^{14}C CONTAMINATION

And How to Conduct Swipe Test

Xiaomei Xu

Radiocarbon in the Earth System
Sep 24-29 2017, Jena

The biggest fear is…

^{14}C tracer contamination

Reported ^{14}C contamination in AMS lab

High-level ^{14}C contamination reported at low background AMS laboratories:

- LLNL: 30,000 times modern (Vogel et al. 1990)
- Arizona AMS lab: 5,000 times modern (Jull et al. 1990)
- XI'AN AMS center: 100,000 times modern (Zhou et al, 2012)

Difficult to clean and costly both in time and effort

Prior knowledge and test

—SWIPE FIRST!

^{14}C prep begins before you collect the sample

We see cases with ^{14}C contamination ALL the times!

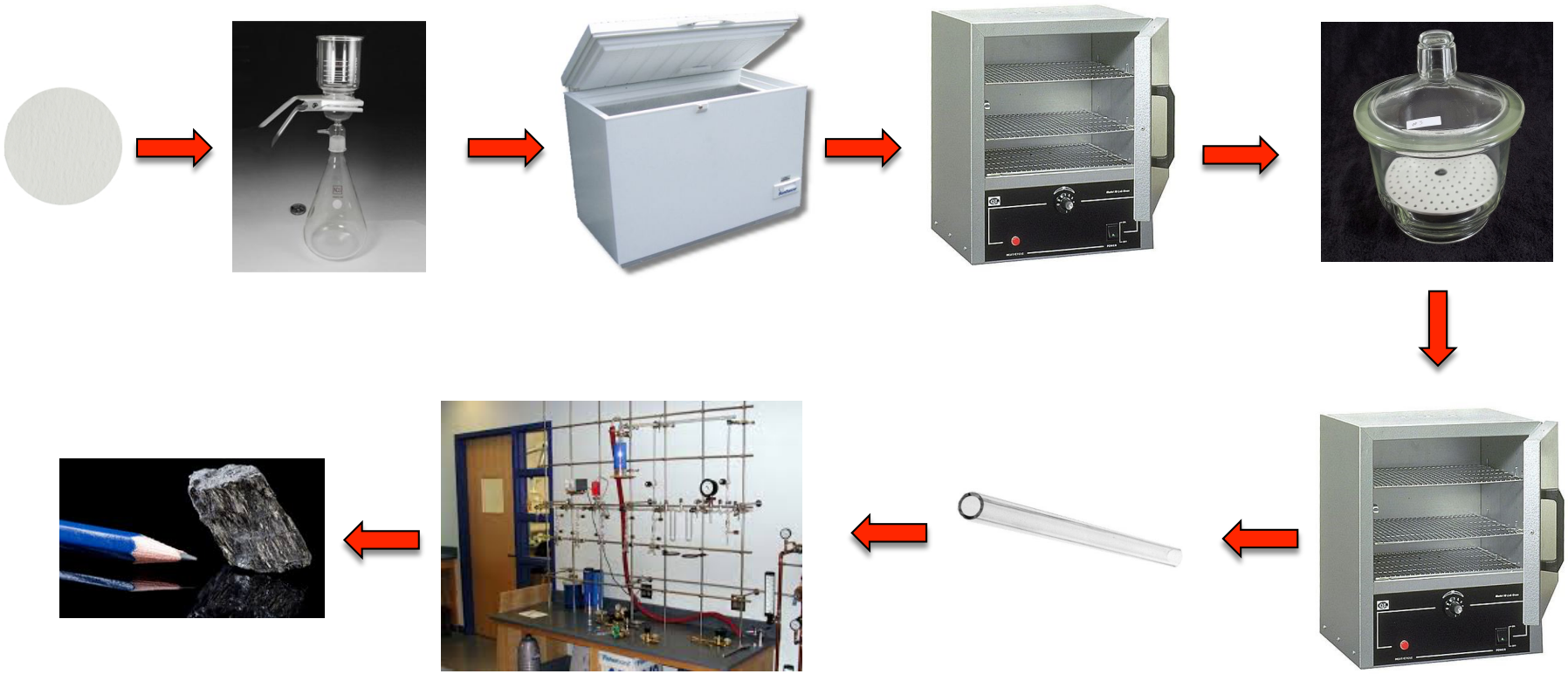
Common places contaminated with ^{14}C tracer	Be extra careful of shared equipment!
▫ Biology & medical labs ▫ Agricultural schools ▫ Any labs that study photosynthesis ▫ Ships! ▫ Labs that use stable isotope tracers	– Ovens – Fridges – Centrifuges – Fume hoods – Coolers – Purchasing/inheriting used equipment – swipe it first

Oftentimes verbal confirmation that lab space/equipment is tracer free is not enough

Know what you are actually measuring:

Know what you are actually measuring:

Example: Collecting and processing POC samples



$$C_{measured} = C_{POC} + C_{quartz\ filter} + C_{acidification} + C_{freezer} + C_{oven} + C_{combustion} + C_{line\ extraction}$$

Know what you are actually measuring:

How much C could I really possibly be picking up??

Example: Collecting and processing POC samples

Did you pre-bake your filter? → Was this manifold cleaned and baked? → Has this freezer **EVER** stored samples from tracer experiments? → Has this dryer **EVER** been used tracer experiments? → Did you use plastics inside the desiccator? ↓

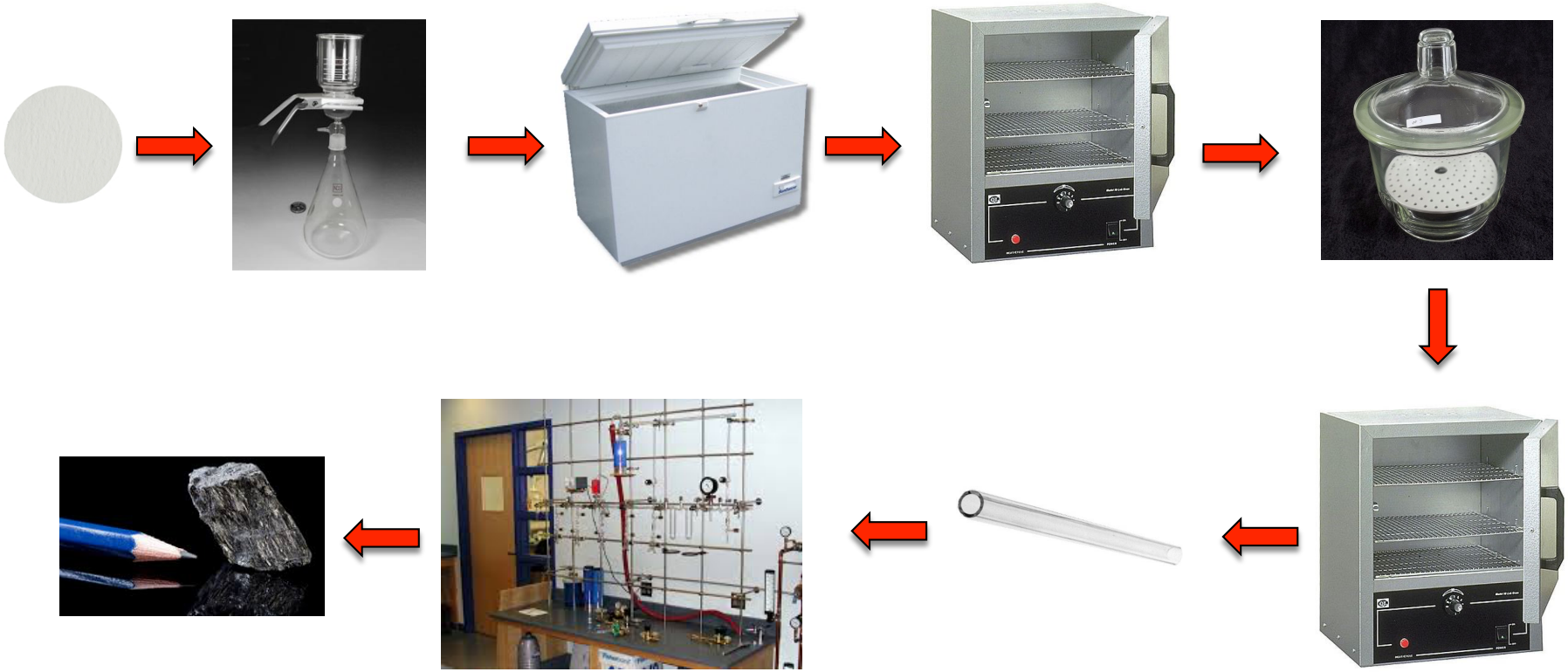
Did you pre-bake your tubes and CuO? ← Was the vacuum line leak tight and clean? ← Was your Fe thoroughly blank tested?

$$C_{measured} = C_{POC} + C_{quartz\ filter} + C_{acidification} + C_{freezer} + C_{oven} + C_{combustion} + C_{line\ extraction}$$

Know what you are actually measuring:

Can you really measure such small amounts?

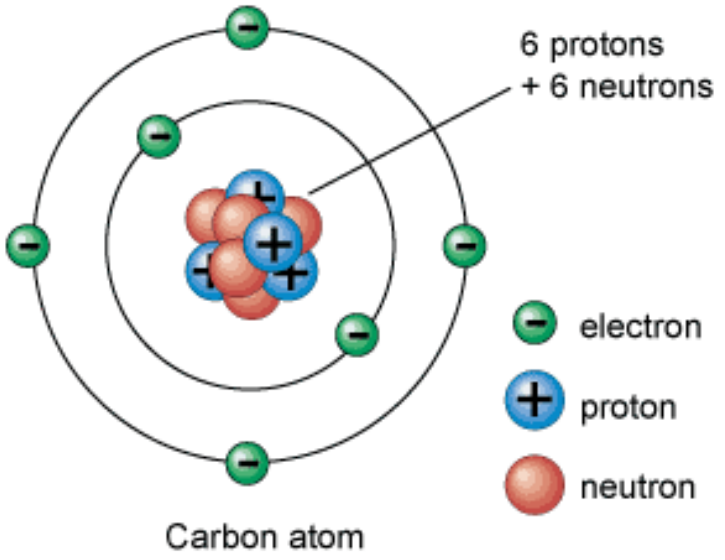
Example: Collecting and processing POC samples



$$C_{measured} = C_{POC} + C_{quartz\ filter} + C_{acidification} + C_{freezer} + C_{oven} + C_{combustion} + C_{line\ extraction}$$

Know what you are actually measuring:

Abundance of C Isotopes



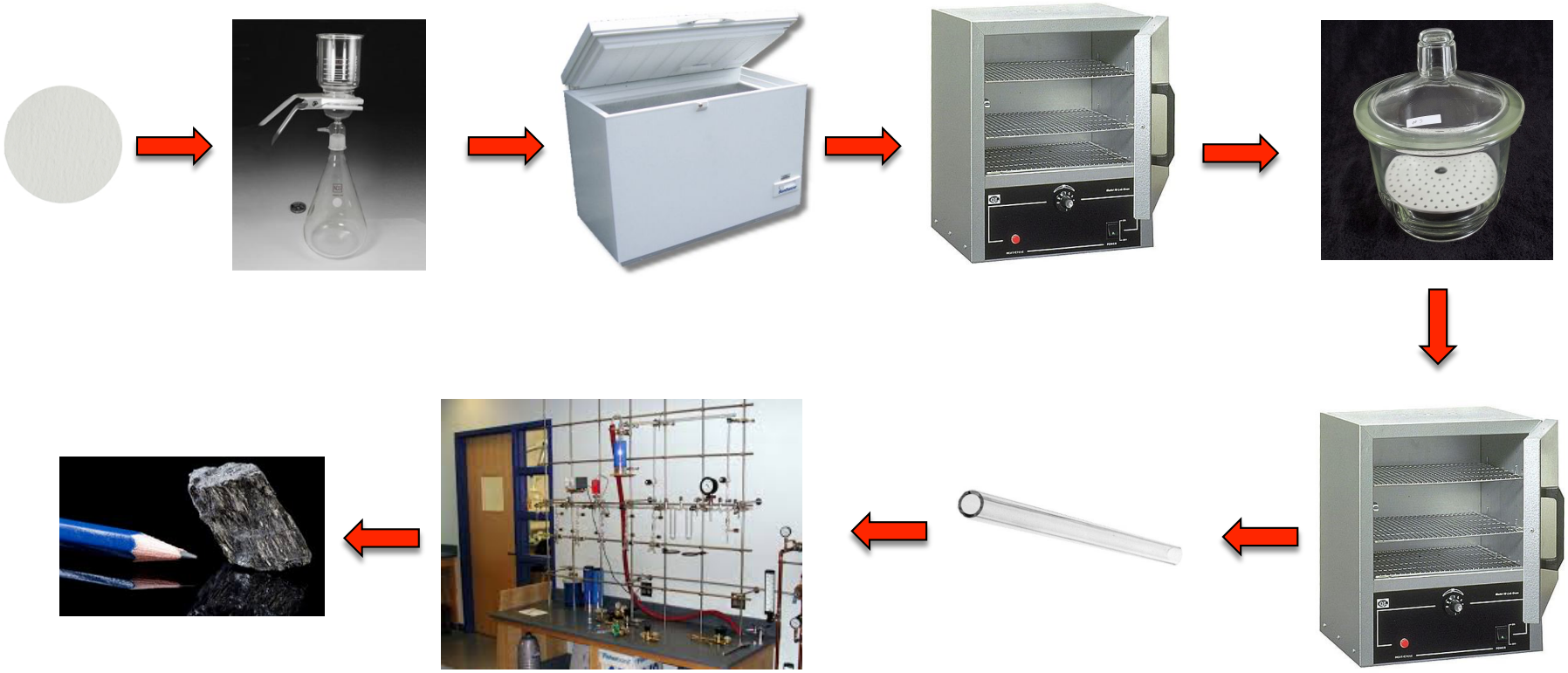
^{12}C	98.91%
^{13}C	1.09 %
^{14}C	**10^{-10} %**

^{14}C is an extremely sensitive measurement!

Know what you are actually measuring:

How can I possibly get an accurate measurement?

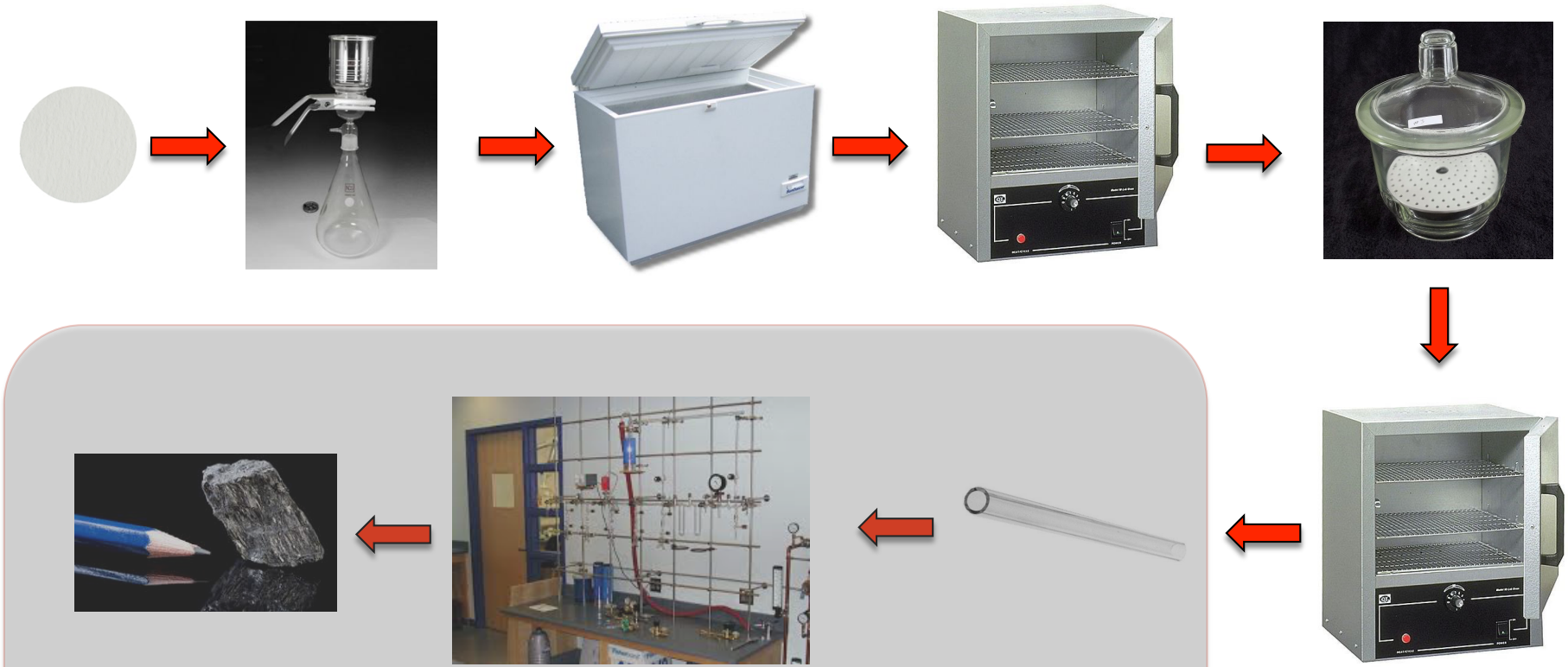
Example: Collecting and processing POC samples



To determine the accuracy of your data, modern and dead standards must be processed along with your samples to assess the size and age of your C blank.

Know what you are actually measuring:

Example: Collecting and processing POC samples

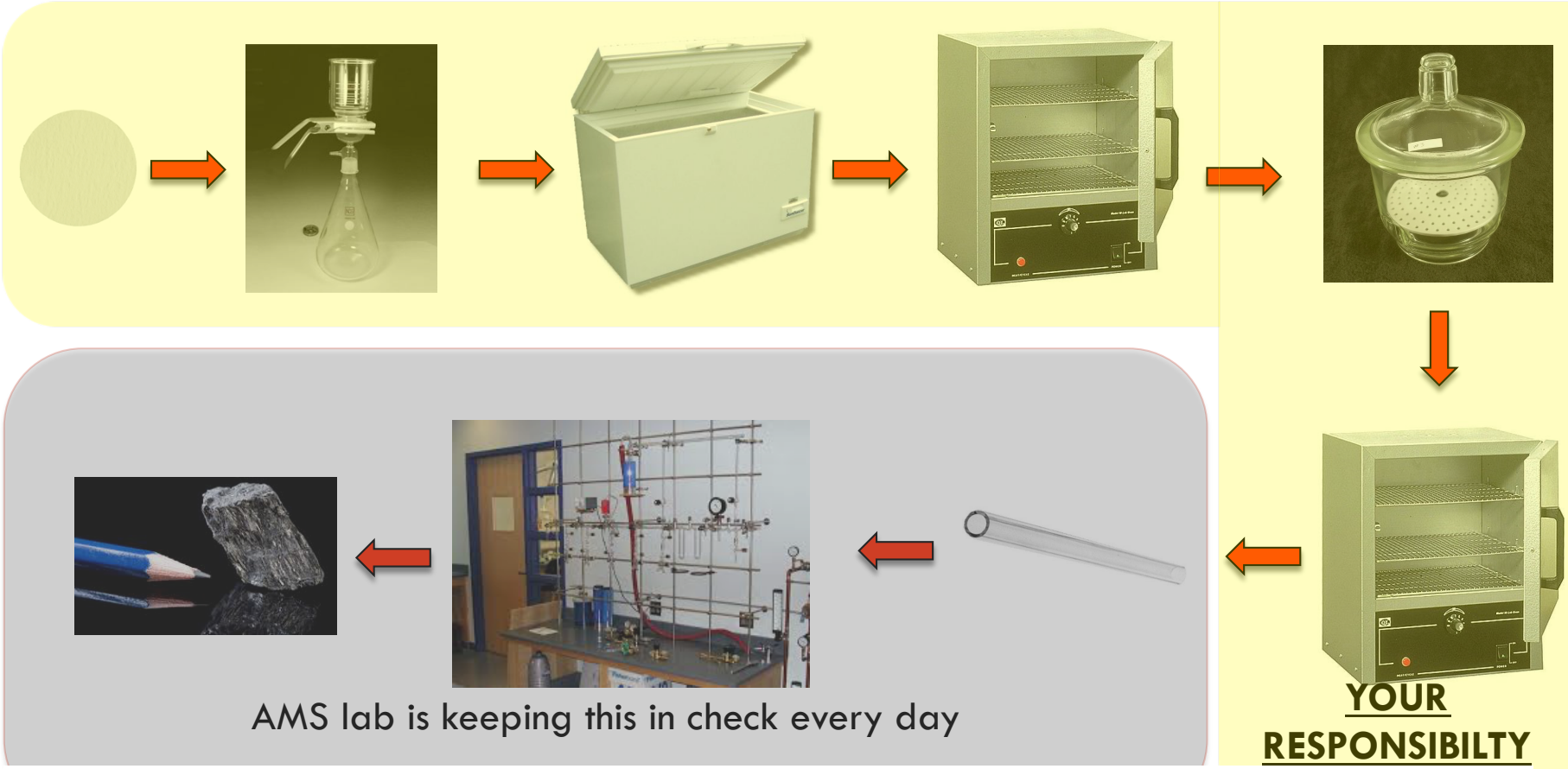


AMS lab is keeping this in check every day

To determine the accuracy of your data, modern and dead standards must be processed along with your samples to assess the size and age of your C blank.

Know what you are actually measuring:

Example: Collecting and processing POC samples



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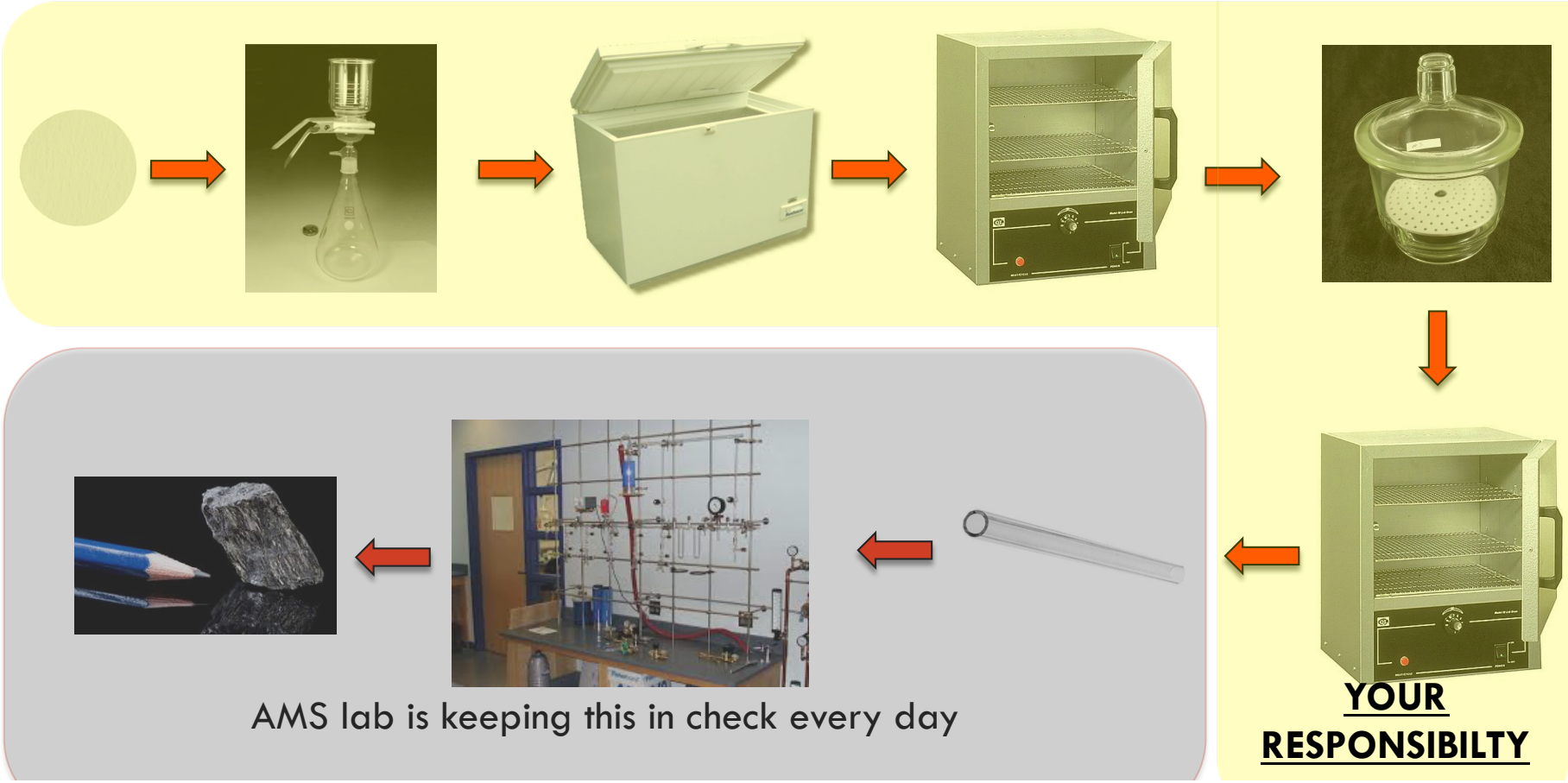
Know what you are actually measuring:

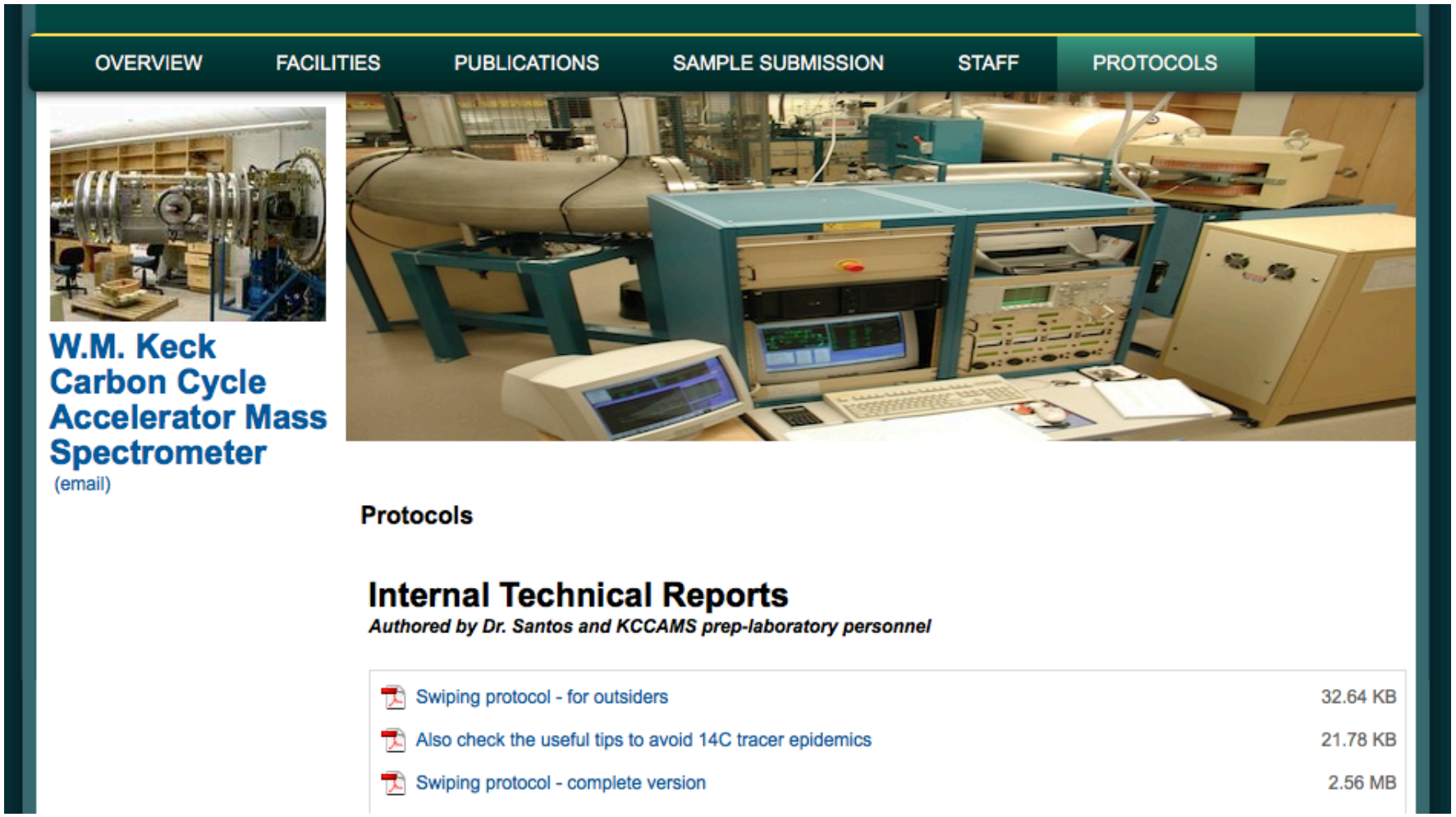
Example: Collecting and processing POC samples



CONTACT US BEFORE YOU COLLECT OR PROCESS YOUR SAMPLES!

How to Swipe

Contact your AMS lab to get a swipe kit, or get your own quartz filters



Places to swipe

Shared Equipment

- Ovens
- Fridges
- Centrifuges
- Fume hoods
- Coolers
- Purchasing/ inheriting used equipment

Shared Spaces

- Door handle
- Light switch
- Counter top
- Sink handle

Swipe Test Sampling

Selecting a 10*10 cm sampling area

↓

Clean the sampling area carefully with paper towel

↓

Wet a pre-baked 25mm diameter quartz filter with methanol

↓

Swipe the quartz filter zigzag on the sampling area

↓

Wait for the alcohol to dry

↓

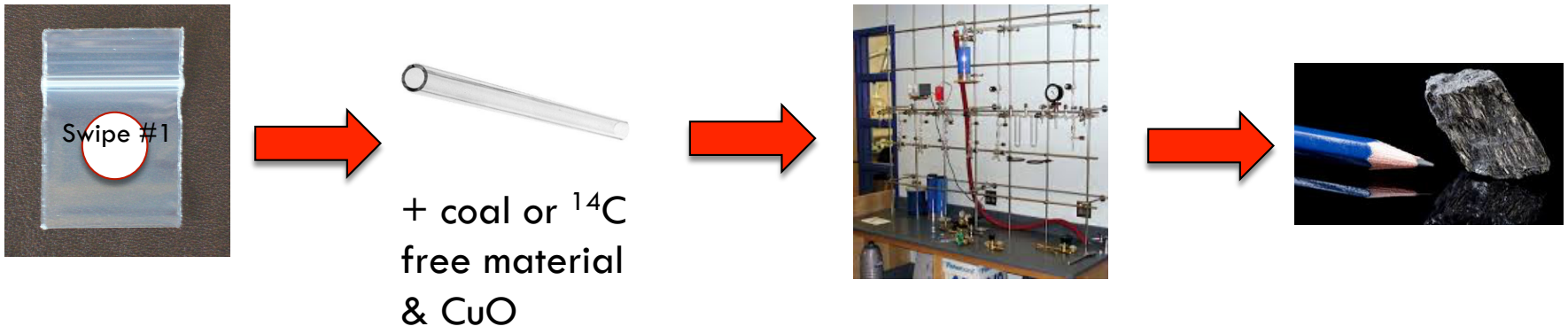
Put it into a plastic ziplock bag and label the bag

↓

Take an extra filter and moisten with alcohol to act as 'swipe blank' before and afterwards

Swipe protocol: https://www.ess.uci.edu/group/ams/protocols

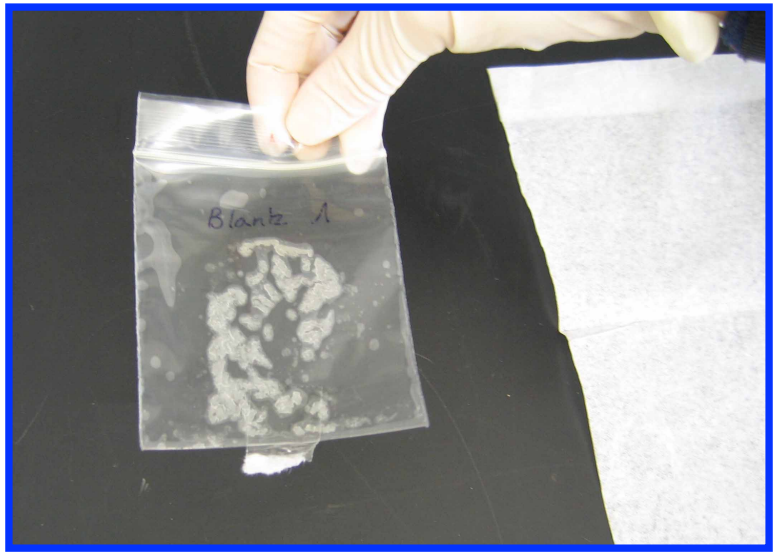
How we process your swipes

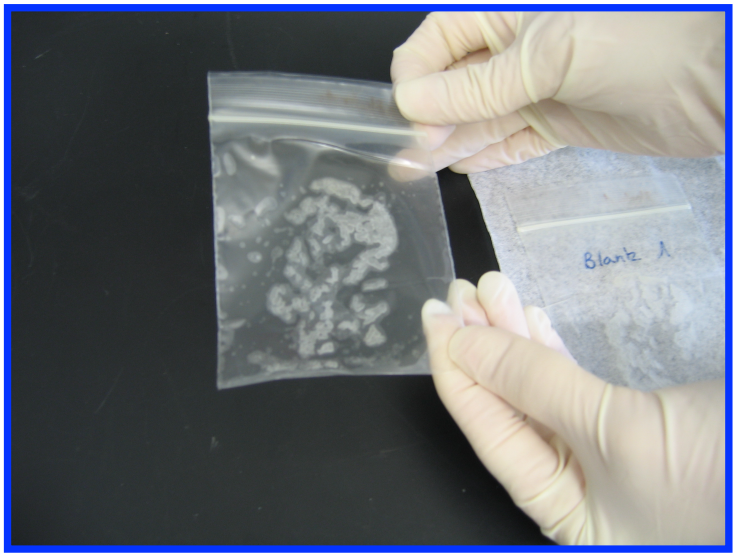


- Coal or ^{14}C free material is added as carrier
- It should only show slight levels of enrichment above background if there is no contamination because you would have swiped trace amounts of C from dirt
- Special prep lab & vacuum line, isolated from the regular lab!

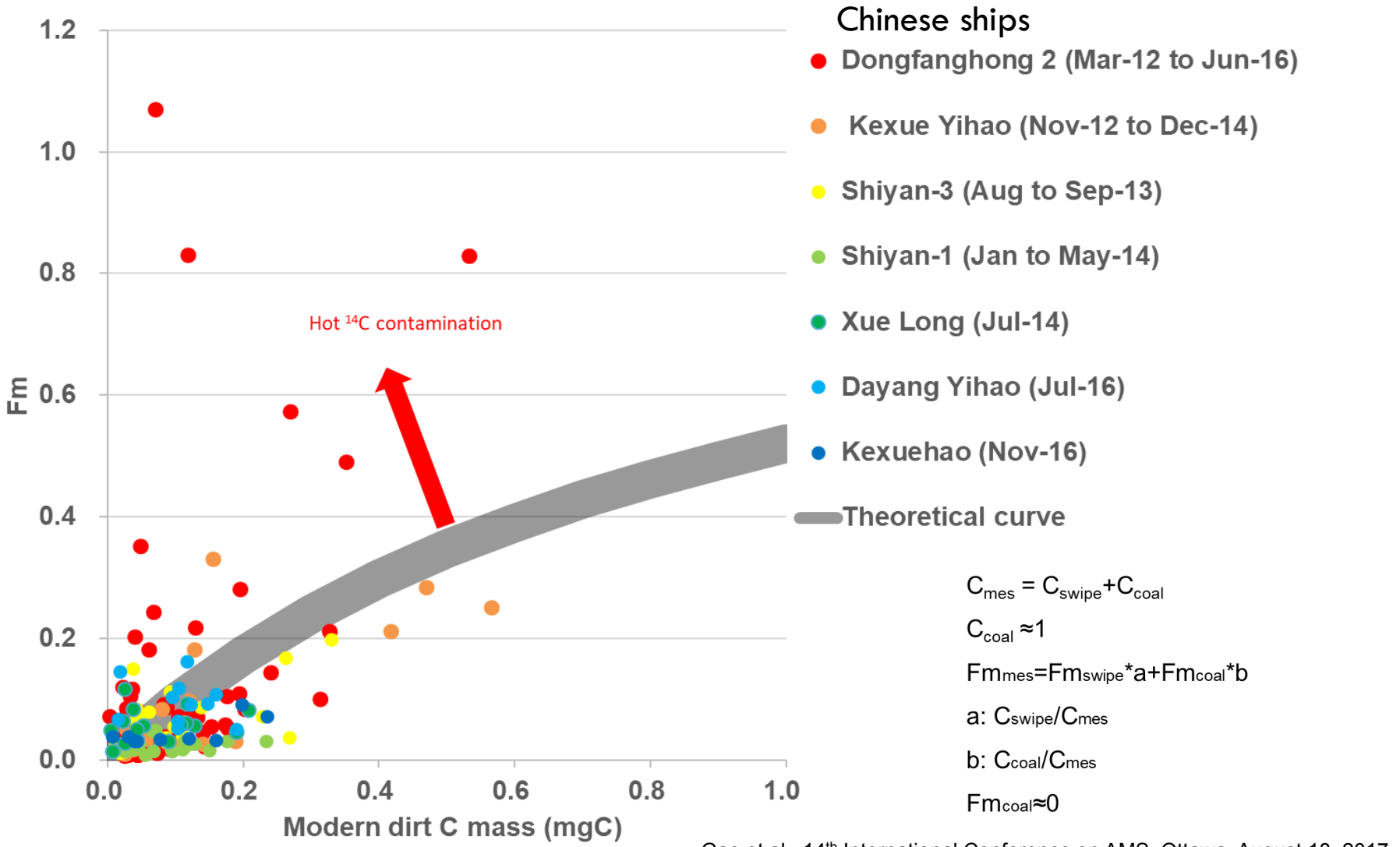
Common mistakes when swiping

1. Too much dirt on the filter
2. Use too much alcohol
3. Alcohol on the outside of the bag erases the label
4. Label smeared - bags stacked together before the label is dry
5. Alcohol contains water?
6. Don't forget filter blanks





Modern Dirt Effect on Swipes – Avoid too much Dirt!



$C_{mes} = C_{swipe} + C_{coal}$

$C_{coal} \approx 1$

$Fm_{mes} = Fm_{swipe} * a + Fm_{coal} * b$

a: C_{swipe}/C_{mes}

b: C_{coal}/C_{mes}

$Fm_{coal} \approx 0$

Gao et al., 14th International Conference on AMS, Ottawa, August 18, 2017

^{14}C Contamination Example 1

KECK CARBON CYCLE AMS FACILITY

EARTH SYSTEM SCIENCE DEPT, UC IRVINE

^{14}C results — Sickman/Lucero swipes — Feb 24 2007

UCIAMS #	Sample name	Other ID	fraction Modern	±	D^{14}C (‰)	±	^{14}C age (BP)	±
	Fume hood		86.0079	1.5768	85007.9	1576.8	-35770	150
	Rm 208 Door handle		177.8736	2.3230	176873.6	2323.0	-41610	110
	Rm 208A Door handle		13.8106	0.0520	12810.6	52.0	-21085	35
	Rm 208A Counter		1.1672	0.0045	167.2	4.5	-1235	35
	Freeze dryer, cleaned		4.2638	0.0163	3263.8	16.3	-11645	35
	Blank 1		0.3328	0.0015	-667.2	1.5	8840	40
	Blank 2		0.3600	0.0012	-640.0	1.2	8210	30
	Trap sent 2/15		0.3868	0.0012	-613.2	1.2	7630	30

Radiocarbon concentrations are given as fractions of the Modern standard, D^{14}C, and conventional radiocarbon age, following the conventions of Stuiver and Polach (Radiocarbon, v. 19, p.355, 1977).

^{14}C Contamination Example 2

UCIT	Sample	D14C (per mill)	error
UCIT17025	Coal and Blank 1	-949.3	0.4
UCIT17028	Coal and Blank 2	-727.1	0.9
UCIT17023	Walk-in cooler	767.4	8.7
UCIT17024	Bench in the grinding room downstairs	1023.7	9.9
UCIT17026	Bench next to scale downstairs (A127)	-208.3	1.3
UCIT17027	Aggregate room Hood 1 (A131)	121463.3	2153
-	Bench next to scale in soils lab upstairs (A235)	-	-
UCIT17029	Bench next to IRGA in soils lab upstairs (A235)	2166.8	15.4
UCIT17030	Coal Trumbore lab	-994.7	0.1

$F^{14}C = 122.4633$

^{14}C Contamination Example 3

KECK CARBON CYCLE AMS FACILITY

EARTH SYSTEM SCIENCE DEPT, UC IRVINE

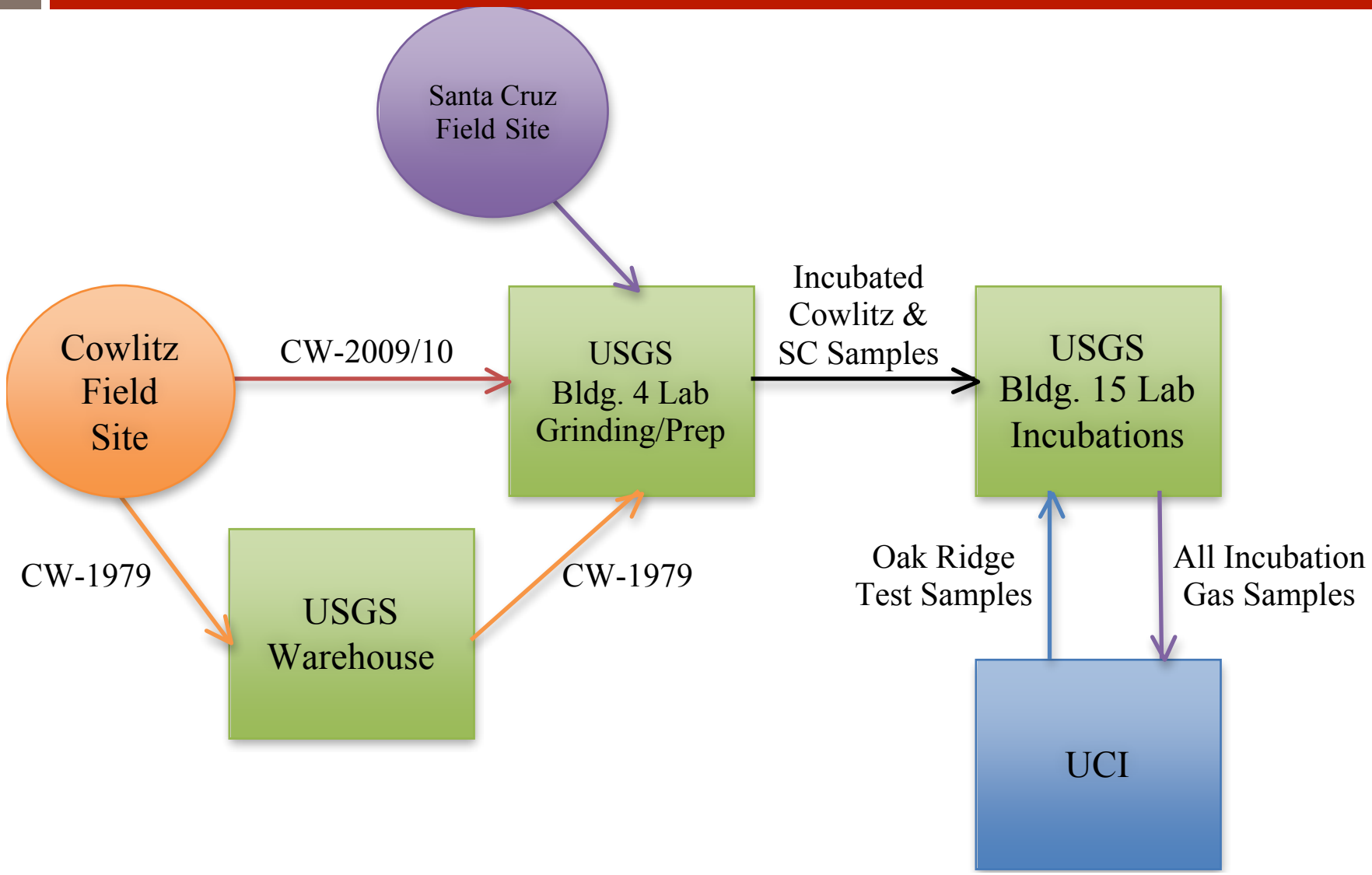
^{14}C results — Corey Lawrence/Jennifer Harder/USGS — **Mar15_12**

UCIAMS #	Sample name	Other ID	Fraction Modern	±	$\Delta^{14}C$ (‰)	±	^{14}C age (BP)	±
	UCIT26759	CO2 Blank 1_Lawrence	0.0554	0.0003	-945.0	0.3	23240	45
	UCIT26760	CO2 Blank 3_Lawrence	0.0451	0.0002	-955.2	0.2	24885	40
	UCIT26761	CW4.200_Lawrence	1.7634	0.0125	750.2	12.5	>Modern	
	UCIT26762	CW8.70_Lawrence	1.3490	0.0057	338.9	5.7	>Modern	
	UCIT26763	CWO1.20_Lawrence	1.7613	0.0125	748.2	12.5	>Modern	
	UCIT26764	CWEE1.24_Lawrence	2.3988	0.0241	1380.9	24.1	>Modern	
	UCIT26765	CWHM2.23_Lawrence	2.9496	0.0331	1927.6	33.1	>Modern	
	UCIT26766	CWHM2.50_Lawrence	4.2441	0.0549	3212.4	54.9	>Modern	
106361	UCIT26767	SCT2.20_Lawrence	1.1628	0.0029	154.1	2.9	>Modern	
106362	UCIT26768	SCT3.22_Lawrence	1.1630	0.0029	154.4	2.9	>Modern	
106363	UCIT26769	SCT5.021_Lawrence	1.1206	0.0025	112.3	2.5	>Modern	

UCIT26761-26766 are likely contaminated. No UCIAMS# were assinged for these samples

To ensure the valuable archived soils are intact we launched an intensive investigation.

Investigation of Contamination – Trace back



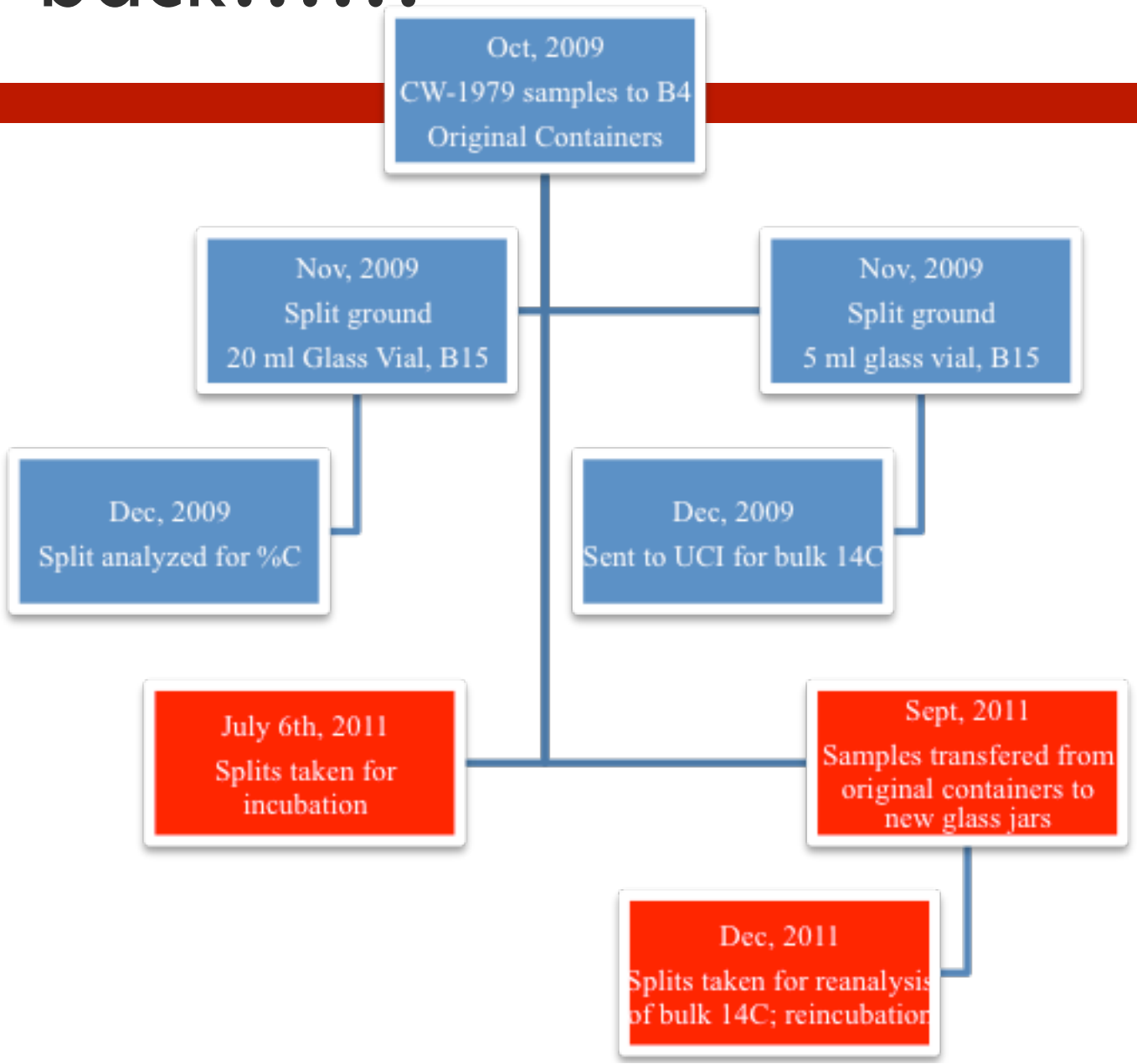
Investigation of Contamination – more Swipes

Date Swiped	Date Analyzed	Description	FM
11/01/11	12/3/11	Bldg. 15 - Corey's Office - Desk and Door Knob	0.2351
11/01/11	12/3/11	Bldg. 15 - Kristen's Office - Desks and Door Knob	0.3237
11/01/11	12/3/11	Bldg. 15 - Settling Lab - Counters and Door Knob	0.2206
11/01/11	12/3/11	Bldg. 15 - Jorie's Small Lab - Counters and Door Knob	0.1249
11/01/11	12/3/11	Bldg. 15 - Jorie's Large Lab - Counters and Door Knob	0.1164
11/01/11	12/3/11	Bldg. 15 - Waldrop's Second Floor Lab - Counter and Door Knob	0.1098
11/01/11	12/3/11	Bldg. 15 - Kuwabara Lab - Counters (H2O and TOC) and Door Knob	0.1181
11/01/11	12/3/11	Bldg. 15 - Chemistry Lab – Counters and Door Knob	0.1888
11/01/11	12/3/11	Bldg. 15 - Incubation Jars - Lids and Glass	0.0875
11/01/11	12/3/11	Bldg. 15 - Blank	0.0949
11/01/11	12/3/11	Bldg. 15 - Balances	0.0864
11/01/11	12/3/11	Bldg. 4 - Counters (Main Counter, Sink) and Door Knob	0.1286
11/01/11	12/3/11	Bldg. 4 - New Glass Archive Jars (Inside and Outside)	0.1171
11/01/11	12/3/11	Bldg. 4 - Blank	0.0827
11/01/11	12/3/11	Bldg. 4 - Sample Spatula used for transferring samples	0.0777
11/01/11	12/3/11	Bldg. 4 - Balances	0.1175
11/01/11	12/3/11	Carrier carbon	0.1022
11/01/11	12/3/11	Carrier carbon	0.0951
11/01/11	12/22/11	Bldg. 4 - Freezer - Inside	0.1633
11/01/11	12/22/11	Bldg. 4 - Sample Boxes (Old and Empty Cowlitz Boxes)	0.1145
11/01/11	12/22/11	Bldg. 4 - Sample Boxes (Current Modern and Archive Samples)	0.0786
11/01/11	12/22/11	Carrier carbon	0.0986
11/01/11	12/22/11	Carrier carbon	0.1012
3/20/12	3/30/12	Bldg. 15 - Rm 2065 light switch/door handle	0.1676
3/20/12	3/30/12	Bldg. 15 - Rm 2065 computer/counter	0.1269
3/20/12	3/30/12	Bldg. 15 - Rm 2065 liquid N dewars/tank	0.1017
3/20/12	3/30/12	Bldg. 15 - Rm 2065 Jen's well/counter	0.1052
3/20/12	**3/30/12**	**Bldg. 15 - Rm 2065 scintillation counter**	**0.9282**
3/20/12	3/30/12	Bldg. 15 - Rm 2065 blank	0.1056
3/20/12	3/30/12	Bldg. 15 - Rm 2067 light switch/door handle	0.098
3/20/12	3/30/12	Bldg. 15 - Rm 2067 counters	0.3647
3/20/12	3/30/12	Bldg. 15 - Rm 2067 blank	0.1043
3/20/12	3/30/12	Bldg. 15 - Rm 2066 outside door handle	0.0987
3/20/12	3/30/12	Tributyrin - Carrier C	0.0996
3/20/12	3/30/12	Tributyrin - Carrier C	0.1065

Play back……

Date	
Sep 2007	Technician for USGS Soils group begins filling liquid nitrogen tank in B15-2065 on a weekly basis.
Aug 2009	Recently collected CW-2009 samples arrive from field and are air-dried in B4.
Oct 2009	Archived CW-1979 samples are obtained from the USGS warehouse and transported to B4 laboratory for processing
Nov 2009	CW-2009 samples are sieved to 2 mm; CW-1979 and CW-2009 are split for %C and $\Delta^{14}C$ analyses of bulk soil; Splits are ground and packaged in glass vials and transferred to B15; Unground CW-2009 samples transferred to new glass jars for storage in B4.
Dec 2009	CW-2009 and CW-1979 samples analyzed in B15 for %C; Splits sent to UCI for $\Delta^{14}C$ of bulk soil
Oct 2010	CW-2010 samples arrive from field and are air-dried in B4; CW-2010 samples are sieved; Splits are taken, ground and transferred to B15 for %C; remaining unground sample transferred to new glass jars for storage in B4.
Dec 2010	Santa Cruz samples arrive in B4 and are air-dried, sieved, and stored in new glass jars (same as those used for Cowlitz samples).
Jul 2011	July 6th, splits of unground Cowlitz samples (CW-1979, CW-2009, CW-2010) are taken from B4 and transferred to B15 in new plastic falcon tubes; July 8th, first batch of incubations started in B15; July 18th, Oak Ridge samples arrive to B15 from UCI;
Aug 2011	Second batch of incubations started in B15
Oct 2011	CO_2 samples collected from incubations (over proceeding months) transferred to UCI and processed for $\Delta^{14}C$
Nov 2011	First round of swipes conducted in B15 and B4
Dec 2011	Splits of unground samples from storage jars in B4 taken and sent to UCI to reanalysis of bulk soil $\Delta^{14}C$; radiocarbon dead CO_2 added to 2 empty incubation jars, allowed to sit overnight, sampled and sent to UCI for $\Delta^{14}C$.
Jan 2012	Reincubation of select Cowlitz soils; Incubation of 3 Santa Cruz soils; radiocarbon dead CO_2 added to 3 incubation jars and allowed to sit during the duration of Cowlitz/Santa Cruz incubations.
Feb 2012	Reincubation sampled and CO_2 sent to UCI for $\Delta^{14}C$
Mar 2012	Second batch of swipes conducted in B15

Play back……


Oct, 2009
CW-1979 samples to B4
Original Containers
Nov, 2009
Split ground
20 ml Glass Vial, B15
Nov, 2009
Split ground
5 ml glass vial, B15
Dec, 2009
Split analyzed for %C
Dec, 2009
Sent to UCI for bulk 14C
July 6th, 2011
Splits taken for
incubation
Sept, 2011
Samples transfered from
original containers to
new glass jars
Dec, 2011
Splits taken for reanalysis
of bulk 14C; reincubation

As a result

- Had to go back and process many batches of soils to figure out which batches, when and where contamination happened
- Eventually determined that the contamination event was only a short time span and swipe test showed it was spatially limited
- However because that could not definitely prove that some samples were not contaminated, they had to assume all were, and many samples had to be discarded

Despite the lab having:

- Limited access with key code entry doors
- Zero tolerance for sharing equipment
- Strict clean procedure

Contamination still happened!

Remediation

If contamination is present, take immediate action to remove it. Do not use the area contaminated until it's proved clean.

- Replace furniture, equipment and tools seriously at risk of contamination.
- Floors, walls, ceiling, cabinets counters can be washed with Radiac Wash
- Everything returning back into the lab should be cleaned with Radiac Wash; If could not be cleaned – discard
- Cover common used counter areas with Al foil which is periodically switched out
- Establish routine procedures to avoid re-contaminating the area. Re-swiping after cleaning

Contaminated field sites

Field Site

- Toolik Lake and most of the research station, AK, US
- Abisko Watershed, Sweden
- Several buildings at Colorado State Univ., Fort Collins, US

Others (Urban)

- Incinerators burn biomedical waste
- Nuclear Power Plants
- Waste water treatment plants – affect the groundwater
- Urban rivers

…….

Special Precautions for natural ^{14}C sampling on shipboard

Before sampling

- Discover as much **history** of the workplace onboard as possible;
- **Swipe** the workplace and check for ^{14}C contamination: work bench, door knobs, light switches, instrument lids, fridges……

During sampling

- **Avoid** using any lab previously used for ^{14}C-tracer study/detected with high ^{14}C contaminations;
- **Cover** the whole workplace/ tools with plastic or foil sheets and change them periodically;
- Always wear **gloves** and change them frequently when handling sample

Post sampling

- **Swipe** outside of the boxes before allowing things back to the lab

SWAB Reports – cursory view

- Yearly Report (over 30 years), conducted by the Tritium Laboratory, University of Miami
- Monitoring of US Shipboard Contamination
- Scrub a surface with Sponge and water, squeeze out the water, measure ^{3}H and ^{14}C on a scintillation counter

Swab Report

- Get yearly report for free: http://www.rsmas.miami.edu/groups/tritium/swab/comments-on-swab-reports/
- Conduct swipe test to be sure even for low-level contamination

REPORT FOR SWAB # 639

LOCATION: Palmer Station, Antarctica
VESSEL: Palmer Labs

DATE: 18 June 2012
TECHNICIANS: J. O'Reilly, G. Tilbury

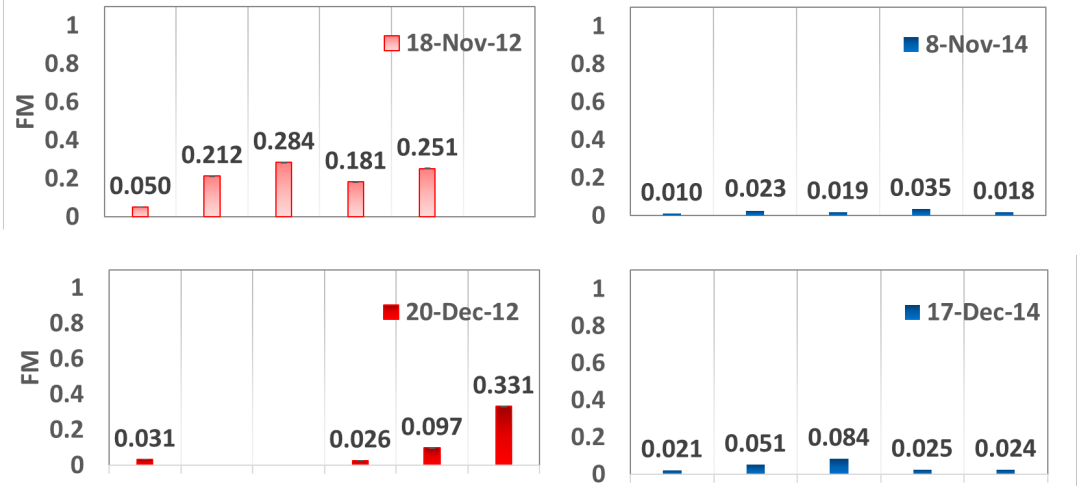
Sample #	Sample Identification	^{3}H dpm/m^{2} activity	±	error	^{14}C dpm/m^{2} activity	±	error
1	1st Vial Bkgnd	0	±	0	0	±	0
2	Initial bucket blank	0	±	0	0	±	0
	Lab 4						
3	Floor in front of Lab 4	41	±	64	0	±	0
4	Door handle area (inside)	382	±	70	0	±	-1
5	Door handle area (outside)	26	±	70	0	±	0
6	Light switch	2	±	31	3	±	34
7	Refrigerator face and handle	*2057	±	129	0	±	0
9	Clean benchtop	182	±	48	*130	±	38
10	Lid handle on PE LSC (left)	*1918	±	122	*51	±	13
11	Lid handle on PE LSC (right)	*2576	±	132	*671	±	49
12	Left side of right benchtop	*3274	±	119	*5046	±	123
13	Center of right benchtop	*3614	±	150	*1464	±	68
14	Right side of right benchtop	*6053	±	191	*2305	±	82
15	Floor in front of R benchtop	*7416	±	228	*226	±	20
18	Left side of left benchtop	**28,817	±	447	*366	±	16
19	Right side of left benchtop	**20,632	±	373	*332	±	17
21	Left side of floor in front left benchtop	**24,746	±	411	*349	±	16
22	Center of floor in front left benchtop	***118,170	±	922	*1596	±	31
23	Right side of floor in front left benchtop	*8027	±	236	*106	±	10
			±	78	0	±	0

Criteria for SWAB Results

Category	Tritium (dpm/m2)	C14 (dpm/m2)	Recommendations
A	< 500	< 50	No action
B*	500 – 10,000	50–10,000	Needs cleaning before natural tracer work. No health hazard. Does not apply to Radiation Vans.
C**	10,000 – 100,000	10,000 – 50,000	Must be cleaned before any use. Includes Radiations Vans.
D***	>100,000	>50,000	May be a health hazard. Notify local Radiation Safety Official.

Swipe test on R/V Kexue Yihao, China

R/V Kexue Yihao（1980 – 2016.05）
Chinese Academy of Sciences



Whole batch of Seawater samples (>200) were contaminated and had to be discarded.

Waste of time (>1 month working onboard) and money!

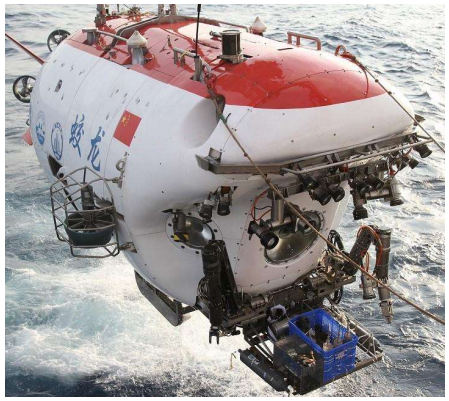
Seawater samples collected during Nov-Dec, 2012

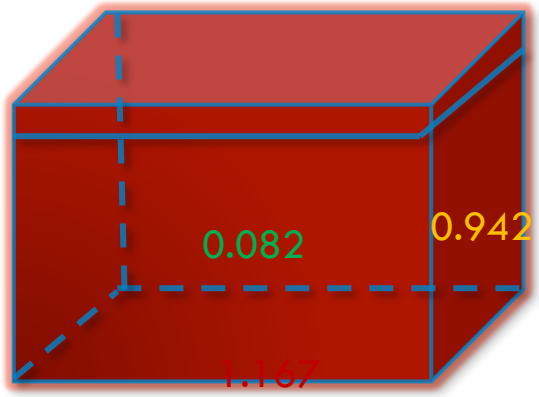
Lab No#	Field No#	Depth (m)	Fm	±
QAS1345	WP165-P11-1-C	2975	1.775	0.008
QAS1346			1.787	0.008
QAS1347			1.795	0.008
QAS1351	WP137-P14-1-F	2000	1.059	0.007
QAS1352			1.052	0.005
QAS1353			1.051	0.015

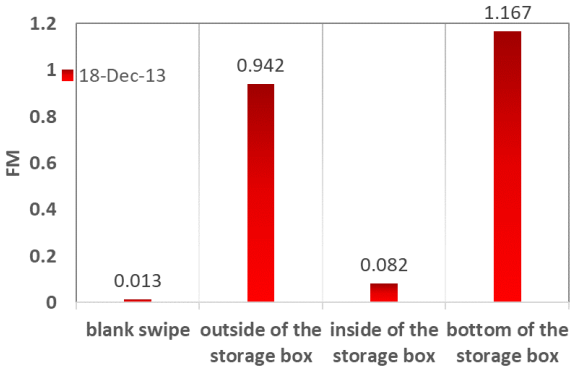
Deep water Fm value~0.78(~2000 yrs)

Swipe test on storage box from Jiaolong, China

Storage box carrying pore water and seawater samples from **Jiaolong** and **R/V Xiangyanghong 09**

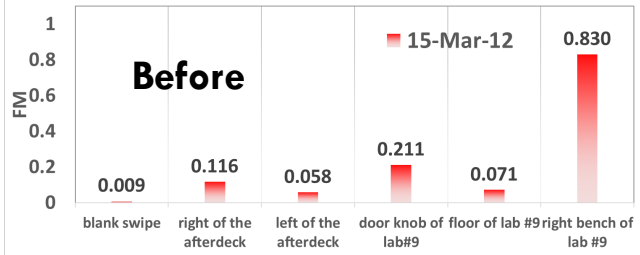


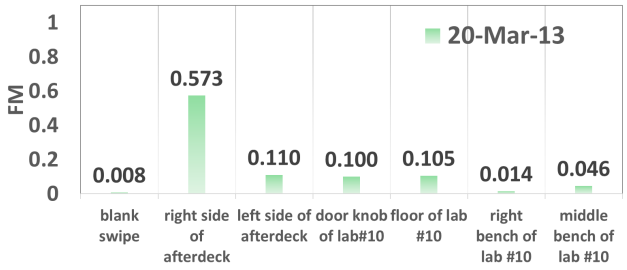


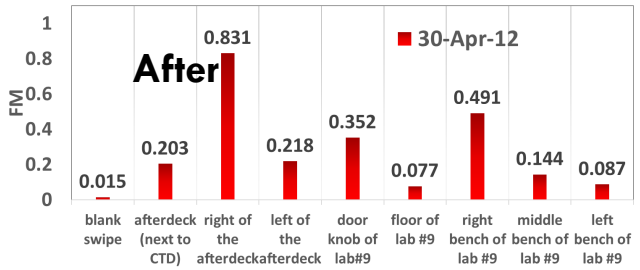


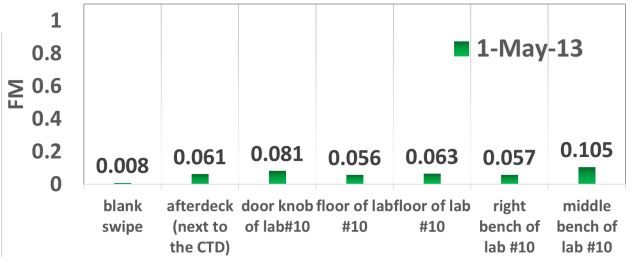
Lab No#	Field No#	Depth (m)	Fm	±
QAS2090	CTD01-2-1	3365	2.936	0.010
QAS2091	CTD01-2-2	3315	3.669	0.012
QAS2092	CTD01-2-3	3215	4.252	0.015
QAS2108	CTD01-2-9	800	3.372	0.011
QAS2109	CTD01-2-10	500	3.309	0.011
QAS2110	CTD02-1	2785	4.602	0.020
QAS2111	CTD02-2	2735	4.156	0.013
QAS2112	CTD02-3	2585	3.835	0.012
QAS2113	CTD02-4	2535	3.337	0.011
QAS2114	CTD02-5	2435	2.892	0.011

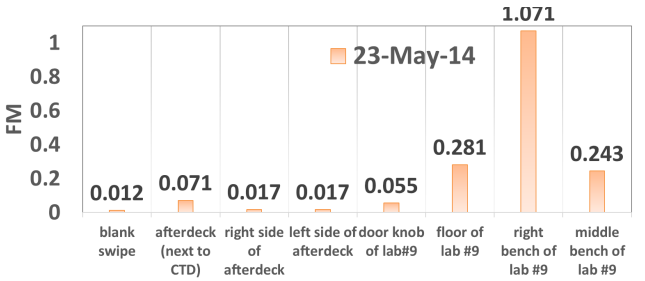
Swipe test on R/V Dongfanghong 2, China

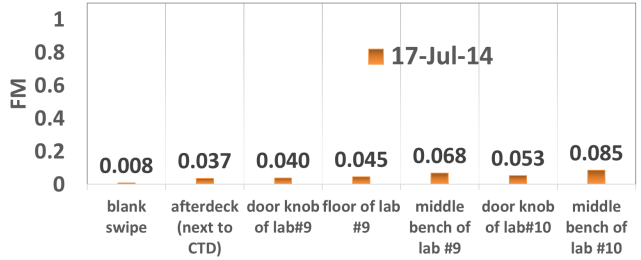




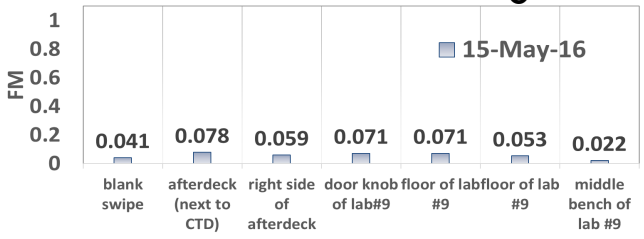


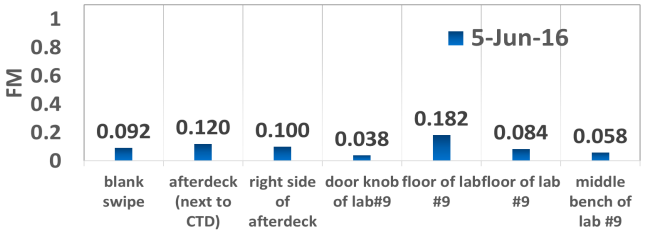






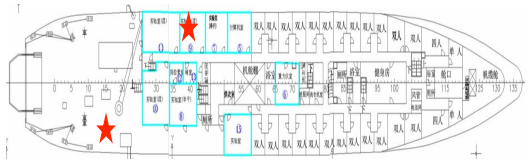
After lab redecorating





R/V Dongfanghong 2 (1996 – Present)

Ocean University Of China



Contamination may be avoided if precautions are taken

Useful Tips to Avoid ^{14}C Contamination

- **Inform** your personnel of the importance of the ^{14}C tracer contamination precautions. **Be emphatic.**
- **Cover** heavily used **areas with heavy duty aluminum foil**. If contamination or suspicion of contamination occurs, the foil can be easily replaced.
- **Avoid direct contact** of samples, tools, glassware, chemicals etc. Use trays on top of benches and hood areas. Place them in plastic trays. They are cheap and can be replaced any time.
- Designate separate sets of tools to be used in each lab or area, and avoid sharing them.
- If you can, try to keep equipment and chemicals inside your lab area and space, rather than moving them between labs. You can buy chemicals in small quantities, or split them from a large volume to be used in separate labs.
- Borrowed or shared equipment should also be used wisely. Designate a separate container to be the communication between your lab space and the equipment in question. Ascertain information about the equipment history before using it.
- Maybe, adopt the use of gloves. Replace them when moving from one procedure to another, or between lab spaces.
- Campus cleaners can carry contamination from one building to another through the cleaning tools (baskets, brooms, etc). Do the lab cleaning yourself.
- Last, but not less important. **Always use common sense.**

Summary

- Knowing the history of the field sites, labs and equipment prior sampling
- **Swipe to be sure.** Swipe protocol: https://www.ess.uci.edu/group/ams/protocols
- Be careful with common places contaminated with ^{14}C tracer
  - Biology & medical labs
  - Agricultural schools
  - Any labs that study photosynthesis
  - Labs that use **stable isotope tracers**
- Avoid sharing - Establish routine procedures to avoid contaminating
- Special precautions on ships
- If contamination is present, take immediate to clean it. Re-swiping after cleaning

Always use common sense!

You don't want to be on our Blacklist!

You can't be too careful!

This is appropriate…

Thank you for your attention!