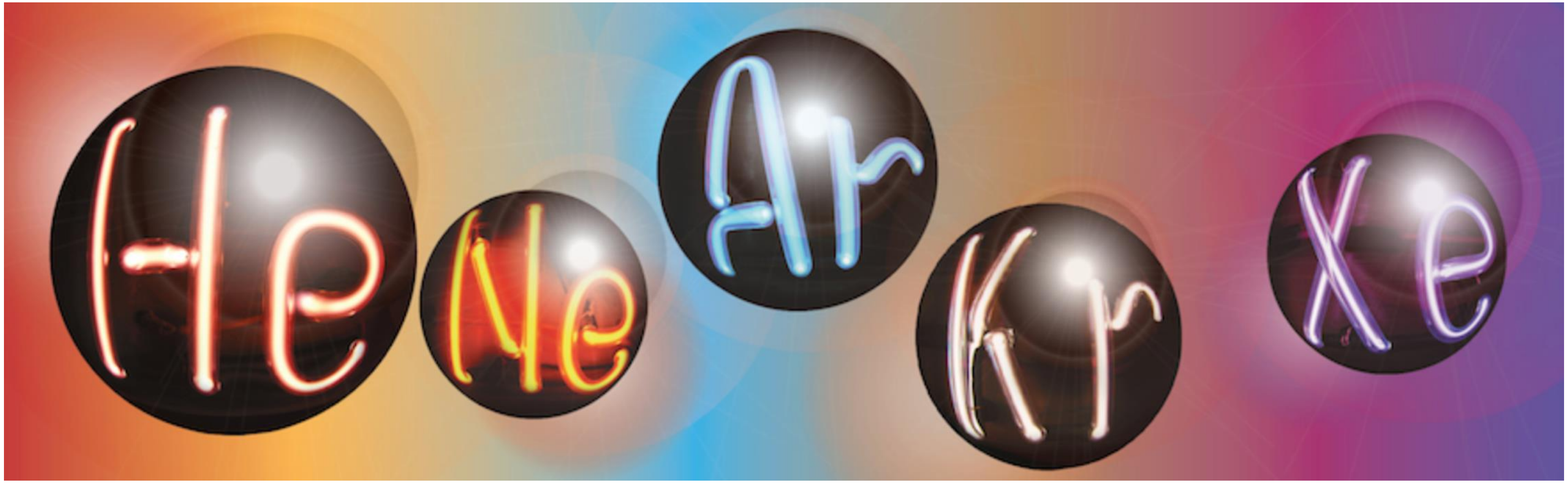
Production and characterization of WLS coatings for the photon detection system of noble liquid detectors



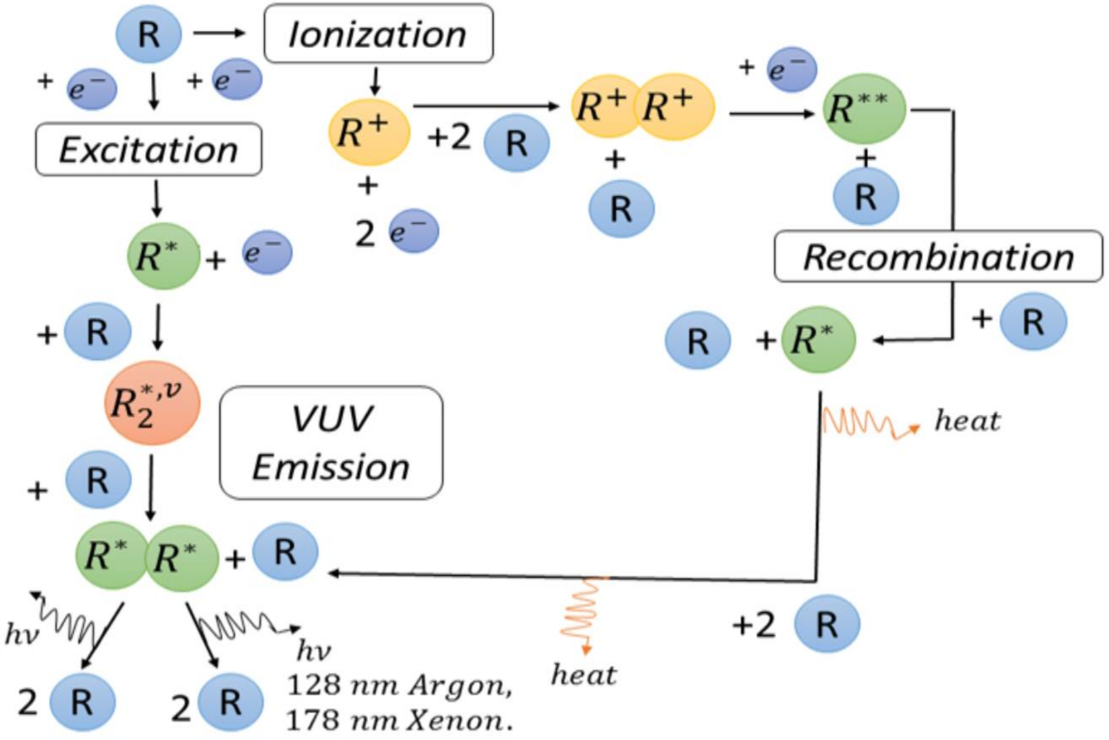
N. Canci – on behalf of DUNE Collaboration

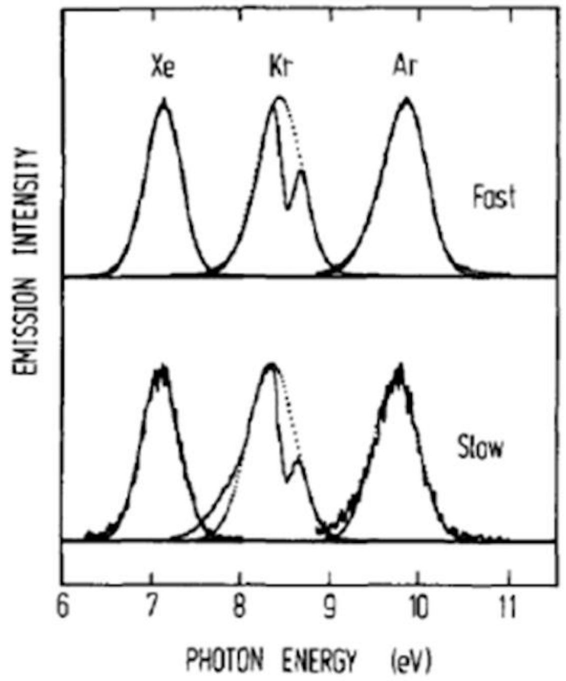
Outlines

- Scintillation Light in Noble Liquids
- Wavelength-shifting Materials
- DUNE Experiment Photon Detection System
- WLS Vacuum Deposition Design
- Description of the evaporation system
- Description of installation
- WLS Coating Process
- Evaluation and characterization of the WLS coated substrates
- Conclusions

Scintillation Light in Noble Liquids

- Charged particles crossing the noble liquid volume produce excitation and ionization followed by recombination, both leading to the emission of vacuum ultraviolet (VUV) light
- Noble liquid scintillation light emitted in the VUV range must to be converted to longer wavelenghts to be detected by «conventional» photosensors



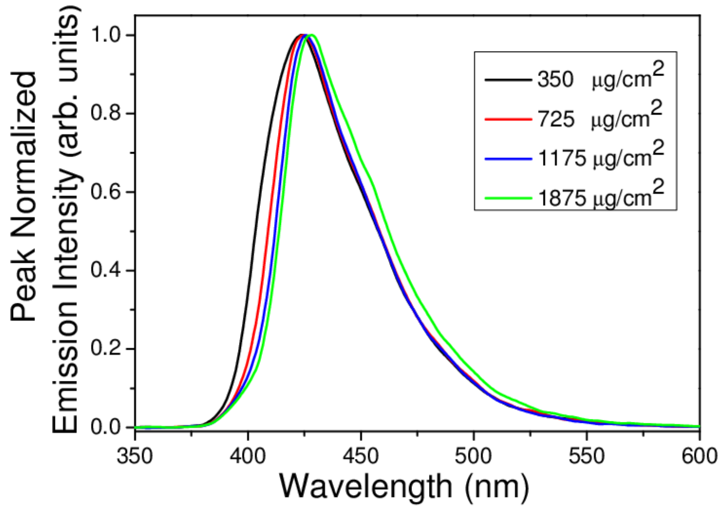


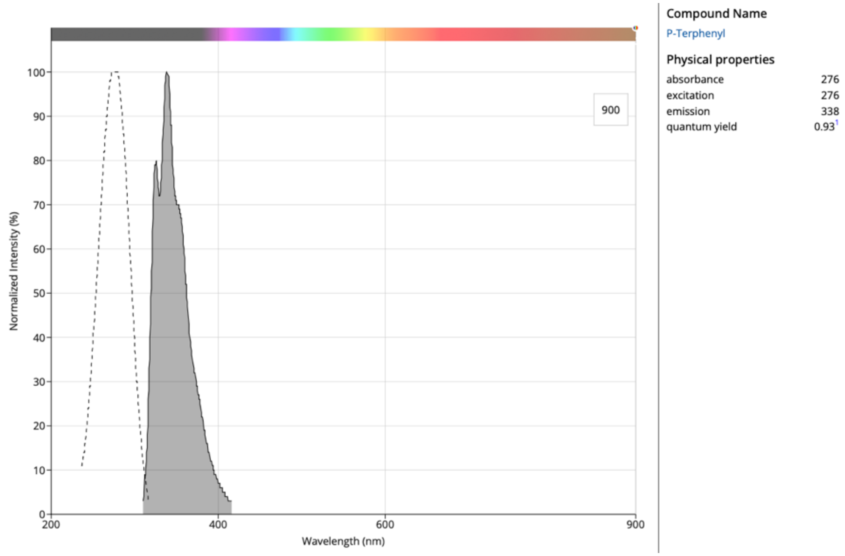
- Argon: λ=128 nm, τ_S=6 -10 ns, τ_T=1500 ns
- Xenon: λ=175 nm, τ_S=3 ns, τ_T=27 ns

Wavelength-shifting Materials

- The use of wavelength-shifting (WLS) coatings is of primary interest in the field of liquefied noble gas detectors (such as argon and xenon) employed as active media in neutrino physics, dark matter experiments and rare event search
- WLS materials, such as TetraPhenyl-Butadiene (TPB) and p-Terphenyl (PTP), are especially used to convert VUV scintillation light to the visible range, enabling efficient detection by conventional photosensors
- WLS coatings are particularly important for large area detectors, since only a fraction of the detector surface can be directly instrumented with photodetectors

TPB emission Spectrum: 420 nm

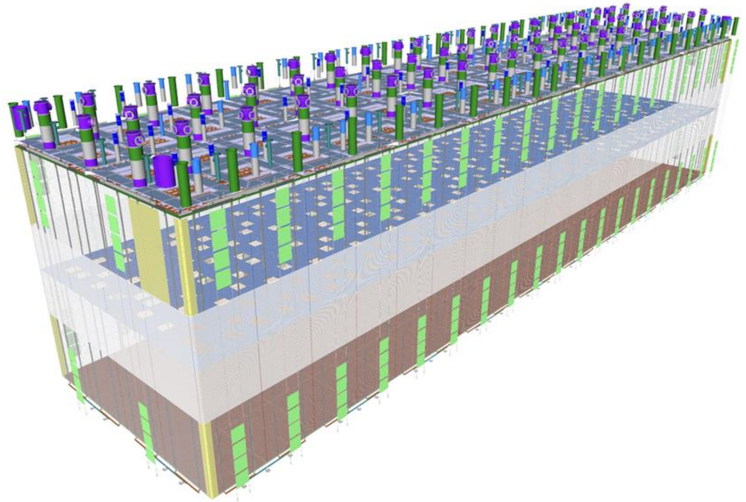


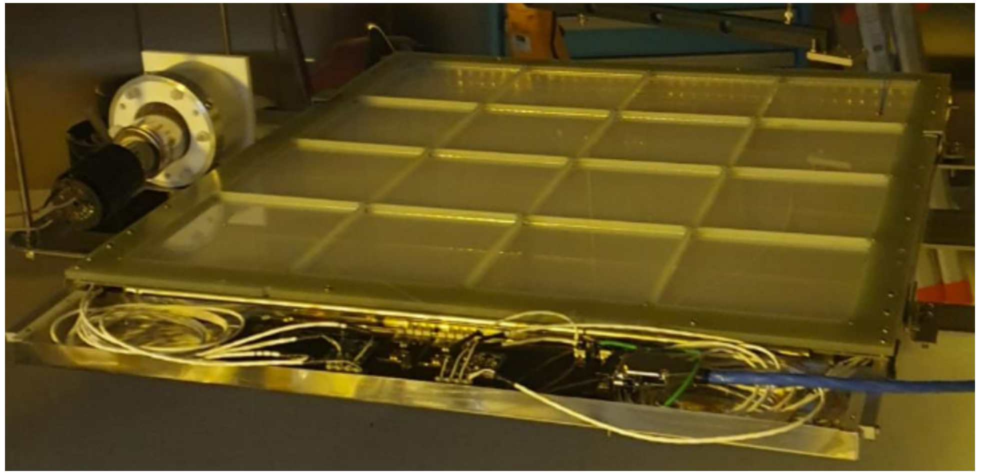


PTP emission Spectrum: 350 nm

DUNE Photon Detection System

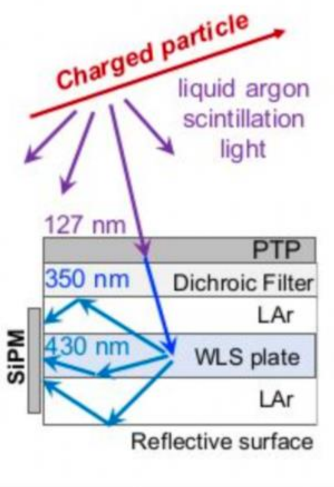
- Deep Underground Neutrino Experiment (DUNE) for neutrino detection is based on the Liquid Argon TimeProjection Chambers (LArTPCs) technology
- DUNE Far Detectors are provided by Photon Detection System (PDS) to detect the VUV photons to provide precise timing information for neutrino interactions, improve calorimetric energy measurements and energy resolution, and act as a trigger for non-beam events
- The PDS of the FDs is based on the novel technology called X-ARAPUCA
- The PDS of the FD-VD will use the Megacell X-ARAPUCA, a 60×60 cm² X-ARAPUCA unit equipped with 16 filters per side and 40 SiPMs mounted along each side wall for light readout

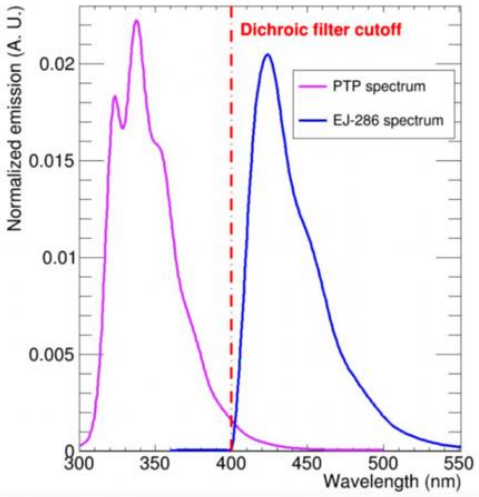




WLS Deposition of Optical Filters

- In the X-ARAPUCA photon detection system, the external surface of the optical filter coated with PTP used as wavelength-shifting material
- A thin film of PTP (200-500 μg/cm^2) deposited on the external surface of the optical filter windows to convert 128 nm VUV to 350 nm visible light and achieve high photon conversion yield
- Evaporation systems designed and constructed for the deposition of PTP onto surfaces of optical windows





- European site for PTP coating on filters (for Vertical Drift Far Detector) hosted in Naples
- Evaporation of filters of XA-VD Modules for cathode (320 double sides) and membrane (320+32 single side)
- Evaporation system hosted in a ISO-6 Clean Room

WLS Vacuum Deposition Design

Evaporation chamber designed considering the following parameters:

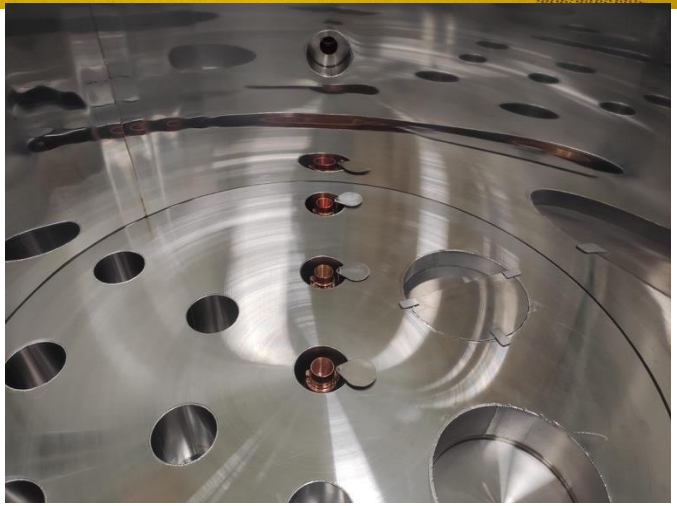
- Size/dimensions and shape of the chamber
- Uniformity of the coating
- Vacuum level
- Number of samples and/or materials deposited per cycle

- Cylindrical symmetry chambers widest used to ensure higher uniformity
- Height of the chamber impacting on the uniformity and on the number of evaporation sources
- Transverse dimensions important requirement for hosting the number and the size of parts to be coated
- Relevant time (order of hours) requested for large evaporators to reach the vacuum level matching the required mean free path allowing for the evaporated molecules to coat the substrate

Vacuum
Evaporation
System for
Ultra
VIolet
Optics

Evaporation System Overview

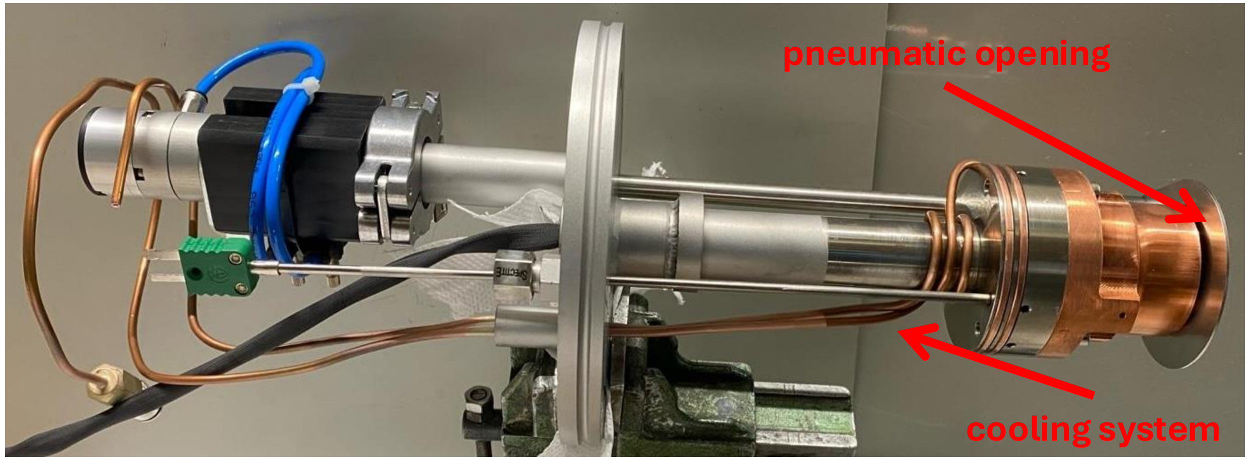
- Stainless Stell cylindrical vacuum chamber:
    - 120 cm diameter - 70 cm height
    - Electropolished inner parts
    - Removable dome flange
- Helium leak test performed with leak rate $<10^{-10}$ mbar/l/s
- Motor with rotating feedthrough and disk holder
- Three (3) crucibles+control units
- Thickness measurement unit
- Pumping system (designed to have a vacuum level of the order of $\sim 5\times10^{-5}$ mbar in ~1 h)
    - Primary dry pump (40 m^3/h)
    - Turbopump (2300 l/s)



Crucibles and Control System

Knudsen cells

- About 30 cm^3 volume/each
- Stainless steel body on DN 100 ISO-K flange
- Copper crucible designed to host fused silica crucibles for high purity coatings
- 1000 W heating modules
- Working temperature at ~230-250°C (maximum temperature 500°C)
- Electro-pneumatic opening and cooling system



Integrated control system including:

- Pump system control
- Vacuum monitoring
- Crucible temperature controls
- Pneumatic caps for opening/closing crucibles
- Disk motor switch and speed regulator
- Thickness monitoring

Deployment of Substrates

Substrate positioning system designed to facilitate efficient handling, precise placement, and stable support of coated parts

- **Full removable dome with crane**:

  allowing safe and convenient access to the chamber for installation, cleaning, and maintenance

- **Dome holding structure**:

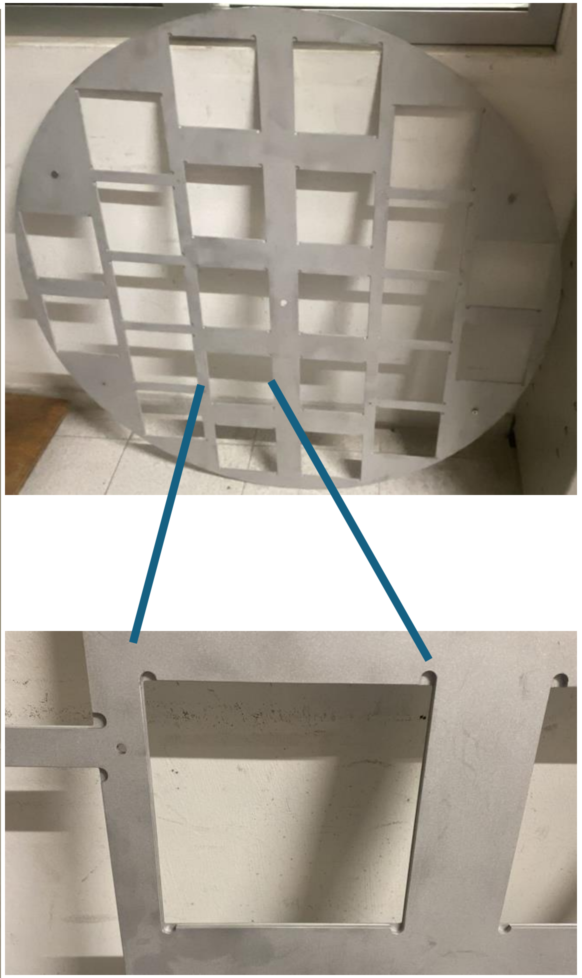
  securing the dome during operation, ensuring stability to withstand mechanical loads and unloads

- **Dedicated substrate supporting frame with slots for substrates**:

  frame to precisely holding multiple samples or substrates simultaneously, ensuring uniform treatment and repeatability of production batches

- **Versatile interface**:

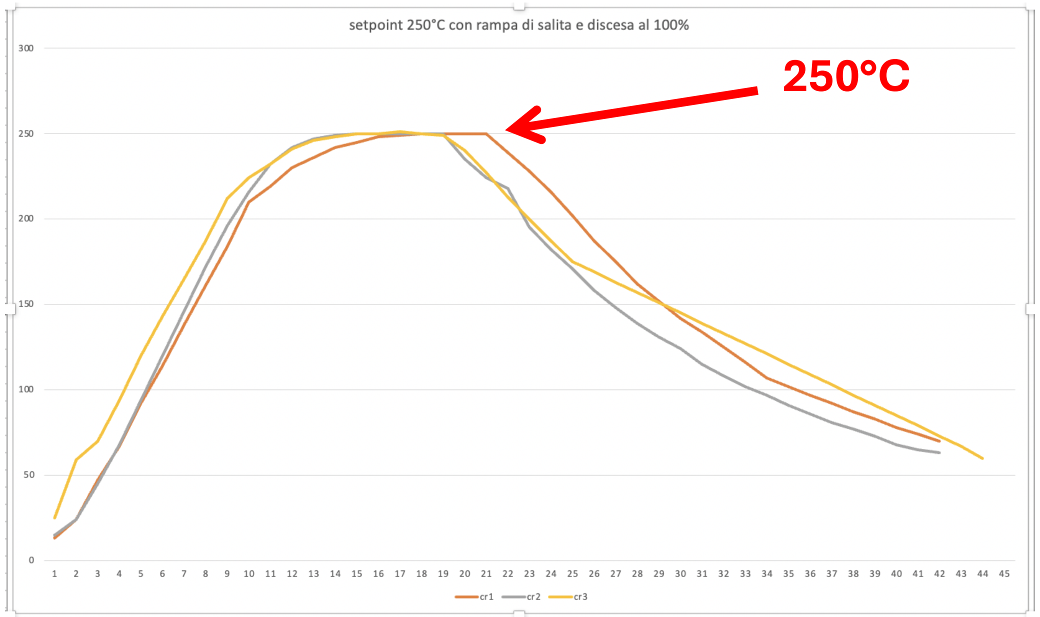
  system offering adaptability to accommodate various holders and support frames, enabling flexibility for different substrate sizes, shapes and configurations

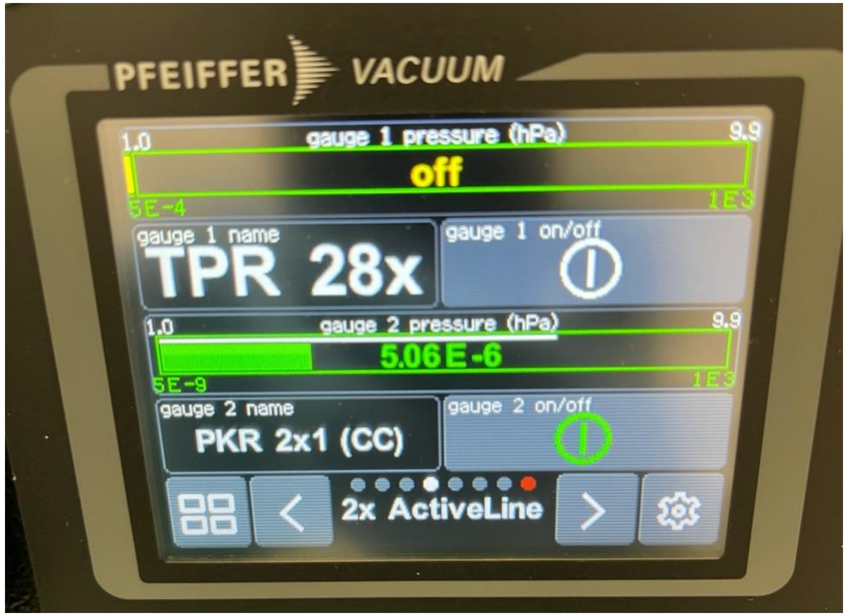


Evaporation System Installation

- Installation and test of the pumping system and vacuum tightness
- Installation and test of the Knudsen Cells:
  - PID controlled temperature
  - Set point stability of the temperature checked

- Finalization of tests for some additional controls and small hardware adjustment
- Commissioning already started
- First evaporation expected soon for fine tuning of paramters of the process
- Coating of the optical filters foreseen for the begining of 2026





Vacuum Evaporation Operations

Phases of operations

- **Preparation**
  - Mounting of the filters on a proper holder
  - Weighting of the reference samples
  - Mounting of the holder on the dome flange

- **Coating**
  - Vacuum phase till ~5×10^{-6} mbar
  - Starting and monitoring of the evaporation
  - End of coating, breaking of the vacuum and back filling with gaseous nitrogen
  - Opening of the flange and removal of the coated filters and samples

- **Quality Control**
  - Visual inspection of the coatings and samples
  - Placement of the filters in appropriate shipping boxes
  - Quality check of the reference samples

Characterization of Coatings

- Visual check of the overall deposition process
- Check of 10% of coated samples for each evaporation process

- **Measurement of mass densities** with precision scale (O(10^{-5} g))
- **Conversion yield test**: spectrofluorimeter measurements compared wrt a reference sample
- **Adhesion test**: controlled-slow immersion of samples/substrates in liquid nitrogen
- **Photo-conversion test**: measurement of the conversion efficiency by illuminating the reference samples with UV light

Low Temperature Tests

Detailed and controlled procedure to ensure integrity of the coatings under cryogenic conditions

- **Adhesion Test**:

adhesion test for the coated samples using acontrolled low-speed motorized system moving at ≤5 mm/min to evaluate the bonding of the PTP to the substrate

- **Immersion in cryostat**:

samples immersed for ~60 minutes in a cryostat filled with liquid nitrogen (~77 K) to assess thermal, mechanical, and structural stability

- **Extraction Handling**:

after dipping, substrates carefully extracted into a nitrogen gas–saturated ambient environment to prevent moisture-induced damage or degradation of the coating or substrate during warming

- **Conversion Efficiency Testing**:

conversion efficiency measurement of the coated samples, before and after liquid nitrogen dipping, to evaluate possible alterations of functional performances (optical and/or chemical conversion efficiency)

Conversion Efficiency Measurements

Systematic procedure to measure the photon conversion efficiency (PCE) of coated samples and evaluate the effect of immersion in liquid nitrogen (LN)

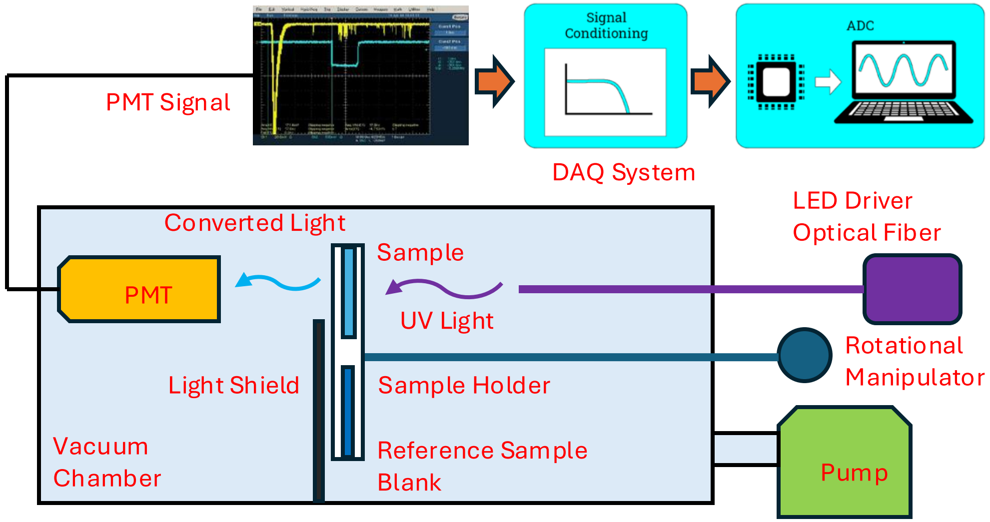
Controlled environment setup to characterize PCE of coated samples using a PMT and UV illumination

Measurement of relative photon conversion efficiency in comparison with blank, reference samples, same saples before and after the LN dipping

Process to ensure reproducible measurements of photon conversion efficiency and photodetector performance:

- **PMT Stabilization**: (~15 minutes) to ensure stable gain and minimal noise, after powering on the PMT
- **Chamber Evacuation**: to ~10^{-2} mbar to reduce moisture and prevent contamination potentially affecting optical measurements
- **Single Electron Response (SER) Acquisition and PMT Gain Tuning**: calibration of the PMT gain to optimize sensitivity and ensure accurate photon counting
- **Sample Illumination**: illumination of samples with 260 nm UV light from an LED driver to convert UV photons into visible photons
- **Signal Acquisition**: intensity or count of converted photons recorded by the PMT, capturing the quantitative photon output from each sample
- **Relative Efficiency Measurement**: photon conversion efficiency calculated by comparing the signals from coated samples with blank/reference samples and by comparing the same sample's performance before and after LN dipping

Concept Design of the PCE System



Conclusions

- Large volume noble gases detectors are mainly focused on neutrino physics and rare event search
- Deep UV scintillation light from noble gases needs to be shifted to be detected by photosensors, namely photomultiplier tubes and silicon devices
- TetraPhenyl-Butadiene and p-TerPhenyl are the most common wavelength shifting materials, able to efficiently convert the scintillation light respectively to 420 and 350 nm
- Large evaporation system (VESUVIO) designed, contructed and installed at INFN-Naples for PTP coating of optical filters substrates of the PDS devices of FD-VD of DUNE experiment
- Evaporation system designed to host (quite) large parts, have the flexibility for coating different materials (glass, quartz, scintillation bars, acrylic, metals, alloys, phototosensors, etc.) and allow 2 complete deposition cycles per day
- Commissioning of the coating station already started, finalization of tests for some additional controls ongoing, fine tuning of the parameters for the process expected soon
- Complementary quality controls and characterization of the coatings trough several methods (measurement of mass densities, adhesion test in LN, photo-conversion yield measurements) will be performed
- Some set-ups already designed, constructed and/or ready for operations, other systems in progress
- Dedicated vacuum evaporation systems are an integral part of LAr detector preparation, used to apply WLS layers precisely and ensure optimal light collection and timing for neutrino physics experiments and rare events search
- Proper evaporation and handling procedures are essential for maximizing the stability and conversion efficiency of the coatings enhancing the overall photon detection system performance in large liquid argon detectors