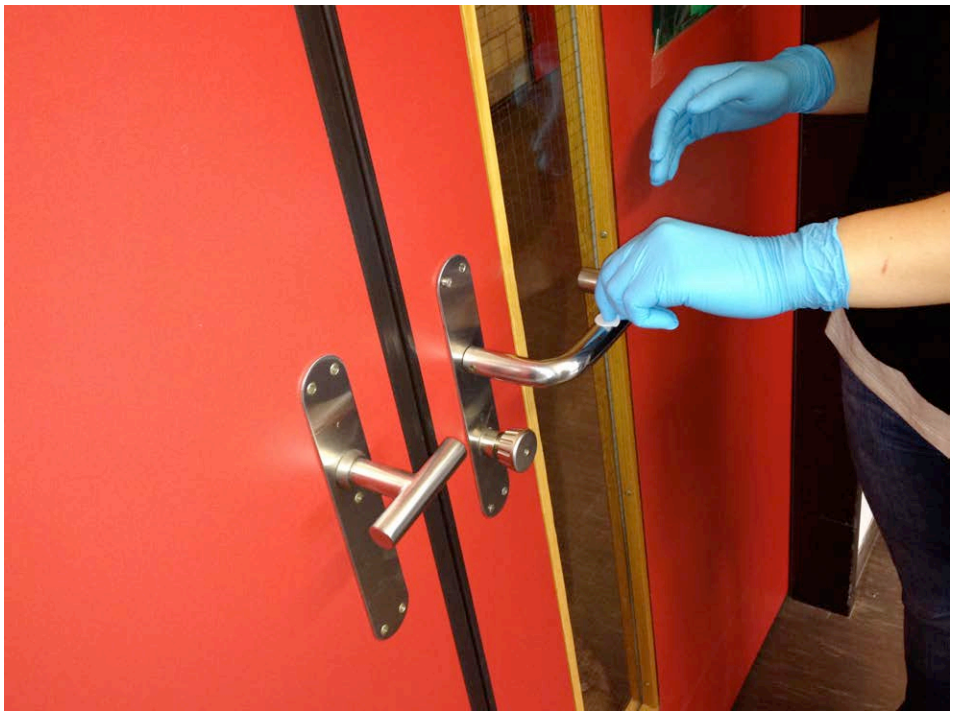
- Dispose of oxidant in a special waste container
- Vials that are not contaminated can be reused
- Acid wash all vials and caps, combust at 450°C for 2-3h

Photos:

1. A Sampling kit:

2. Moistening a filter in IPA

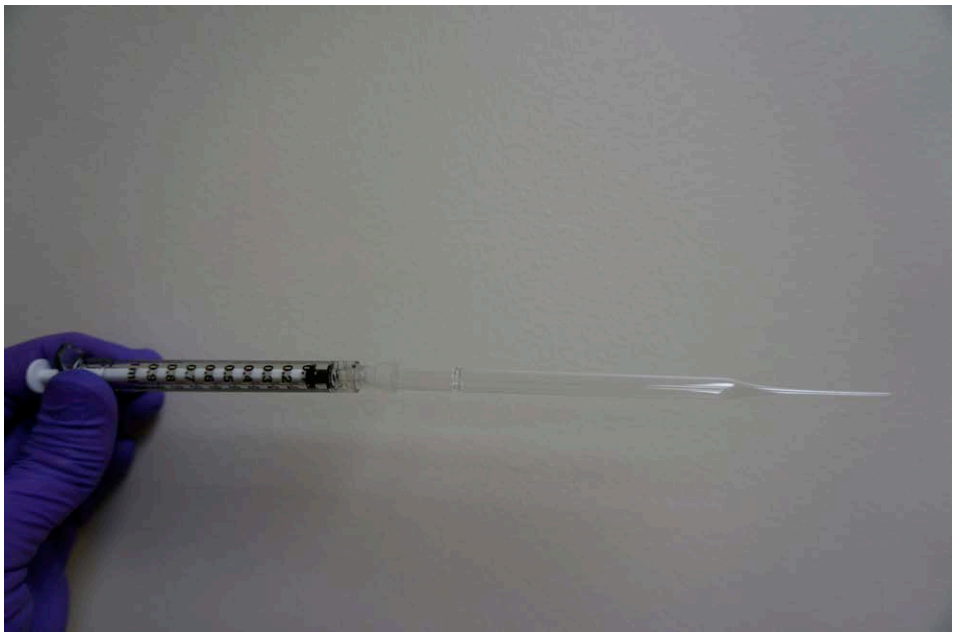
3. Swabbing a door handle:



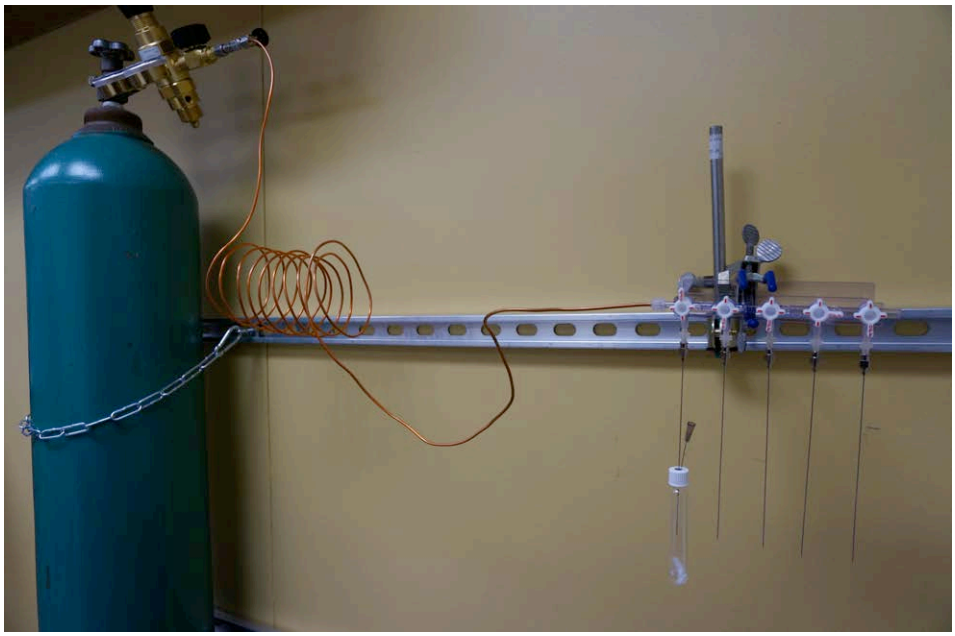
4. A dedicated preparation area away from natural abundance laboratories

5. Drying the swabs and vials in an oven

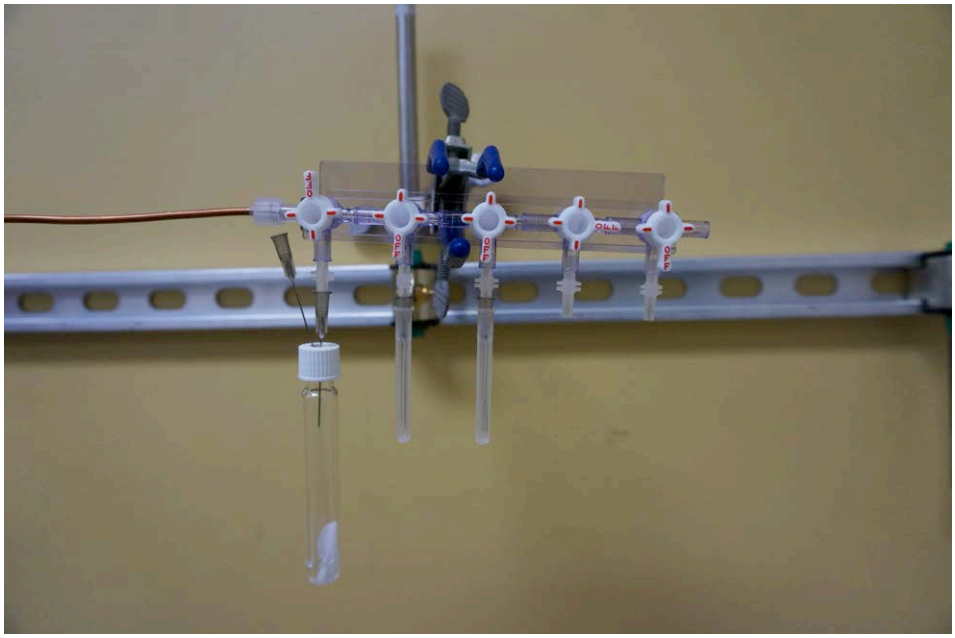
6. Syringe for dispensing 1 mL of persulfate solution



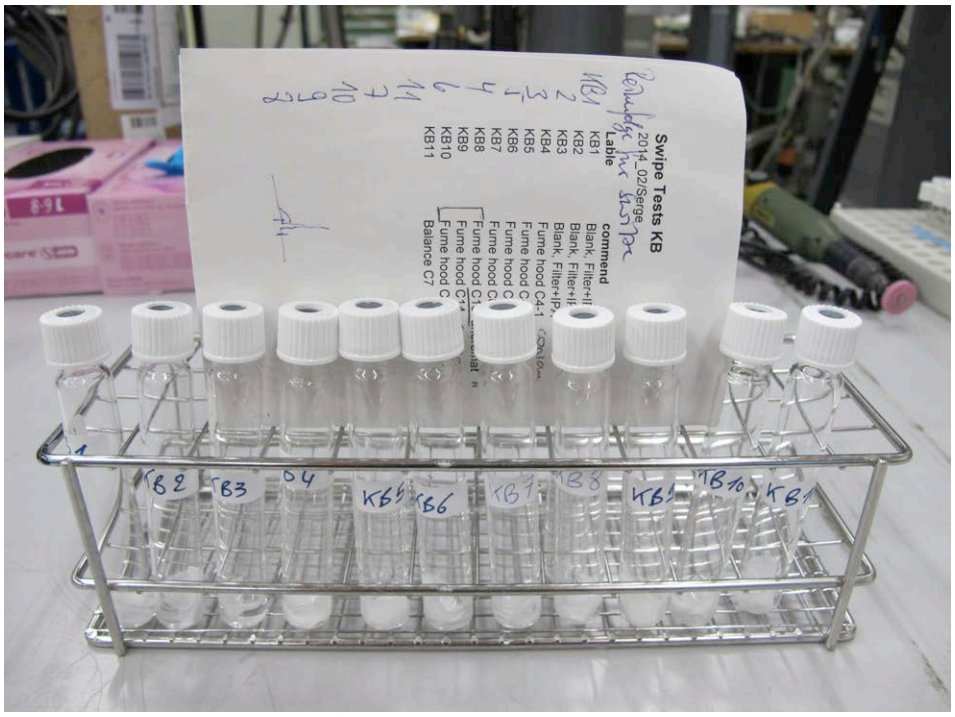
7. A helium purge station



8. Purging vials with Helium using disposable tips



9. Prepared sample waiting for analysis



10. Analysis using the CHS-GIS-MICADAS