

Lead materials and chemical reagents – What's their lead-210 background?

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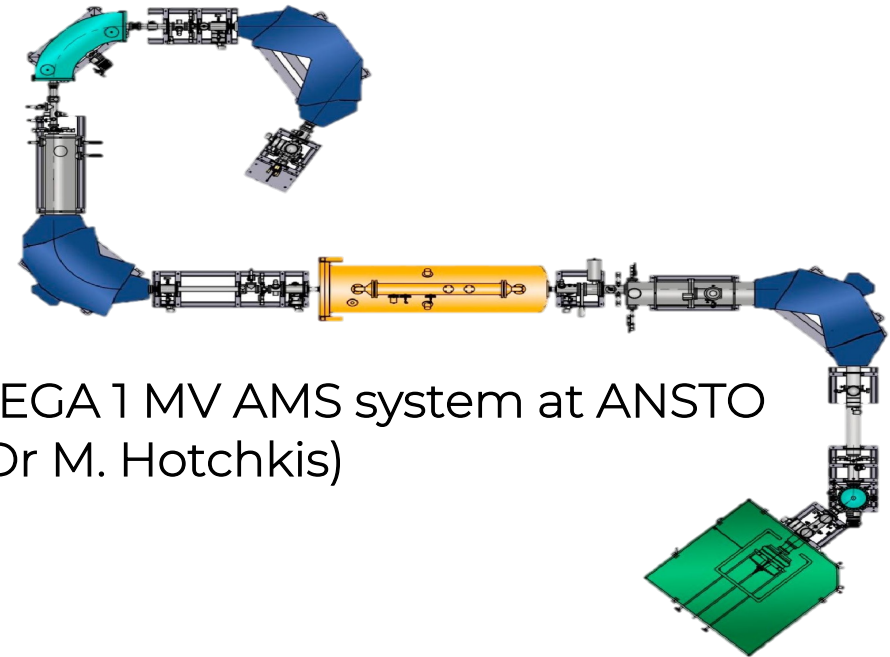
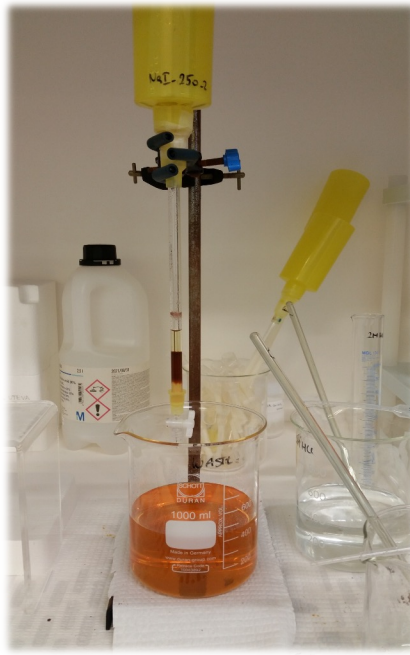
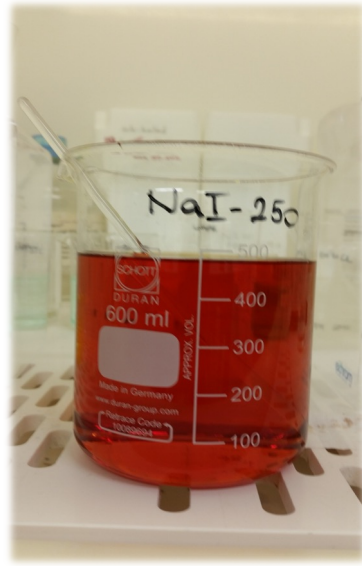


What do we want to do, why & how?

Goal: Characterise and quantify radioimpurities in detector material (NaI(Tl)) - ^{40}K , U & Th decay series, ^{210}Pb , ^{129}I , etc.

Why: Mimic signals in region of interest, limit sensitivity

How: Combination of analytical chemistry and Accelerator Mass Spectrometry.



VEGA 1 MV AMS system at ANSTO (Dr M. Hotchkis)

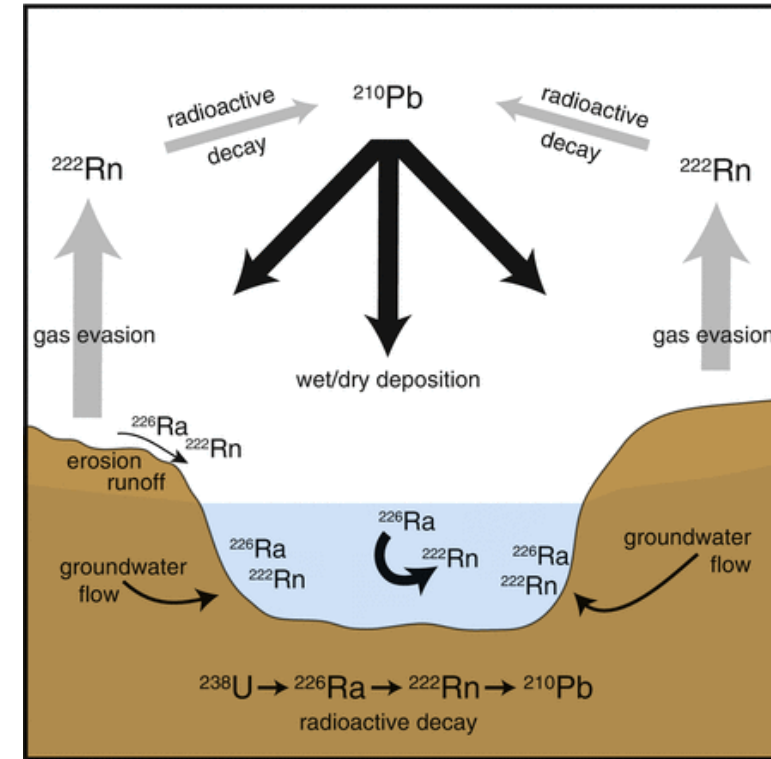
Challenges & Strengths

AMS: is a relative measurement (isotopic ratio, $^{210}\text{Pb}/^{208}\text{Pb}$), carrier.

Pb-210: decay product of U-238 \rightarrow omnipresent.

NaI: Expected $^{210}\text{Pb} < 3.0 \times 10^{-2}$ mBq/kg ($^{210}\text{Pb}/^{208}\text{Pb} = 1.33 \times 10^{-14}$) [1].

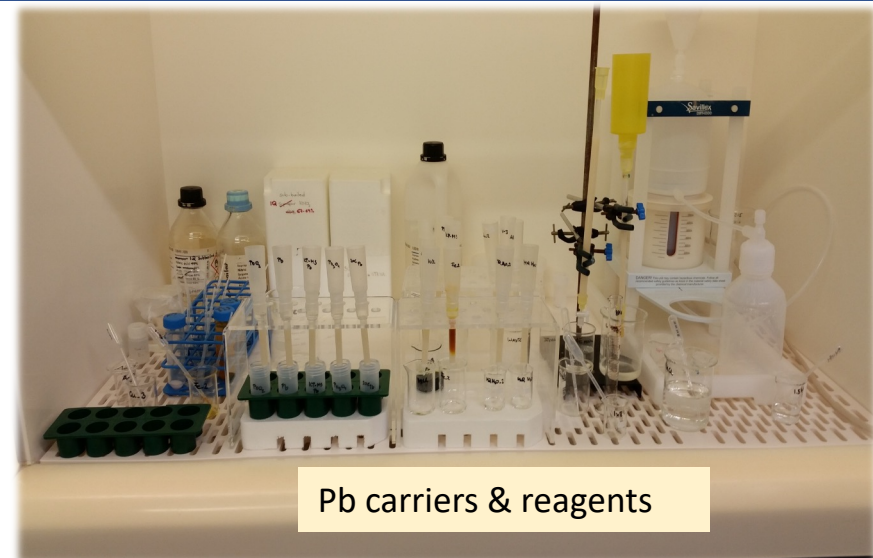
We need to: Screen potential Pb carriers and chemicals/reagents to be used.



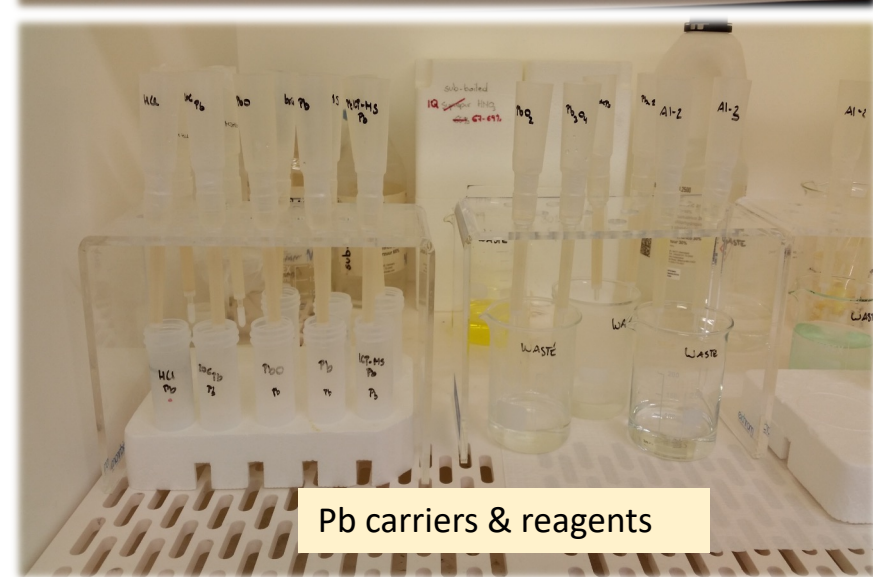
Currently, we are able to measure $^{210}\text{Pb}/^{208}\text{Pb}$ ratios down to 1×10^{-14} [2].

Pb carriers and chemical reagents

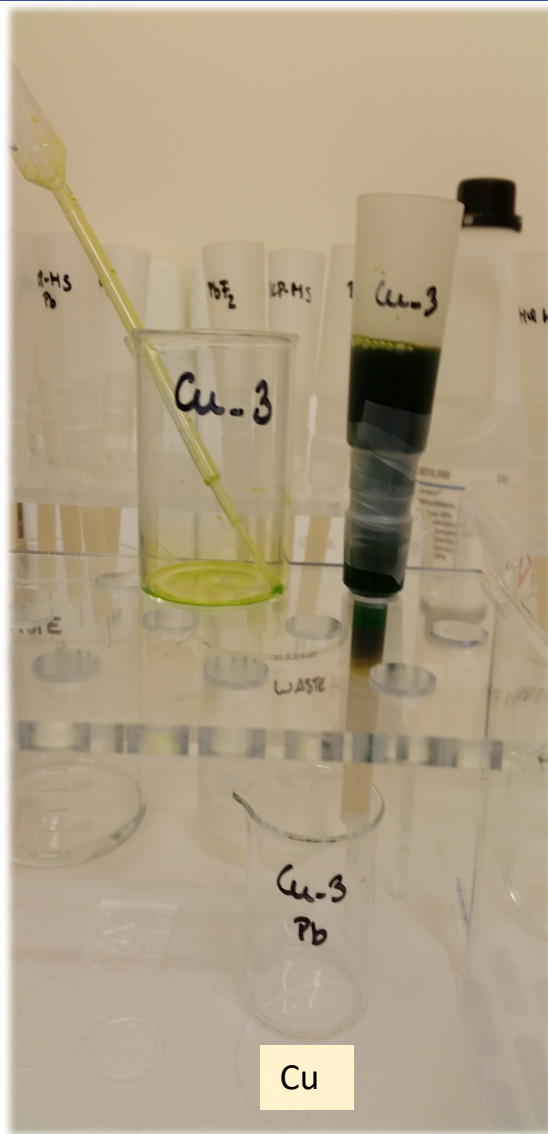
Pb compound	Pb compound	Chemical reagent	Reagent
Pb (metal powder)	Pb_20001 (roof)	AE resin (1-X8, 100-200 mesh, Cl ⁻ form)	Ag (Ag powder)
PbO	²⁰⁴ Pb & ²⁰⁶ Pb	HCl (Suprapur [®])	NaI-250
PbO ₂	ICP-MS Pb	Fe (Fe wire)	Chemical blank
Pb ₃ O ₄	brick	Al (Al cathodes)	Ag, AgF & AgF ₂ [*]
PbF ₂	LNGS material	Milli-Q water	SbF ₃ [*]
U. Chicago Spanish lead	Hampton Court Palace roof	Sub-boiled Milli-Q water	
PNNL ancient lead	LANL stock & Chicago stock	Cu (Cu cathodes)	



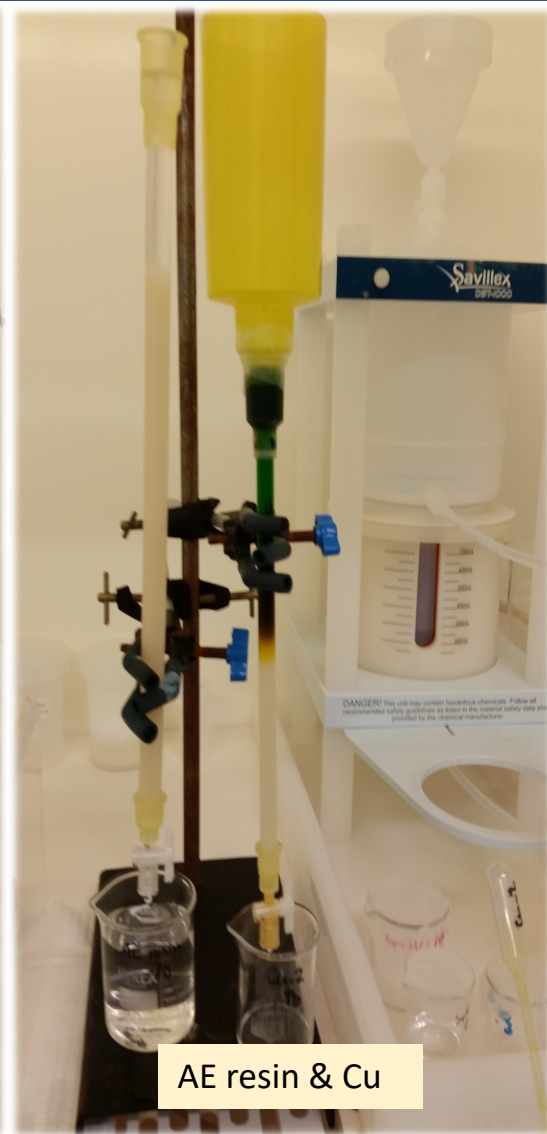
Pb carriers & reagents



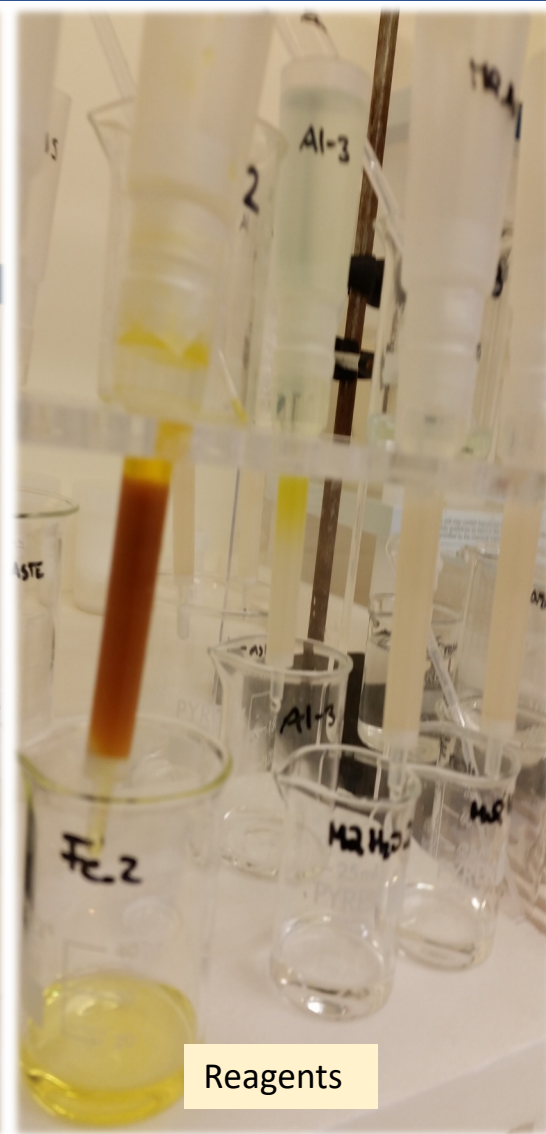
Pb carriers & reagents



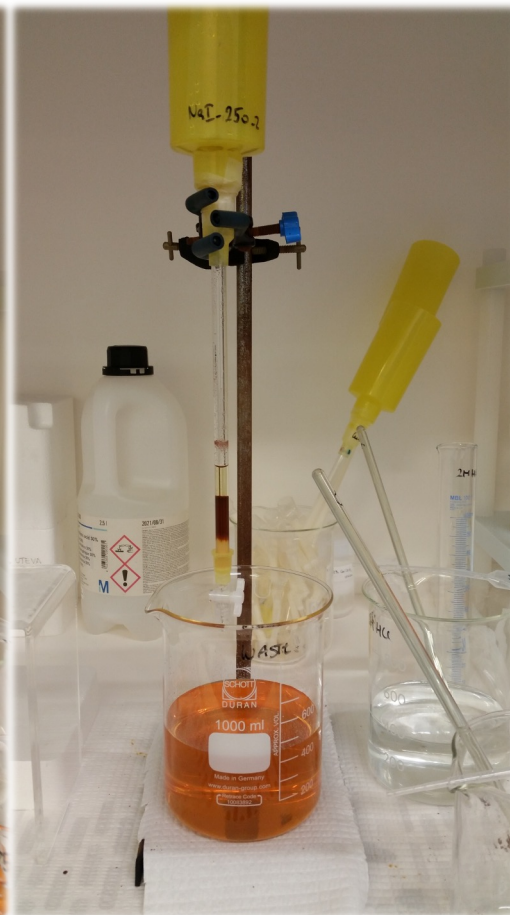
Cu



AE resin & Cu



Reagents



250 g NaI in 2M HCl

6M HCl \rightarrow Pb

Results – Pb carriers

Pb compound	^{210}Pb [cts]	$^{210}\text{Pb}/\text{Pb}$	Pb compound	^{210}Pb [cts]	$^{210}\text{Pb}/\text{Pb}$
Pb (metal powder)	0		ICP-MS Pb	$7^{+3.30}_{-2.75}$	9.6×10^{-14}
PbO	$1^{+1.75}_{-0.63}$	1.2×10^{-14}	brick	0	
PbO ₂	$1^{+1.75}_{-0.63}$	1.1×10^{-14}	LNGS	0	
Pb ₃ O ₄	$4^{+2.78}_{-1.66}$	4.8×10^{-14}	U. Chicago Spanish lead	$4^{+2.78}_{-1.66}$	9.1×10^{-15}
PbF ₂	$4^{+2.78}_{-1.66}$	3.9×10^{-14}	PNNL ancient lead	$3^{+2.30}_{-1.90}$	2.7×10^{-15}
Pb_20001 (roof)	$1^{+1.75}_{-0.63}$	7.3×10^{-15}	Hampton Court Palace roof	$4^{+2.78}_{-1.66}$	2.5×10^{-15}
^{204}Pb	0		LANL stock	$3^{+2.30}_{-1.90}$	1.7×10^{-15}
^{206}Pb	$1^{+1.75}_{-0.63}$	9.1×10^{-15}	U. Chicago stock	33 ± 6	2.3×10^{-14}

NaI: Expected $^{210}\text{Pb} < 3.0 \times 10^{-2}$ mBq/kg \rightarrow $^{210}\text{Pb}/^{208}\text{Pb} = 1.33 \times 10^{-14}$ [1]
 \rightarrow $^{210}\text{Pb}/\text{Pb} = 7.0 \times 10^{-15}$.

Results – chemical reagents

Chemical reagent	^{210}Pb [cts]	$^{210}\text{Pb}/\text{Pb}$	Chemical reagent	^{210}Pb [cts]	$^{210}\text{Pb}/\text{Pb}$
AE resin (1-X8, 100-200 mesh, Cl ⁻ form)	0		Sub-boiled Milli-Q water (1 L)	0	
HCl (Suprapur [®])	$2^{+2.25}_{-1.26}$	5.4×10^{-14}	Sub-boiled Milli-Q water (10 L)	$1^{+1.75}_{-0.63}$	1.7×10^{-13}
Fe (Fe wire)	$1^{+1.75}_{-0.63}$	7.7×10^{-14}	Ag (Goodfellow 99.99%)	$2^{+2.25}_{-1.26}$	1.3×10^{-14}
Al (Al cathodes)	$2^{+2.25}_{-1.26}$	1.1×10^{-12}	NaI-250	-	-
Cu (Cu cathodes)	-	-	Chemical Blank	$2^{+2.25}_{-1.26}$	2.6×10^{-13}
Milli-Q water (1 L)	0		PbF ₂ + Ag (1:10)	27±5	4.1×10^{-14}
Milli-Q water (5 L)	$5^{+2.81}_{-2.25}$	2.6×10^{-13}	PbF ₂ + AgF (1:10)	$15^{+4.32}_{-3.68}$	1.9×10^{-14}
Milli-Q water (10 L)	22±5	7.4×10^{-13}	PbF ₂ + AgF ₂ (1:10)	29±5	2.0×10^{-14}
Milli-Q water (20 L)	$6^{+3.28}_{-2.18}$	3.6×10^{-13}	PbF ₂ + SbF ₃ (1:10)	$4^{+2.78}_{-1.66}$	2.9×10^{-14}

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Milli-Q water: ~0.2 mBq/L

Sub-boiled water: ~0.09 mBq/L

Optimising the chemical procedure

Large mass of NaI is challenging for chemical extraction of Pb (85 % is I).

Tests using **Anion Exchange resin 1-X8**

(100-200 mesh, Cl⁻ form, 2 ml) and

Sr resin (100-150 μm, 2 ml).

Anion Exchange resin → poor retention,
i.e., pre-concentration (Fe/Pb co-ppt)

Sr resin → high retention for Pb but resin is bleeding.

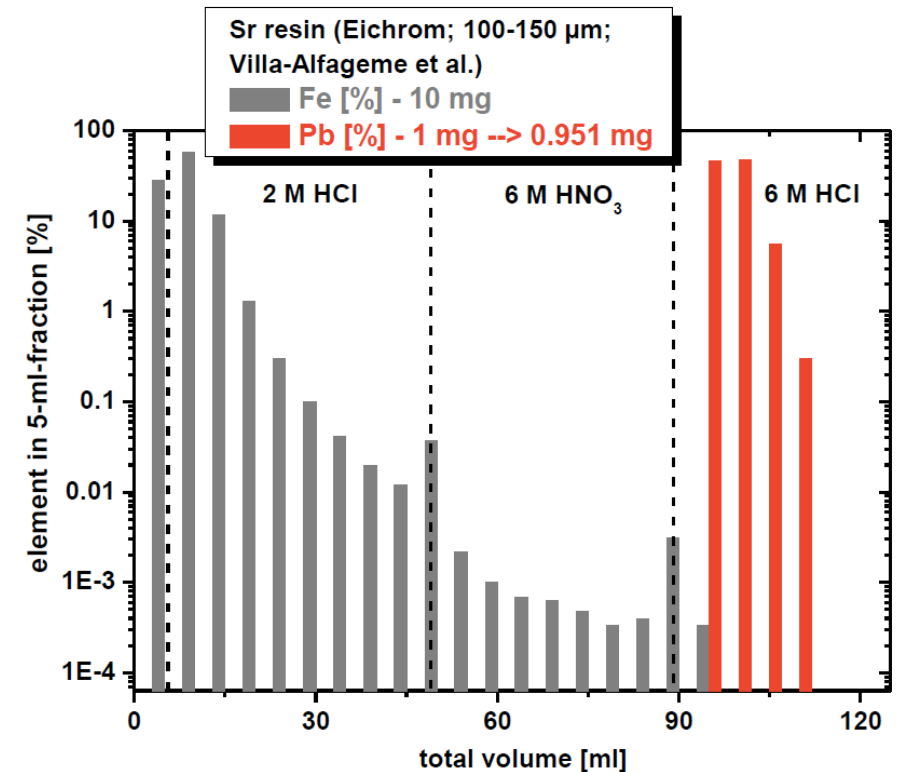
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Thank you for your attention!

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Any questions?

Michaela Froehlich

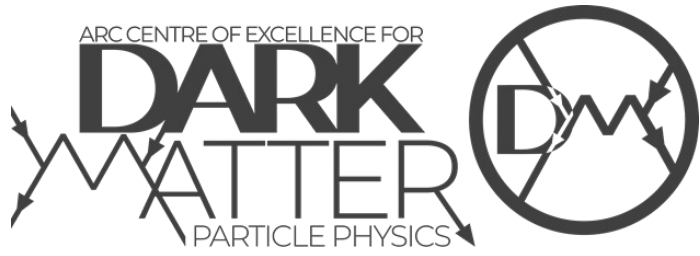
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