





### A Scintillator Purification Plant and Fluid Handling System for the SNO+ Experiment



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# **Deep Underground Laboratories**



LRT2015, Seattle, 18-Mar-2015

### **SNOLAB – At Creighton Mine**



### Surface Facility



km overburden 5000mwe)



<u>63/11/21/</u>



# The SNO+ Experiment





# **Scintillator based on LAB**

Linear alkylbenzene (LAB) for the liquid scintillator solvent:

- Chemical compatibility with acrylic
- High light yield
- High purity available
- Safe
  - Low toxicity
  - □ High flash point 140°C
  - Boiling point 278-314°C
  - Environmentally safe (bio-degrades)
  - Low solubility in water 0.041 mg/L
- Inexpensive
- Suitable density  $\rho = 0.86 \text{ g/cm}^3$

□ PPO for the wavelength shifter:

- Common "whitening" or marker dye
- Low toxicity
- Melting point 72°C, boiling point 360°C





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Cepsa Plant – Bécancour, QC

### **SNO+ in SNOLAB**



New SNOLAB expansion



### **SNO+ in SNOLAB**





□ Neutrino-less double beta decay (Natural Te loaded – <sup>130</sup>Te)

- Determine if neutrino is Dirac or Majoranna type
- CP violation in neutrinos
- Most sensitive measurement of absolute neutrino mass

□ Neutrino physics (+ solar and earth science)

- Solar neutrinos (pep, CNO)
- Geo antineutrinos
- Reactor antineutrinos
- Supernova neutrinos
- Nucleon Decay

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# **Target backgrounds for SNO+**

□ SNO+ follows the experience of Borexino and KamLAND. We assume the same background levels as BX since we have, as baseline, adopted the same purification processes, materials and cleanliness considerations. We have then advanced and added to these processes for adaptation to the SNO+ experiment. SNO+ also gains from the SNOLAB cleanroom environment and experience.

#### □ Target levels:

- **Th:**  $10^{-17}$  g/g (about 3 cpd for <sup>208</sup>Tl and <sup>228</sup>Ac)
- U (Rn): 10<sup>-17</sup> g/g (about 9 cpd for <sup>214</sup>Bi and <sup>210</sup>Bi)
- <sup>40</sup>K: 1.3x10<sup>-18</sup> g/g (about 23 cpd, good spectrum fit)
- <sup>85</sup>Kr, <sup>39</sup>Ar: < 100 cpd (pileup effects)
- Caveat: SNO+ reuses the original SNO acrylic vessel, which may lead to additional background due to bulk and surface leaching of U and Th daughters. The SNO+ plants are designed with high capacity throughput to purify these potential loads.
- Target level for U chain due to Rn ingress leads to vacuum leak tightness requirement 10<sup>-6</sup> mbar.L/sec (total combined from all sources).
- $\Box$  Kr, Ar target levels also require <~10<sup>-6</sup> mbar.L/sec air leak rate (integrated plant).

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# **Scintillator plants**











## **Considerations for Plant Design**

- Require capacity to purify full detector volume in ~4 days, for reduced equilibrium level of potential <sup>222</sup>Rn and <sup>224</sup>Ra sources (both ~4 day halflives). Also, due to mining company logistical and access issues, we require ability to turn-over the detector within 5 days.
- Purification processes are equilibrium stage processes with require high temperatures to gain stage efficiencies, and tall columns for more stages.
- □ Being underground we are severely constrained by space and height.
- □ Limited electrical power available. Plant has ~0.75 MW installed, but limited to ~0.5 MW operating (SNOLAB has two 1500 kVA MPCs).
- ❑ Limited cooling capacity available. SNOLAB has 320 tons (1.1 MW), of which about 400 kW needed for climate control (13 AHUs) and 400 kW for other experiments, leaving 300kW baseline (can peak higher).
- □ Limited  $LN_2$  boil-off available due to logistics to ship  $LN_2$  dewars underground. Limited to about 5 kg/hr for cover gas and stripping.
- Plant and equipment must meet code for earthquake and near-field seismic events due to mining "rock-bursts".
- □ LAB is combustible and must have all risk of fire mitigated due to unique life-safety hazards being located within a mine.

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### **SNO+ Simplified Process System**



SNOLAB SNO

## **Purification strategies:**



#### Multi-stage distillation

- Initial LAB cleanup for high radio-purity and optical clarity
- Dual-stream PPO distillation for scintillator recirculation

#### □ Pre-purification of PPO concentrated solution

N2 sparging, batch water extraction, followed by "flash"-distillation

#### **Pre-purification of Te-salt solution and "loading agent" chemicals**

- PH-controlled precipitation or thermal re-crystalization of Te-salt solution
- Thin-film evaporation or silica-gel purification of "loading agent" chemicals

#### Steam/N2 stripping under vacuum

Removes Rn, O<sub>2</sub>, Kr, Ar and provides LAB humidity control

#### □ Water extraction (liquid-liquid extraction)

- Provides high-flow recirculation polishing stage
- Effective for ionic metals (K, Pb, Ra) and partially effective for Th and Po
- Cannot be used with Te-loaded scintillator, however equipment can be used to unload Te

#### □ Functional metal scavengers (on silica gel matrix)

- High-flow columns effective for Pb, Bi and Ra
- Can be regenerated with acid wash
- Processing and assay of the acid wash provides a method for radio-assay of scintillator

#### Filtration

Ultra-fine (0.05μm) filtration for "ultra-fine" suspended contamination

## **Distillation: benchtop vs column**

"Bench-top" (laboratory scale) distillation is quite different from continuous multistage tower distillation. A small batch distillation cannot show the efficacy of the fractionating distillation process.





Distillation is also an equilibrium stage process – but you need the stages!!!

$$V_{n+1}y_{n+1} = L_n x_n + D x_D \Longrightarrow y_{n+1} = \frac{L_n}{V_{n+1}} x_n + \frac{D}{V_{n+1}} x_D$$

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### Laboratory LAB batch distillation tests





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### **Distillation Column Design**





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# **Distillation Challenges**

- Although heavy metals are expected to have very low volatility, organic optical contaminants *c* can have K-values close to LAB. Need to maximize stages.
- Total column pressure must be reduced as low as possible, to avoid thermal reactions with LAB at higher temperatures. Instead of conventional downcomers, we use dual-flow tray to minimize stage dP and maximize efficiency.
- For low particle generation, the column internals to avoid bolted hardware. A novel pre-tension technique using cotterpins is employed for tray hold-down.
- Limited power and cooling underground requires maximum heat process heat recover. We use high-pressure steam generator to extract heat from primary LAB condenser.



$$\alpha_{A} = \frac{K_{A}}{K_{LAB}} = \frac{y_{A} / x_{A}}{y_{LAB} / x_{LAB}} = \frac{y_{A} (1 - x_{A})}{x_{A} (1 - y_{A})}$$











Clean and install trays





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Dual-Stream Fractional Vacuum Distillation SNALAB SNO



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#### Illustrative Example of Steps for Single Batch-wise Stage of Water-Extraction



### **Counter-Current Water Extraction**



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### Water Extraction Bench-top Spike Tests



- Tests with <sup>224</sup>Ra show a high removal efficiency 98%.
- There is a lead component in LAB, irremovable by WE.





### SCHEIBEL® Column for SNO+





### Water Extraction water loop



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## Gas stripping (N2 and Steam)





# **Stripping Challenges/Solutions**

- Solubility of noble gases (Rn, Ar, Kr) high in aromatic solvent (eg. Henry coefficient for Rn in LAB is ~10, which means Rn partitions ~87% into LAB at 1 atm). Need more stages, and reduced pressure.
- Flow rate 150 LPM, hence need large flow rate of low-radon stripping gas to obtain the required stages. However, logistically it is very difficult to ship sufficient liquid nitrogen into the mine. Solution to use steam as stripping gas, where water is low in Rn from UPW plant degasser/regasser.
- Kremser equation (trace quantity limit):

$$N = \frac{\ln\left[\left(1 - L/mV\right)\left(x_0/x_N\right) + L/mV\right]}{\ln\left[mV/L\right]} \approx 4 \quad \text{Stages}$$

For  $m \approx 80$  at 150 Torr, with 3 kg/hr N<sub>2</sub> and 10 kg/hr steam, to obtain  $x_0/x_N = 20$  for Rn.

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### Gas stripping (N2 and Steam)



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### **Utility Room Today**



















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### Pipes, Pipes, Pipes





# **Scintillator Plants Status**



### Plant construction

- All Columns, tanks and equipment installed
- All process piping is fabricated and installed
- All instrumentation installed
- Utility piping 90% complete April 2015
- All electrical power installed (new feeder, MCCs, VFDs, etc)
- All civil work completed (steel, firewalls, HVAC, lighting etc)
- Control system junction boxes and cabling about 50% completed
- Insulation (hot and cold) and heat tracing May 2015
- Completed installation of fire protection and suppression July 2015

### Pre-Commissioning

- Helium leak checking 95% complete March 2015
- Passivation and cleaning June 2015

### Commissioning

- Operating controls narrative completed (controls, alarms, interlocks etc)
- DCS graphical pages completed
- All control strategies configured
- Water commissioning and instruments configuration (Sept 2015)

# **Conclusions**

- SNOLAB SNO
- The SNO+ scintillator purification plant advances and further develops the techniques and methods used in Borexino and Kamland. The plant has increased capabilities and throughput, both to extend the scientific capability, and due to the severe constraints due to location within the mine. The plant high throughput (150 LPM) provides capability to mitigate substantial background loads from the detector AV, while also extending the SNO method and techniques for full-detector ex-situ radioactivity assaying.
- ❑ The system comprises distillation (LAB and PPO), steam stripping, water extraction, metal scavenger columns, and batch loading equipment for preparation and pre-purification of PPO and Te solutions.
- Plant construction is completed (few minor outstanding items), and the helium leak checking and passivation & cleaning is now in progress.
- Plant control system (DCS) configuration is well advanced and soon ready for commissioning.
- □ Review process underway for insurance and Vale permissions to operate.

### **SNO+ Collaboration**







Queen's Alberta Laurentian **SNOLAB** TRIUMF

BNL, AASU U Penn, UNC **U** Washington UC Berkeley/LBNL Chicago, UC Davis

Oxford Sussex QMUL Liverpool Lancaster



LIP Lisboa LIP Coimbra

**TU Dresden** 



### **SNOLAB Future Planning Workshop 2015**

#### Dates: 24-25 August 2015

The objective of the Future Planning workshop is to provide SNOLAB with a road-map of potential large scale projects that may require its experimental areas on a five to ten year timescale. It is understood that projects will be at various stages of development, and this is not a final selection meeting. Following discussion with the Experiment Advisory Committee, SNOLAB resources could be made available on request to potential projects, to help develop plans and determine suitability and achievability.

Registration is open for presentations on the SNOLAB website:

http://www.snolab.ca/science/meetings-and-workshops/workshops/2015-future-planning-workshop

<u>www.snolab.ca</u>  $\rightarrow$  SCIENCE  $\rightarrow$  MEETINGS&WORKSHOPS



# The End

## **Some Plant Process Specifications**

- Materials SS316, Teflon, glass, acrylic
- Pressure 150psi, B31.3 category D (cold) and normal (hot).
- Temperature 240°C (under boiling point, but above flash point)
- Surface preparations and cleanliness Electropolished, passivated (ASTM A967), cleaning to Mil Spec 1246C class 50.
- Leak tightness 10<sup>-6</sup> mbar.L/sec full plant, 10<sup>-9</sup> mbar.L/sec fittings, 10<sup>-8</sup> for vessels and pumps.
- Pumps, valves and fittings:
  - 1"-2" SS electro-polished tubing, fusion welded (GTAW orbital weld)
  - VCR fittings (<=1")
  - Metal gaskets (eg. Helicoflex) for >1"
  - O-rings Teflon Encapsulated Viton (TEV)
  - Diaphragm or bellows valves for leak tightness
  - Mag-drive pumps

Insulation to TSSA and INCO standard for underground

**Codes (electrical CSA, pressure vessel TSSA, ...)** 

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□ The PPO will be dissolved and pre-purified as a concentrated solution at 120 g/L.

- □ To match 19 LPM LAB fill rate 55 kg/day of PPO will be mixed into 450 L LAB.
- Will mix 1 batch per day, which will be water extracted 3 times, sparged and filtered, then transferred to surge tank.

□ The PPO conc solution will distilled in a flash kettle in parallel with the LAB.

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# **Delivery of LAB to underground**



- At 66 tonnes/wk the fill time is 12 weeks

- Bottleneck is railcars (could have up to 6 trucks/week, and distill up to 150 tonnes/wk)

