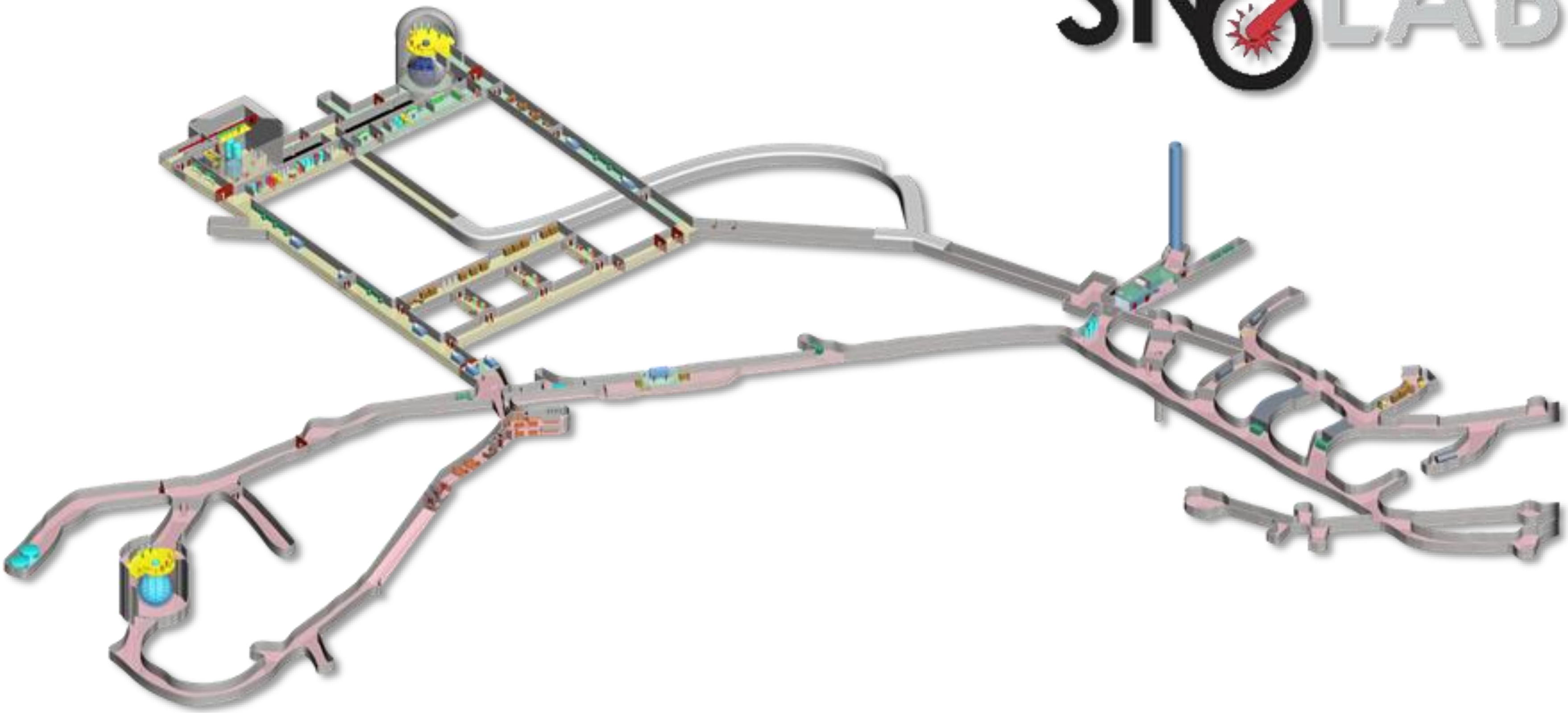
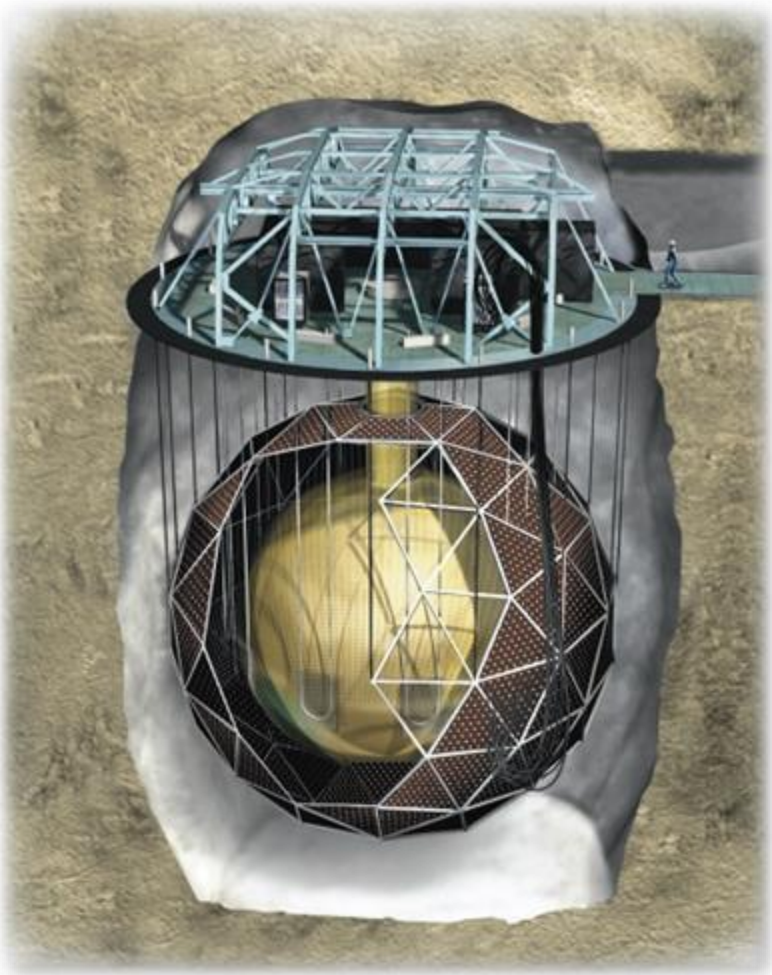
Tellurium purification and deployment for the SNO+ Experiment

Szymon Manecki, SNOLAB Research Scientist,
LRT, October 2nd, 2024

Introduction



SNO+ Physics



- Water Phase (current) *PRD 99, 032008 (2019)* *PRD 105 112012 (2022)*
  - Best limits on invisible modes of nucleon decay
  - Measurement of the 8B solar neutrino flux in SNO+ with very low backgrounds *PRD 99, 012012 (2019)*
  - Highest efficiency (~50%) for neutron detection in a water Cherenkov detector *PRC 102, 014002 (2020)*
  - Detection of antineutrinos from distant reactors using only pure water *PRL 130, 091801 (2023)*
- Scintillator Phase (current)
  - Demonstrating event-by-event reconstruction of the direction of recoil electrons (from solar neutrinos) in a liquid scintillator – this result was also an achievement that hasn't been done before *PRD 109, 072002 (2024)*
  - Being only the second detector to make measurements of the neutrino oscillation parameter Δm_21^2 using antineutrinos from nuclear reactors, an important verification of the previous measurement *arxiv.org/abs/2405.19700*
- Tellurium Phase (upcoming)
  - Developing methods to load tellurium into organic liquid scintillator *NIM, 1051, 168204 (2023)*
  - Developing techniques to purify telluric acid *NIM. A. 795:132-139 (2015)*

Double Beta Decay

- Are neutrinos their own anti-particles?

- 2νββ (Dirac)
  (A, Z) ⟶ (A, Z + 2) + 2e- + 2νe
  ~ 10^{18}-10^{21} years

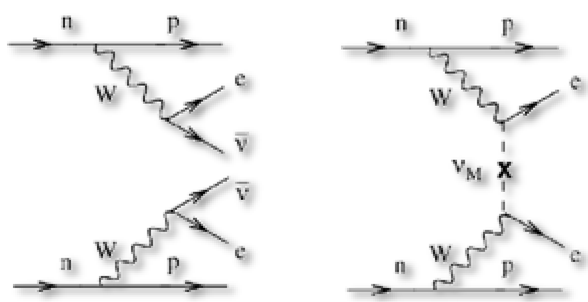
- 0νββ (Majorana)
  (A, Z) ⟶ (A, Z + 2) + 2e-
  > 10^{25} years

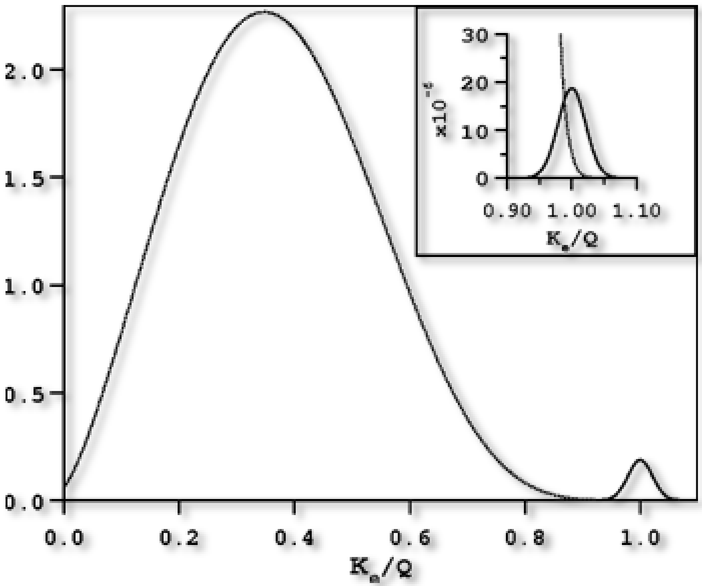
- We measure:

$$\frac{1}{T_{1/2}} = G\, g_A^4\, \mathcal{M}^2 \left(\frac{m_{\beta\beta}}{m_e}\right)^2$$

Phase space factor

Nuclear matrix element





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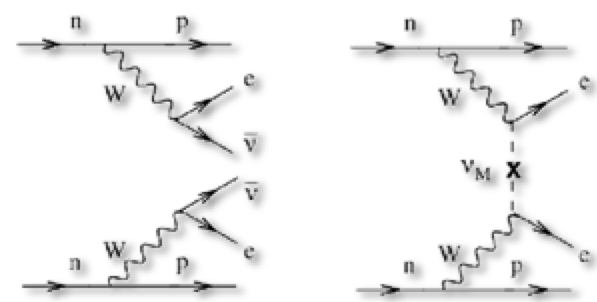
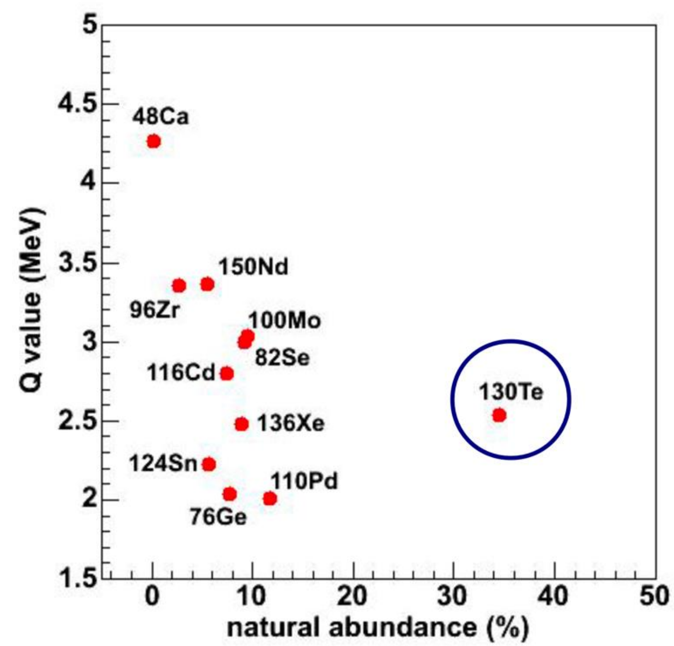
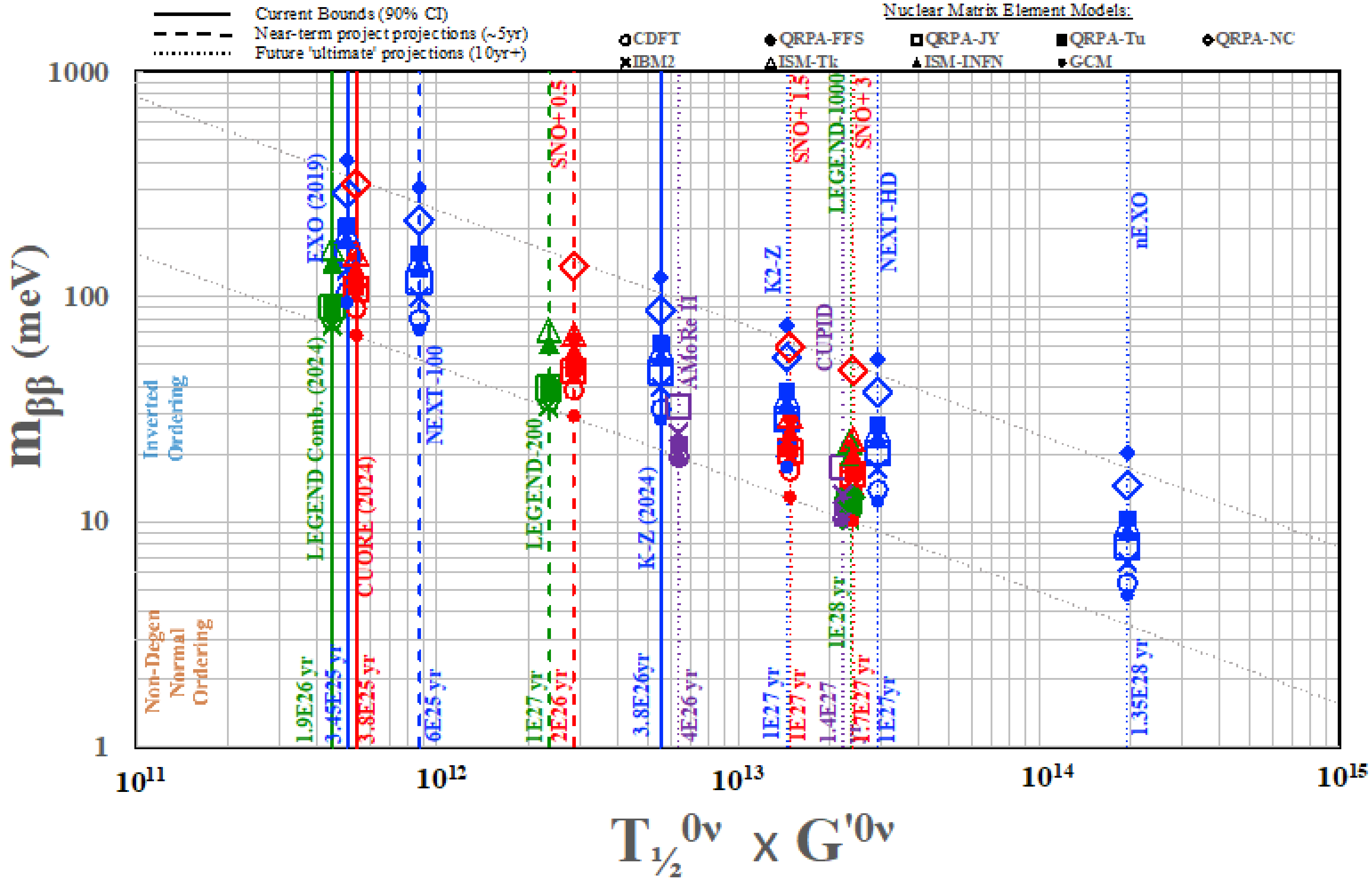
Phase space factor (G)

Nuclear matrix element ($\mathcal{M}$)



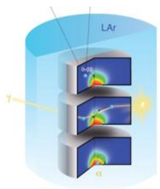


Double Beta Decay



Double Beta Decay

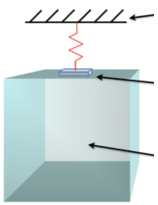
- LEGEND-200
  - 200 kg of ^{enr}Ge HPGe TPC's



- $T^{0\nu}_{1/2}$ lower limits (90% frequentist C.L.)

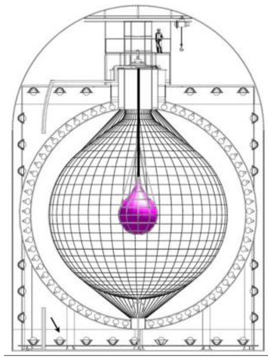
Observed	Sensitivity
$> 1.9 \cdot 10^{26}$ yr	$2.8 \cdot 10^{26}$ yr

- CUORE
  - 750 kg TeO_2 cryogenic calorimeters



Half-life limit: $T^{0\nu}_{1/2} > 3.8 \times 10^{25}$ yr (90% C.I.)

- KamLAND-Zen
  - 800 kg ^{enr}Xe scintillator based detector



Combined $T^{0\nu}_{1/2} > 3.8 \times 10^{26}$ yr

Milan '24 0νββ:
https://agenda.infn.it/event/37867/sessions/29923/#20240618

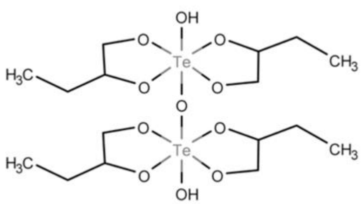
Te Scintillator

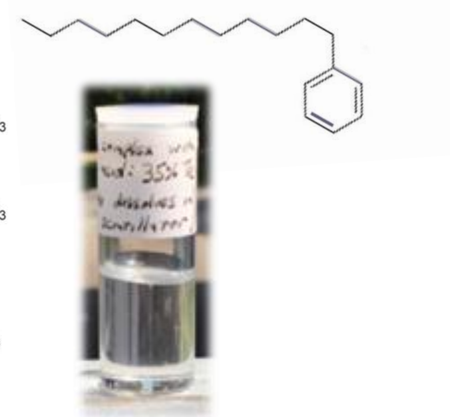
- 780 T Linear Alkylbenzene (LAB)
  + 2 g/L PPO (Primary Fluor)
  + 2 mg/L bisMSB (WS)
  + 6 mg/L BHT (Stb)

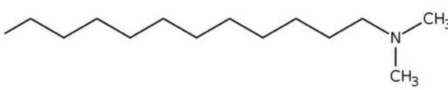
- Tellurium Butanediol (TeDiol)
  0.5% Te in LAB

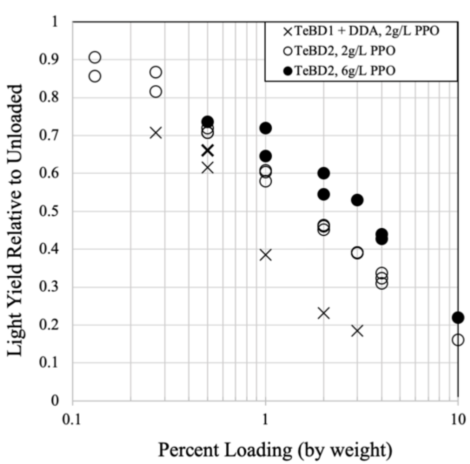
- DDA (stabilizing amine)
  0.2% in LAB

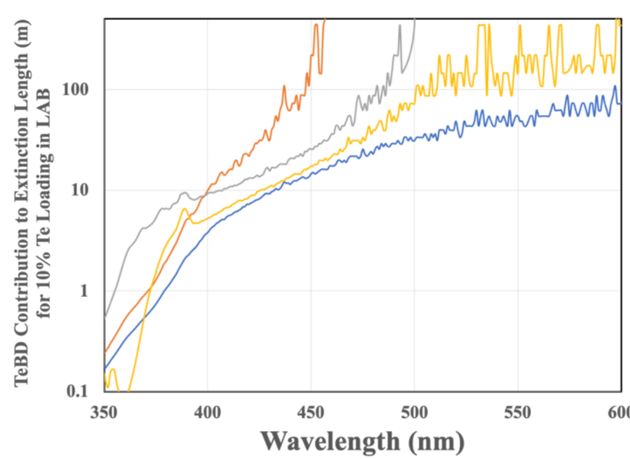
A Method to Load Tellurium in Liquid Scintillator for the Study of Neutrinoless Double Beta Decay
NIM, 1051, 168204 (2023)





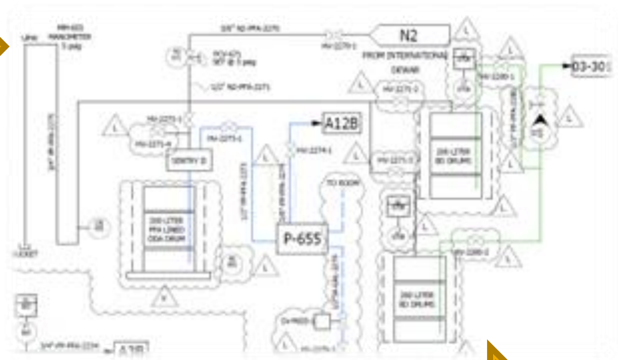


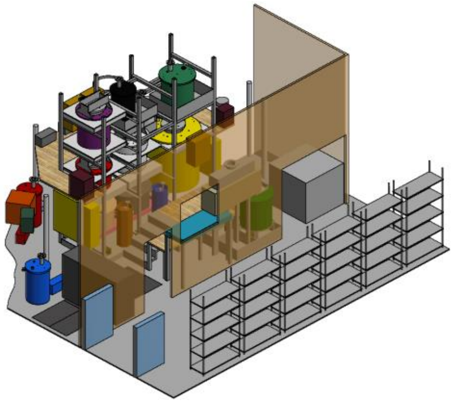




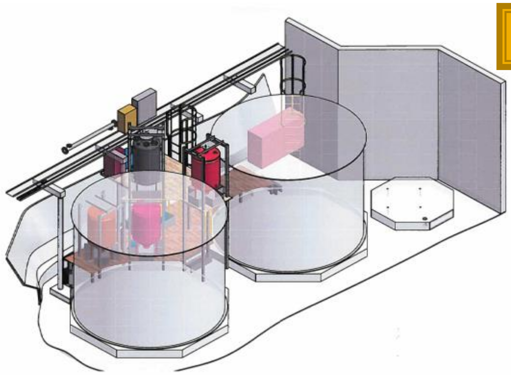
Te Systems

DDA Distillation

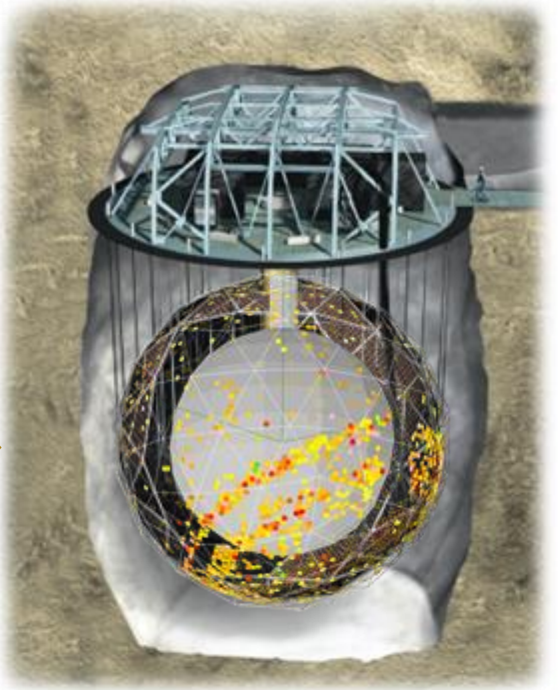




TeA

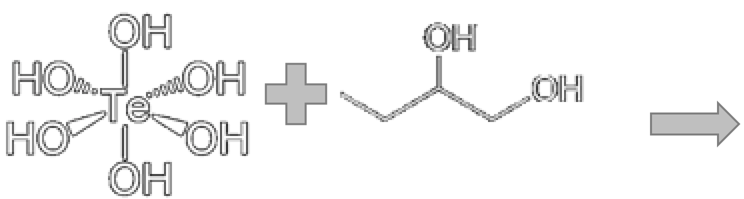


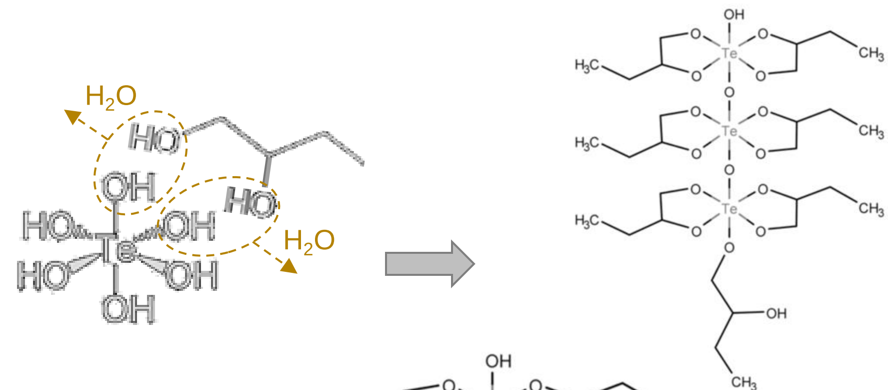
TeDiol

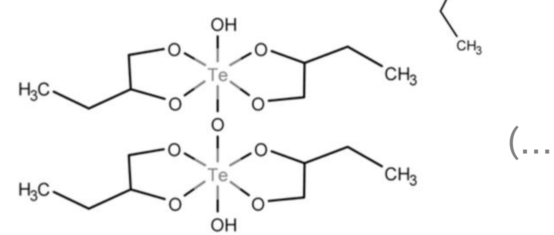


Te Reagents

- LAB-soluble TeDiol complexes are formed in condensation and further oligomerization reactions of Telluric Acid with 1,2-Butanediol







- TeA purification (~8 tonnes)
  - Test Batch operations completed
- 1,2-Butanediol distillation (~14 tonnes)
  - Using very similar parameters to LAB
- DDA distillation (~2 tonnes)
  - U/Th target at ~10^{-15}g/g (expected reduction factor of 1000 from the assayed level has been easily reached with spiked distillation)
  - Expected reduction factors for Co/Na have been achieved, but clean handling post-distillation is going to be important

TeA Purification

- The purification technique relies on solubility of TeA in water based on pH
  - $Te(OH)_6 \leftrightarrow Te(OH)_5O^- + H^+$

    in-soluble soluble
- Insoluble contamination
  - Dissolve in water, and filter
- Soluble contamination
  - Force TeA to recrystallize by adding Nitric Acid, let it precipitate out, and drain the "dirty" liquid

Isotope	t_{exp}=1 yr
^{22}Na	15309
^{26}Al	0.048
^{42}K	565
^{44}Sc	102
^{46}Sc	43568
^{56}Co	2629
^{58}Co	25194
^{60}Co	6906
^{68}Ga	37343
^{82}Rb	18047
^{84}Rb	11850
^{88}Y	390620
^{90}Y	823
^{102}Rh	276189
^{102m}Rh	133848
^{106}Rh	1534
^{110m}Ag	69643
^{110}Ag	939
^{124}Sb	3101138
^{126m}Sb	240
^{126}Sb	358996

Pilot-plant

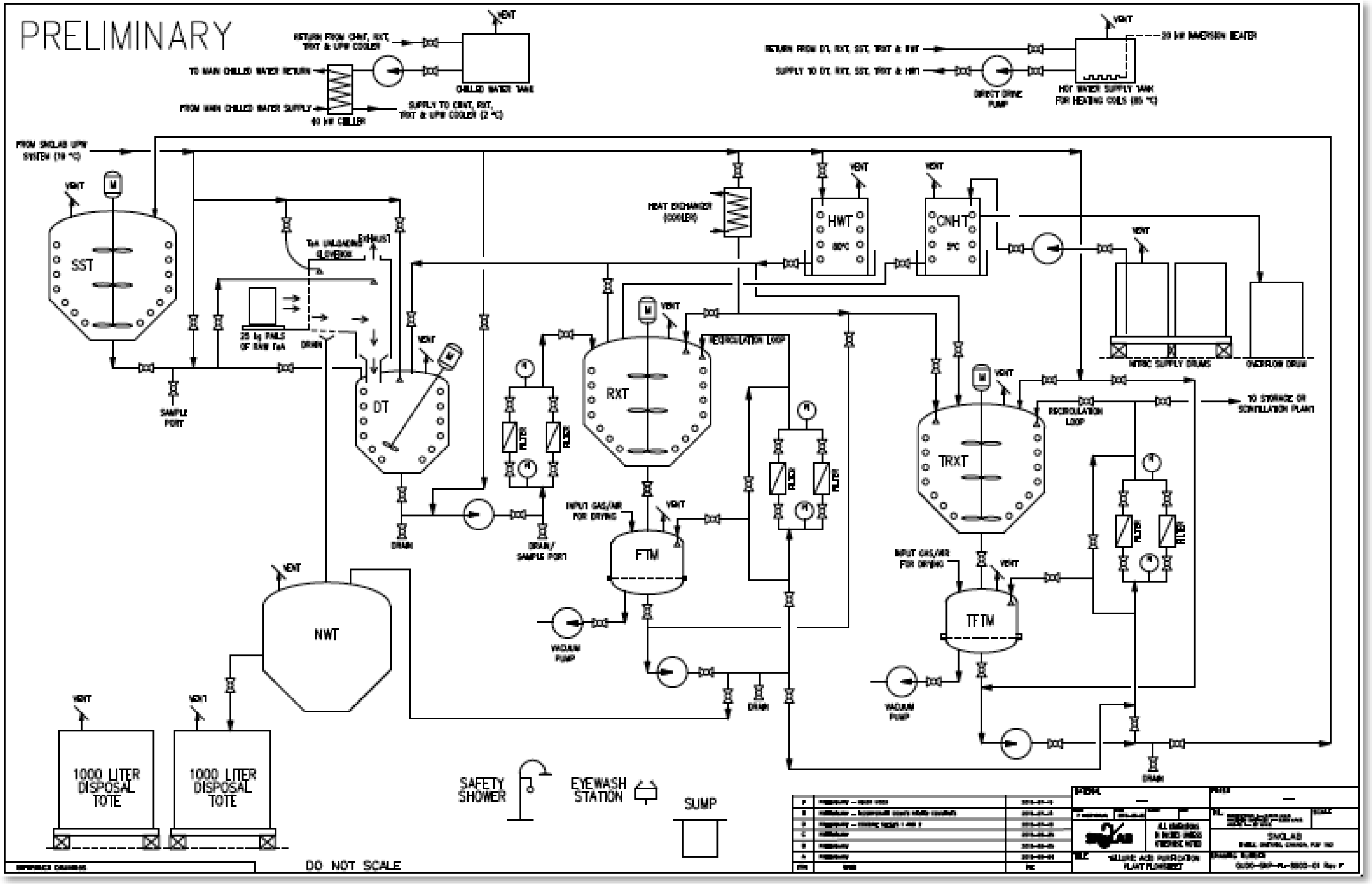
Free purification factor due to underground cooldown

Target (r.f. 10^3):
^{238}U: 1.3×10^{-15} g/g
^{232}Th: 5×10^{-16} g/g

Expected reduction for cosmogenics by: 10^5-10^6

Nucl. Inst. Meth. A. 795:132-139 (2015)

TeA Plant Flow Diagram



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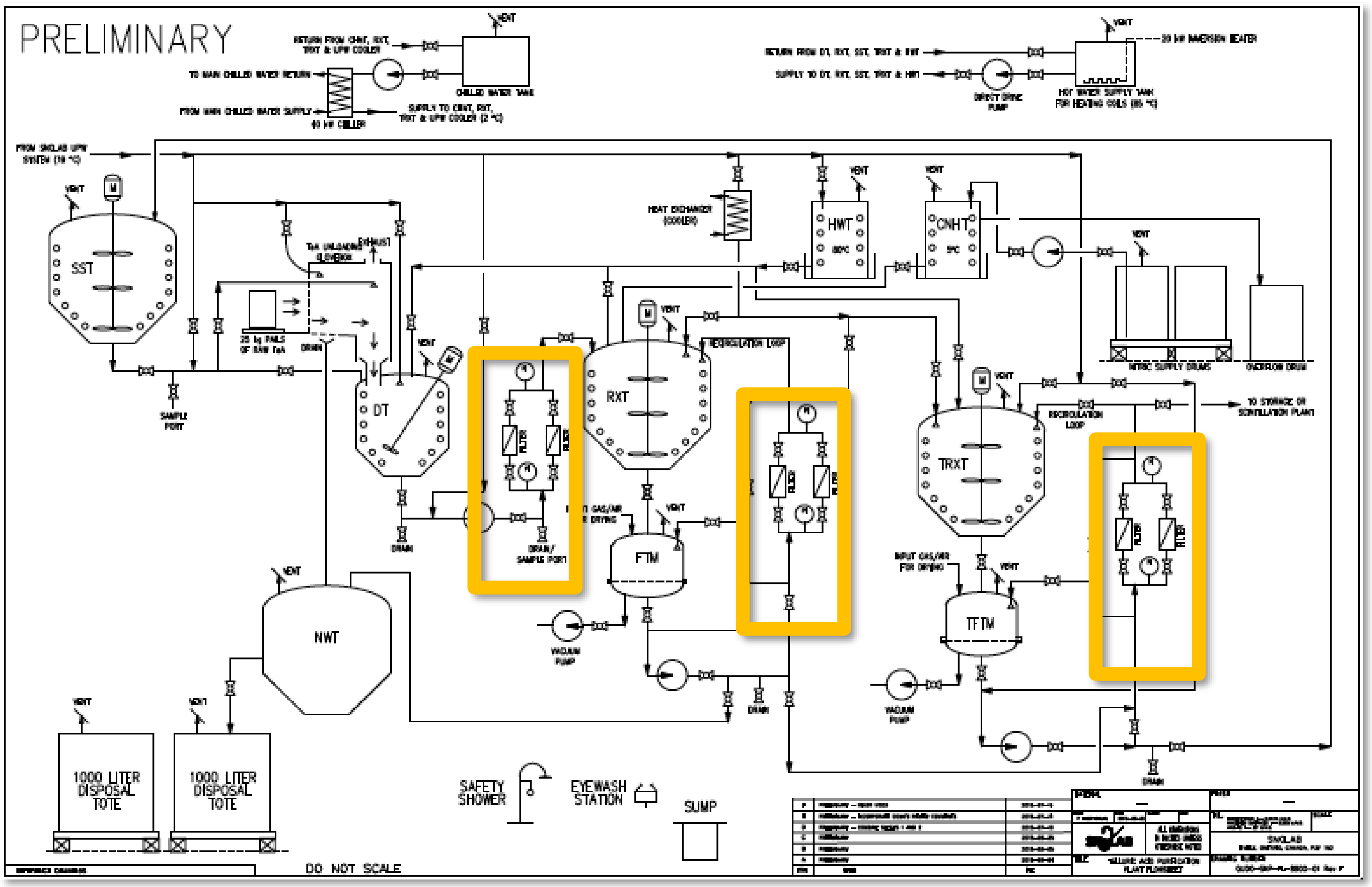

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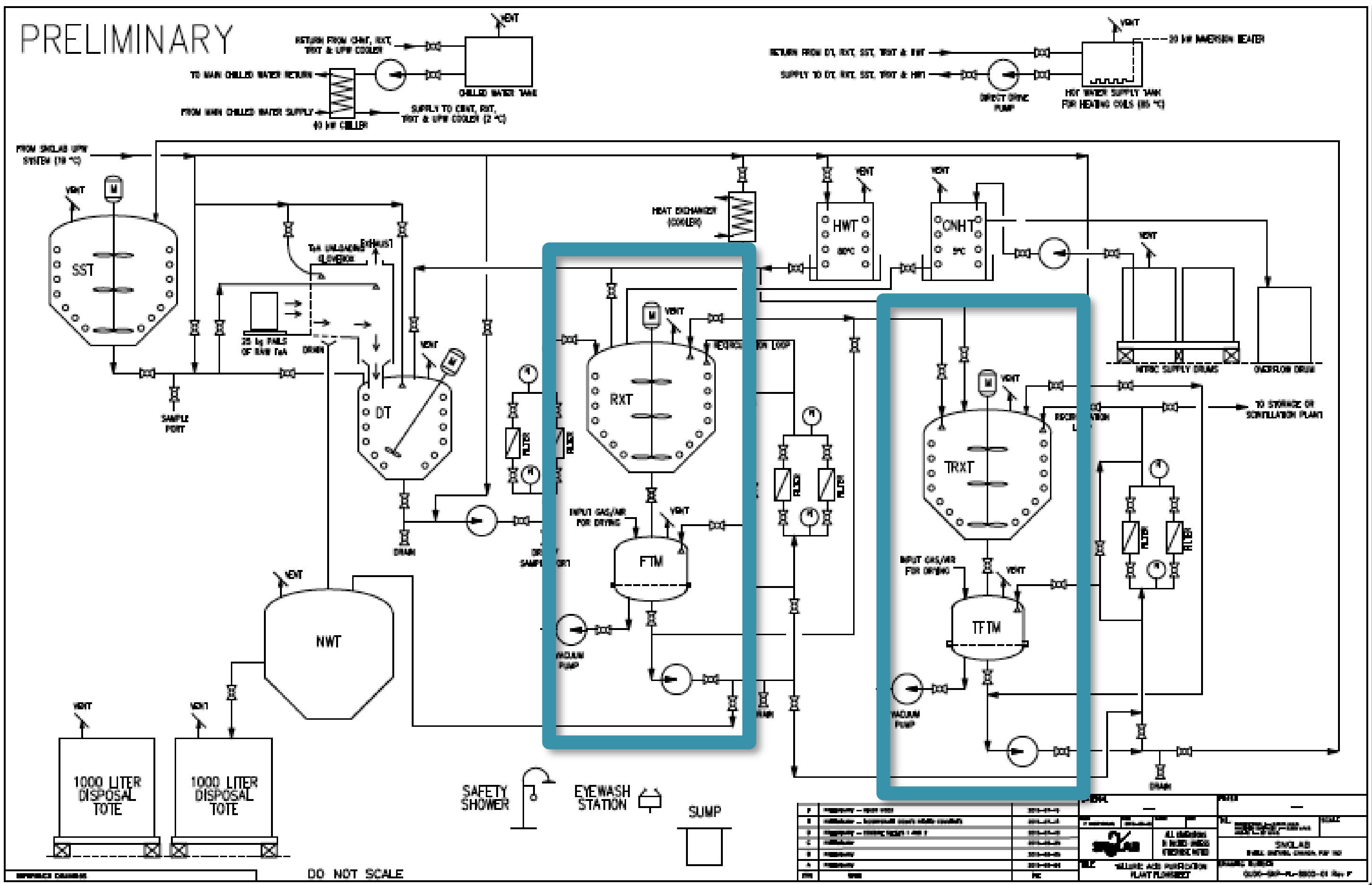

TeA Full Scale Test

- Safety (Process Checklist and Monitoring)
  - Transport and handling of nitric acid and telluric acid
  - Sampling
- Process (Plant and Performance QA)
  - Mechanical, Electrical, Instrumentation
  - Yields and efficiencies
- Physics (Process Purification QA)
  - Purifications factor and ICP-MS analysis

TeA Full Scale Test

- Safe Reagent Handling Underground
  - Nitric acid shipping and logistics
  - TeA loading into the plant
  - Nitric and telluric acid sampling
  - Process systems

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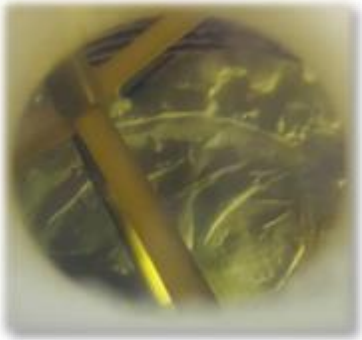


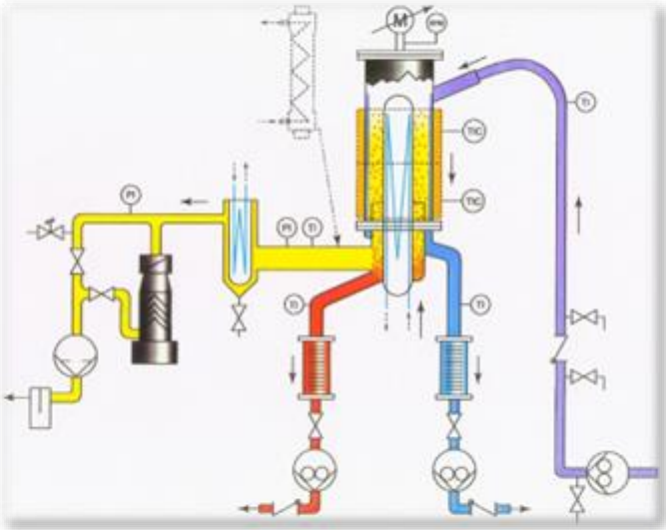
TeA Full Scale Test

- Safe Reagent Handling Underground
  - Redissolved TeA sample after first stages of nitric acid recrystallization will be analyzed with ICP-MS soon
  - We've determined that our TeA is much cleaner than what we've been assuming so far
    (U and Th at the level of a few ppt from two different batches/drums)
  - Alternative nitric acid supply also demonstrated to be better than expected: ~5×10^{-15} gU/g and ~6×10^{-16} gTh/g
  - Ultimately the purification factor is determined by the amount of residual nitric acid in the TeA crystal - which we just demonstrated to be satisfactory

DDA Distillation

- Purification of the DDA will be carried out using Wiped Film Distillation
  - Very efficient method for separating and purifying liquids in chemical processes
  - Particularly useful for thermally sensitive materials with high boiling points.

pope Scientific Inc
Solution Driven.



- DDA assay
  - High Purity Ge (HPGe) detector at SNOLAB
    - $^{238}U = 0.20$ ppb
    - $^{232}Th = 0.82$ ppb
    - $^{40}K < 1.88$ ppm
  - Neutron Activation Analysis
    - Na < 0.1 ppm
    - Br < 30 ppm (cosmogenic activation)
  - $^{14}C/^{12}C$ to confirm its non-biogenic origin
    - Accelerator Mass Spectrometry at uOttawa:
    - Sample #1: ~ 10^{-15}
    - Sample #2: ~10^{-16}

Underground delivery within 1 week to avoid potential cosmogenic activation

DDA Distillation

- Expected reduction factors
  - >1000 for U/Th (40k achieved)
  - >10 for Na/Co (15 for Na and 50 for Co achieved)
- Limited contact with S. Steel
  - All process lines made of Teflon
  - Only 'brief contact' with SS at the distillation plant

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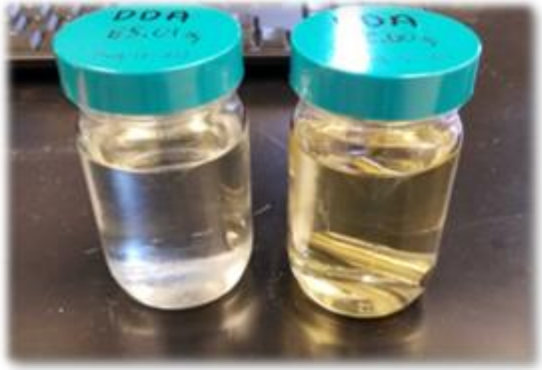

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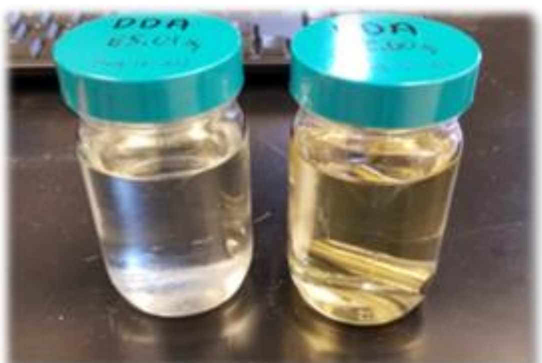
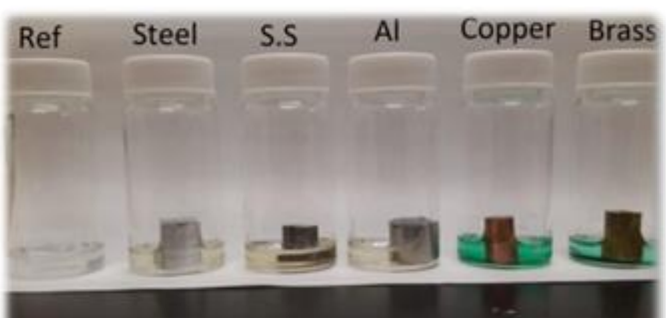
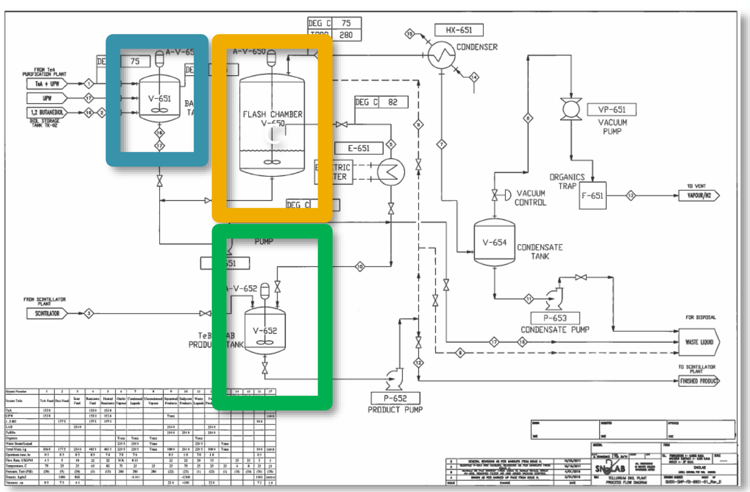




TeDiol Synthesis

- Tellurium synthesis tests have been carried out on a smaller scale, between 8 grams and 1.6 kilograms

- Full, 200-kg scale batch is planned soon



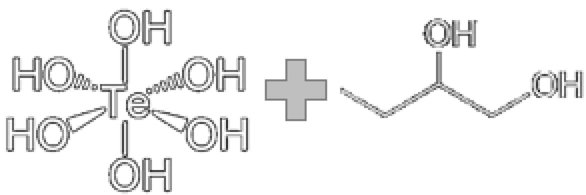
Thank You

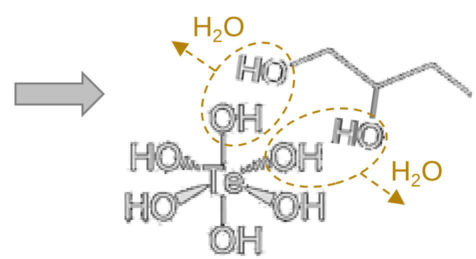

NOTICE
NO ENTRY WITHOUT PERMISSION
2084
2085
2082
2083
V-601 DT
CAUTION
CAUTION
NOTICE
NO ENTRY WITHOUT PERMISSION

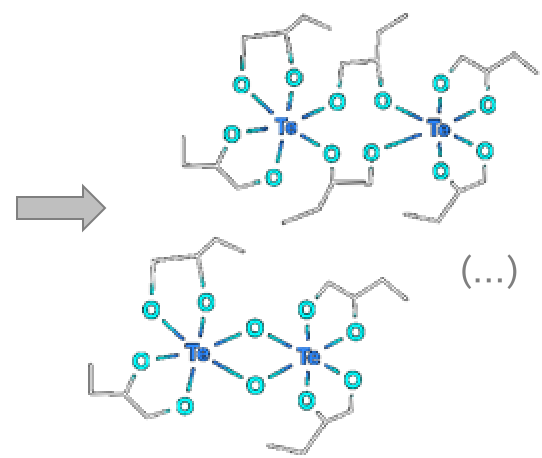
Backup

Te Scintillator

- LAB-soluble TeDiol complexes are formed in condensation and further oligomerization reactions of Telluric Acid with 1,2-Butanediol



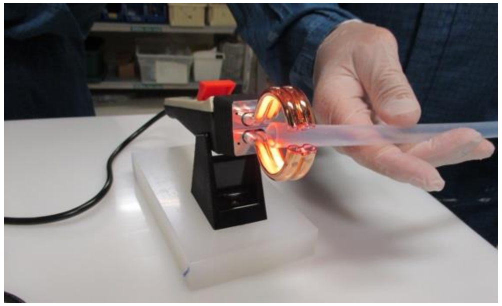




- TeA purification
  - The target 8 tonnes of TeA have already been cooling down underground (50% for > 2 years and 50% for < 2 years)

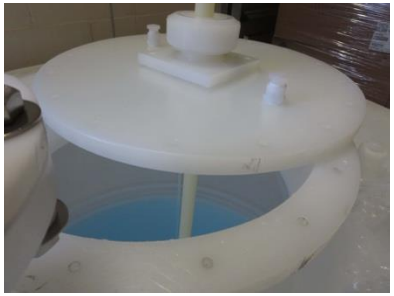
TeA Fabrication & Cleaning

- All wet process lines and vessels constructed with plastic to suppress metals leaching
  - Polypropylene vessels designed and delivered by SeaStar
  - On-site contractors trained in clean PFA piping installation



TeA Fabrication & Cleaning

- SeaStar process vessel leaching (with warm acid solution)



Results from ICP-MS assay of leachate (ppt)

	Soak 1 (2 days)		Soak 2 (4 days)		Soak 3 (4 days)	
	RXT	TRXT	RXT	TRXT	RXT	TRXT
U	1	0.2	<0.05	<0.05	<0.05	<0.05
Th	5	1	1.1	<0.1	<0.1	<0.1
Ca	2700	2000	380	180	<20	<20
Fe	5600	5000	220	170	17	37

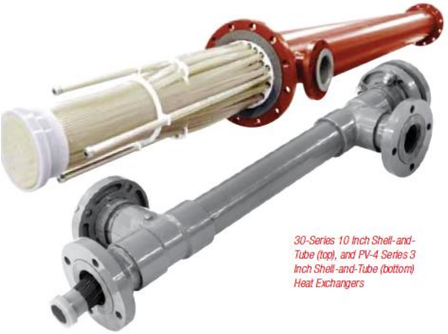
Compare: goal of 0.1 ppt U and <0.05ppt Th in purified TeA. Other measured metals (relevant for cosmogenics) lower than Ca and Fe, <0.1 ppb goal.

Vessels meet our purity requirements!

Further cleaning/leaching with nitric acid after installation will provide additional safety factor.

TeDiol Fabrication & Cleaning

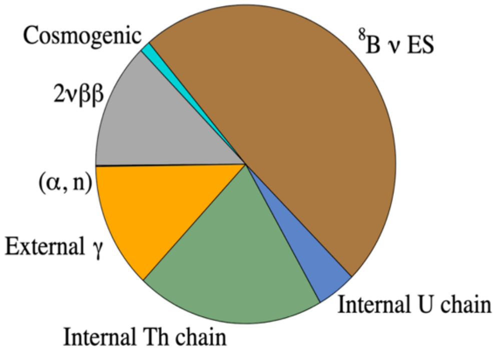
- PFA (PerFluoroAlkoxy) lined FRP (fiberglass-reinforced polymer) tanks for main process vessels
  - Flash chamber, batch tank & product tank
- PFA in-line heater
- PFA condenser
- PFA Centrifugal Pumps
- Nitrogen sparging system
- Stainless steel condensate tank

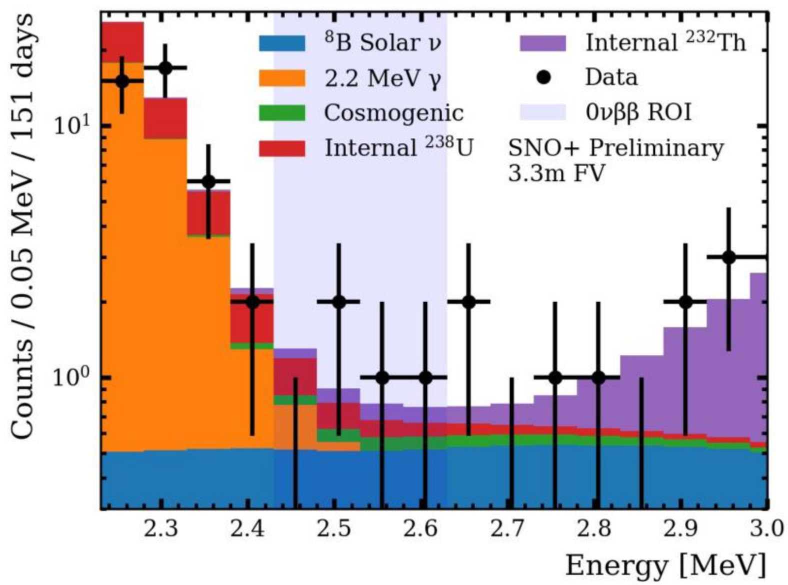


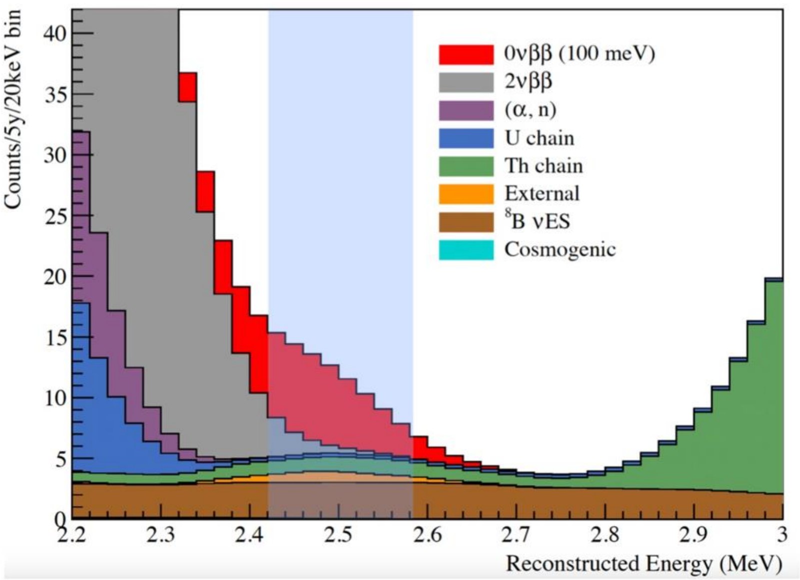
30-Series 10 Inch Shell-and-Tube (top), and PV-4 Series 3 Inch Shell-and-Tube (bottom) Heat Exchangers

SNO+ Te Phase

- With 0.5% Te loading
  - $T^{0\nu}_{1/2} > 2.1 \times 10^{26}$ yrs
  - $m_{\beta\beta} > 45$ meV$_{(IBM-2)}$

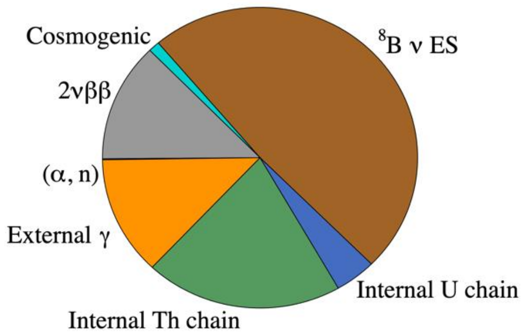


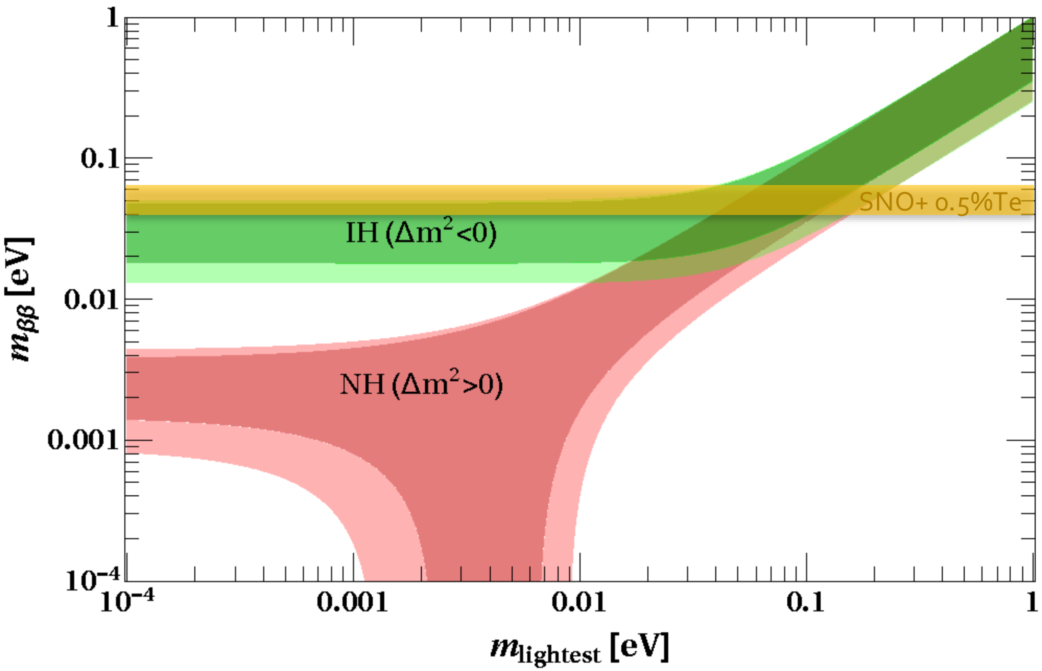




SNO+ Te Phase

- With 0.5% Te loading
  - $T^{0\nu}_{1/2} > 2.1 \times 10^{26}$ yrs
  - $m_{\beta\beta} > 45$ meV$_{(IBM-2)}$





Notes

- SNOLAB
- Intro to DBD (like in the MI talk)

- UG systems
  - Ask if Aleksandra will cover PPO, bisMSB and BHT
  - Cover the why (stability) and how (water-extr.)

- UG TeA and TeDiol Plants
  - Te Purification concept and TeDiol synthesis
- TeA Test Batch
  - Nitric handling, yields, process

- DDA Still
  - Why we need it on surface
  - Metal spike via exposure to metal powder