





Liquid Scintillators for Daya Bay Neutrino Experiment

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on behalf of LS Group, Daya Bay collaboration

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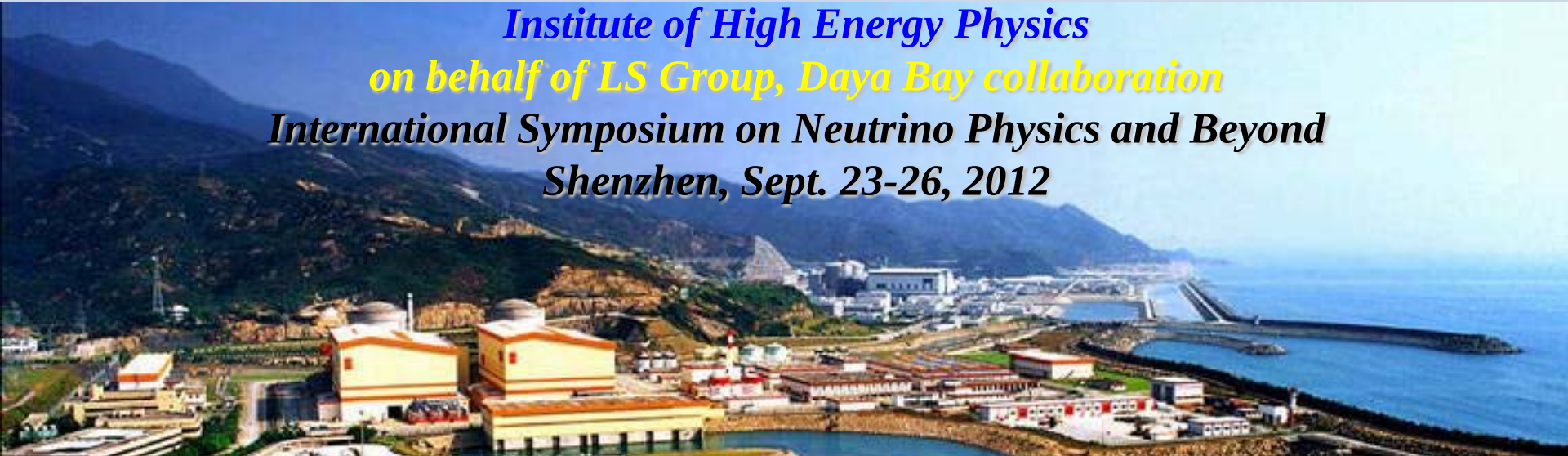
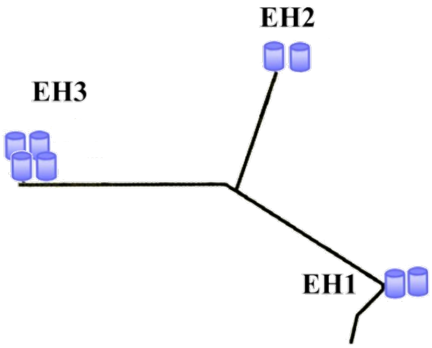


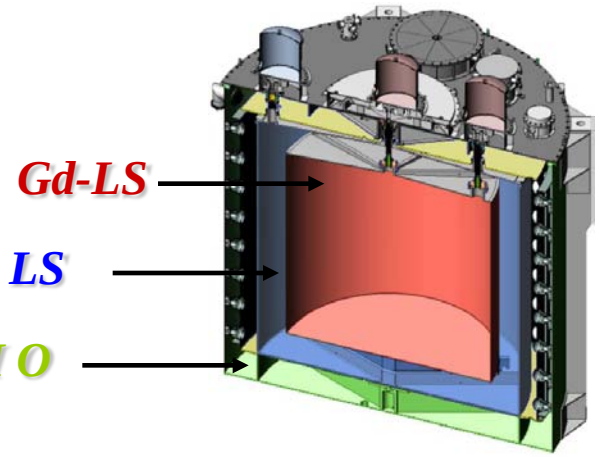
Outlines

- Gd-LS for Daya Bay experiment
- Gd-LS recipe, developments and performance
- Gd-LS Mass production
  - Equipment test runs and Gd-LS Dry Run
  - Purification of raw materials
  - Gd-LS Mass production and characterization
- Pre-study of LS for Daya Bay Experiment phase II

The Daya Bay Detectors

- 8 detectors for experiment
- For each detector:
  - 40 t Mineral Oil
  - 20 t LS
  - 20 t Gd-LS as target
- 160t Gd-LS in total





- Our goal: Production of 185t Gd-LS and 185t LS (20t for backup)

Requirements for Gd-LS

- **Long attenuation length** (high optical transparency)
- **High light yield**
- **Long-term stability**
- **Ultra-low impurity content** (Radioactive contaminants U/Th $\leq 10^{-12}$ g/g)
- **High flash point, low toxicity**
- **Compatible with acrylic vessels**
- **Practical for mass production(185t)**
  - Different from small, fine experiments in the lab

Key points for R&D of Gd-LS

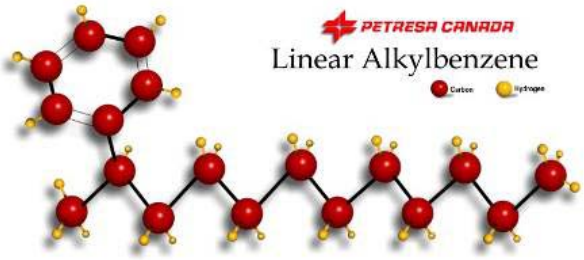
- **Choice of scintillation solvents**
  - Long attenuation length and high light yield
  - High flash point and low toxicity
  - Compatible with acrylic

- **Choice of chelating ligands for Gd**
  - Form stable complex with Gd
  - Good solubility in scintillation solvents
  - Long optical transmission at the PMT sensible region after loaded with Gd

Scintillation solvents

	PC	PCH	DIN	PXE	Dodecane	M. Oil	LAB
Formula	C_9H_{12}	$C_{12}H_{16}$	$C_{16}H_{20}$	$C_{16}H_{18}$	$C_{12}H_{26}$	C_nH_{2n+2} (n~30)	C_6H_5-C_nH_{2n+2} (n~12)
F. P. /^oC	48	99	>140	145	71	215	130
Compatible with Acrylic	N	-	-	-	Y	Y	Y

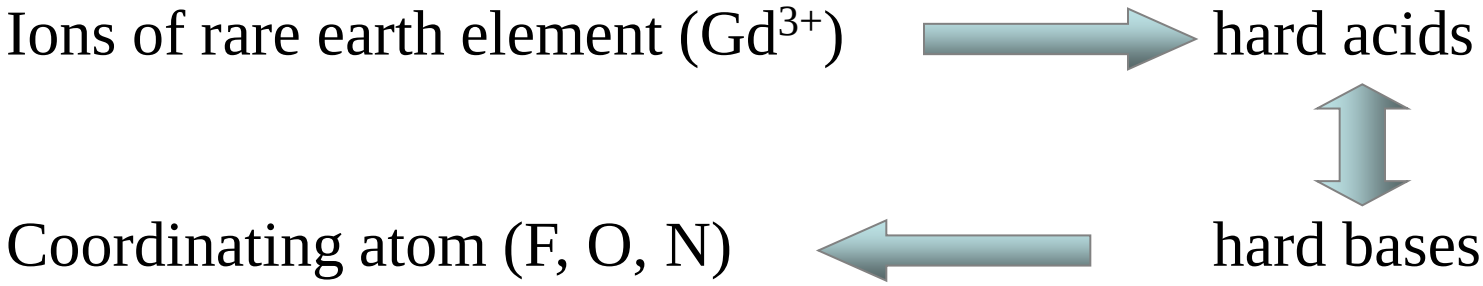
- **Linear Alkyl Benzene (LAB)**
  - Good optical property
  - High flash point: 130^oC
  - Low toxicity
  - Compatible with acrylic



Chelating ligands for Gd

Rule:

Hard acids bind strongly to hard bases and soft acids bind strongly to soft bases.



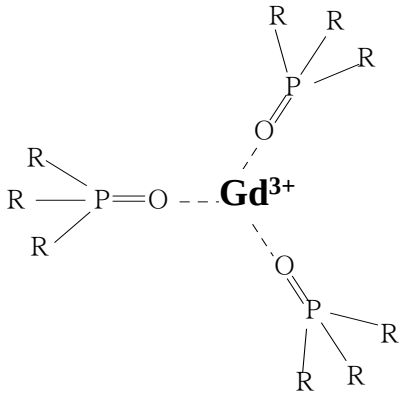
Organophosphorus-compounds

β-diketones

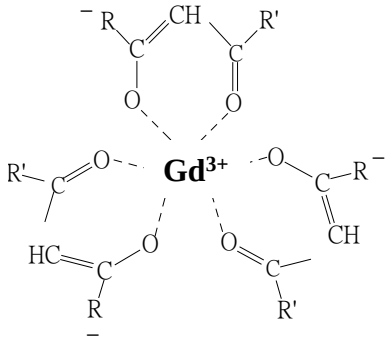
Carboxylic acids

Choice of ligands

- Organophosphorus-compounds
  - Easily complex with Gd, high production yield
  - Increase the absorption around 430nm greatly when doped into scintillator

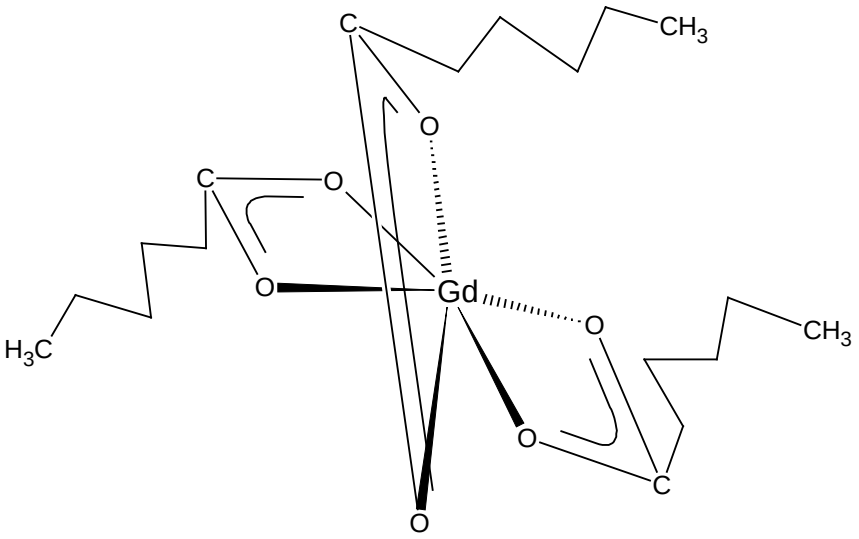


- β-diketones
  - Form a very stable complex with Gd
  - The complex has low solubility in scintillation solvent
  - high absorptions at 430 nm when doped into scintillator



Carboxylic Acids

- 2-Methylvaleric acid (2-MVA) 6C
- 2-ethylhexanoic acid (2-EHA) 8C
- 3, 5, 5-trimethylhexanoic acid (TMHA) 9C



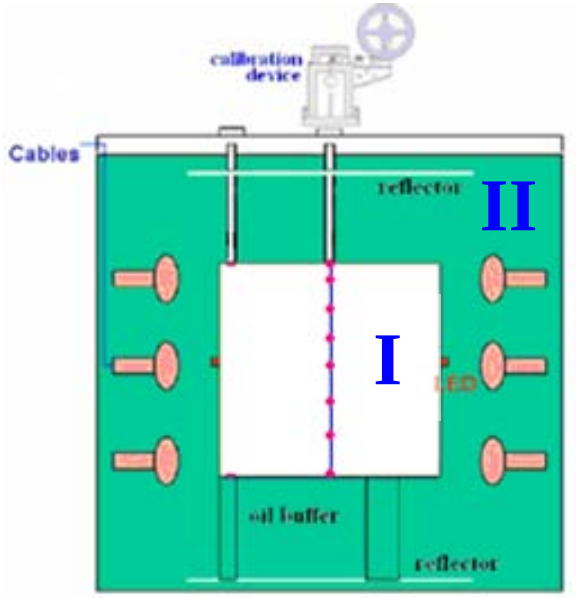
$Gd(TMHA)_3$

Gd-LS Recipe developed by Daya Bay

- Scintillation solvent: LAB
- Fluors: 3g/L PPO, 15mg/L bis-MSB
- Gd was doped as **Gd-TMHA (solid dissolution)**

Prototype of AD at IHEP

- **Motivation**
  - Validate the design principle
  - Test technical details of tanks
  - **Test Gd-LS developed at IHEP**
  - Test calibration procedure and Pu-C source
- **Achievements**
  - Energy response & MC Comparison
  - Reconstruction algorithm
  - Neutron response & Pu-C source
  - Effects of reflectors
  - **Performance of Gd-LS**



I: 600 L 0.1% Gd-LS

II: 5 t mineral Oil

750 L IHEP prototype Gd-LS preparation and characterization

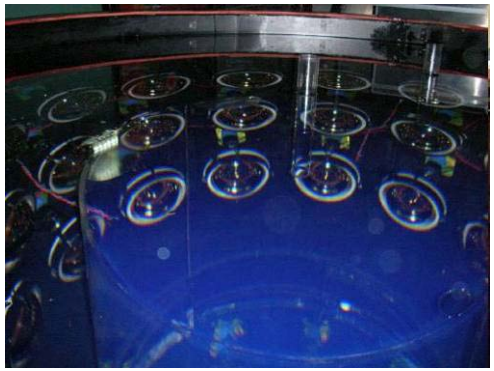
- 750 L 0.1% Gd-LS synthesized in Jan. 2007.
- Characterization of prototype Gd-LS
  - Gd% monitored more than 2.5 years: 0.11%±0.00%
  - Attenuation length measurement

Wavelength /nm	0.1% Gd-LS /m	LAB /m
410	7.3	4.4
430	13.7	14.9
450	13.0	16.8
470	14.1	15.5

  - Relative light yield measurement

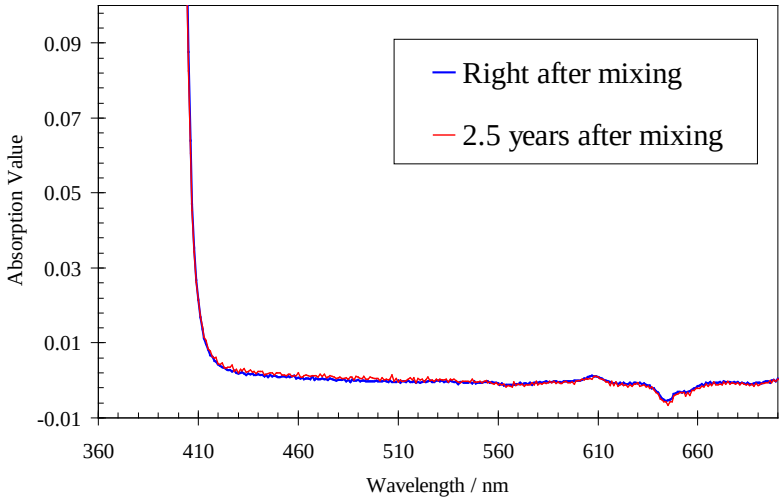
	LS	Gd-LS
R. L. Y.	52.2%	53.0%

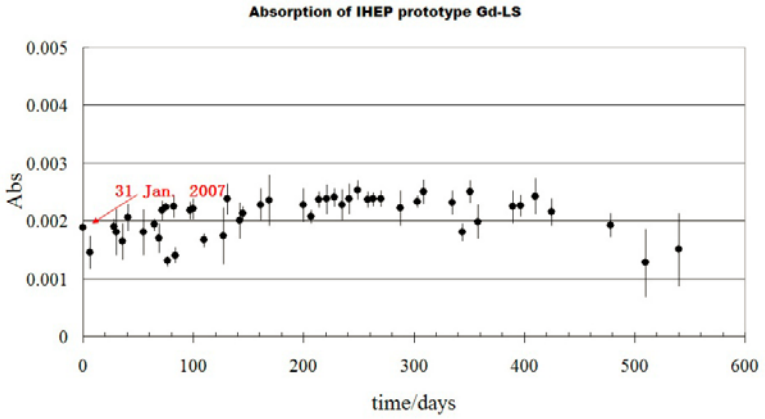
R.L.Y.= L.Y. of liquid scintillator / L.Y. of anthracene



Long-term stability of prototype Gd-LS

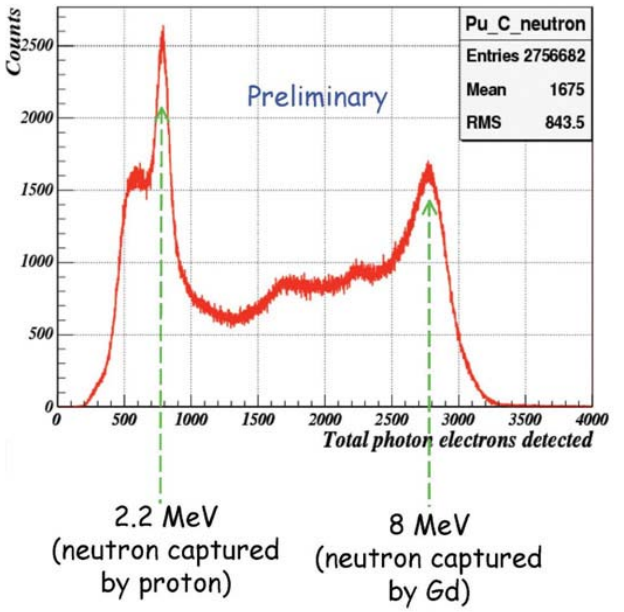
- Monitored by UV-Vis spectrophotometer for 2.5 years





- Neutron capture signal in prototype

(Pu-C Neutron Source)

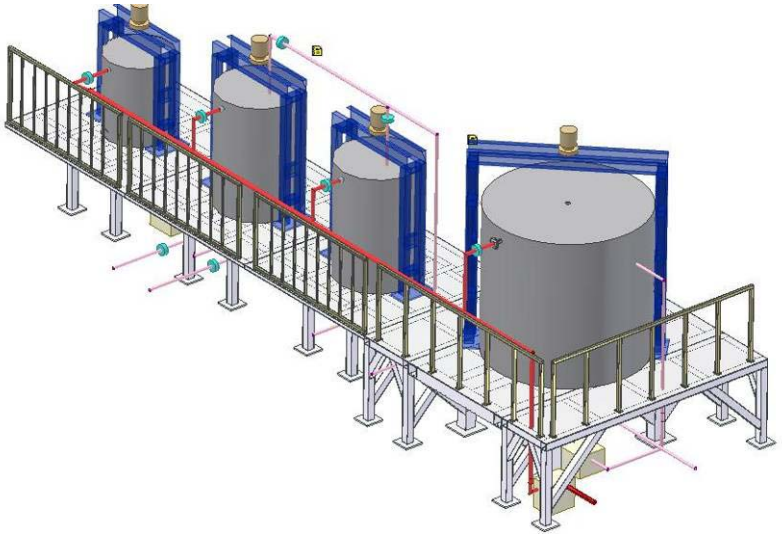


Mass production

- Equipments and flow
  - Equipment test runs to test the equipments and conditions for production, and to optimize the production flow
- Purification of raw materials
- Mass production
  - 185 t Gd-LS, 50 batches, 3.7 t each batch

Equipment test Runs

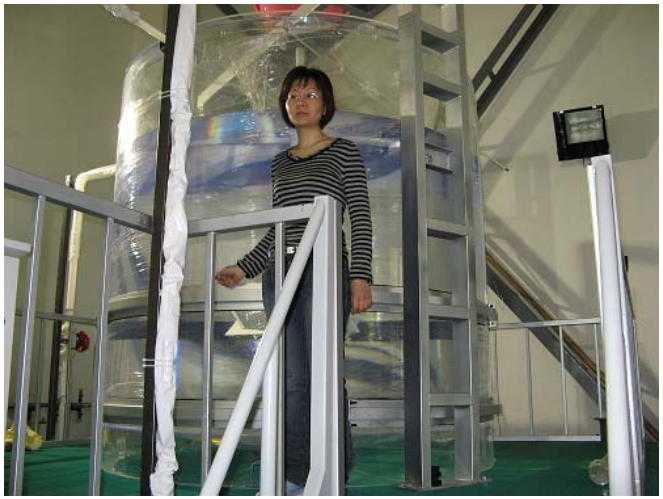
- Equipment for mass production had been built and installed at IHEP before mass production
- 8 equipment test runs had been finished from Jun. 2008 to Apr. 2009 in order to test synthesis of Gd-TMHA solid, the most important and difficult step of production
- Equipments, flows, conditions had been tested and improved through test runs



Dry Run Gd-LS

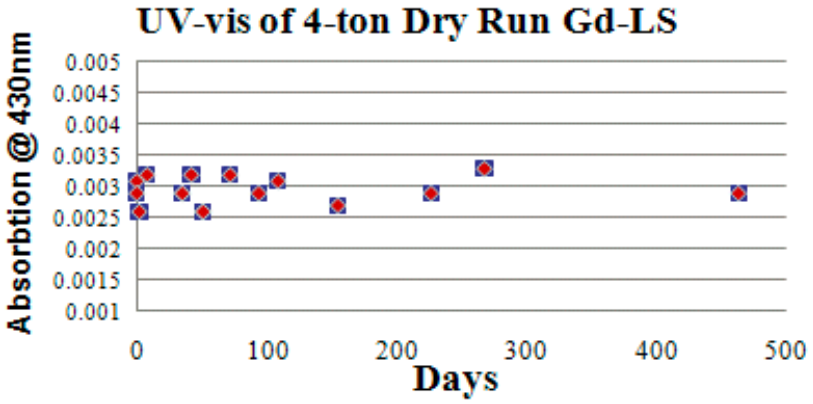
- Then one batch (3.7t) Gd-LS had been produced to test the whole procedure of the production in Mar. 2009.
- Optical properties:
  - Attenuation length of Dry Run Gd-LS at 440 nm： 8.2±0.3 m
  - Attenuation length of LAB at 440 nm ： 15.0±0.7 m
  - Attenuation length of 3g/L PPO in LAB： 7.8±0.3 m

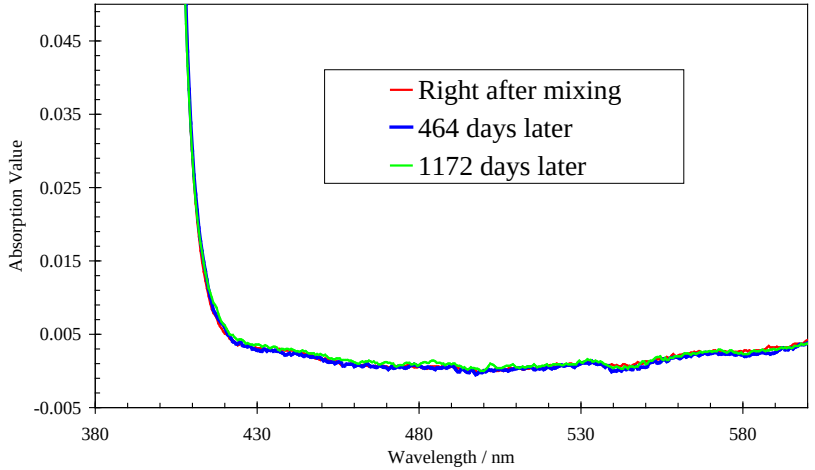
(PPO are not purified)



Dry Run Gd-LS

- Long-term stability, by UV-Vis spectrophotometer





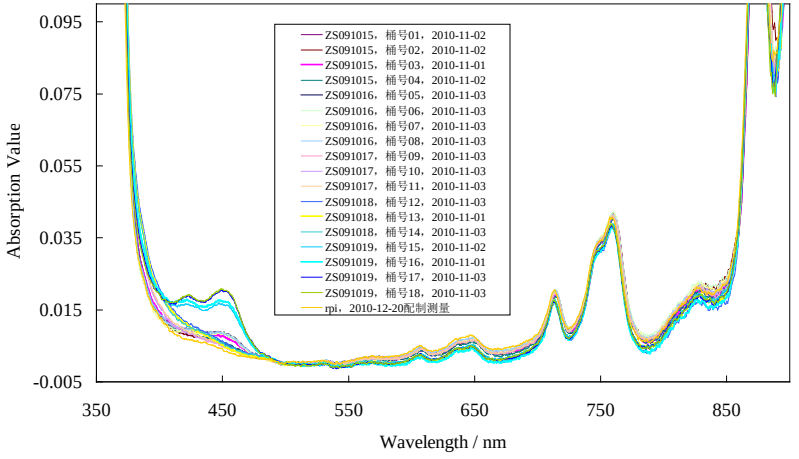
- **Equipment test runs and characterization of Dry Run Gd-LS show we are able to start Gd-LS mass production**

Purification of raw materials

- 388t LAB: specially ordered, need no purification
  - LAB was produced when catalyst performance is the best
  - Narrow fractionation temperature range to 2°C
  - Remove heavy portion
  - Fluoroplastics pipes and nylon bags for filling and transportation

Purification of raw materials

- Bis-MSB: scintillation grade, need no purificaiton
- PPO: ——provided by *JINR, Russia*
  - purification of PPO is contract to a chemical company in Wuhan, China.
  - Purified PPO had been used for production only if it pass our QA/QC tests.
  - 1761 kg PPO had been tested on-site, 391kg disqualified PPO had been returned back for further purification

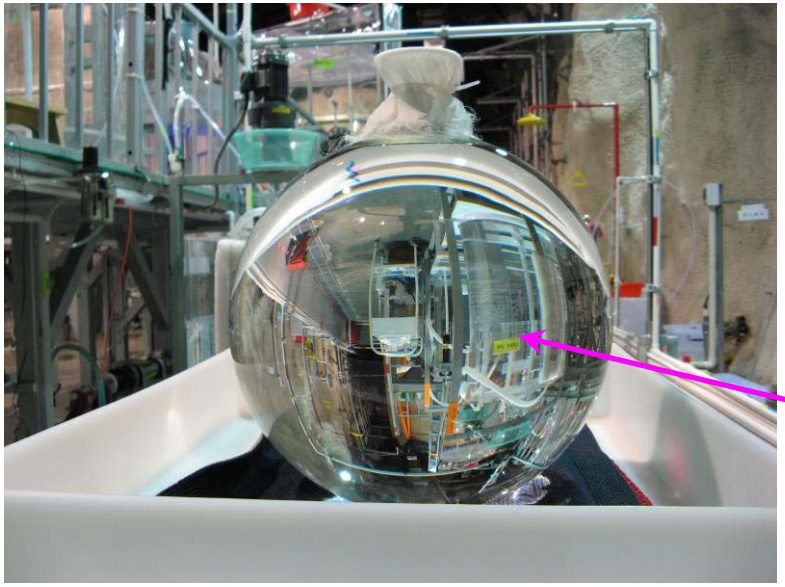


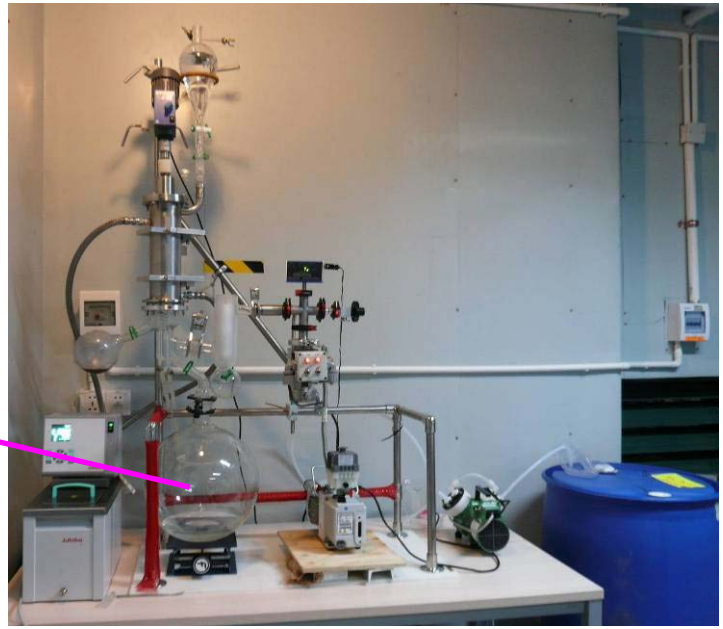
Purification of raw materials

- $GdCl_3 \cdot xH_2O$
  - 670kg $GdCl_3 \cdot xH_2O$ had been purified
  - Co-precipitation:
    - Dissolution
    - pH adjustment， precipitation
    - Filtration
  - QA/QC on-site

Purification of raw materials

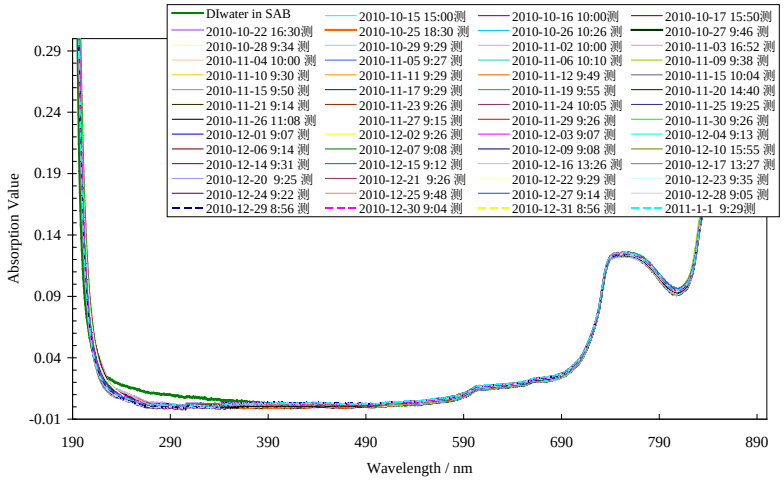
- TMHA
  - Vacuum distillation, more than 1 batch per day
  - QA/QC by UV-Vis spectrophotometer





Purification of raw materials

- Gd-TMHA solid had been synthesized in pure water
- Water purification system built on-site for production
- Deionized water had been monitored by UV-Vis spectrophotometer and Resistivity meter for quality insurance



185t Gd-LS production

- 185t Gd-LS production (50 batches):
  - Produced underground in Hall 5 inside Daya Bay NPP

Production Procedures

- **Strict QA/QC** for every step of production
  - Deionized water, QA/QC
  - Purification of $GdCl_3$ and TMHA, QA/QC
  - Preparation of Gd and TMHA reactant solutions, QA/QC
  - Gd-TMHA synthesis, QA/QC;
  - Dissolution of fluors, QA/QC
  - Mixing of Gd-LS, QA/QC, pump into storage tank
- 50 batches Gd-LS had been prepared smoothly without any accident

185t Gd-LS prodution

- 1st batch started on Oct. 17th, 2010
- 50th batch finished on Jan. 10th, 2011

Gd-LS in Storage Tanks

- 50 batched Gd-LS were storage in 5 acrylic tanks
- N_2 protection for each tank.
  - Storage Tanks are relatively sealed, N_2 goes into the top area of 5 storage tanks without disturbing the liquid.
- ~60h circulation for each tank to make Gd-LS uniform

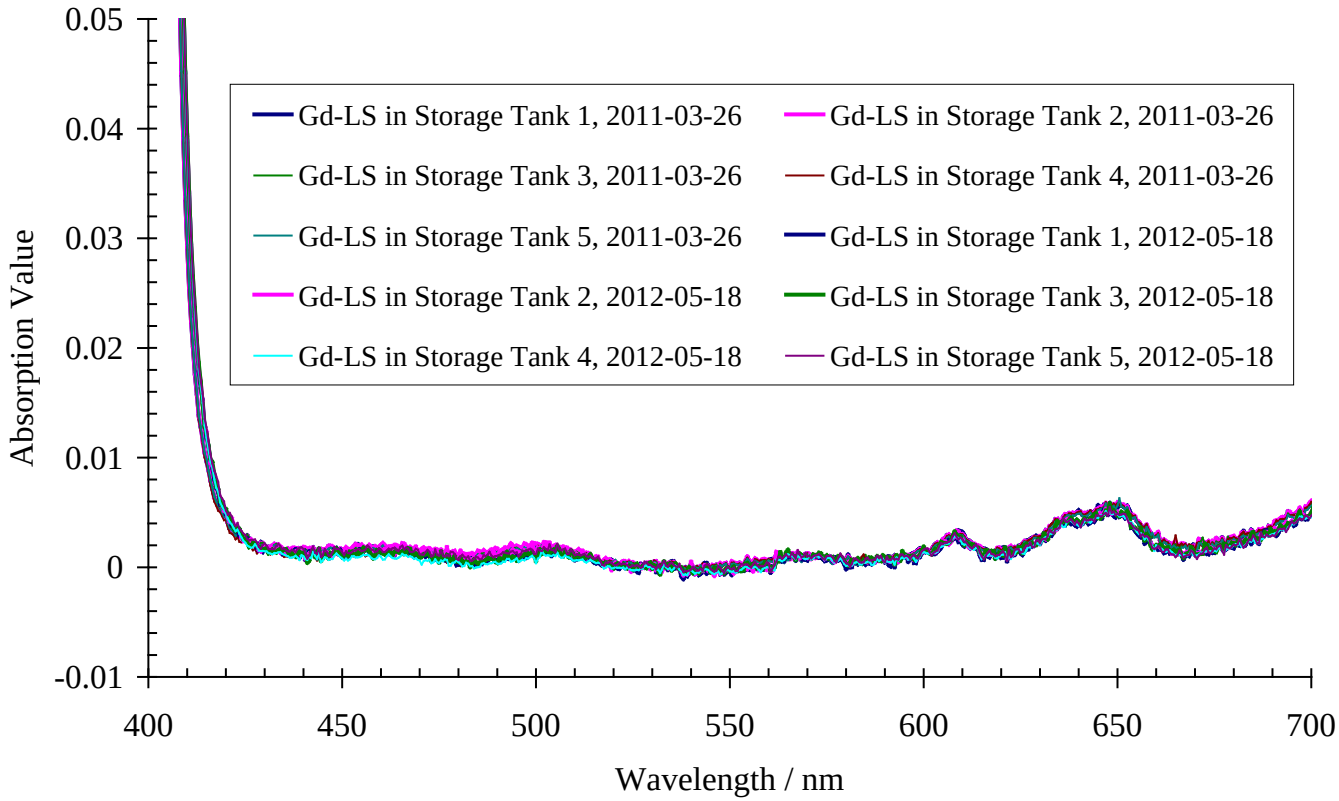
Characterization of Gd-LS

- Gd concentration, density, water content and UV-Vis spectra are consistent for 50 batches Gd-LS
- **Attenuation length at 430nm is ~16m （requirement: >10m）**

Measurement	#1	#2	#3	#4	#5
att. length	**15.7m**	**16.0m**	**15.9m**	**15.8m**	**16.0m**

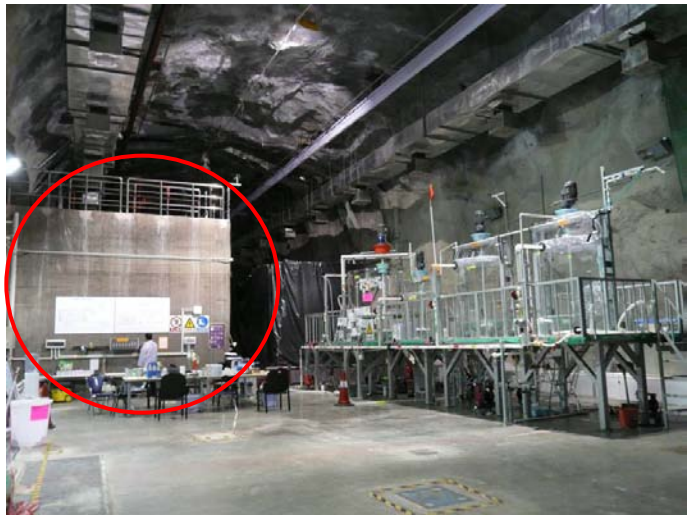
Characterization of Gd-LS

– long-term stability by UV-Vis



LS mass production

- 50 batched, simple mixing of LAB and fluors (PPO& bis-MSB)
- Use Gd-LS production equipment, need to be cleaned. No Gd detected after cleaning
- Stored in 2 nylon bags which are placed in 2storage pools.
- QA/QC



Quick Summary

- 185t Gd-LS/LS production for Daya Bay experiment had been performed successfully followed by strict QA/QC procedure
- Monitoring of Gd-LS in storage tanks and data taking of ADs show an excellent properties of Gd-LS.

LS for Daya Bay II

- Larger detectors, ~30m
- Need ~20,000t LS
- Attenuation length of scintillation solvent is important
  - Purification of scintillation solvent (LAB) is crucial
    - Goal: A.L.(LAB):30~40m
    - Daya Bay I: A.L.(LAB):<20m
  - Purification of fluors (PPO&bis-MSB)

Study of LAB at IHEP

- LAB had been purified by different methods
- Al_2O_3 is the best, but still can't meet our requirement
- What we do next:
  - Synthesis LAB by using high quality materials
  - Further purification of LAB
  - Purification of fluors

LAB	Abs/430nm	A.L.@430nm/m
Raw	0.0011	14.2
V.D.	0.0011	19.5
Silica gel	0.0010	18.6
Al_2O_3	0.0005	22.3