第一届中微子、原子核物理和新物理研讨会(νNN2025)

Development and Progress of Te-Doped Liquid Scintillators for 0νββ Detection

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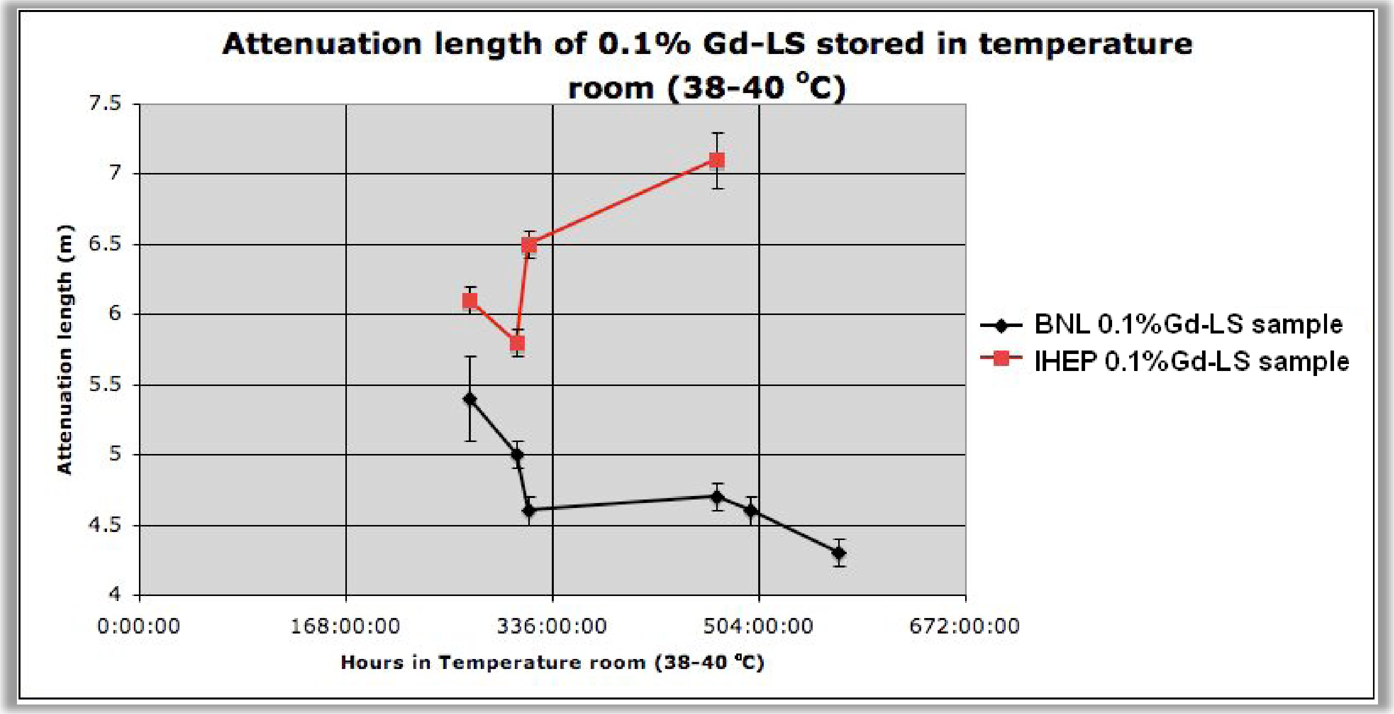
Liquid Scintillator research @ IHEP: A Historical Overview

- **2005**： Initiating doped liquid scintillator R&D, focusing on Gd-LS for Daya Bay Neutrino Experiment
- **2007**： Daya Bay Collaboration officially adopts IHEP’ s Gd-LS recipe *(NIM A 2008, 584: 238-243)*
  - Decision Basis: Stability tests of Gd-LS AL degradation at high-temperature conditions conducted and independently verified by Third-Party Lab (HKU)



Liquid Scintillator research @ IHEP: A Historical Overview

- **2010.10~2011.1**： Successful production of 185 t Gd-LS (Served as Chemistry Lead)

NIM A, 763(2014)82–88

Liquid Scintillator research @ IHEP: A Historical Overview

- The Daya Bay Experiment successfully concluded data collection, made breakthrough achievements and garnered multiple distinguished honors
- **Gd-LS fulfilling its mission with Outstanding Performance**

2013 CAS Outstanding Science and Technology Achievement Prize, 13/20

Daya Bay Reactor Neutrino Experiment Team - Medal of the National Innovation Award (2017)，14/16

Second Prize of Beijing Science and Technology Award (2015), 2/10

Award-Winning Project: Development of Gadolinium-Doped Liquid Scintillator for the Daya Bay Neutrino Experiment

- Other Applications of Gd-LS：
  - Sold 2.56 tons Gd-LS to a Russian research institute for USD 65,000
  - Neutron background measurement in JinPing underground Laboratory , China Institute of Atomic Energy
  - Provided technical consulting services on Gd-LS property measurement for Tsinghua University, along with technical support during the development of neutron detectors.

Liquid Scintillator research @ IHEP: A Historical Overview

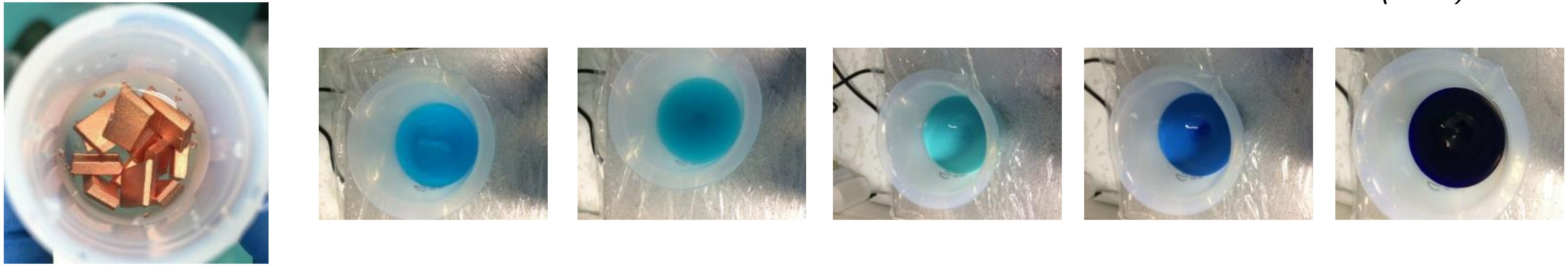
- **2014 till now**: JUNO LS, in charge of ultra-high-purity raw materials for 20 kt LS as well as other chemical-related research for JUNO
  - **60 t PPO** (2,5-Diphenyloxazole), **72kg bis-MSB** (1,4-bis(2-methylstyryl)benzene), **…**
    - Completed chemical supply according to the JUNO schedule, with quality far exceeding expectations
    - Optical purity and Th&U reaches internationally leading level, Th(U) content is several orders of magnitude lower than comparable samples
    - Can be directly used in LS mixing to meet the requirements of JUNO
    - The superior quality of ton-scale PPO earned the Third Prize of Hubei Provincial Science and Technology Progress Award (2022, 3/9)

MIN A 1041 (2022) 167323

Liquid Scintillator research @ IHEP: A Historical Overview

- **2016-2018**: Conduct another research on low-background measurement techniques for ultra-low background detectors
  - Developed a co-precipitation approach to measure trace amount of U(Th) in copper to **sub-ppt level** for the EXO collaboration

NIM A 941 (2019) 162335



- **2017**: Initiate Te-LS Research

Motivation for Te-LS: JUNO's potential on 0νββ searches

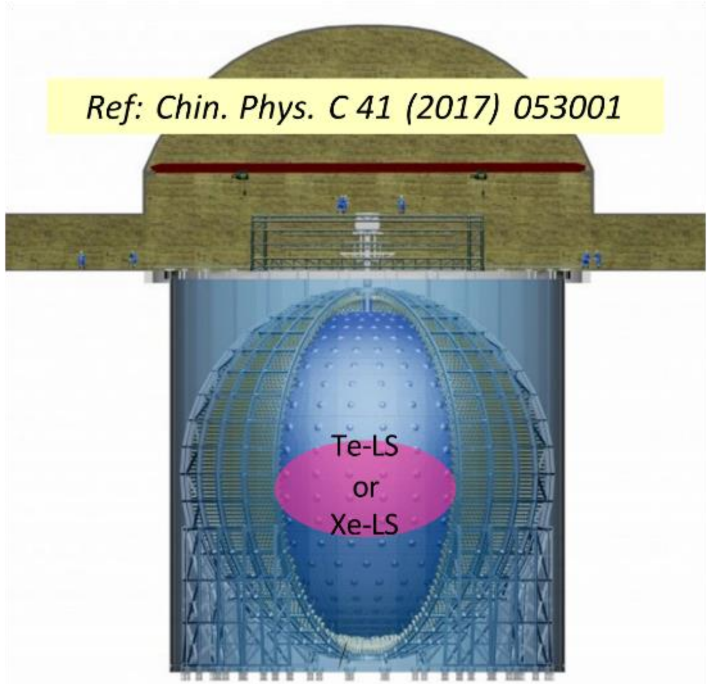
- **Future Prospects:** JUNO CD could be utilized to explore 0νββ in future studies.

  Potential to explore normal mass ordering parameter space of Majorana neutrino mass (~ meV)

- **Research Foundation:**
  - Our team possesses over a decade of expertise in:
    - Developing doped liquid scintillators
    - Investigating ultra-low-background raw materials
- **Strategic Significance:**

  Conducting Te-LS research at IHEP holds both:
  - Substantial technical groundwork
  - Important scientific value

- JUNO offers a unique opportunity to search for 0νββ after completion of mass ordering measurements (~2030)
  - Large target mass: 20 kton → LS 100-ton scale isotope (Te or Xe) loading
  - Ultra-pure LS shielding → Low background
  - Energy resolution < 3% @ 1 MeV



Concept of the experiment

Research History of Te-LS based on JUNO LS recipe at IHEP

- **Phase I: Exploratory Stage (2017-2019)**
  - Initiated experimental studies on Te-doping technology for LS
    - 2017: inorganic Te + surfactant
    - 2018: liquid-liquid extraction
    - 2019: organic Te-diol compounds
- **Phase II: Technical Breakthroughs (2020-2024)**
  - 2020: Developed an azeotropic distillation approach, enabling >5% Te mass loading in LS
  - 2021: Abs at 430 nm remains essentially unchanged when doped with 0.6% Te
  - 2022~2023: A novel room-temperature synthesis approach was established to decrease safety risks
  - 2024: Established synthesis and purification protocol for Te-LS production
- **Phase III: Application-Oriented Development (2025-present)**
  - 2025: Deployed 100 kg Te-LS demonstration at IHEP
  - Lab studies: optimization of light yield, long-term stability test (>3 years), improve Te-LS transparency characterized by AL, develop methods for removing radioactive impurities, …
  - 20 t Te-LS demonstration …

Te-LS @IHEP

Te-LS for 0νββ: An internationally recognized challenge

Excep. **high** Te-doping conc.(3~5%)； **No Impact** on LS Optical Perf.， **Ultra-low** rad. background， High-quality retention **>10 years**

- × Liquid-Liquid Extraction
- ✓ Organic Te-compound
- ×surfactant
- × Inorganic Te nanoparticles

TA + 1,2-hexanediol

Technical Route 1: Azeotropic distillation approach

Innovation Highlights: water-free reaction → enhanced solubility and stability

Advantages： Te>>5%， Uniform Transparent, and Stable

Patent： *ZL 202011370855.5*

Publication： *NIM A 1049 (2023) 168111*

Technical Route 2: Room temp. Syn. approach

Innovation Highlights:

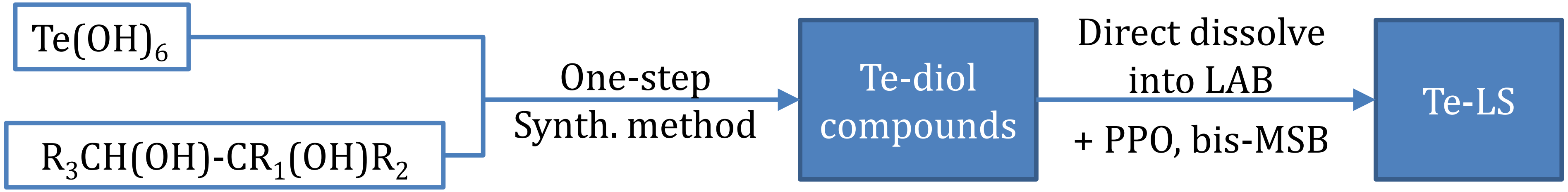
No heating required, R.T. Synthesis

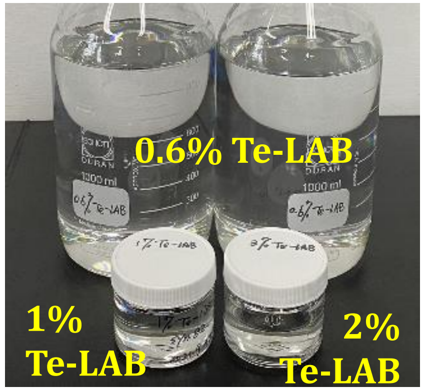
Advantages： Performance unchanged, greener & more scalable

Patent： *applied*

Publication： *in preparation*

Route 1: Azeotropic distillation approach





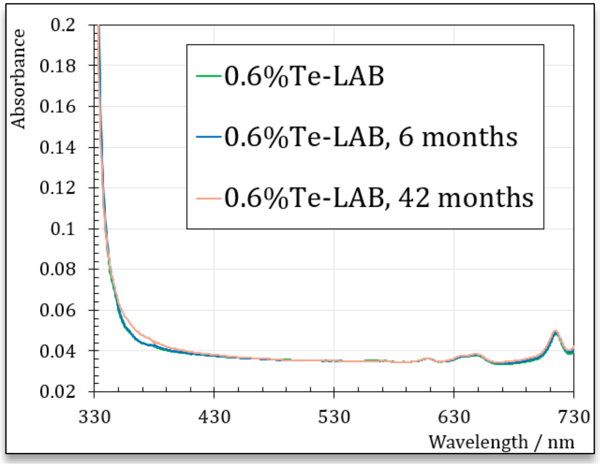
- Characteristic of the approach: Water-free environment for the synthesis
  - No extra water introduced into the reaction system
    - Telluric acid in the solid state was used directly instead of being dissolved in the water
  - Water generated in the reaction was taken out continuously by azeotropic distillation
    - Acetonitrile was chosen as an azeotropic solvent because of its relatively lower boiling point (81.6 ◦C) and higher water content (16%) in azeotrope

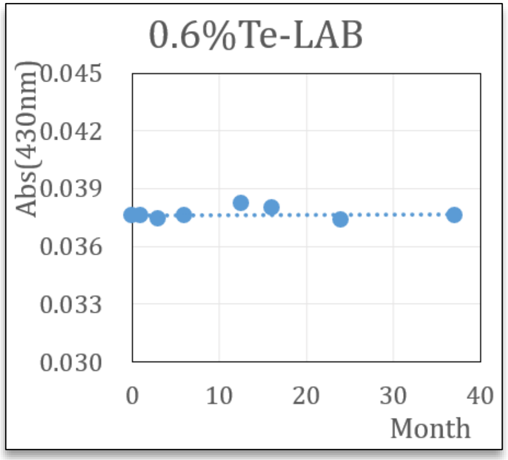
SNO+
TA + 1,2-butanediol, Aqueous-phase heating reaction. Partial Te samples exhibited instability, with Te compounds precipitating upon moisture exposure.
2017 *J. Phys.: Conf. Ser.* **888** 012084

Y. Ding, et al., NIM A 1049 (2023)

Route 1: Azeotropic distillation approach

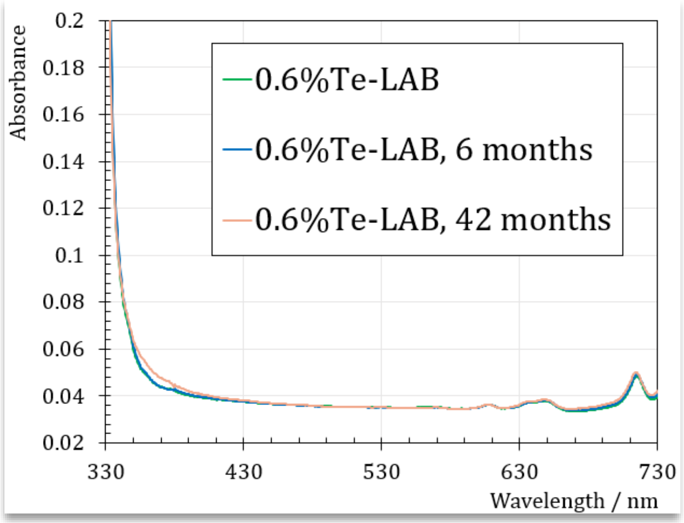
- **Advantages**
  - **Ultrahigh Te Solubility**: Te product is miscible with LAB, capability of Te loading in JUNO LS: > 5%
  - **Exceptionally High Transparency & Long-Term Stability**: 0.6% Te loaded samples shows very limited influence on the absorbance of scintillation solvent around 430 nm and remained the same within the equipment sensitivity (10^{-4}) for 3.5 years
  - **A one-step synthesis**: simple, the product can be used for preparing Te-LS directly without post-processing, easily scalable for mass production
  - **Broad applicability to diverse diols**: a serials of diols with more sophisticated structures can be used since the reaction is greatly facilitated by the removal of water

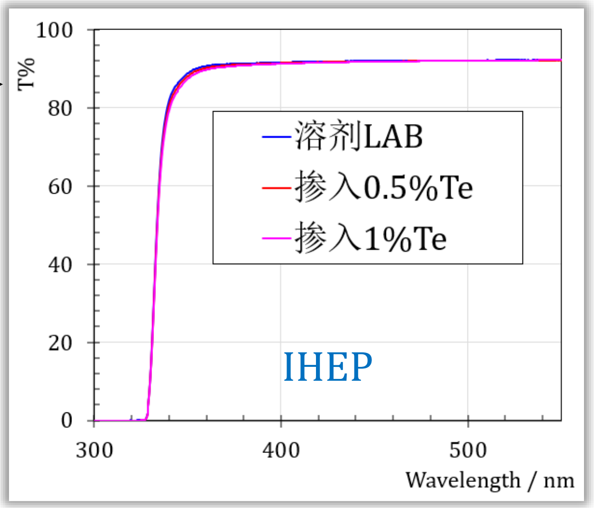


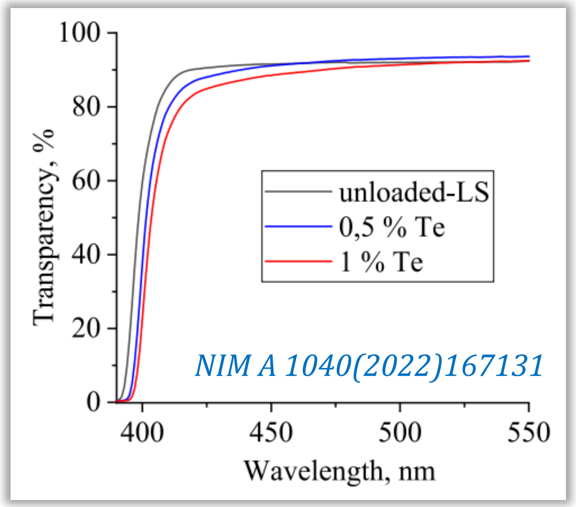


Best diol + Azeo. Dist. Approach +purified raw materials → Ultra-High Optical Quality Te-LS

- **Best-in-Class Transparency Among Peer Samples**
  - 0.6% Te , no visible difference (ΔAbs<0.002 for λ>370 nm) compared to the purified LAB (A.L. > 20m)



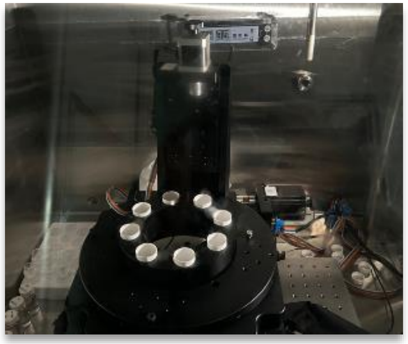




- **Long-term stability**
  - Absorption spectroscopy confirms stability exceeding **3.5 yrs**
  - No comparable data reported internationally

- **L.Y.**:
  Preliminary optimization→Significant enhancement

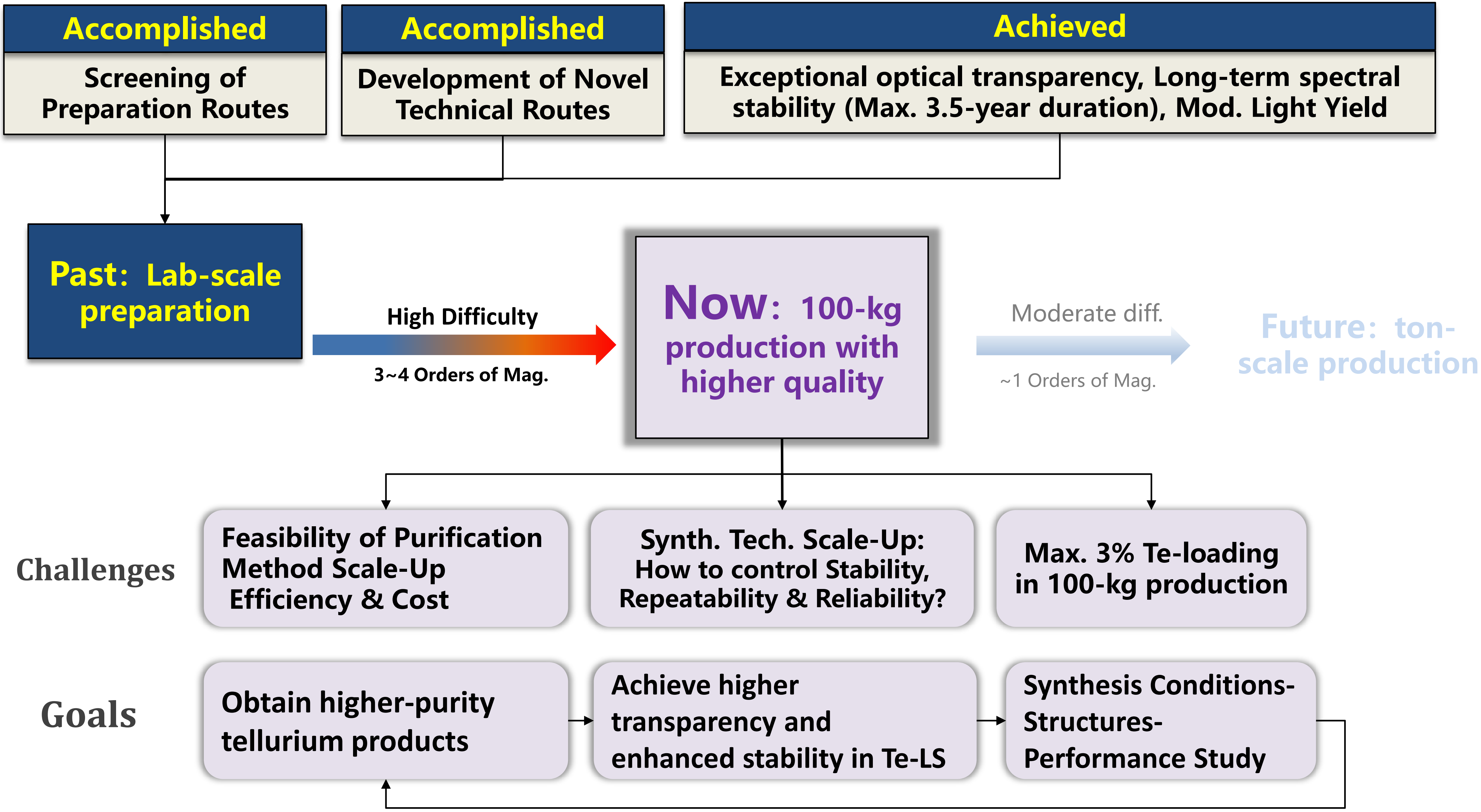
	R.L.Y.
Pure LS	100%
0.5% Te-LS (Before Opt.)	53%
0.5% Te-LS (After Opt.)	**77~83%**



Route 2: Room temperature Synthesis approach

- **Disadvantage** of Azeo. Dist. Approach: safety risks associated with large-scale heating and distillation of low-boiling-point solvents (acetonitrile, 81.6 ◦C)
- Developed a **Room-Temperature Synthesis Approach**
- Characteristic of the approach: one-step synthesis **at room temperature**
- Excellent properties of Te-samples:
  - Exceptional optical transparency (ΔAbs(430nm)/%Te $\leq$ 0.0003)
  - Long-term spectral stability exceeding or approaching 1 year till now for both **1% and 3% Te** formulations
  - Briefly, the Te samples obtained by this method outperform even those produced by the Azeo. Dist. Method in terms of performance
- A green, efficient alternative for large-scale Te-LS production for next-generation neutrinoless double-beta decay experiments

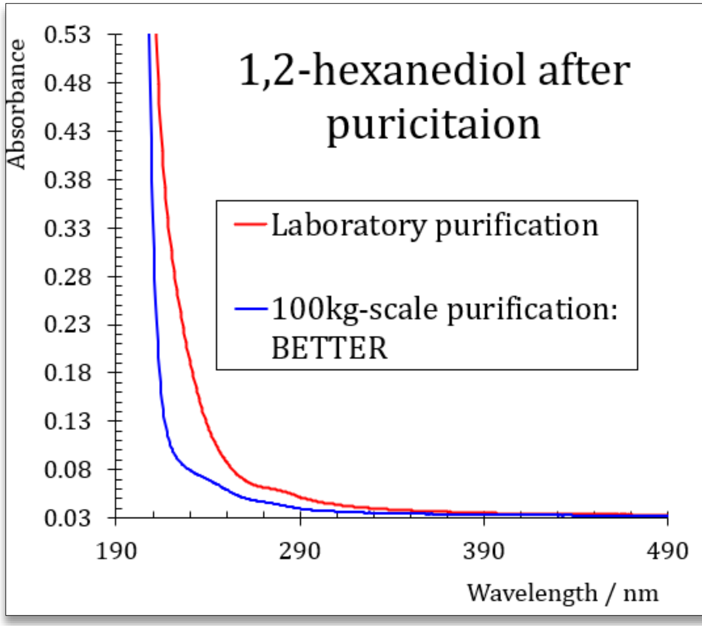
Patent applied, Manuscript to be submitted



Accomplished
Screening of Preparation Routes
Accomplished
Development of Novel Technical Routes
Achieved
Exceptional optical transparency, Long-term spectral stability (Max. 3.5-year duration), Mod. Light Yield
Past: Lab-scale preparation
High Difficulty
3~4 Orders of Mag.
Now: 100-kg production with higher quality
Moderate diff.
~1 Orders of Mag.
Future: ton-scale production
Challenges
Feasibility of Purification Method Scale-Up Efficiency & Cost
Synth. Tech. Scale-Up: How to control Stability, Repeatability & Reliability?
Max. 3% Te-loading in 100-kg production
Goals
Obtain higher-purity tellurium products
Achieve higher transparency and enhanced stability in Te-LS
Synthesis Conditions-Structures-Performance Study

Progress of 100-kg production

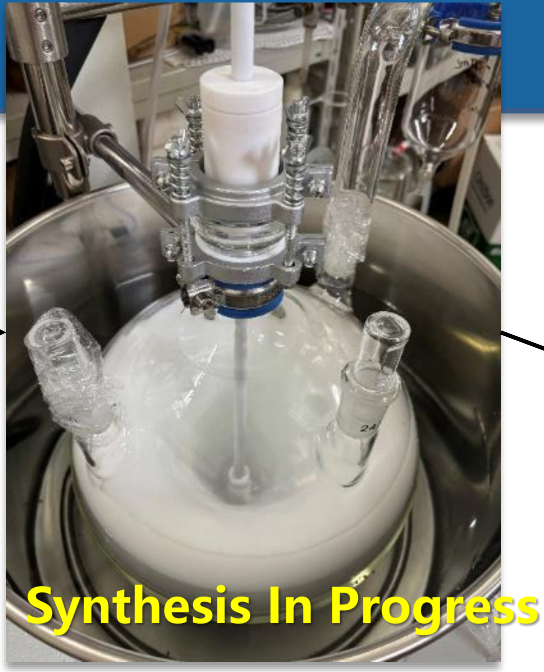
- Bulk purification methods for raw materials with corresponding QC standards and protocols was developed
  - Completed extensive research on optical purification methods for raw materials. Considering both purification efficiency and cost, a combined purification approach was adopted
  - Purification highlight: **Dramatic cost reduction with superior purity compared to expensive lab purification**

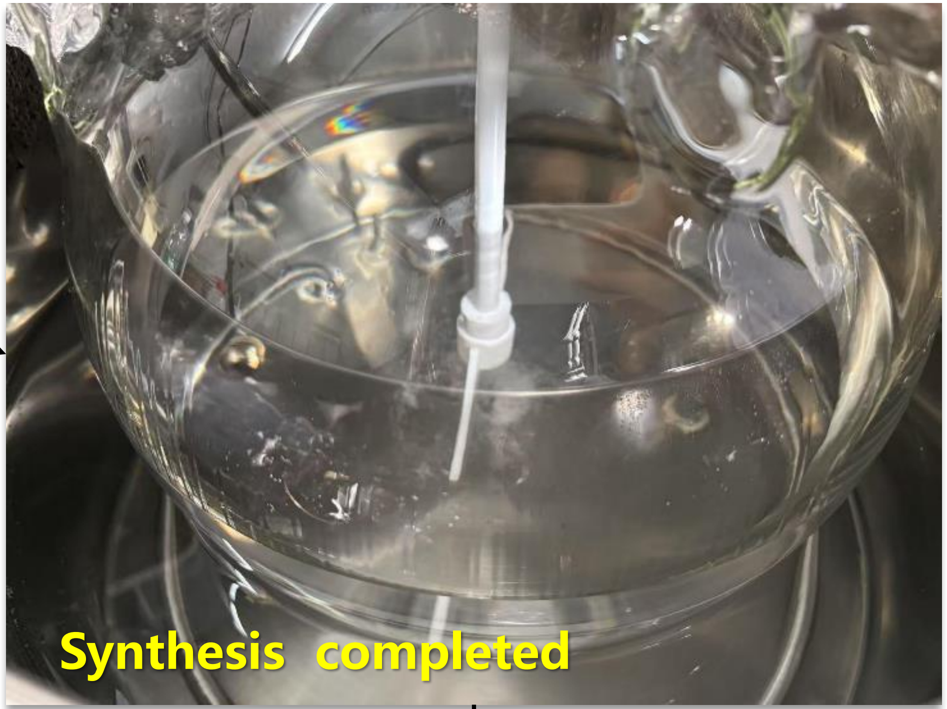


- Dual-Temperature (Room Temp./High Temp.) Reactor was designed and customized

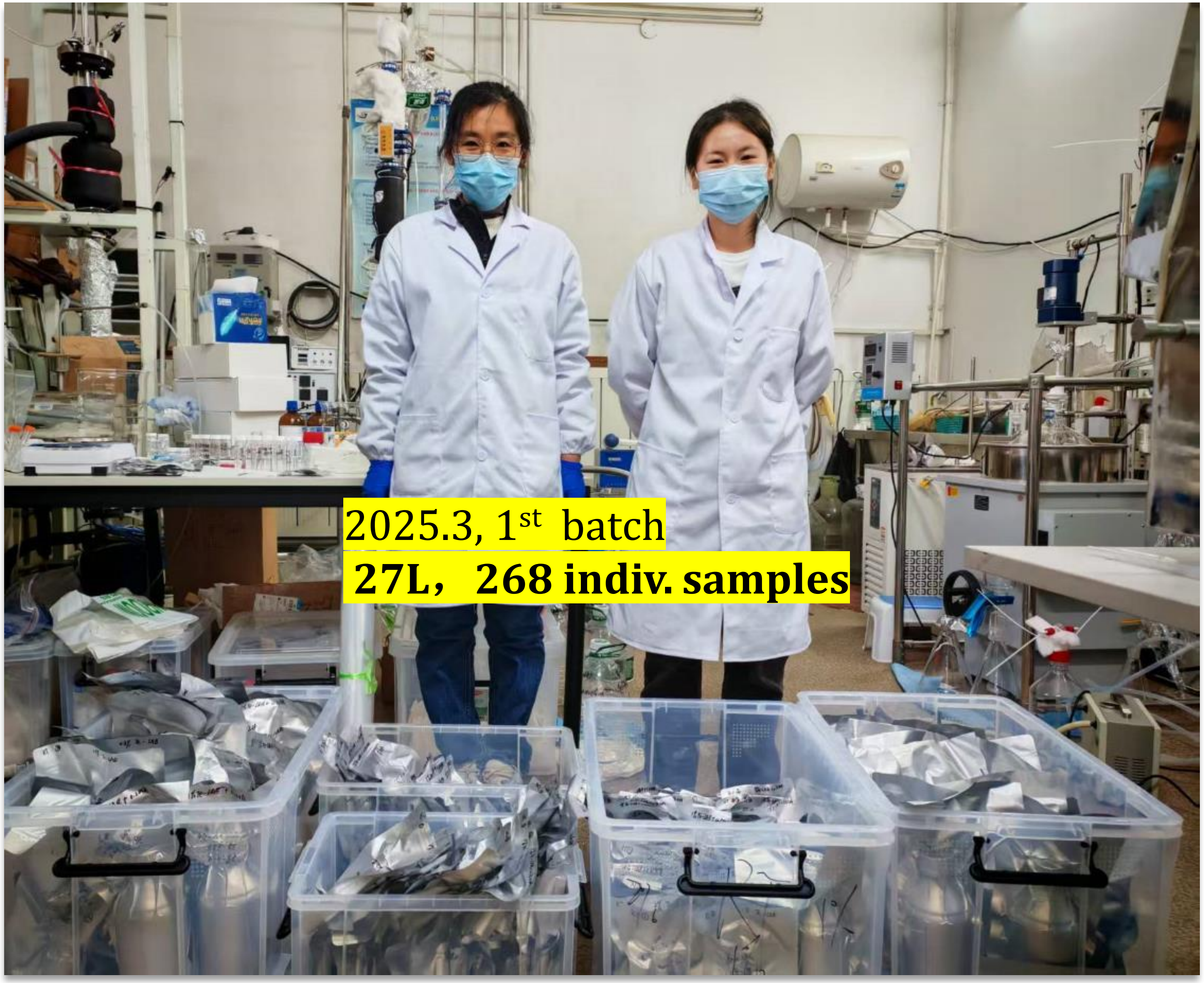
- Start 100kg-scale production in 10 batches

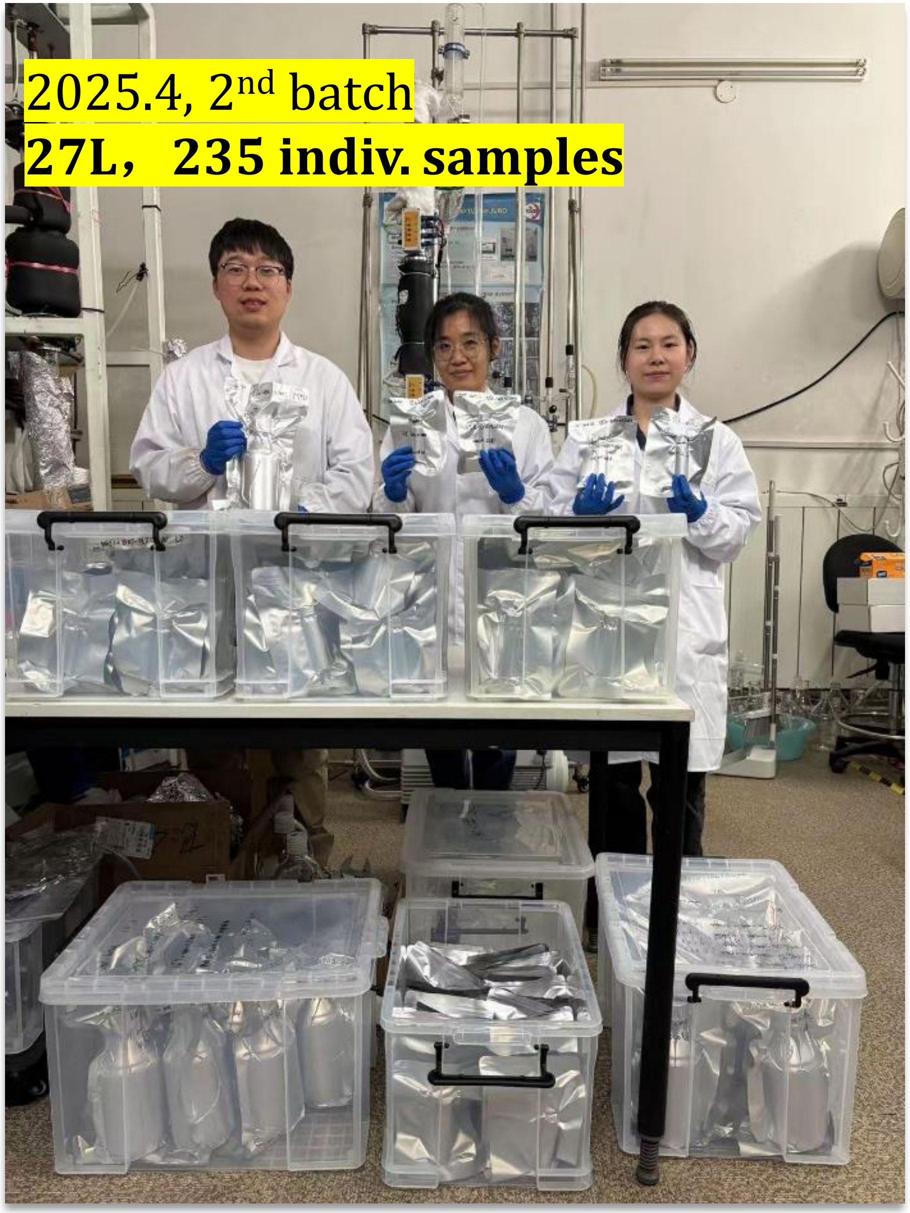
Production Steps





Hundreds of Te samples w/ diff. formulations were prep. for mult. research purposes

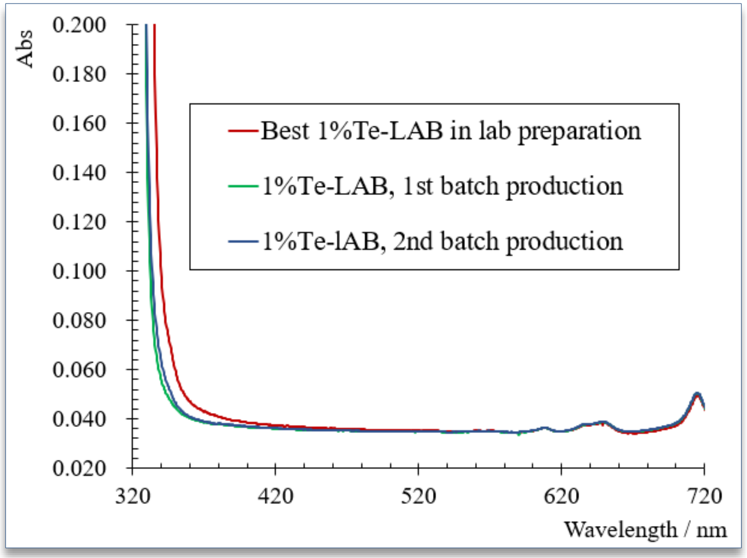




Progress of 100-kg production

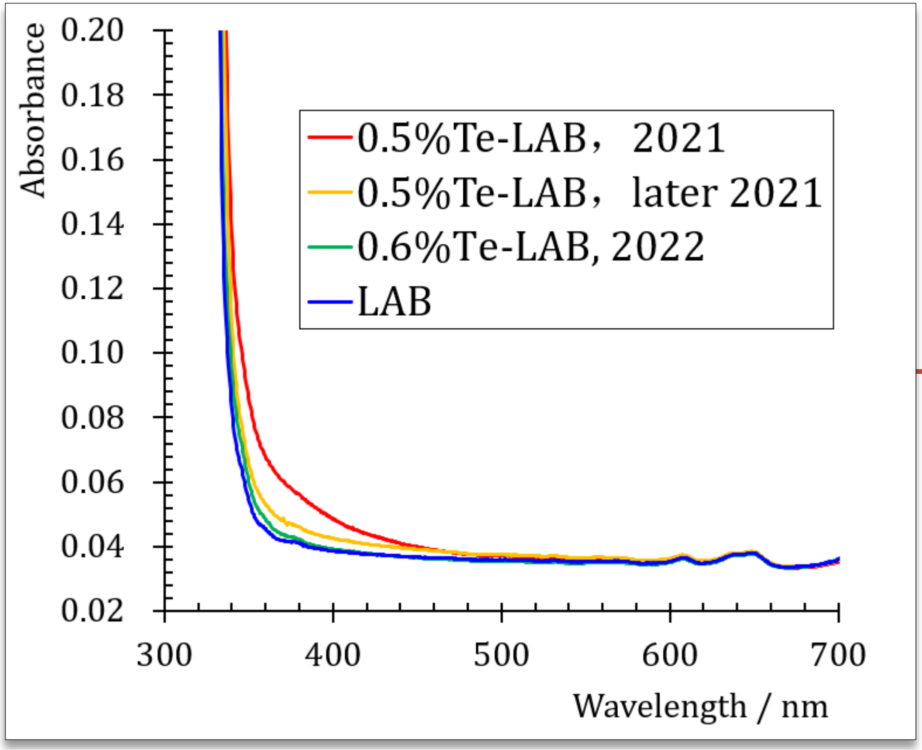
- 2025.3~4, 2 batches production has been finished successfully
  - Pause, sample measurements, and design subsequent synthesis based on results to study the impact of synthesis conditions on performance
- 2025.7: 3^{rd} batch production, still ongoing

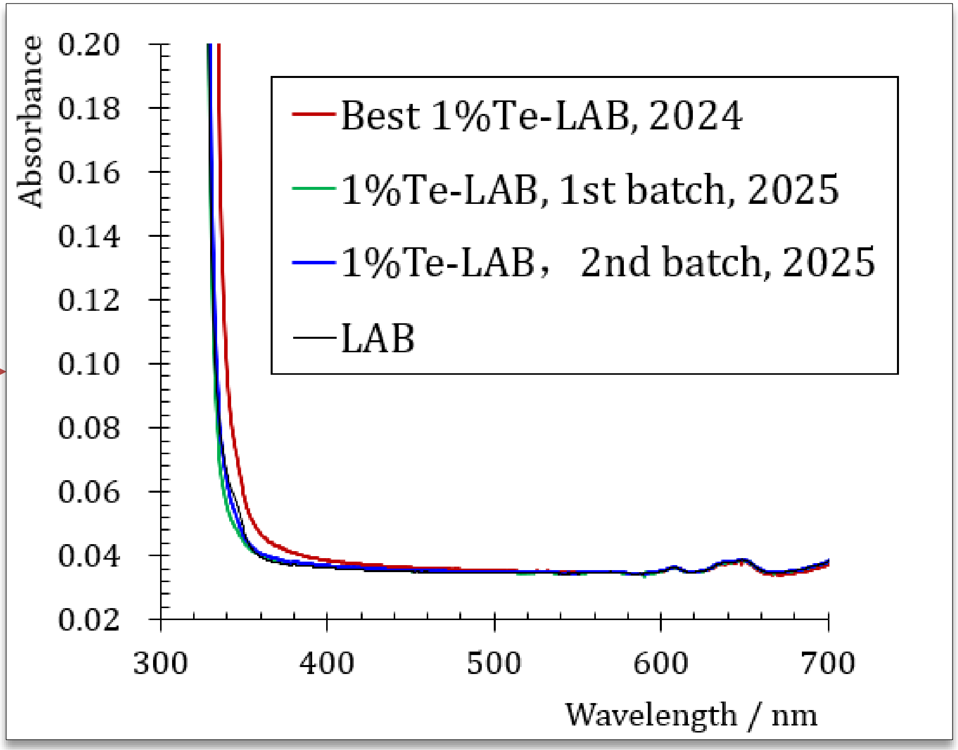
- 100-kg production Highlight: **Scale-Up with Enhanced Performance**
  - >50 kg higher-transparency Te-LAB samples were obtained
  - Te-LAB samples from 1^{st} and 2^{nd} production exhibit absorption spectra far superior to the best lab samples.
  - L.Y. and A.L. measurement is undergoing



Extensive Efforts in Enhancing Transparency and Stability

- **After years of dedicated efforts, key technologies for enhancing transparency and stability have been mastered**





- There is still a long way to go considering further increasing Te concentration

Summary: Current Research Status & Future Research Plan

0.5%Te-LS，Good L.Y.

L.Y. Enhancement in High Te-Doping LS

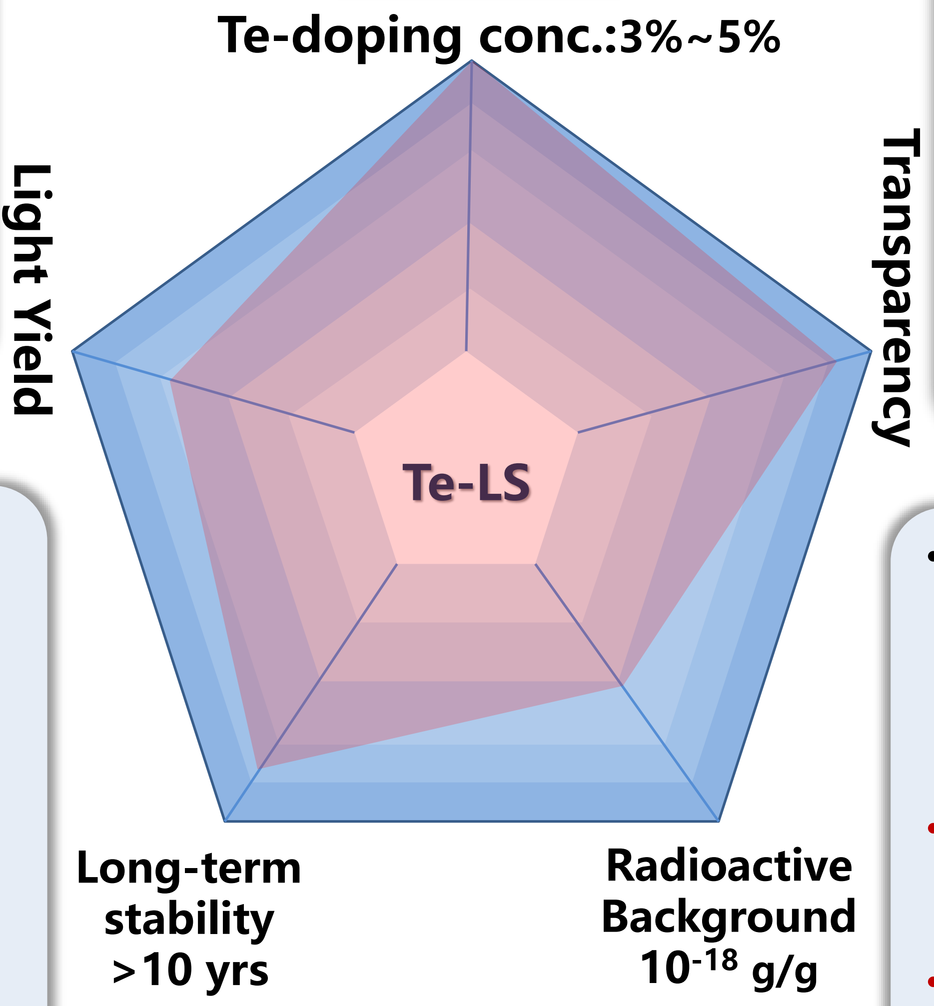
- Exploring the mechanism of Te impact on LS LY
- Modify fluors to enhance L.Y.

1% Te，Good Long-Term Stability

Long-Term Stability at High Te-Doping Levels

- **Index**：Abs+L.Y.
- **Protocol**: R.T. + Aging tests
- **Internal Factors**： Explore relationship btwn. synthesis conditions, product Structure, and stability
- **External Factors**：Functional auxiliary reagents

Achieved



1%Te，exceptionally high transparency

Transparency Study at High Te-Doping Conc.

- **External Factors**：Optical Purification
- **Internal Factors**：Impact & Opt. of Te Comp. Comp. & Struct.

- **Purification Strategy Confirmed**: Raw material purification only to ensure direct product availability for preparation
- Establish quant. anal. methods for trace rad. impur. in raw mater.
- Develop purif. methods for rad. impur. in raw mater.

Thank you for your attention!