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Cleaning of SNOGLOBE

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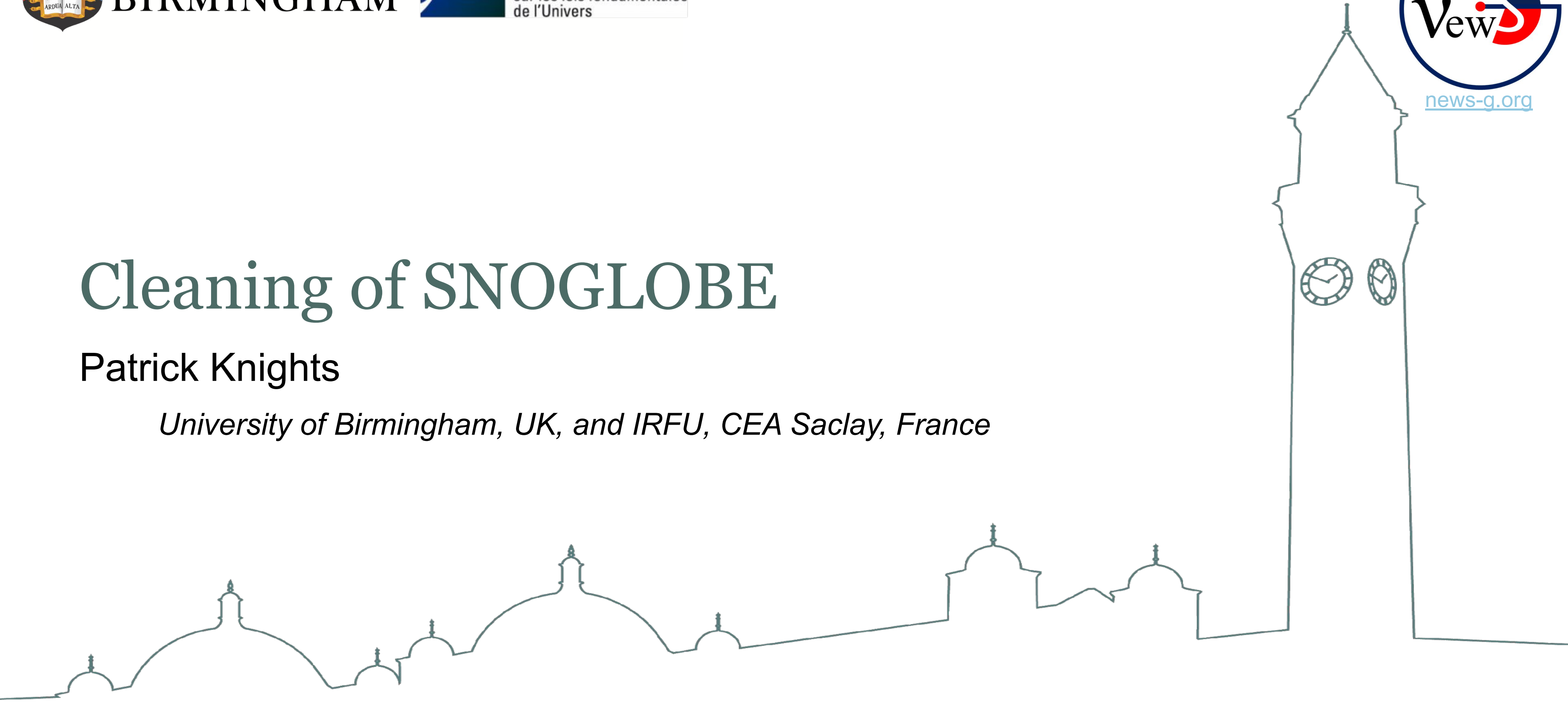
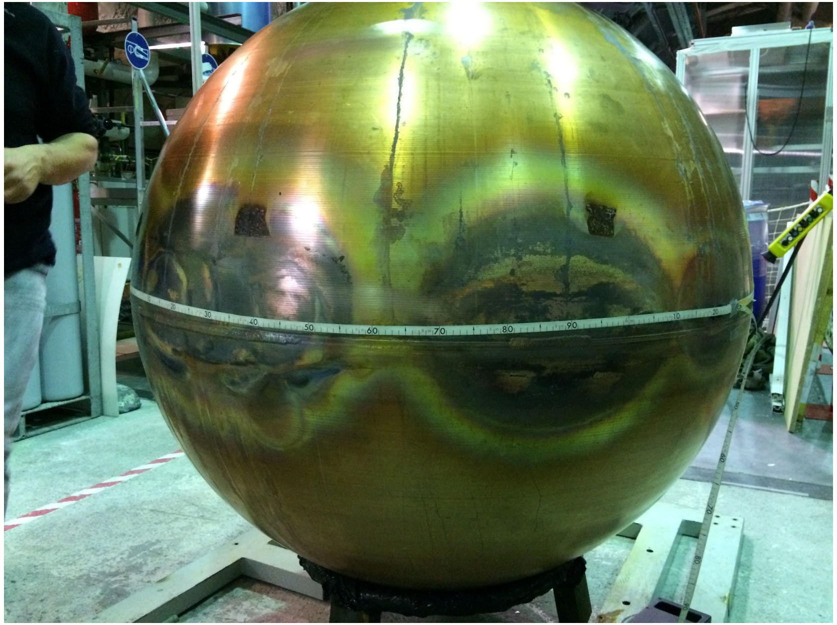


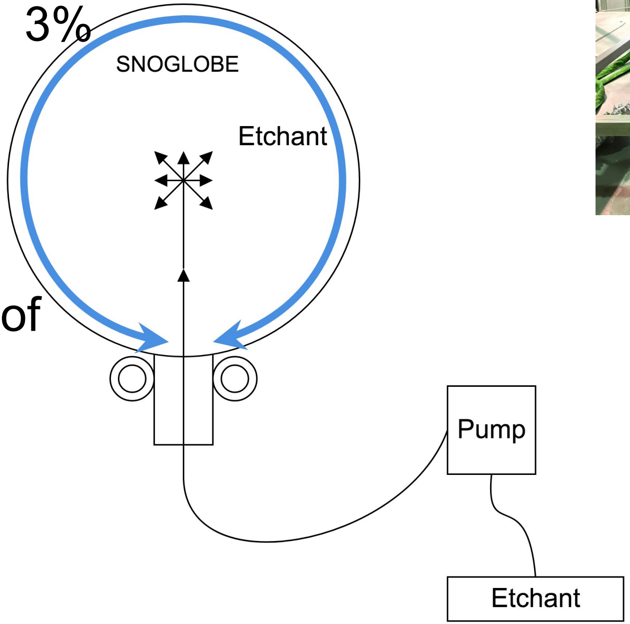
Internal Cleaning After Welding

- During welding and pressure testing, inner surface was exposed to air and water
- Cannot be certain how the sphere was handled by company
- Decided on internal cleaning effort to supress surface contamination
  - ☐ Etching and passivation



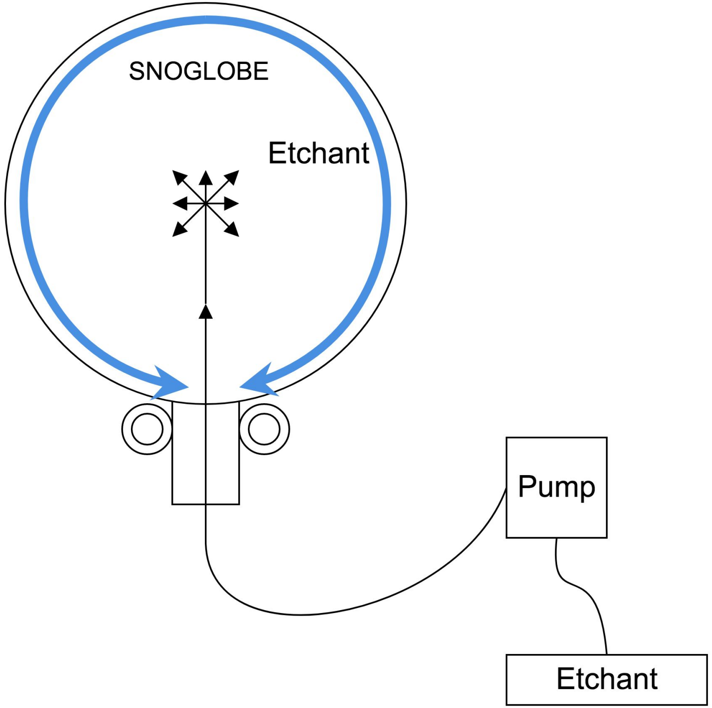
Etching of Internal Surface

- Cleaned by chemical etching using 3% H_2O_2, 1 % H_2SO_4 in water
- Samples of etchant collected during operation
  - ☐ Spectrophotometer used to measure copper concentration of etchant
  - ☐ ~2 µm of copper was removed
  - ☐ Etching took ~5.5 minutes



Internal Cleaning After Welding

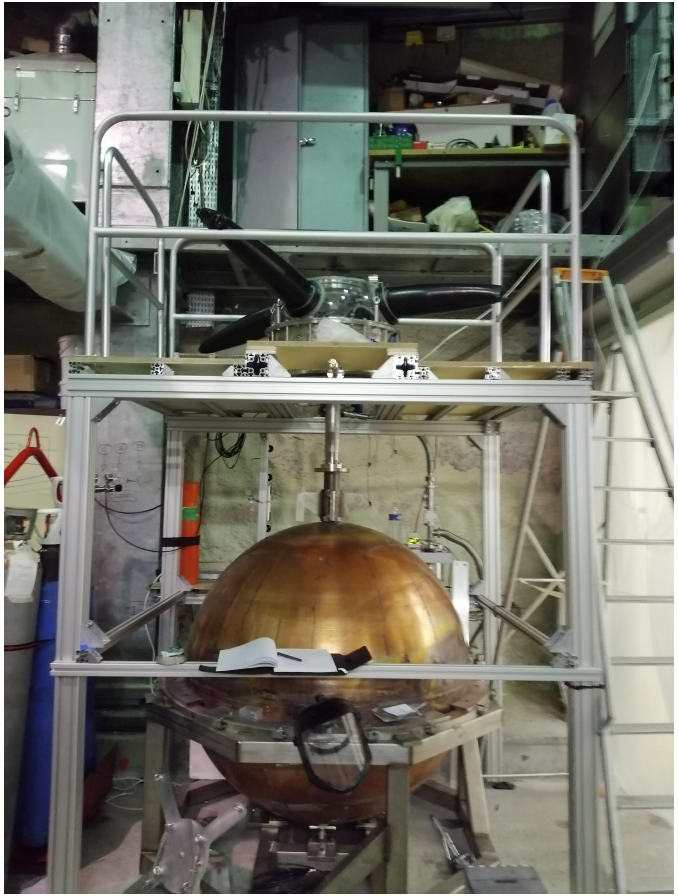
- After rinsing, surface was passivated using 1% citric acid solution
- Volume was flushed with nitrogen
  - Sphere was sealed and vacuum pumped after operation to minimise further contamination
- Sphere has remained sealed since
  - Either in vacuum, nitrogen overpressure or filled with operating gas



Current State of External Surface

- External surface needs to be cleaned in LSM for two main reasons
  - ☐ Contribution to background
  - ☐ Risk of contamination of the inner surface of the shielding

- External surface has **several points for concern**
  - ☐ Copper deposited on the outside
  - ☐ Rough parts of copper, trapping dirt
  - ☐ Burnt stickers/tape
  - ☐ Tape and adhesive from tape



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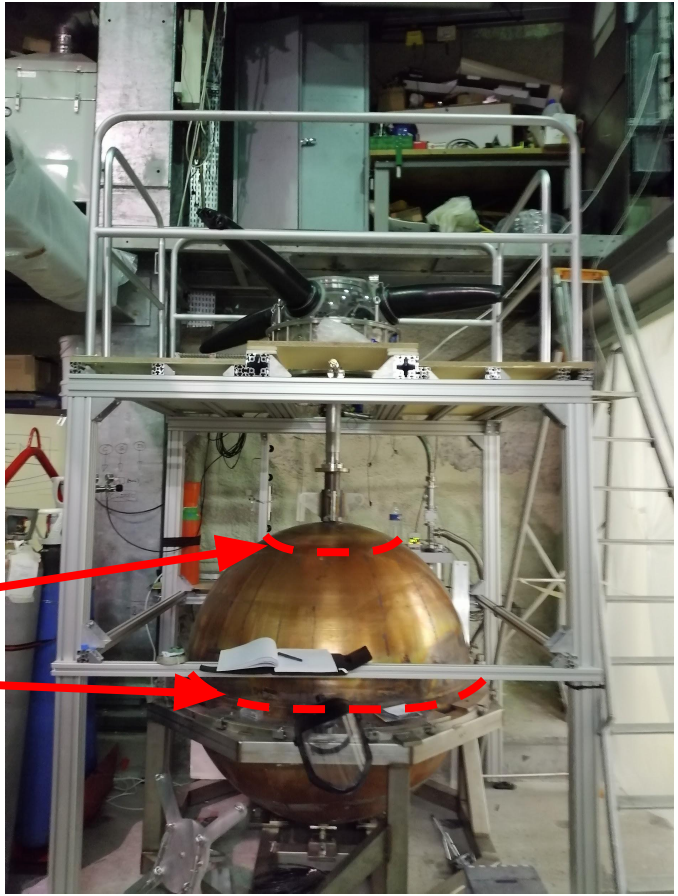

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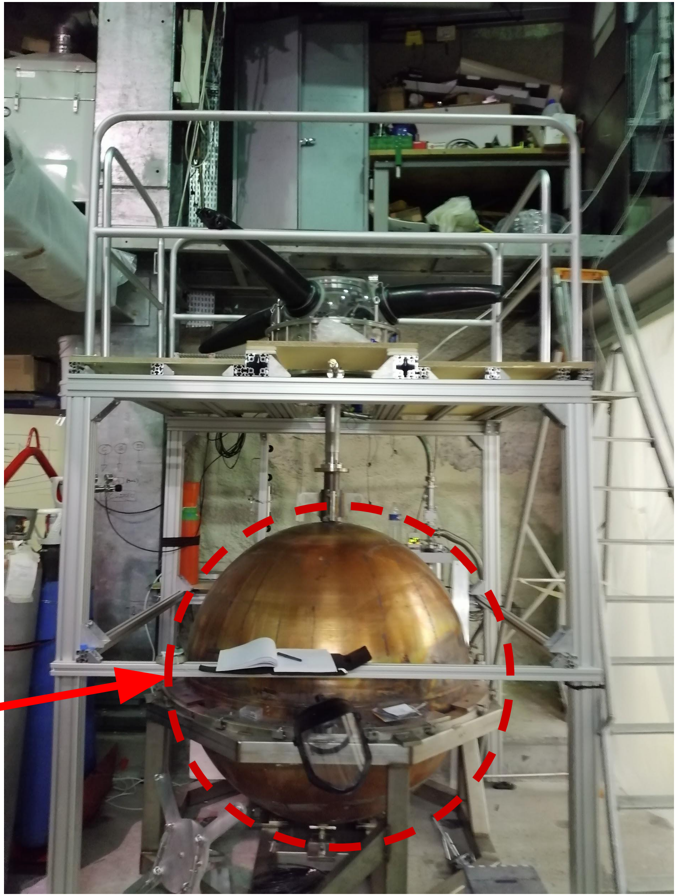
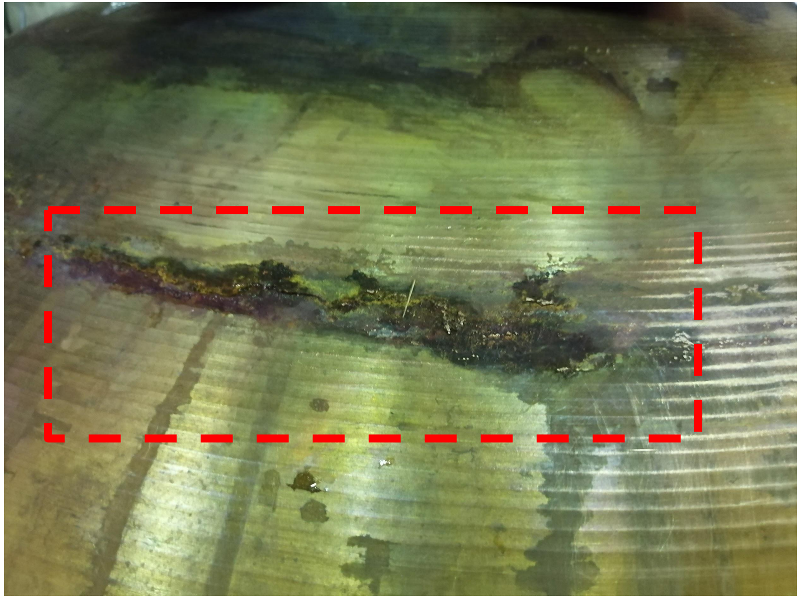
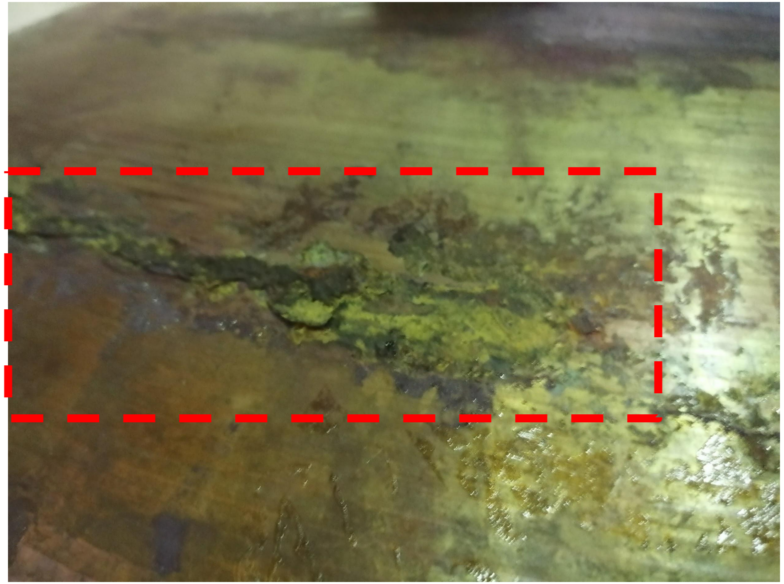


Extra Copper Deposited on Outside



- Deposited due to leak of electrolyte during cleaning
- Extra copper is not a problem, but the trapped dirt is



- **Solution**: Sand away enough copper to allow surface to be cleaned

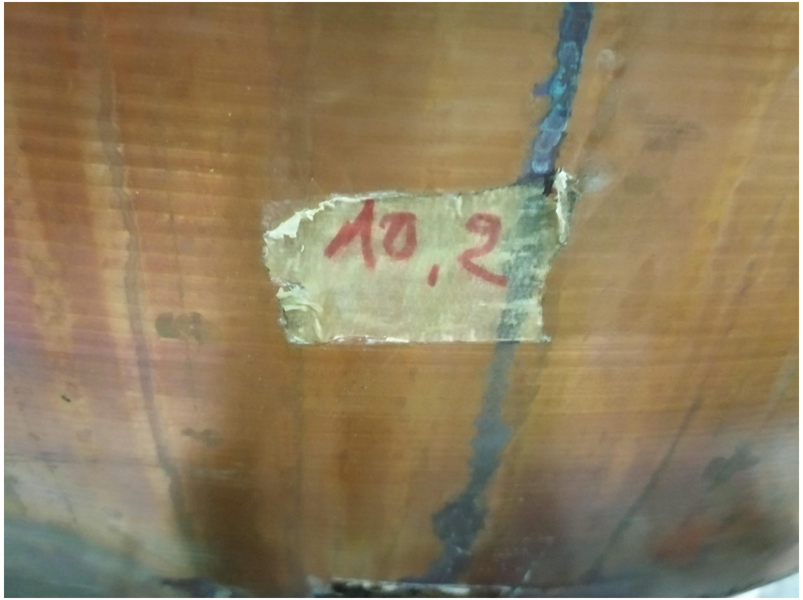
Rough Copper Surface at Equator

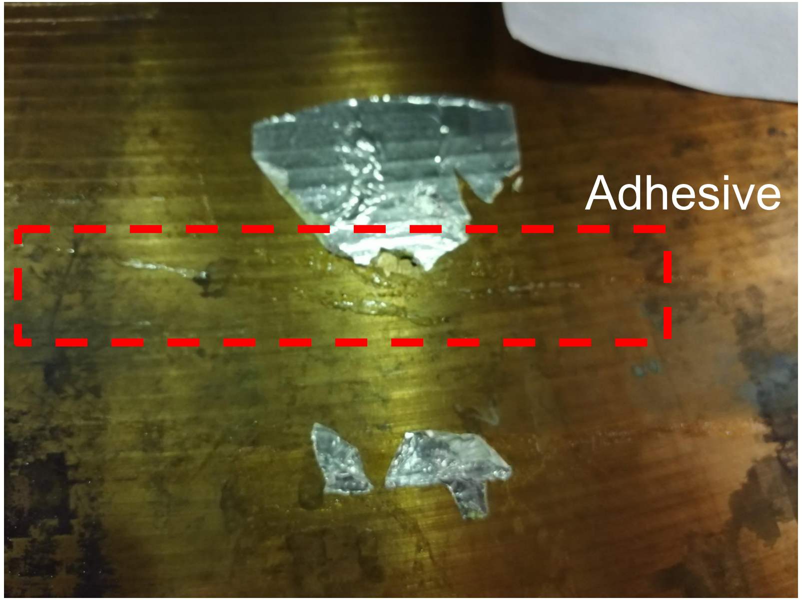
- Due to electrolyte leak, copper removed in ‘groves’ at equator
  - ☐ Only a problem if it is trapping dirt
- **Solution**: Clean well with hard-bristled brush and Micro90 (detergent). Ensure etchant gets into groves. Perhaps sand particularly rough parts

Burned Stickers/Tape

- Looks like tape was burned to the outside of the sphere
  - Probably during welding or in baking
- **Solution**: Sanding. I wasn't able to remove it with alcohol or acetone

Tape and Adhesive from Tape

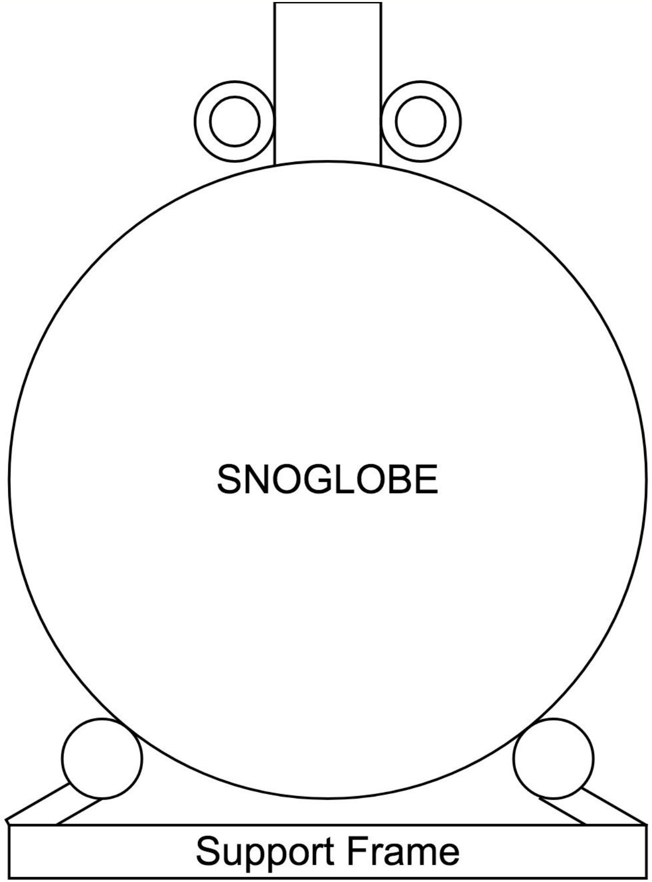




- Tape that could not be removed by hand. Also left some adhesive
- Tried to clean with alcohol and acetone, but not successful
- **Solution**: With hard scrubbing they will come off, or Eric Hoppe recommended MEK (methyl ethyl ketone) or MIBK (methyl isobutyl ketone) solvents

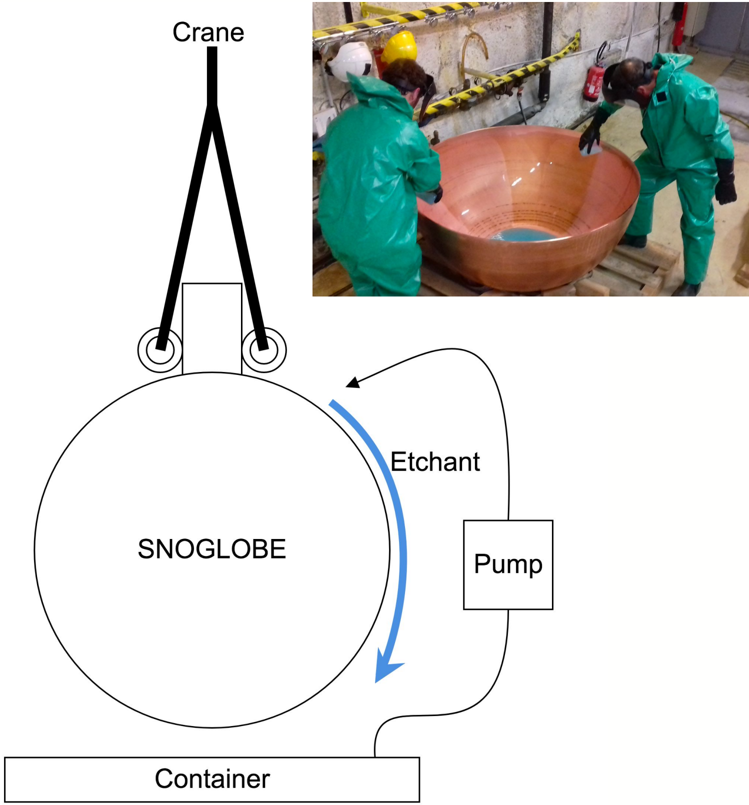
Cleaning Procedure - Sanding

- Sphere will be moved to entrance of lab
  - Too much dust produced to do sanding in sphere's current location
- Sanding should be completed before other cleaning
  - Should be done with fine-grit silicon carbide paper where possible
- Should also clean the tape/adhesive residue at this stage with solvent or scrubbing

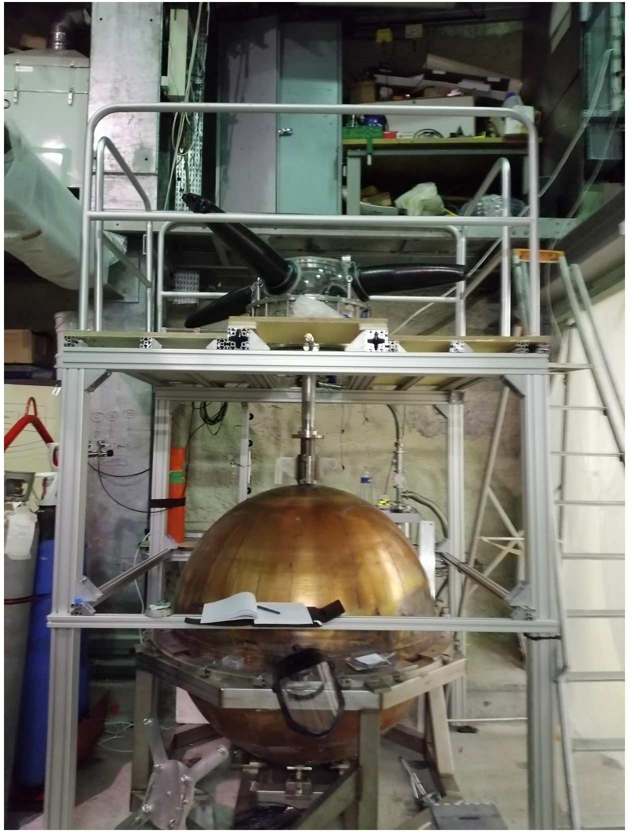


Cleaning Procedure - Detergent and Etching

- Surface should then be washed with Micro90 detergent (1%) in water (balance)
  - Use hard bristle brush to remove all trapped dirt/oil
- Rinse
- Chemically etch surface with 3% H_20_2, 1 % H_2SO_4 in deionised/demineralised water
  - Pump will be used to circulate the etchant
- Should be rinsed afterwards
- Passivate surface with 1% Citric acid solution



Summary



- Internal surface was chemically etched and passivated following the welding
  - Not exposed to air since (and won't be ever again!)
- External surface require some attention
  - Several points of concern for cleanliness
- Operation to perform the external cleaning will take place next week
  - All required materials are avaliable in LSM
- Full procedures are available on the TWiki: https://www.snolab.ca/news-projects/private/TWiki/bin/view/Main/DetectorSurface

Additional Material

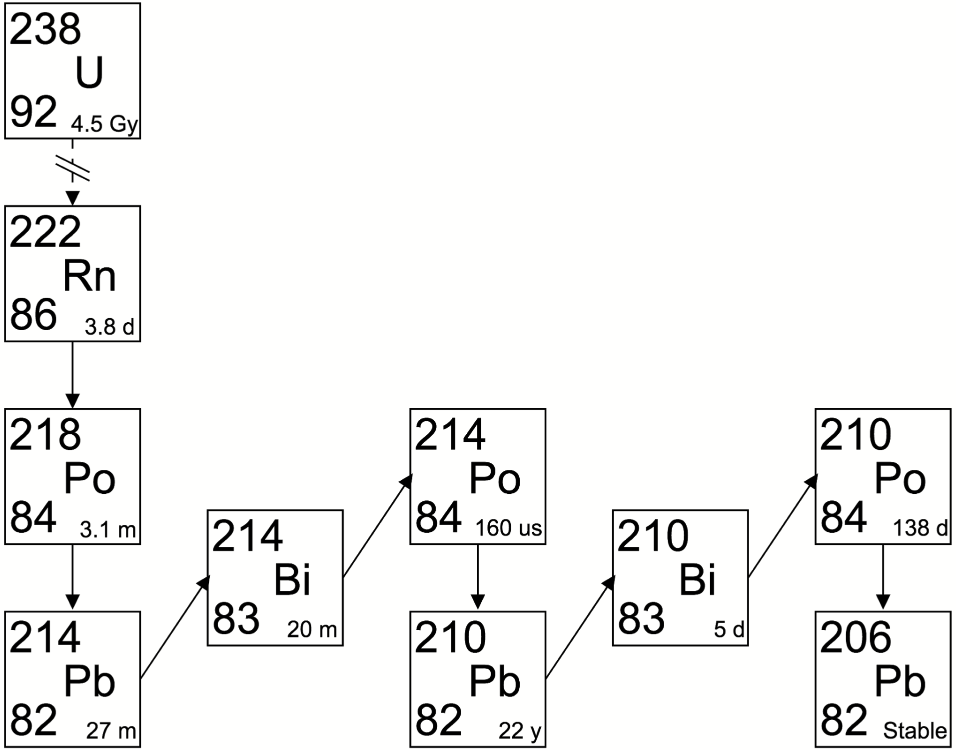
Copper as a Construction Material

- **Copper** is common material of choice for such experiments:
  - ☐ Strong enough to build limited-pressure vessels or support structures
  - ☐ Commercially available at high purity
  - ☐ Low cost
  - ☐ **No long-lived radio-isotopes**
    - Longest is ^{67}Cu, $t_{1/2}$= 62 hours
  - ☐ Possibility to **electrochemically purify**

SEDINE,
DM Detector
(NEWS-G)

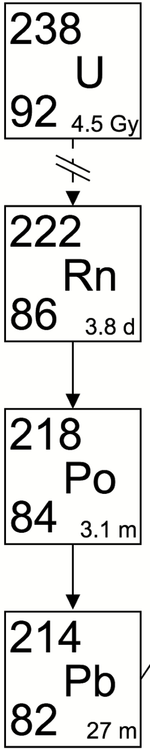
https://news-g.org/

Background Contributions in Copper

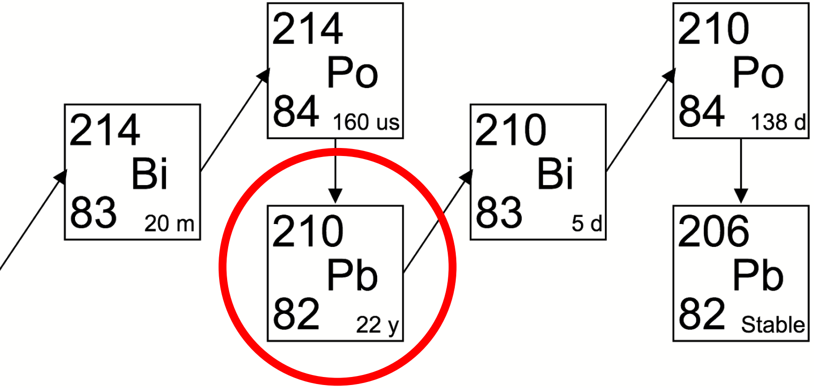


- $^{63}Cu(n,\alpha)^{60}Co$ by fast neutrons from cosmic muon spallation
- ^{238}U and ^{232}Th decay chain – traces naturally found and deposited by ^{222}Rn
- ^{238}U and ^{232}Th contamination:
  - Commercial copper ~1-10 µBq/kg

^{210}Pb Measurements



XIA UltraLo-1800

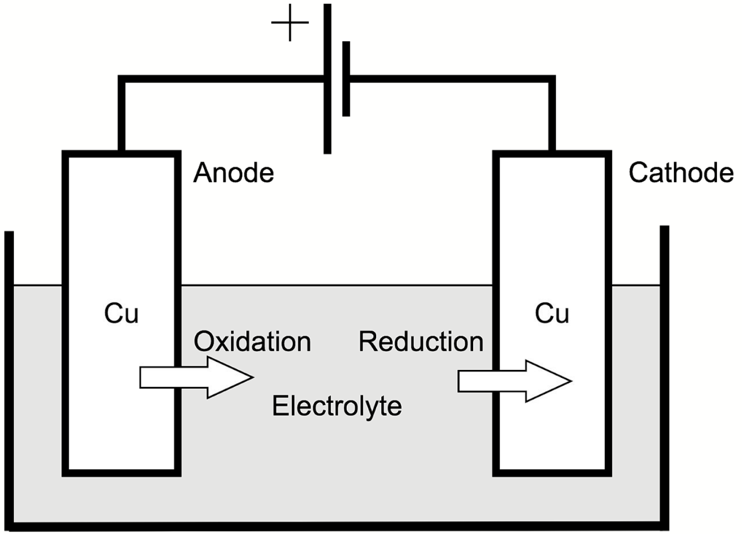


- ^{238}U content **measured directly** by mass spectroscopy
  - **Used to infer daughter isotopes**' quantities in material
- But **^{222}Rn** deposits daughter nuclei on surfaces during production, mixing into bulk copper
  - ^{210}Pb has a 22-year half-life – **amount may be larger than that inferred from ^{238}U**
- Recent method to measure ^{210}Pb from ^{210}Po α-decays

See XMASS collaboration: doi.org/10.1063/1.5018989
https://www.xia.com/ultralo-theory.html

Electrolytic Cell and Electroplating

- **Electrolysis** governed by **oxidation** and **reduction** reactions
- Reduction of ions requires supply of electrons → **Current**
- Also requires energy → **potential difference**
- **Ions reduced at cathode**: material build-up
  - ☐ Supplied current drives reaction
  - ☐ Deposited mass proportional to current:

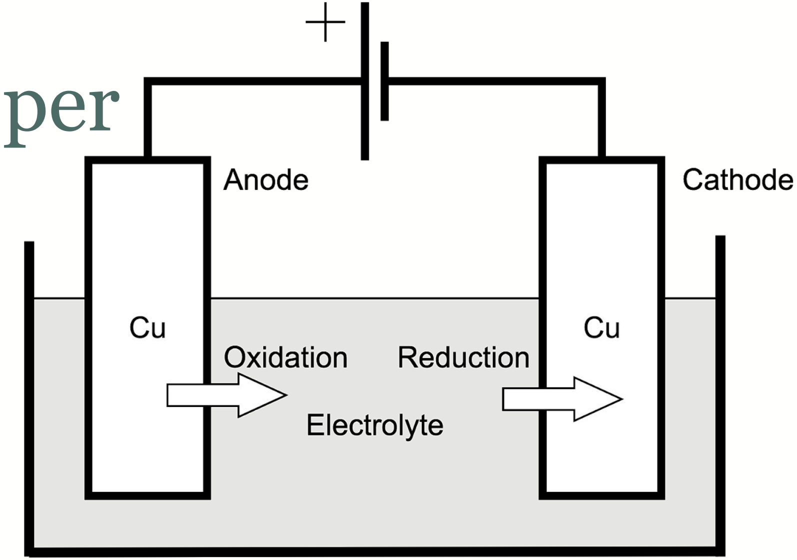


$$M = \frac{m_r \int I(t)\mathrm{d}t}{zF}$$

M - mass deposited
m_r - molar mass
$I(t)$ - current
z - number of electrons transferred
F - Faraday Constant$(= eN_A)$

Electroplating Ultra-Pure Copper

- Some ions reduce more readily than others → **reduction potentials**
- Copper benefits from **‘electrowinning’** - high reduction potential +0.34 V
- Reduction potential of:
  - ☐ Uranium (U^{3+}): -1.80 V
  - ☐ Thorium (Th^{4+}): -1.90 V
  - ☐ Lead (Pb^{2+}): -0.13 V
- Lower than copper → **copper refined during electroplating** if electrode potential is low enough



Example: Electroforming at PNNL

https://www.pnnl.gov/science/highlights/highlight.asp?id=1434

Why don't impurities plate too?

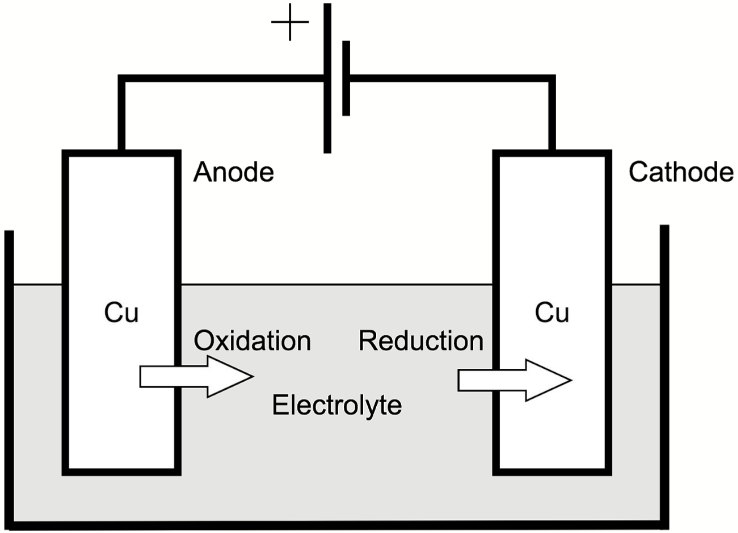
- Reaction that proceeds determined by standard cell potential:

$$E^0_{cell} = E^0_C - E^0_A$$

- Related to change in Gibbs Free Energy:

$$\Delta G^0 = -zFE^0_{cell}$$

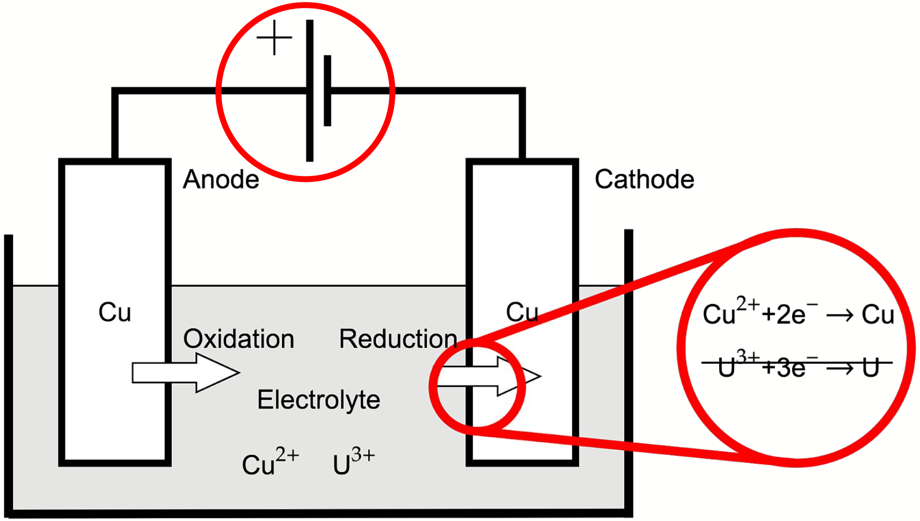
- If $\boldsymbol{\Delta G^0 < 0}$, then reaction is spontaneous
- If $\boldsymbol{\Delta G^0 > 0}$, then extra energy is needed



E^0_{cell} - standard cell potential
E^0_C - standard reduction potential at cathode
E^0_A - standard reduction potential at anode

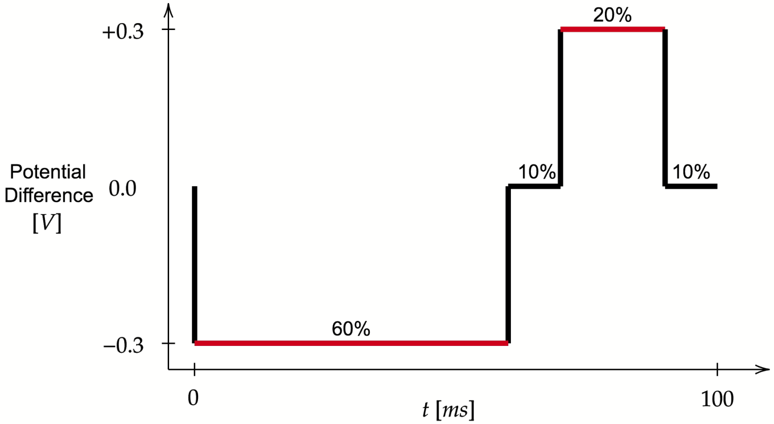
An Example: Uranium Contamination

- Example of **electrolyte containing U^{3+} and Cu^{2+} ions**, with a Cu anode:
- To reduce U^{3+} to U
  - E^0_{cell} = -2.14 V → Requires energy
- To reduce Cu^{2+} to Cu
  - E^0_{cell} = 0 V → In **equilibrium**
- Cu^{2+} reduction is energetically favourable to U^{3+} reduction
- Potential difference required to drive reaction and overcome energy losses



Pulse Plating for better characteristics

- Voltage application: DC or pulsed
- ‘**Pulse plating**’ or ‘pulse-reverse plating’ uses waveform for electroplating
  - Relaxation period and sometimes with a ‘polishing’ section
- Demonstrated benefits over DC plating:
  - **Raised areas reduced** by reverse bias section of pulse
  - **Greater uniformity of deposit** – relaxation period allows diffusion of ions
  - **Higher density** deposit



Hoppe, E.W. et al. J Radioanal Nucl Chem (2008) 277: 103. https://doi.org/10.1007/s10967-008-0716-5
M.S. Chandrasekar, Malathy Pushpavanam, Electrochim. Acta, 53, 8, 2008, pp 3313-3322, https://doi.org/10.1016/j.electacta.2007.11.054.

Radiopurity Results for Electroformed Copper

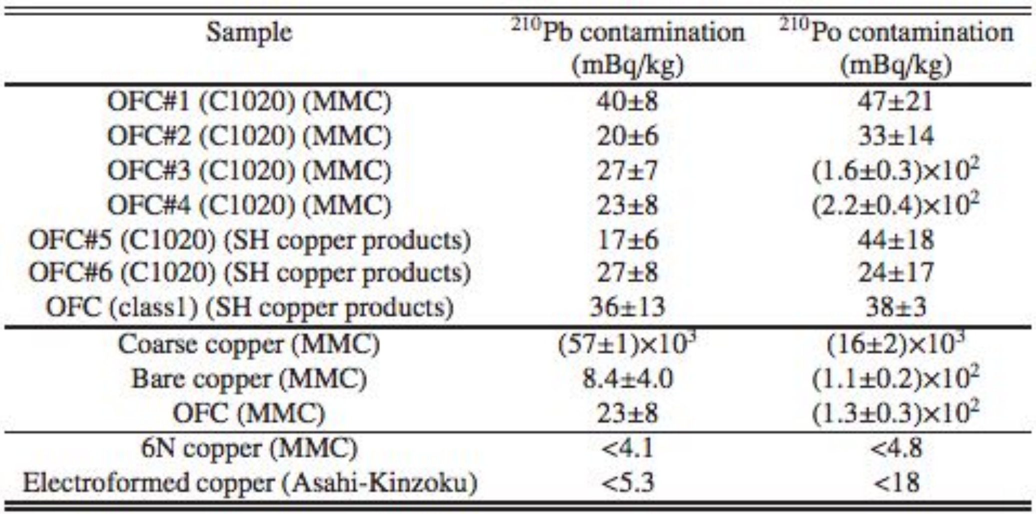
Copper Type	^{232}Th [ppt] (*μBq/kg*)	^{238}U [ppt] (*μBq/kg*)	Source
C10100	0.46±0.06 (*1.19*±0.25)	0.21±0.06 (*2.54*±0.74)	Majorana Demonstrator, (PNNL) 10.1016/j.nima.2016.04.070
Electroformed	<0.029 (*<0.11*)	<0.008 (*<0.10*)	Majorana Demonstrator, (PNNL) 10.1016/j.nima.2016.04.070
Electroformed	0.035±0.004 (*0.14*)	<0.050 (*<0.06*)	CES, LSC 10.1063/1.5018987

- Current ^{238}U and ^{232}Th contaminations below sensitivity of most sensitive assay technique - ICP-MS
  - Bounds are just upper limit – value may be much lower

^{210}Pb in Electroformed Copper

- Electroformed copper shows factor ~5 or more reduction in ^{210}Pb compared to Oxygen free copper (OFC)
  - Inferred from measurements of ^{210}Po α-particles
- Measurements of electroformed copper were limited by background

See XMASS collaboration: doi.org/10.1063/1.5018989



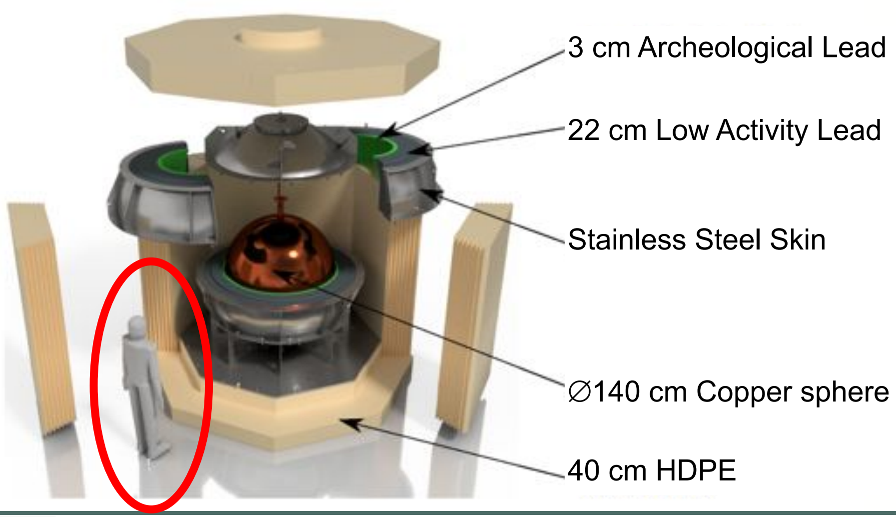
Sample	^{210}Pb contamination (mBq/kg)	^{210}Po contamination (mBq/kg)
OFC#1 (C1020) (MMC)	40±8	47±21
OFC#2 (C1020) (MMC)	20±6	33±14
OFC#3 (C1020) (MMC)	27±7	$(1.6\pm0.3)\times10^2$
OFC#4 (C1020) (MMC)	23±8	$(2.2\pm0.4)\times10^2$
OFC#5 (C1020) (SH copper products)	17±6	44±18
OFC#6 (C1020) (SH copper products)	27±8	24±17
OFC (class1) (SH copper products)	36±13	38±3
Coarse copper (MMC)	$(57\pm1)\times10^3$	$(16\pm2)\times10^3$
Bare copper (MMC)	8.4±4.0	$(1.1\pm0.2)\times10^2$
OFC (MMC)	23±8	$(1.3\pm0.3)\times10^2$
6N copper (MMC)	<4.1	<4.8
Electroformed copper (Asahi-Kinzoku)	<5.3	<18

XIA UltraLo-1800

https://www.xia.com/ultralo-theory.html

NEWS-G - Next Generation

- Next NEWS-G detector is a ∅140 cm Cu sphere
  - ☐ Aurubis C10100 4N (99.99% pure) copper
- To be installed in LSM, Spring/Summer 2019 for first tests
- Shipped to SNOLAB, Autumn 2019



https://news-g.org/

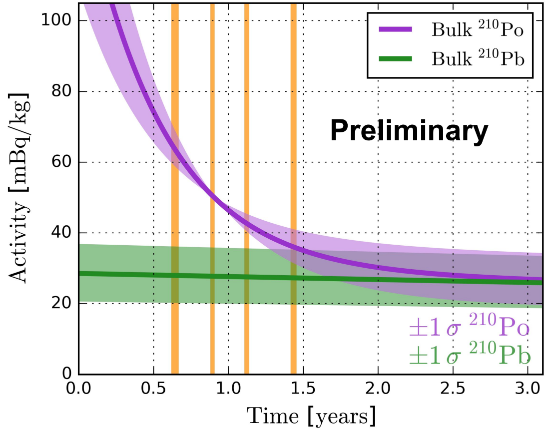
Test of hemisphere spinning

^{210}Pb in NEWS-G Copper

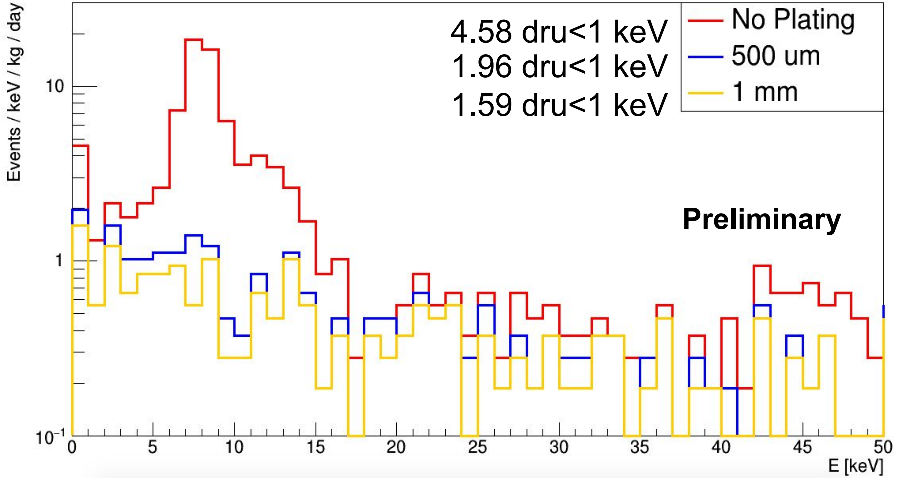
- Collaborative agreement between XMASS and NEWS-G **to perform four measurements of ^{210}Po**, every three months
- Preliminary analysis indicates ^{210}Pb at 28.5±8 mBq/kg
  - ☐ Similar to other samples
- Geant4 simulation indicated that this gives **4.58 dru*<1 keV**
- Reduced to 1.96 dru if 0.5 mm of pure copper is electroplated onto the inner surface
  - ☐ Other sources ~0.6 dru

*1 dru = 1 count/keV/kg/day

Best Estimate of ^{210}Po & ^{210}Pb from two measurements of ^{210}Po



10^7 decays of ^{210}Pb and ^{210}Bi in Cu Walls in 2 bar Ne+10% CH_4



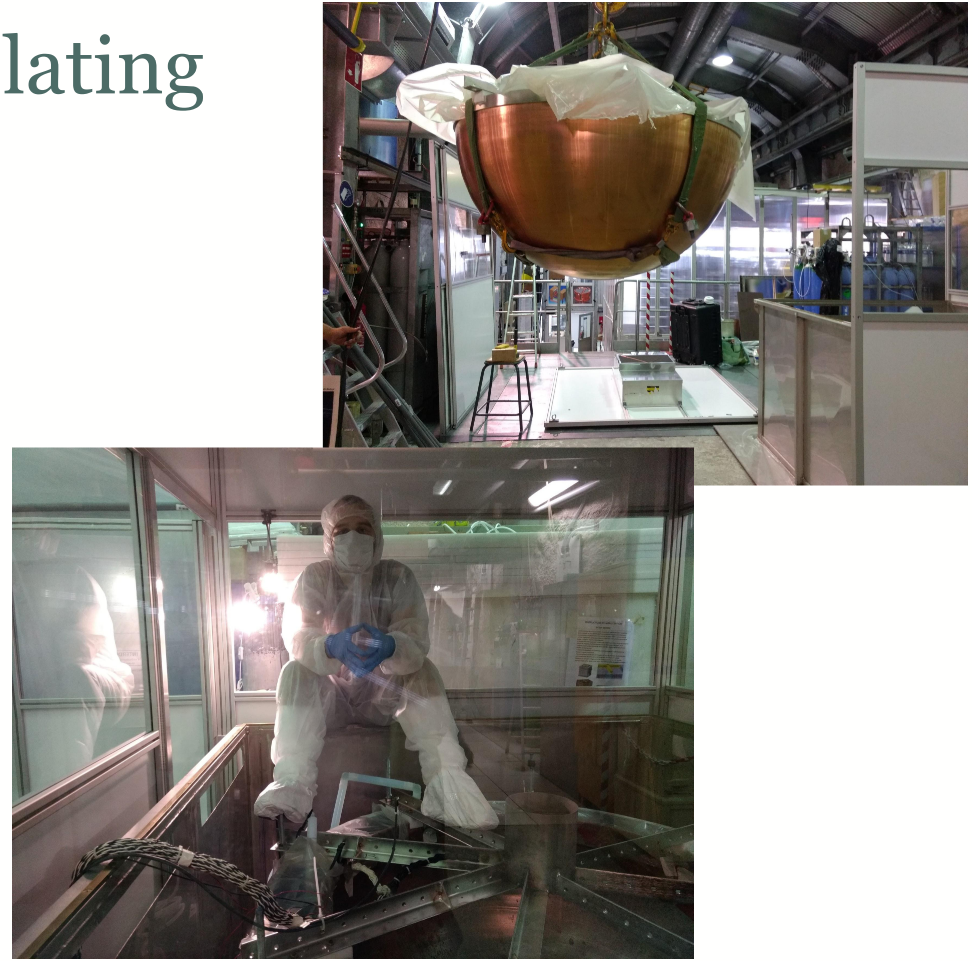
Preparation of Surface

- Operation performed in **LSM**
- Sanded to remove rough parts
- Cleaned surface with **Micro90 detergent**
- Chemically etched using 3% H_2O_2, 2% H_2SO_4 in deionised water
    - Shown to be effective etchant while less aggressive than alternatives such as nitric acid
- Same treatments for **copper anode**

More on surface preparation: https://doi.org/10.1016/j.nima.2007.04.101

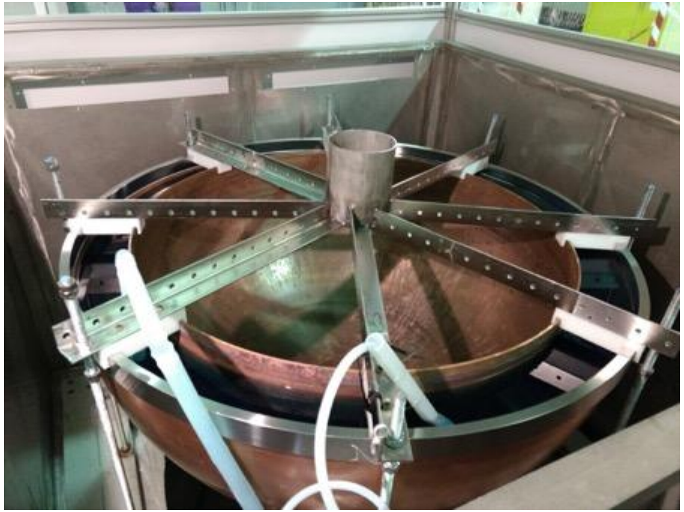
Installation for Electroplating

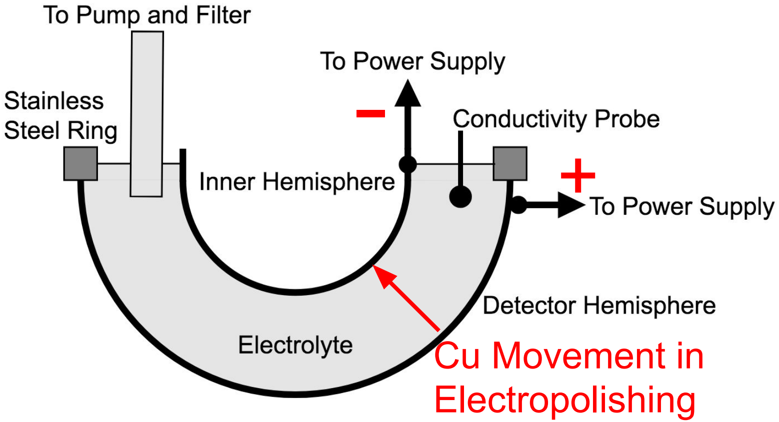
- Hemisphere moved to enclosed **clean area** where electroplating was carried out
- Electrolyte of H_2SO_4, H_20 and $CuSO_4$
- Pump and 1 μm particulate filter installed
  - Provides mechanical mixing and filtration
- Anode installed into hemisphere, separated by 12 cm



Electropolishing

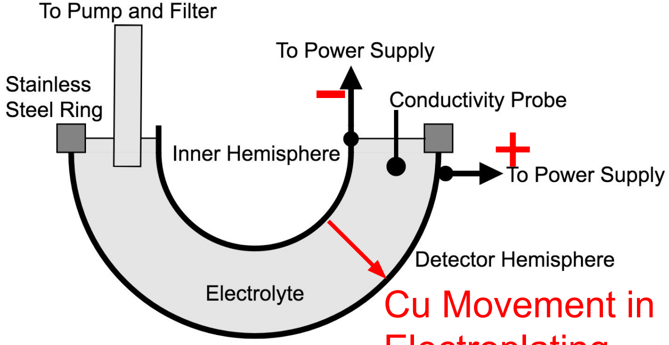
- Electropolishing serves several purposes
  - Removes layer without chemical or mechanical attack
  - Preferentially removes raised areas on surface
  - **Increases concentration of $CuSO_4$** in electrolyte
- **Pulse plating** used
- Estimated first (second) hemisphere 21 µm (28 µm) polished, based on current measurements



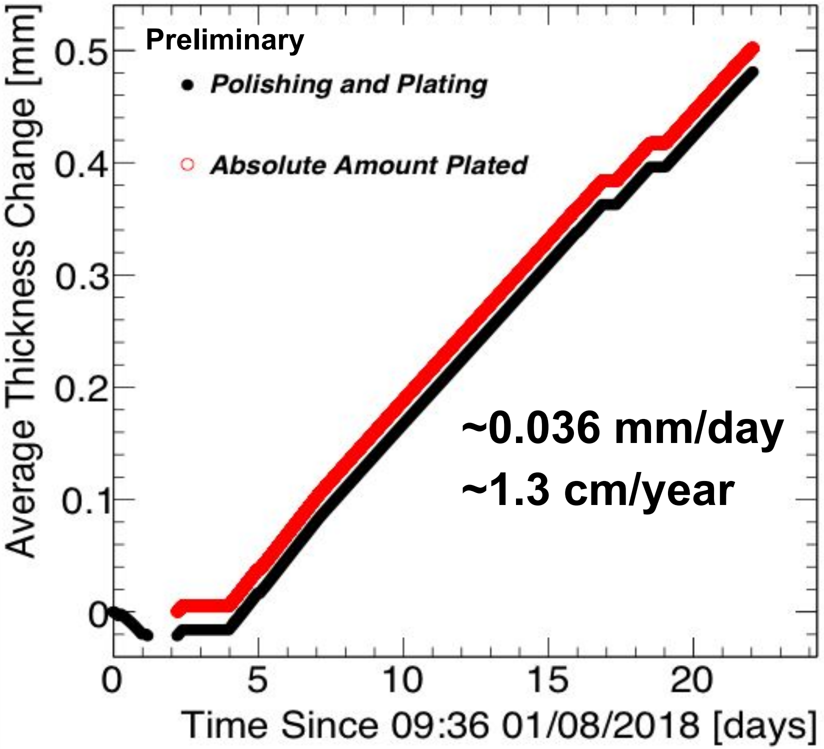


Electroplating

- Electrode potential difference of **~0.3 V used**
  - Established value for copper
- Plating continued for ~15 days
- In total estimate first (second) hemisphere had **502 μm (540 μm)** plated, based on current measurements

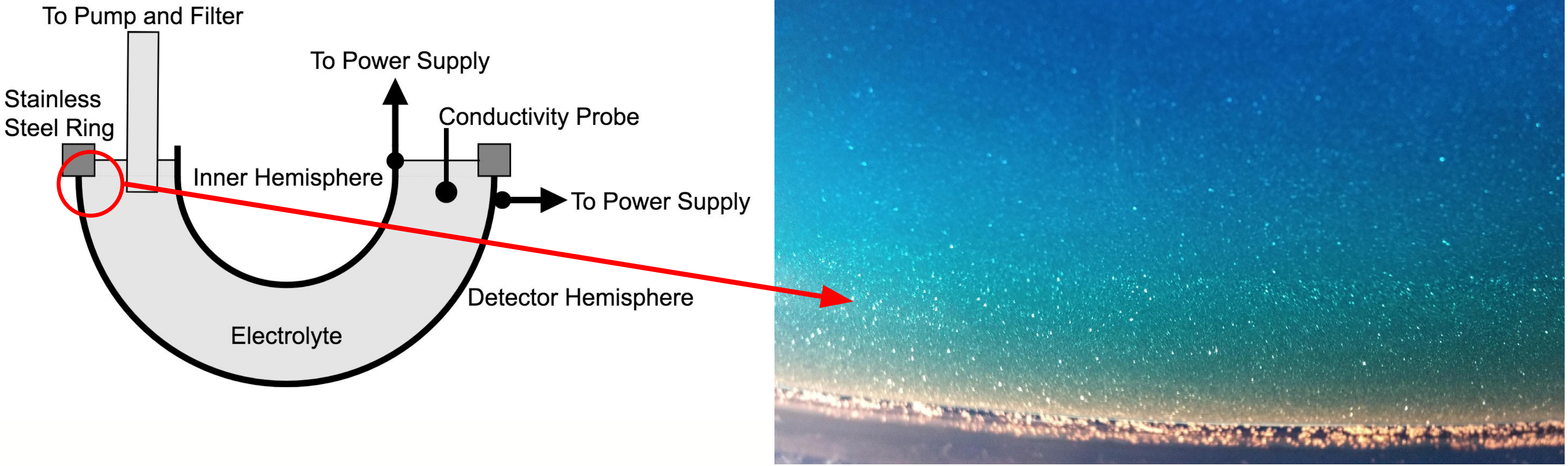


Cu Movement in Electroplating



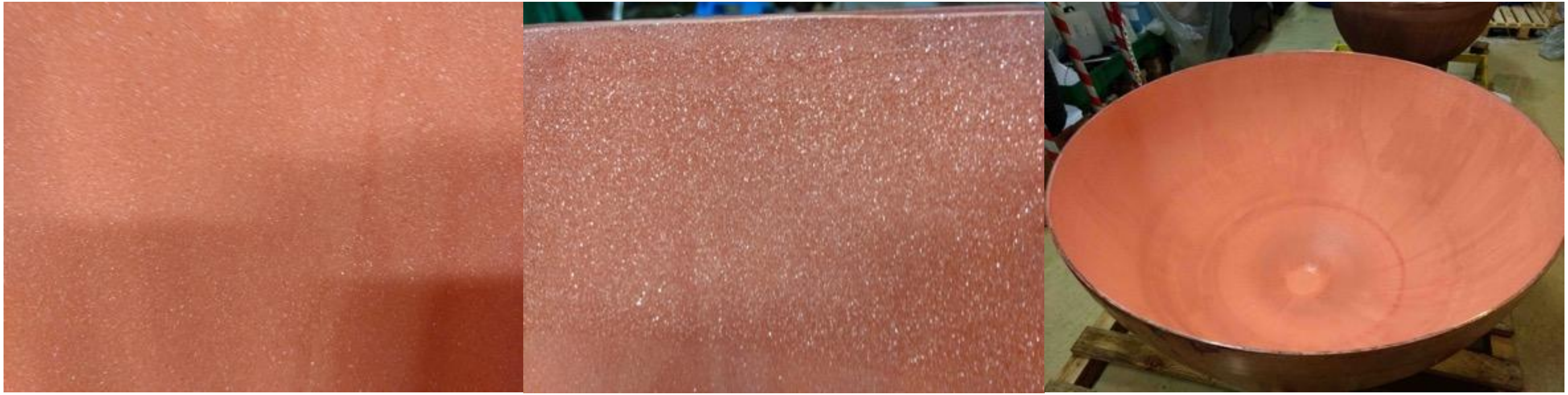
~0.036 mm/day
~1.3 cm/year

Electroplating - Results



- Copper depositing on hemisphere surface

Electroplating Results



- Layer of Cu deposited on surface
  - ☐ Awaiting results of analysis to verify purity
- Rinsed and passivated with citric acid before hemispheres welded together
- Final chemical etch performed on intact sphere
- Detector being installed in LSM for first tests and commissioning

Cosmogenic Activation of Copper

- Produced by ^{63}Cu(n,α)^{60}Co with fast neutrons from cosmic muon spallation
- ^{60}Co $t_{1/2}$ =5.3 years
- Saturation activity of 60Co 340^{+82}_{-68} μBq/kg*

*Baudis, L., Kish, A., Piastra, F. et al.
Eur. Phys. J. C (2015) 75: 485.
https://doi.org/10.1140/epjc/s10052-015-3711-3

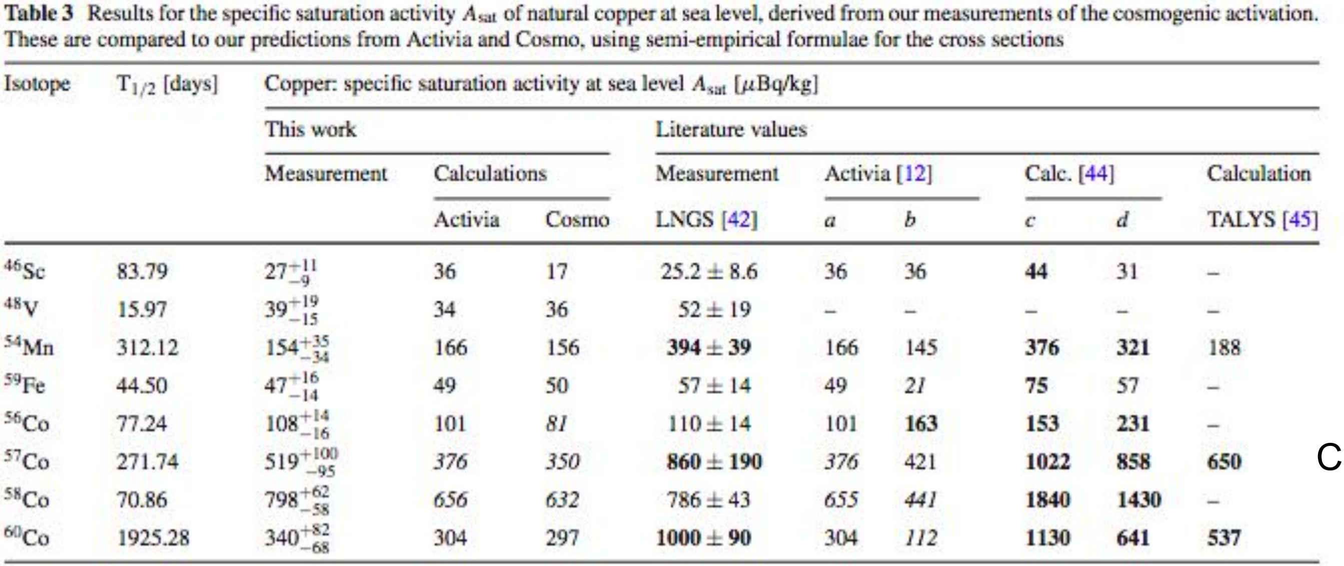


Table 3 Results for the specific saturation activity A_{sat} of natural copper at sea level, derived from our measurements of the cosmogenic activation. These are compared to our predictions from Activia and Cosmo, using semi-empirical formulae for the cross sections

Isotope	$T_{1/2}$ [days]	Copper: specific saturation activity at sea level A_{sat} [μBq/kg]								
		This work			Literature values					
		Measurement	Calculations		Measurement	Activia [12]		Calc. [44]		Calculation
			Activia	Cosmo	LNGS [42]	*a*	*b*	*c*	*d*	TALYS [45]
^{46}Sc	83.79	27^{+11}_{-9}	36	17	25.2 ± 8.6	36	36	**44**	31	–
^{48}V	15.97	39^{+19}_{-15}	34	36	52 ± 19	–	–	–	–	–
^{54}Mn	312.12	154^{+35}_{-34}	166	156	**394 ± 39**	166	145	**376**	**321**	188
^{59}Fe	44.50	47^{+16}_{-14}	49	50	57 ± 14	49	*21*	**75**	57	–
^{56}Co	77.24	108^{+14}_{-16}	101	*81*	110 ± 14	101	**163**	**153**	**231**	–
^{57}Co	271.74	519^{+100}_{-95}	*376*	*350*	**860 ± 190**	*376*	421	**1022**	**858**	**650**
^{58}Co	70.86	798^{+62}_{-58}	*656*	*632*	786 ± 43	*655*	*441*	**1840**	**1430**	–
^{60}Co	1925.28	340^{+82}_{-68}	304	297	**1000 ± 90**	304	*112*	**1130**	**641**	**537**

Co^{2+} Reduction Potential=-0.28 V

Standard Conditions

- Temperature of 298.15 K (25.0°C)
- Each gaseous reagent has a partial pressure of 1 atm (rarely 100 kPa)
- Effective concentration of 1 mol/L for each aqueous species