# Synthesis and Characterization of Organic Tellurium Compounds for SNO+ Liquid Scintillator


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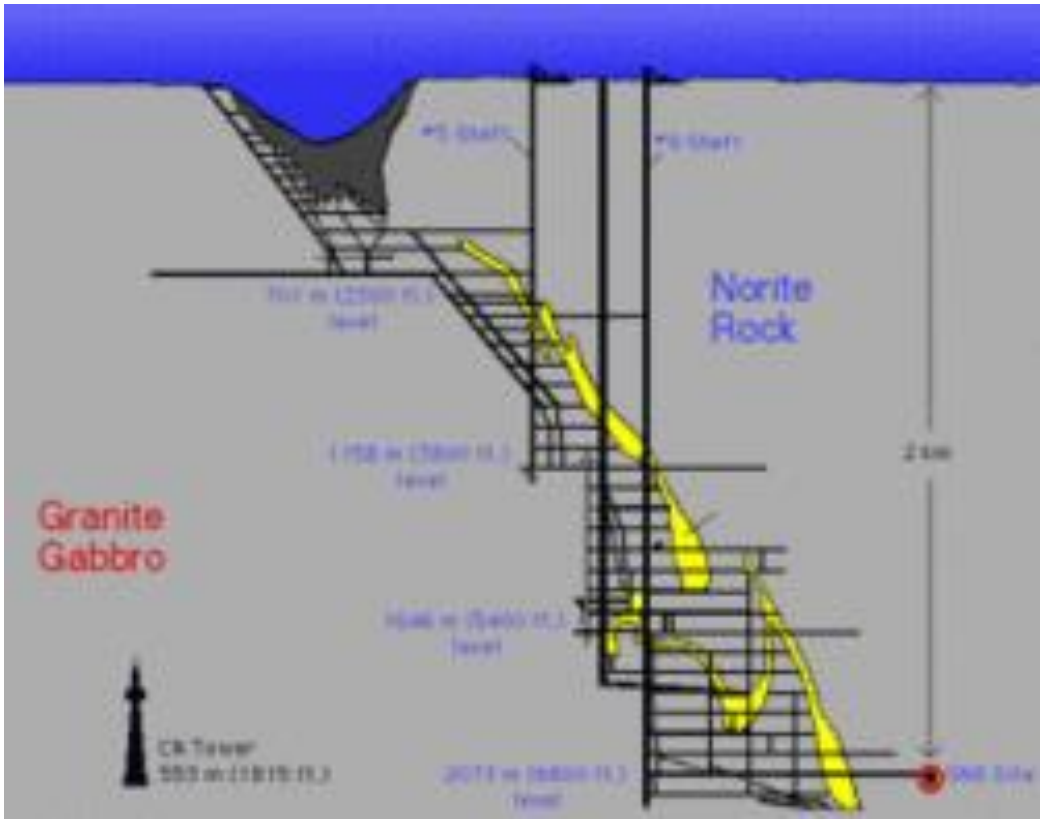
**Queen's University**

**For SNO+ Collaboration**


**LSC2017 Conference**

- Large multi-purpose liquid scintillator detector based in the Creighton Mine, Sudbury, Canada;
- Situated in a clean lab, SNOLAB, at 2039m depth;





Alberta
Laurentian
Queens
SNOLAB
TRIUMF

TUD

UNAM

LIP

Lancaster
Liverpool
Oxford
QMUL
Sussex

Armstrong Altantic
UCBerkeley/LBNL
BNL
UCDavis
UChicago
UNC
Norwich
UPenn
Washington

# SNO+ Collaboration

# SNO+ Detector

New Calibration systems

Norite + granite/gabbro

SNO heavy water replaced by 780 tonnes of liquid scintillator

Improved electronics

New hold-down rope system

7kt ultra pure water shield

~9300 PMTs (54% coverage) 18 m diameter

Liquid scintillator will be loaded with varying amounts of double-beta isotope

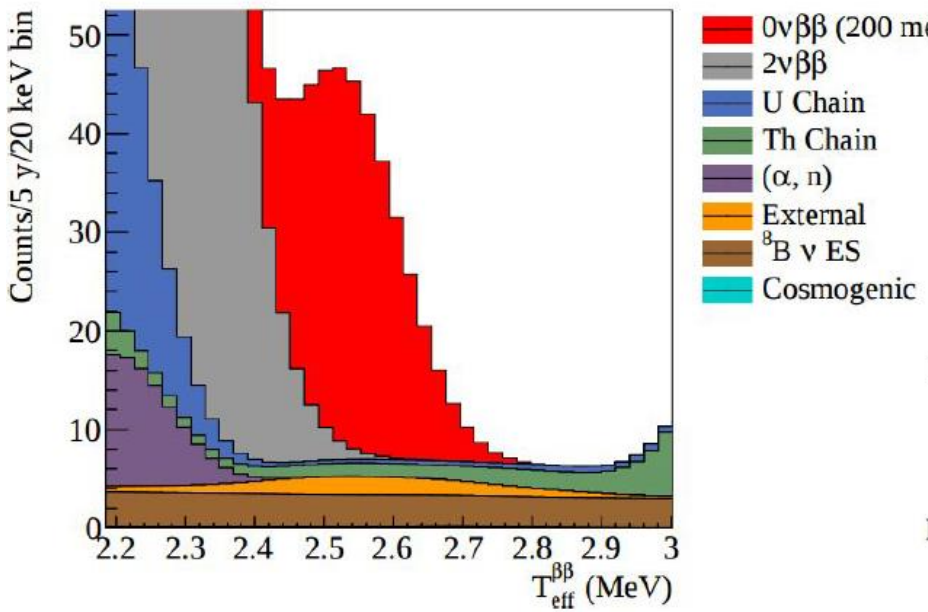
12 m diameter 5cm thick acrylic vessel (AV)

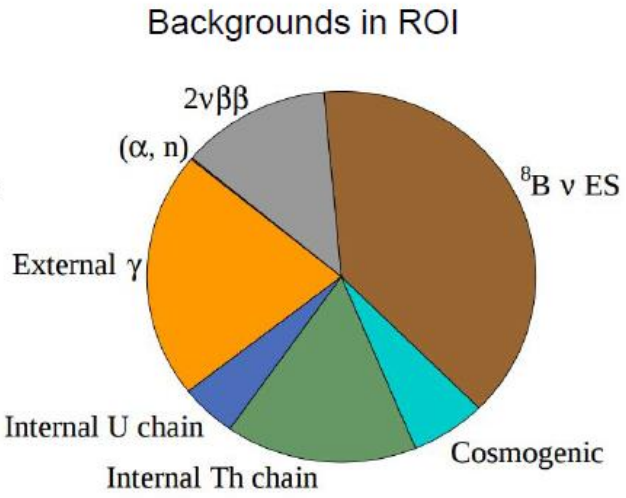
"for the discovery of neutrino oscillations, which shows that neutrinos have mass"

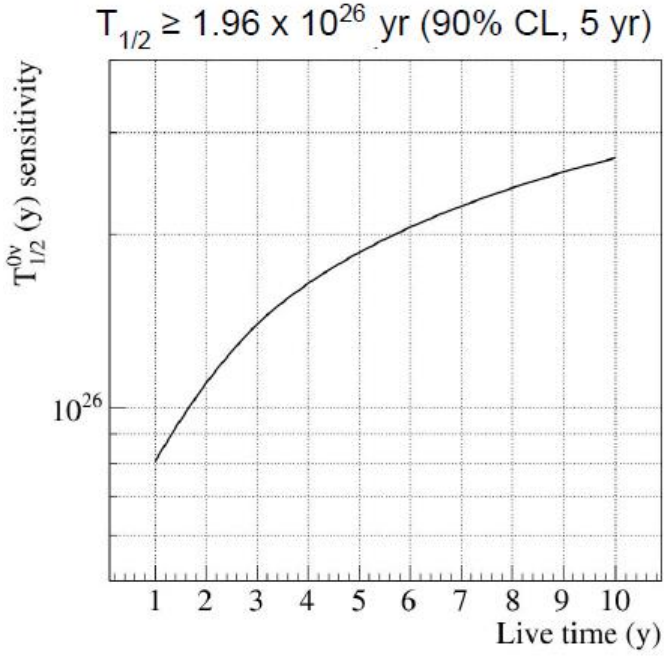
## Art McDonald Nobel Lecture Dec 2015

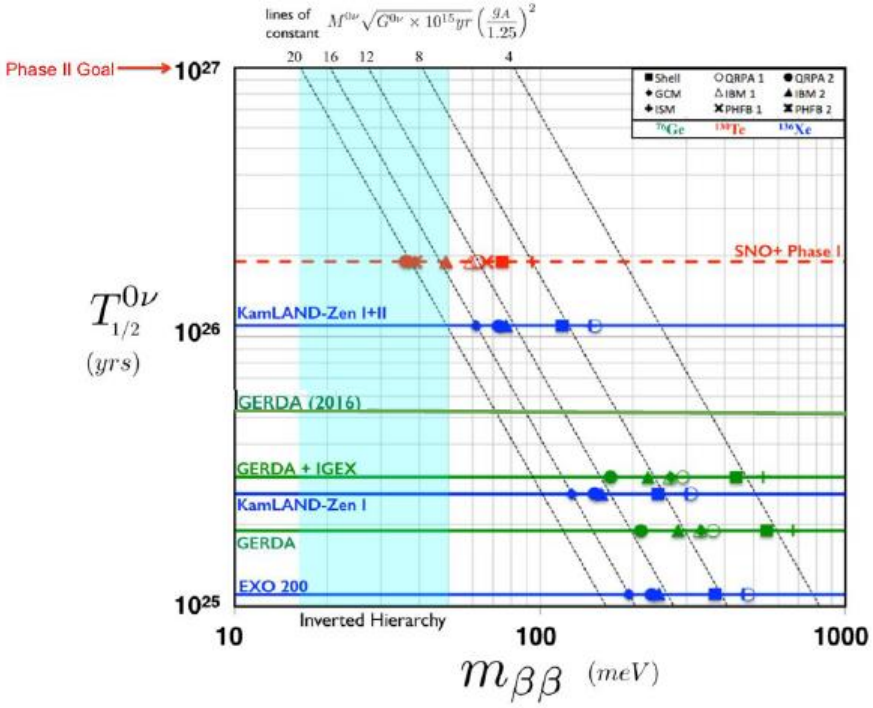
**SNO+ experiment is in construction to replace the heavy water with an organic liquid (Linear Alkyl Benzene) loaded with Tellurium-organic compound. Tellurium is an ideal element to observe "neutrino-less double beta decay" a very rare radioactive process that will test whether neutrinos are their own anti-particles and if so, could tell us the absolute mass of all neutrino types. This is relevant to theories where neutrinos have a strong role in the conversion of anti-matter to matter in the early Universe.**

# Neutrinoless double-beta decay



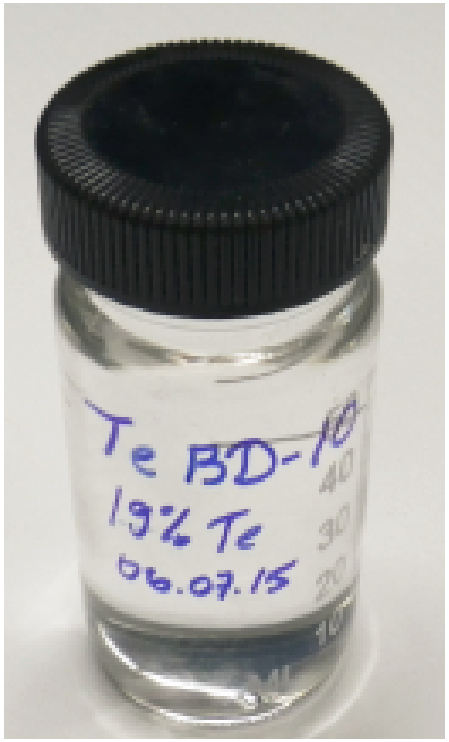

*S. Andringa et al.* (The SNO+ Collaboration), *Current Status and Future Prospects of the SNO+ Experiment*, Advances in High Energy Physics, Volume 2016, Article ID 6194250, 21 pages

# Loading Tellurium into SNO+ Scintillator

- 780 tonne detector and high $^{130}$Te isotopic abundance gives large isotope mass:
  - 0.5% (w/w) Te in Phase 1 is 3.9 tonne of Te or 1330kg of $^{130}$Te;
  - Could increase to percent-level loading in future phases;

- Tellurium can be dissolved into **Linear Alkyl Benzene** (LAB) scintillator as an organic tellurium compound (TeBD)

**Linear Alkyl Benzene**




**TeBD & LAB mixture**

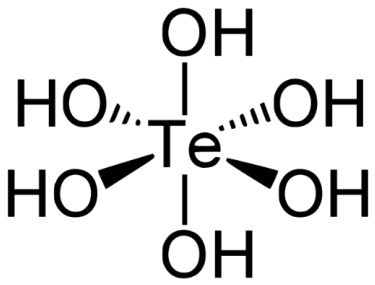
# TeBD Synthesis

## The Polyol-Tellurate Complex Formation Reaction

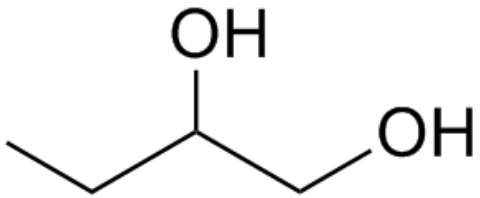
I. Thermodynamics of Telluric Acid Ionization and of Complex Formation

By Herbert R. Ellison, John O. Edwards and E. A. Healy

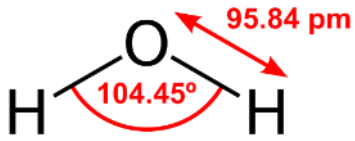
Received April 3, 1961




Telluric acid




1,2-butanediol

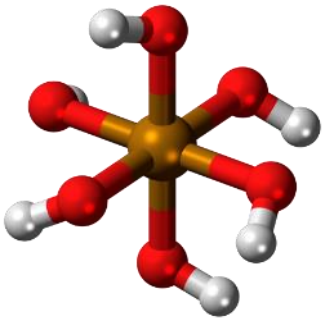


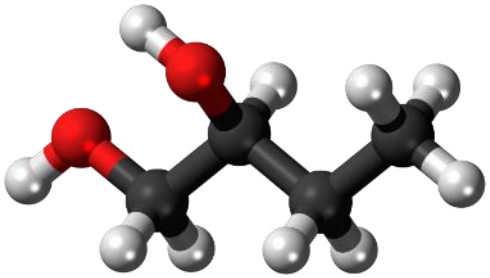

$\Delta$

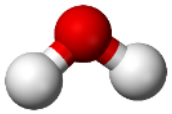
$H_2O$

\+

TeBD Complex







(Dissolved in LAB)

Oxford University

# Telluric Acid Purification

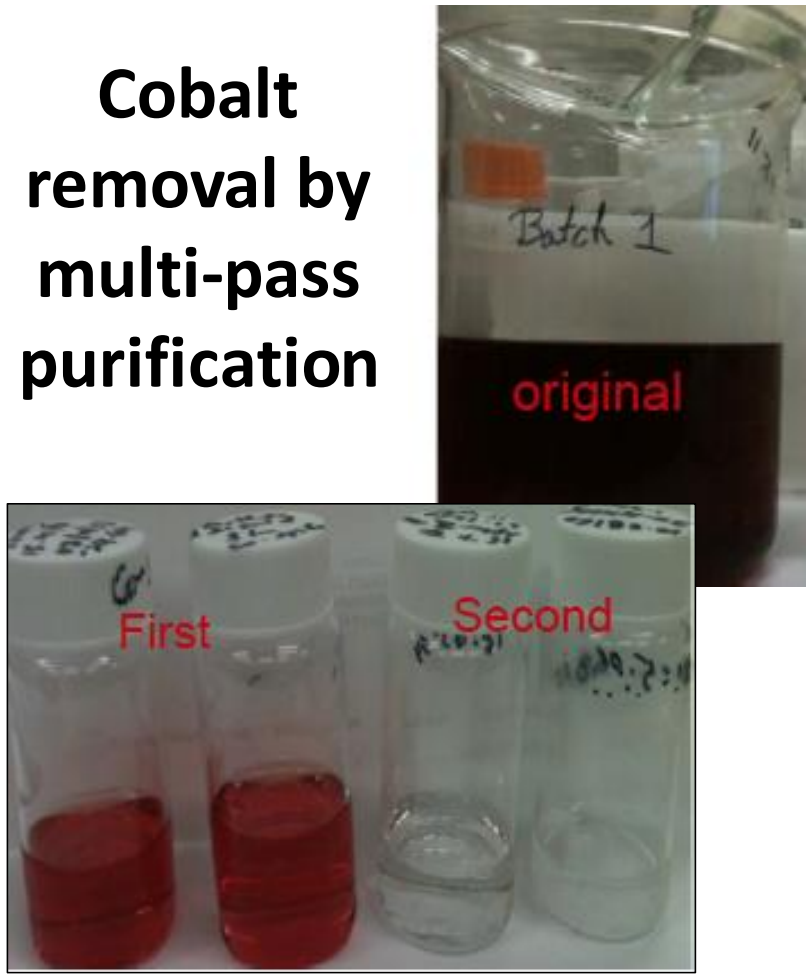
- Telluric acid obeys the following equilibrium:

$$Te(OH)_6 \leftrightarrow Te(OH)_5O^- + H^+$$

Insoluble Soluble

  - pH determines the equilibrium state
- Purification basics:
  1. Dissolve telluric acid in water and filter it
     - Removes water insoluble impurities
  2. Add nitric acid to force the telluric acid to recrystallize/precipitate, pump away the liquid, rinse
     - Removes acid soluble impurities
- By "tuning" the process pH's, this can be quite specific to telluric acid – most other chemicals are removed with high efficiency

**Cobalt removal by multi-pass purification**




See S. Hans et. al. NIM A **795**:132-139 (2015).

# TeBD Synthesis Scaleup Roadmap

**Step 1:**
~8g TeBD synthesis, ~30% [Te];

Scaling Factor (X20)

**Step 2:**
~160g TeBD synthesis, ~33% [Te];

Scaling Factor (X10)

**Step 3: Prototype**
~1,600g TeBD synthesis, ~33% [Te];

Scaling Factor (X150)

**Step 4: UG Plant**
TeBD production matches Te purification;

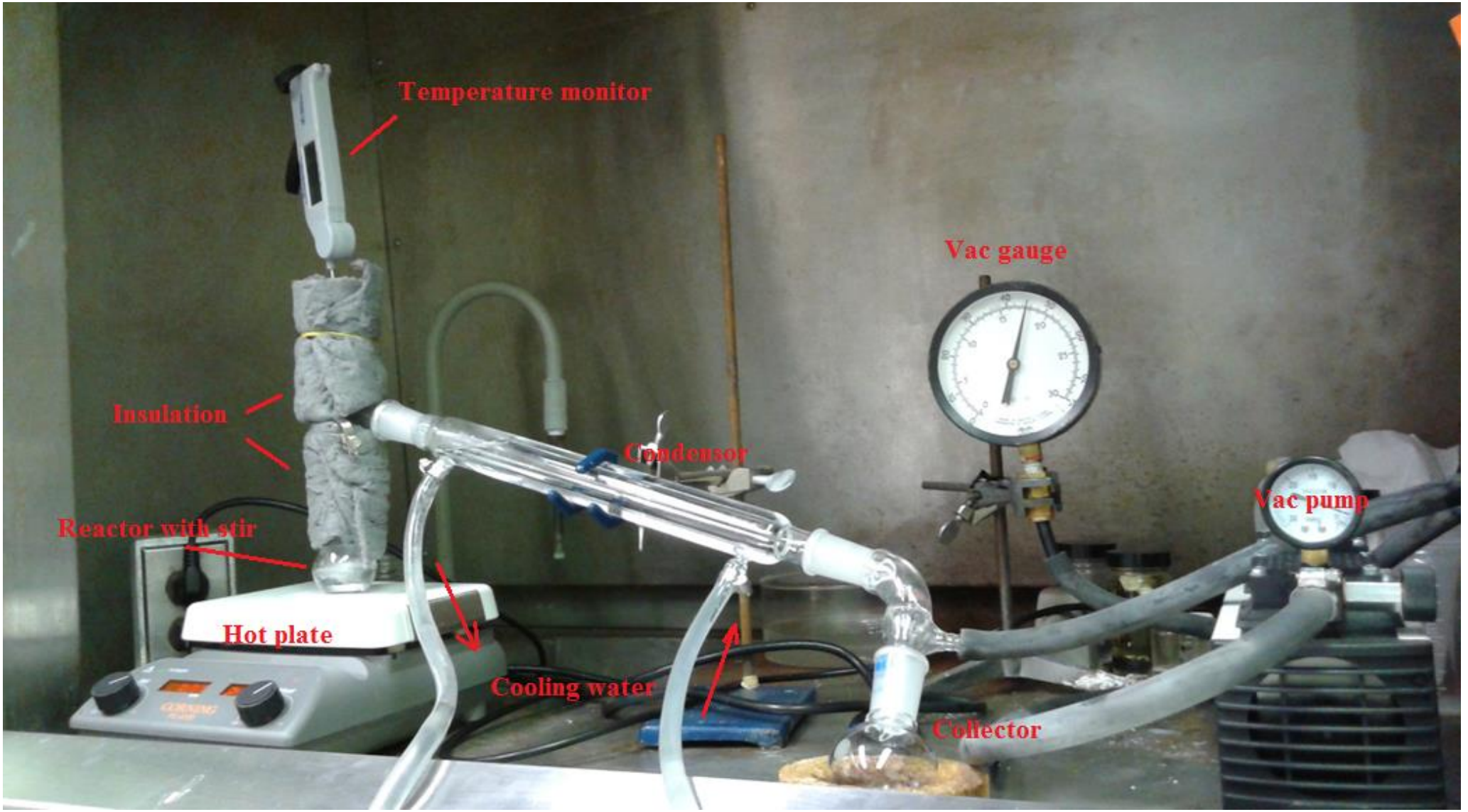
# **Step 0:** TeBD Open-system Synthesis;

Early synthesis without precise control of process parameters

**Main Challenges:**

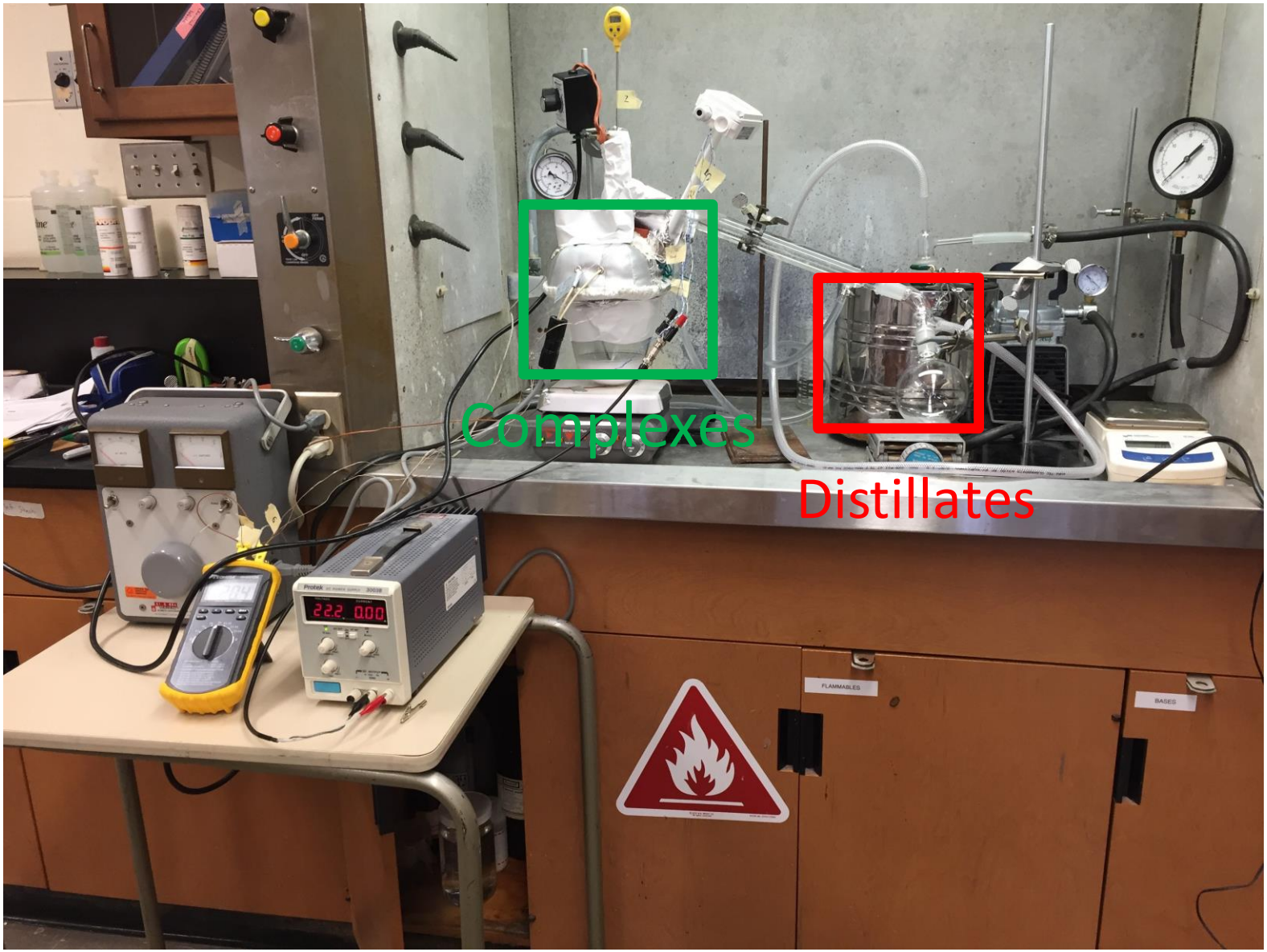
- Very limited literature available;
- Reaction mechanism, pathway, and parameters;
- Lab testing systems & scaling up potentials;

# Step 1: ~8g TeBD Closed-system Synthesis



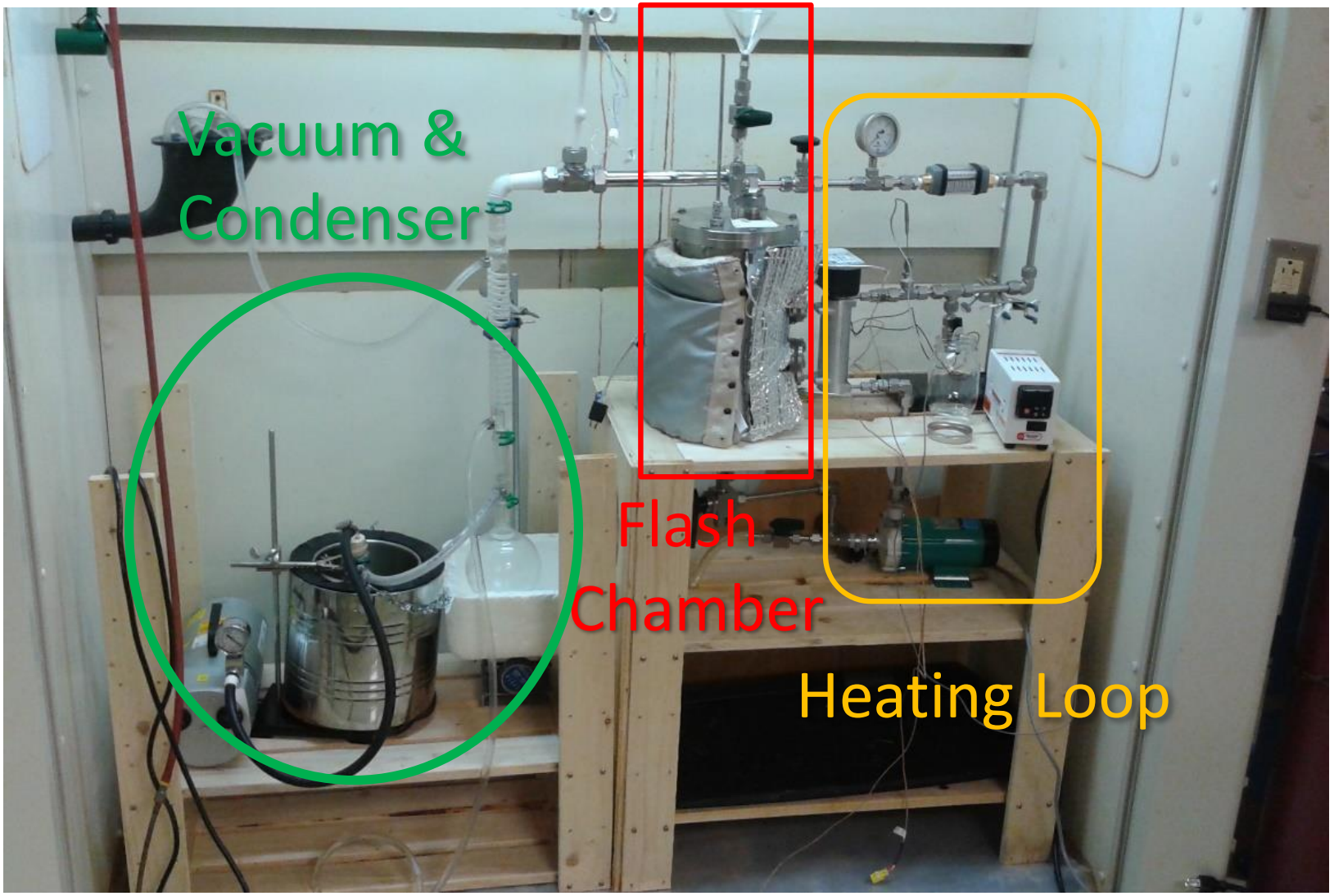

- Closed-system synthesis: More practical for UG production;
- Parameters are investigated for process development & scale-up;
- Duplicated at different institutes, detailed SOP was developed;

# Step 2: ~160g TeBD Closed-system Synthesis




- Scale-up study, also to meet analytical testing demand;
- Modification on temperature/pressure control;
- With ‘Endpoint’ indications;

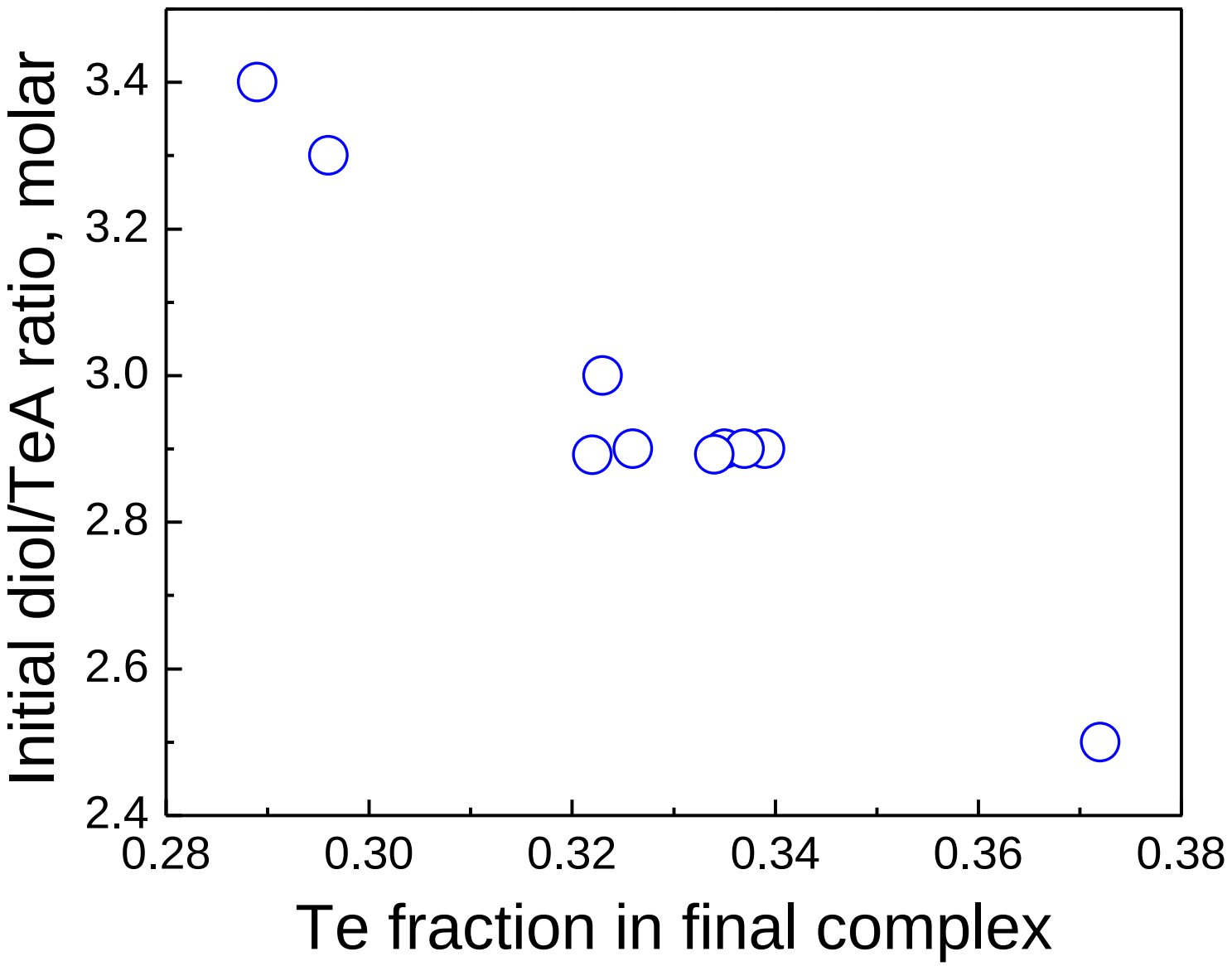
# Step 3: ~1.6kg TeBD Synthesis Prototype




6g TeBD
+
6g LAB

- "Flash chamber" with pressurized heating loop;
- Demonstrated robust synthesis parameters;

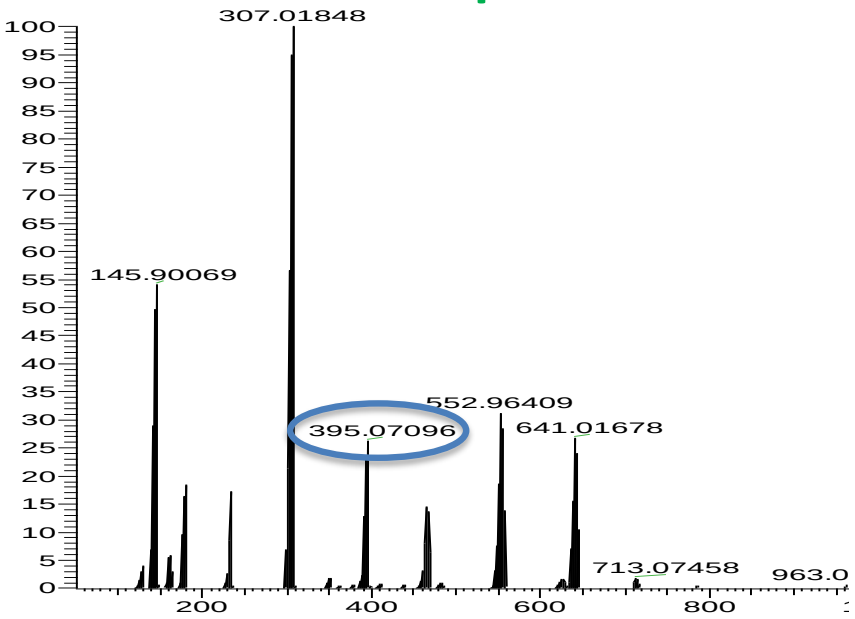
# TeBD Synthesis Parameters

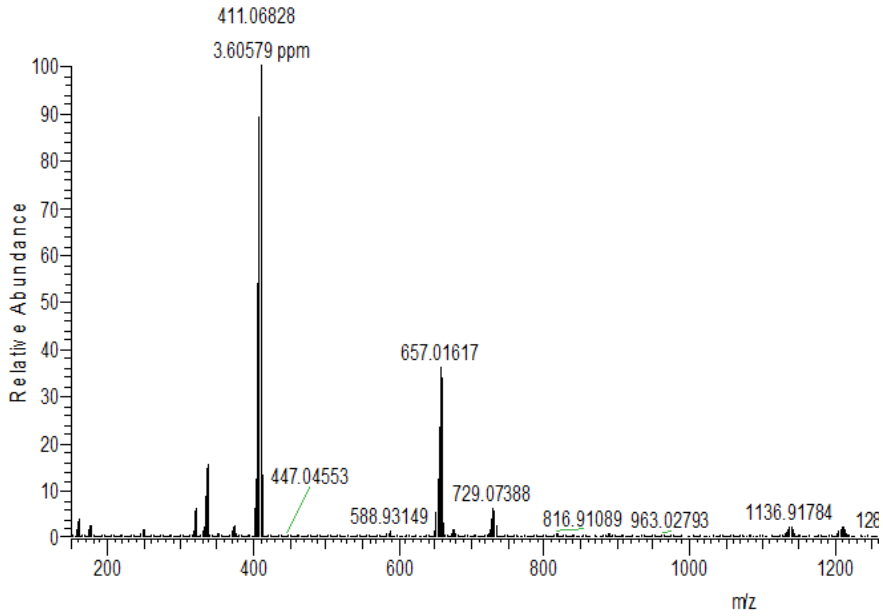


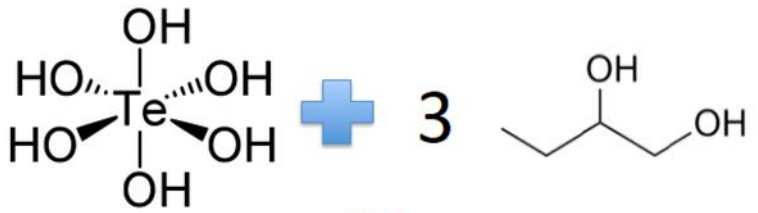

- An example of systematic study of synthesis parameters.
- TeBD complexes from above batches can be dissolved in LAB.

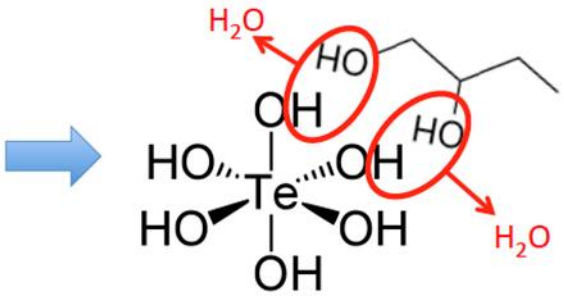
# Understand of the Synthesis Process

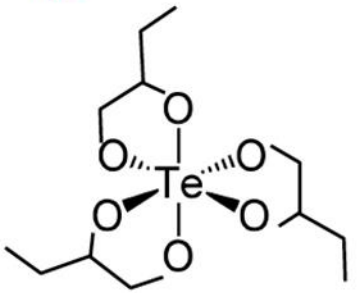
## Mass Spectrometry of TeBD Complexes



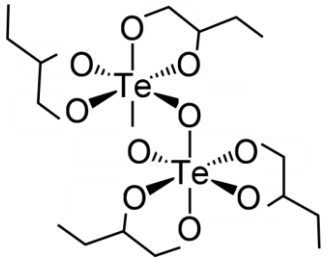



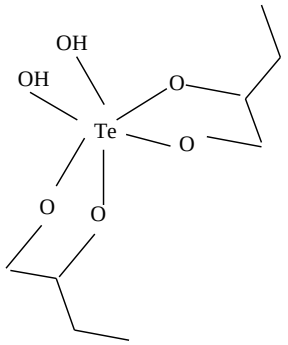




+ 6 $H_2O$

Also show:

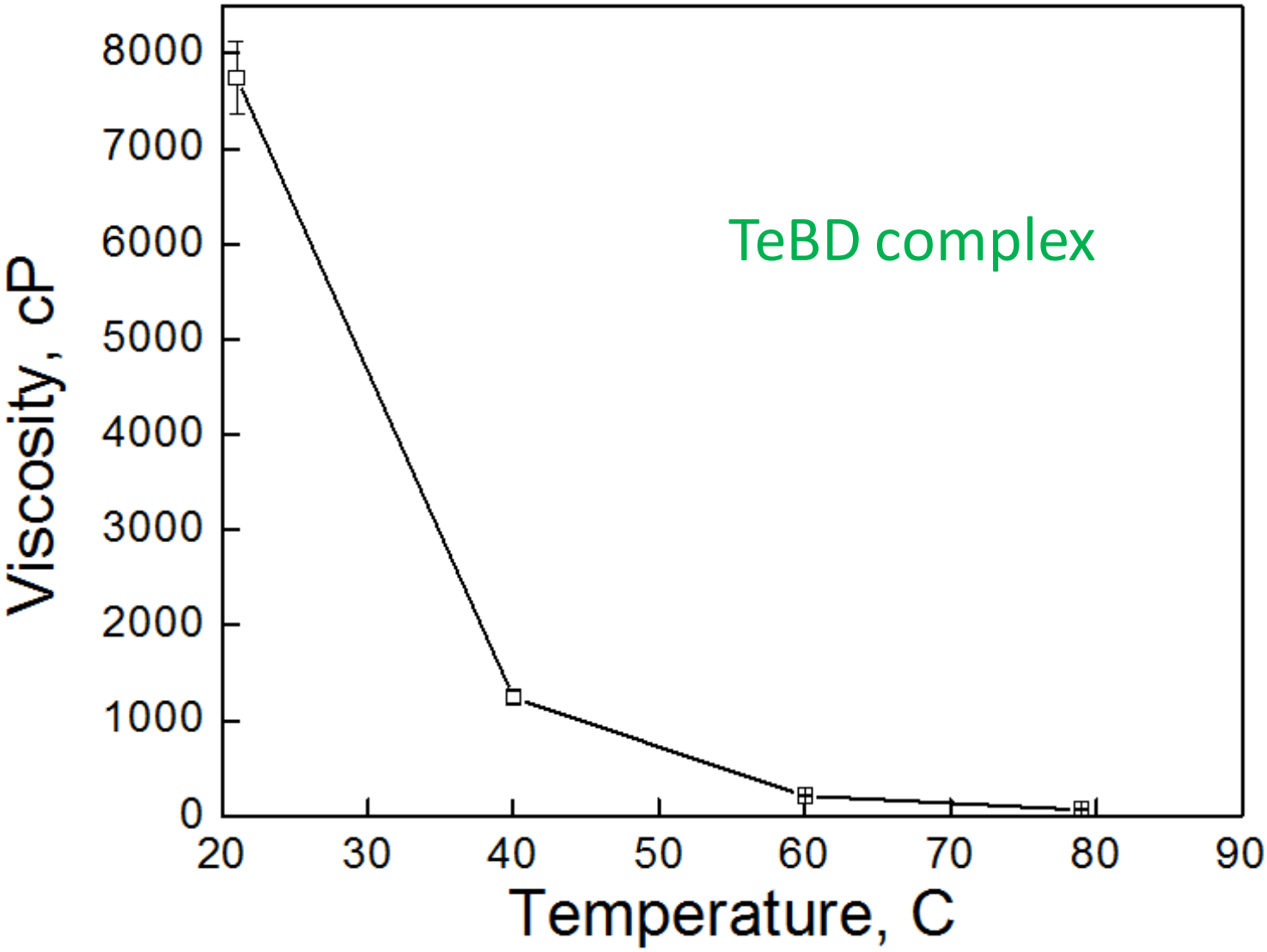



etc.

# Understand of the Synthesis Process

## Viscosity of TeBD, TeBD w/ LAB

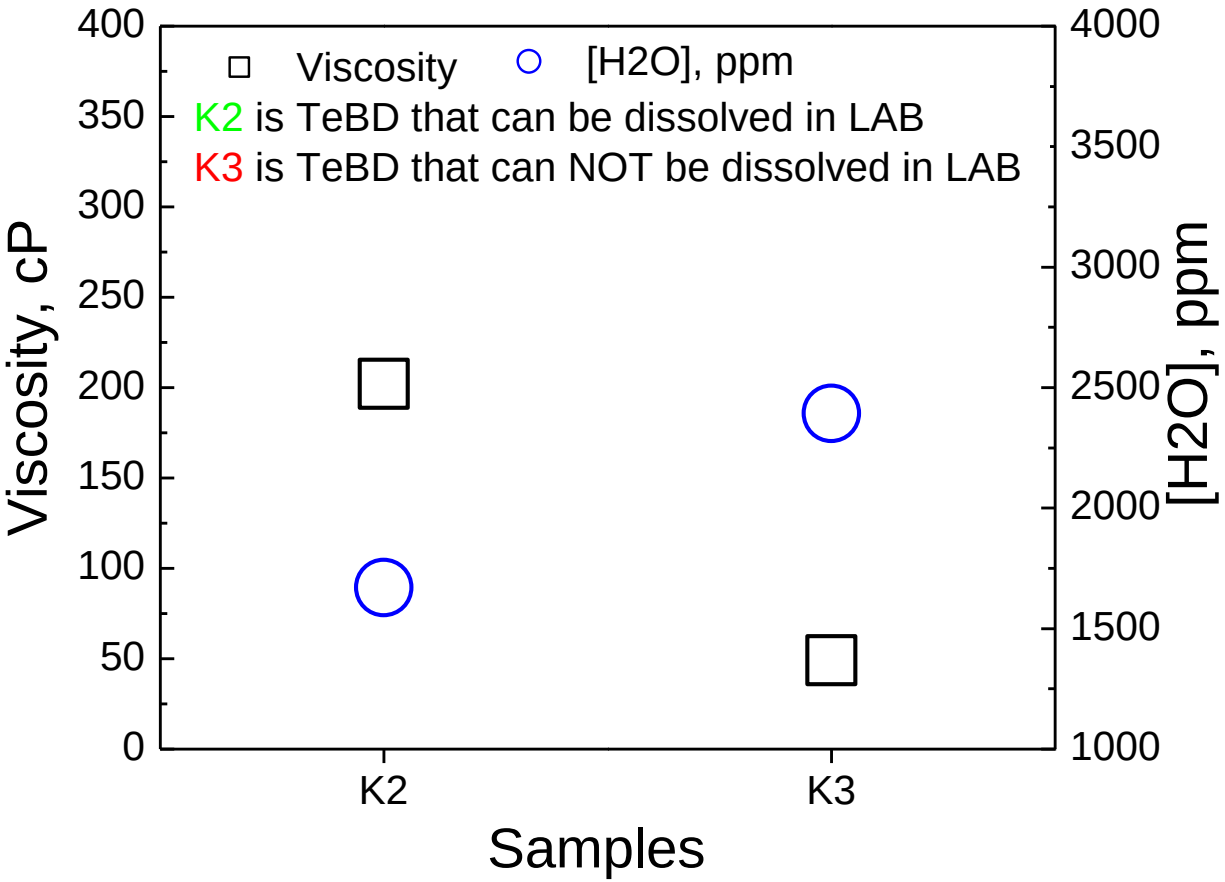



Diluted TeBD complex with LAB (~15%Te); (~22cP @ 21C)

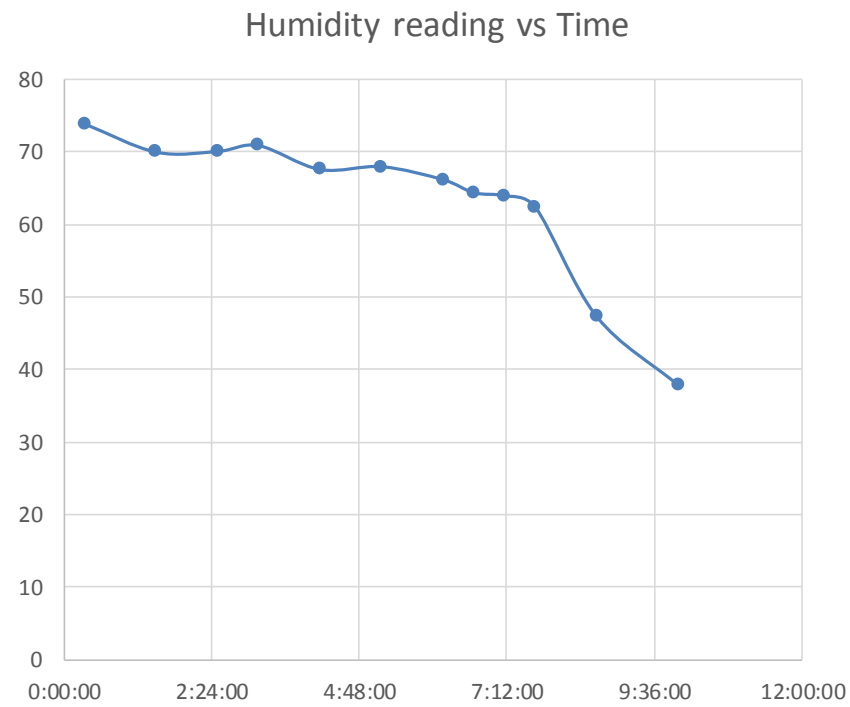
Diluted TeBD complex with LAB (~0.5%Te); (~5cP @ 21C)

# TeBD Synthesis 'Endpoint' Indication

## Humidity, Viscosity, Sample mixing, etc.




$[H_2O]$ data from Karl Fischer Titration



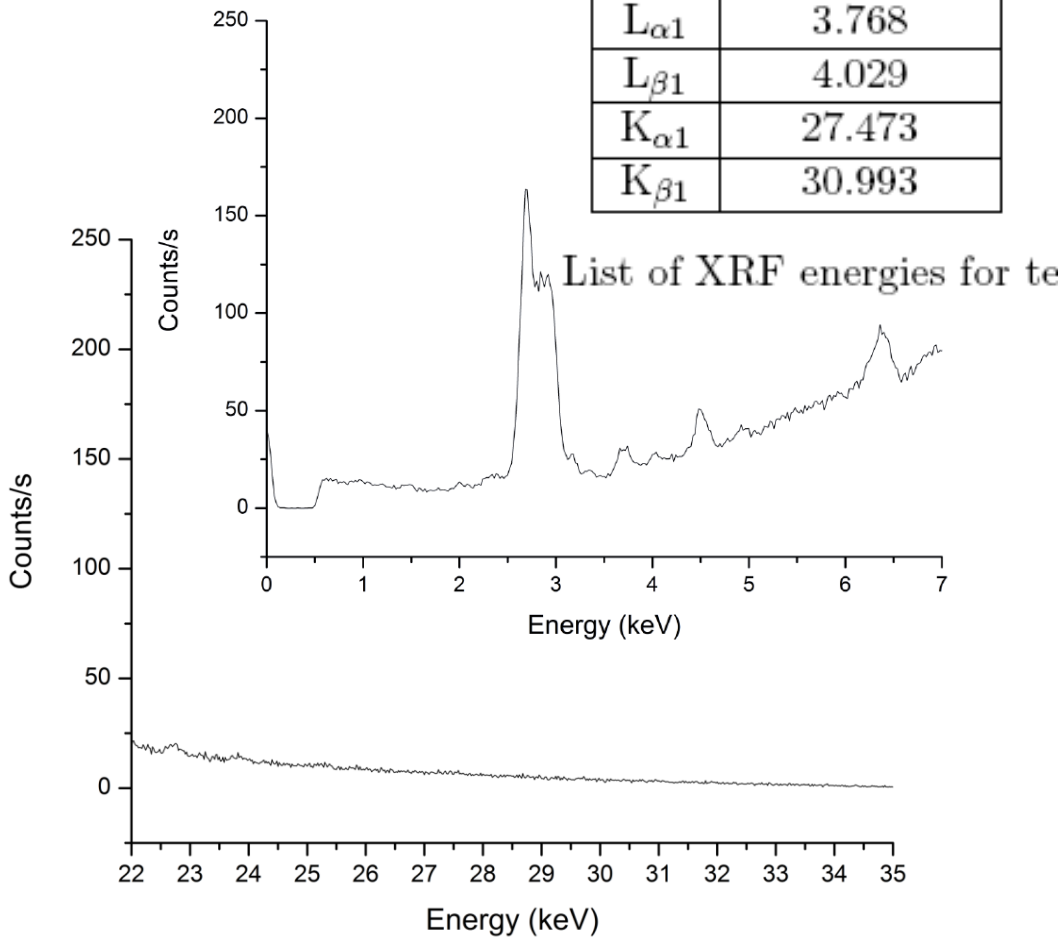

Humidity curve from actual synthesis run

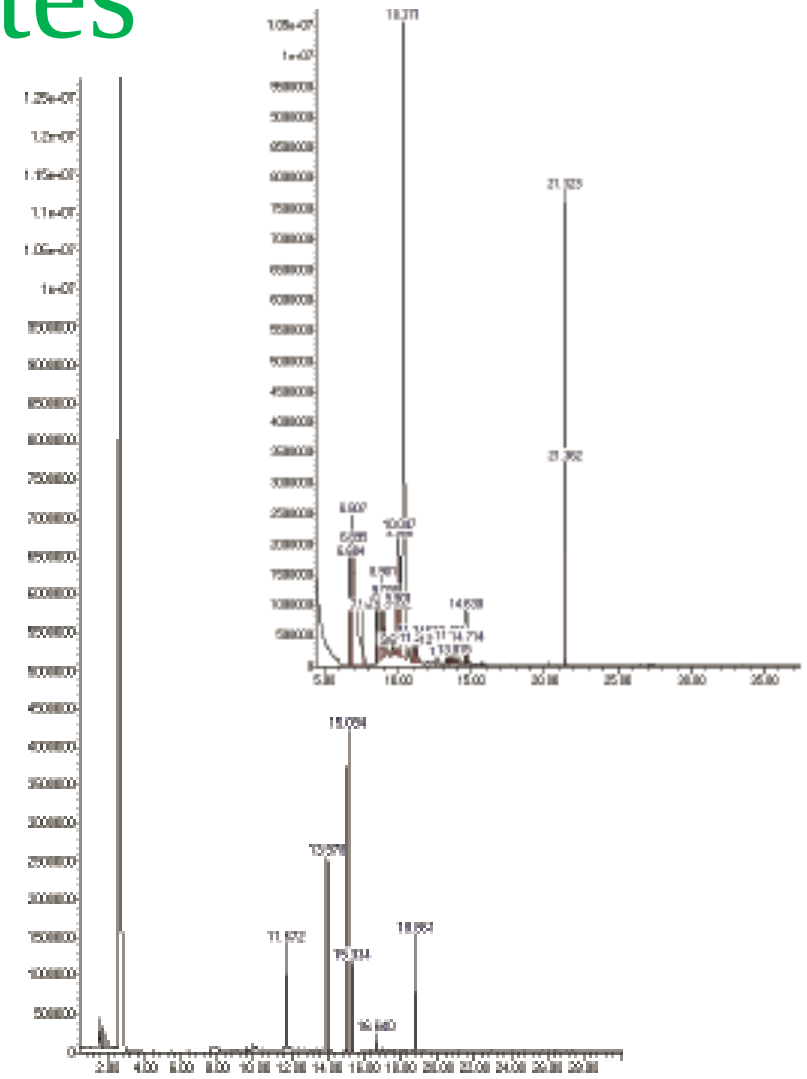
# Understand of the Synthesis Process

## Distillates

| Line | Energy(keV) |
|---|---|
| $L_{\alpha 1}$ | 3.768 |
| $L_{\beta 1}$ | 4.029 |
| $K_{\alpha 1}$ | 27.473 |
| $K_{\beta 1}$ | 30.993 |

List of XRF energies for te



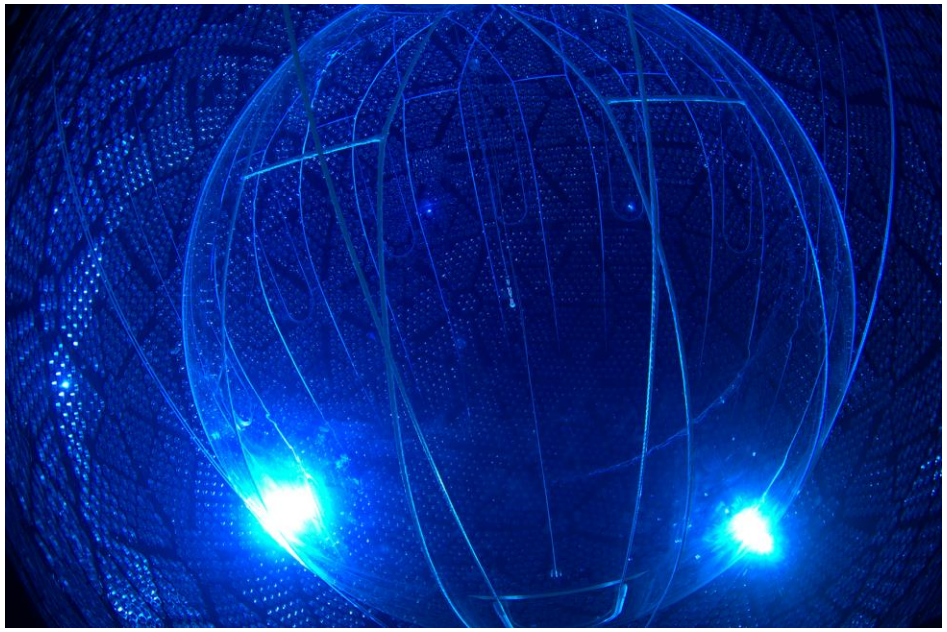



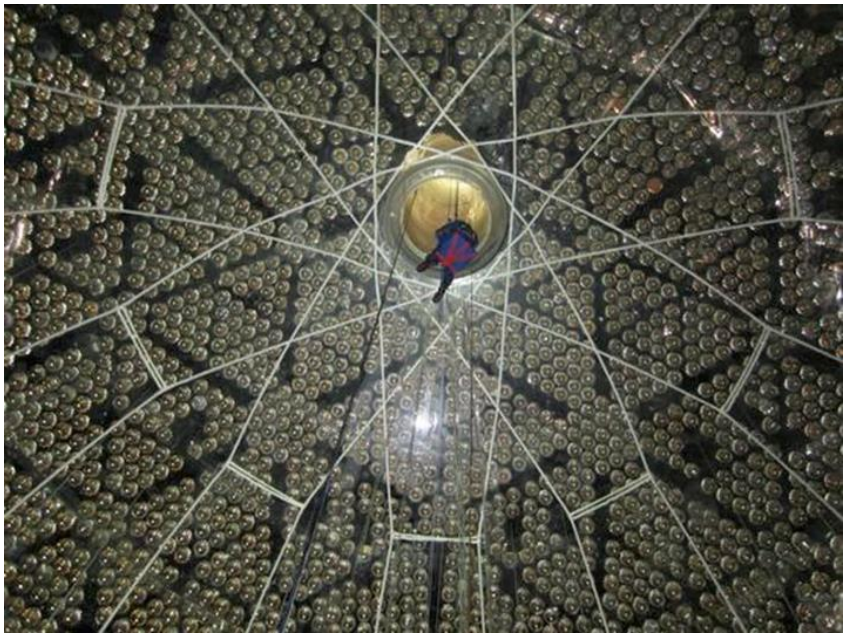
[Te] is under detection limits.

By-products can be identified by GCMS.

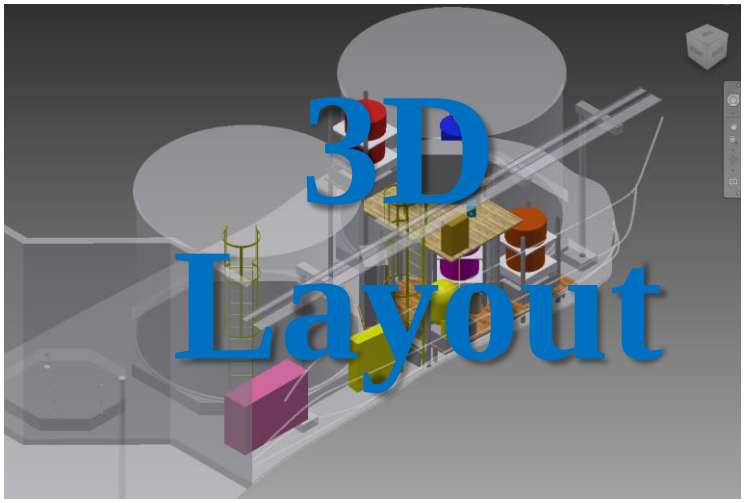
# Step 4: Underground TeBD Plant

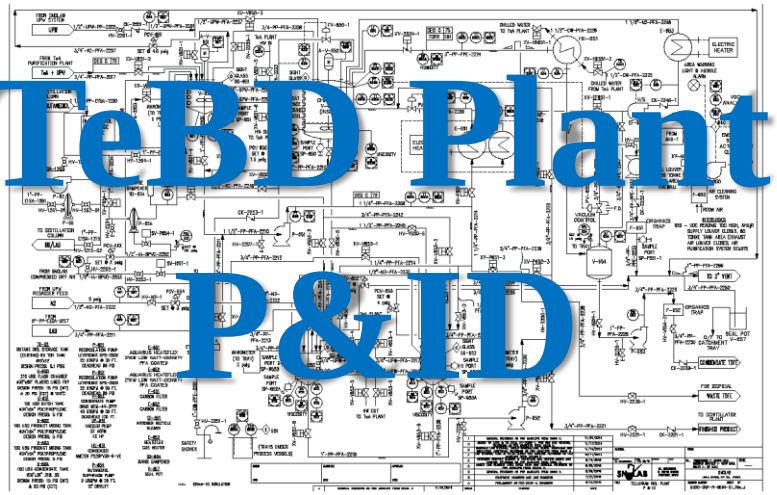
- The underground TeBD synthesis plant will be designed to produce tellurium organic compounds at a 250-kg batch scale (~85 kg tellurium per batch);
- To achieve the 0.5% Te loading in the scintillator, about 45 batches will need to be processed.





# TeBD Synthesis Plant

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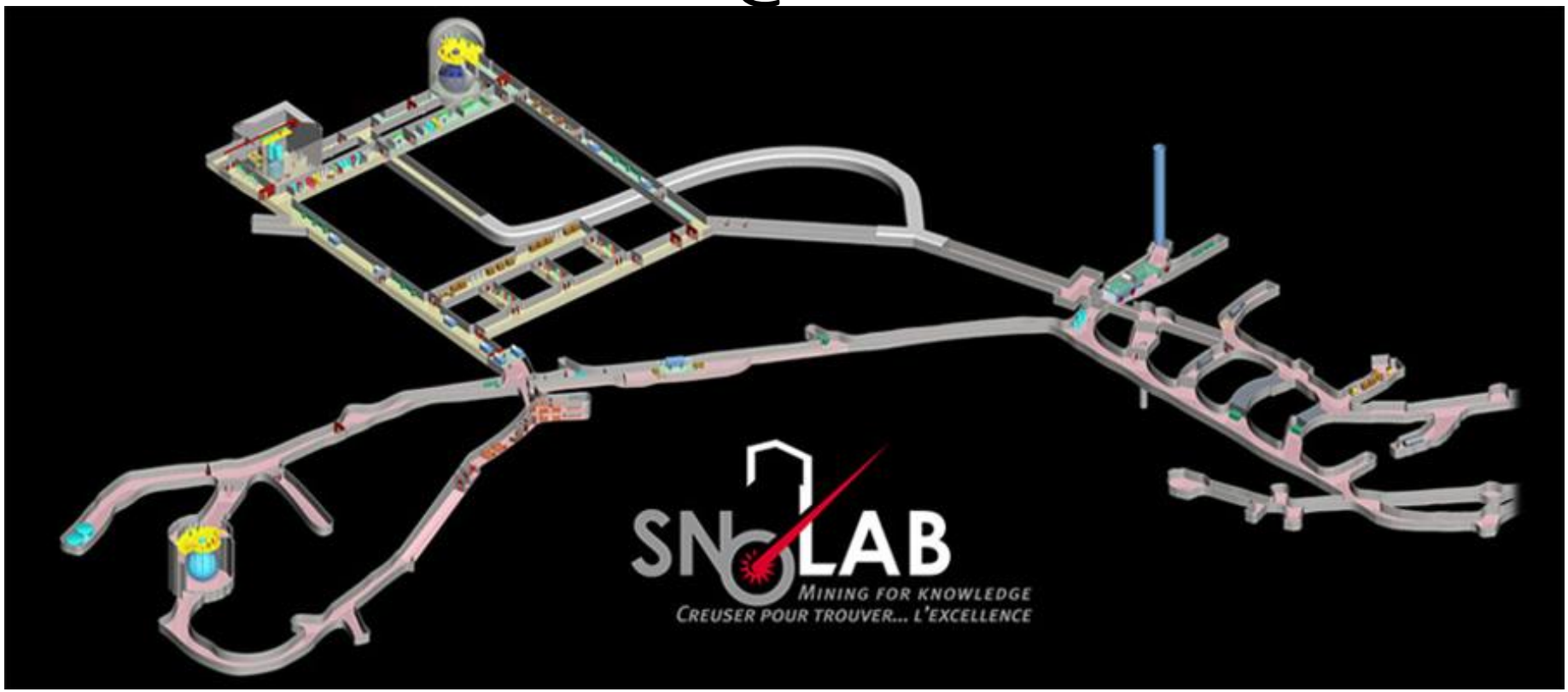

# Summary

- SNO+ is a project that is a follow-up to SNO;
- The primary objective of SNO+ is to search for the neutrinoless double-beta decay of $^{130}$Te;
- Liquid scintillator is made from linear alkyl benzene;
- The detector will be loaded with tellurium:
  - 0.5% natural tellurium in 780 tonnes of liquid scintillator (1330 kg of $^{130}$Te);
  - Te loading technology development and scaleup;
  - Underground (2km deep) TeBD synthesis plant;