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JUNO Detector: Liquid scintillator purification with distillation and stripping plants

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Summary. — JUNO (Jiangmen Underground Neutrino Observatory), currently under construction in China, is a multipurpose underground liquid scintillator detector, designed to study some of the fundamental neutrino parameters. The central detector of JUNO consists of a huge acrylic sphere filled with 20 kton of organic liquid scintillator (LS) mixture, whose optical and radio-purity properties are crucial to achieve the experimental goals. For this purpose, before filling the detector, the scintillator will be purified through a sequence of 5 purification processes: alumina filtration, vacuum distillation, mixing plant, water extraction and gas stripping. The distillation of the solvent is fundamental to remove heavy and high-boiling radioactive metals, such as ^{238}U, ^{232}Th and ^{40}K, while the stripping process has been proven effective to remove gaseous impurities like Ar, Kr and Rn. Two dedicated plants have been designed and built to perform the distillation and the stripping purification processes on JUNO LS. These plants have been recently commissioned at JUNO site to test and verify the purification efficiency on several LS samples and to optimize the operating parameters of the plants. Some preliminary results (absorption spectra) on LS will be presented.

1. – JUNO Experiment

The Jiangmen Underground Neutrino Observatory (JUNO) is an underground neutrino experiment under construction near Kaiping, in South China [1]. JUNO will detect neutrinos from several sources [2] and study some of the fundamental neutrino parameters. Among the extensive scientific program, the main goal is the determination of the neutrino mass ordering by detecting reactor antineutrinos from two nuclear power plants, located at about 53 km. JUNO foresees to measure the neutrino mass ordering at 3σ level in 6 years. JUNO is composed by an acrylic vessel of 35,4 m in diameter as a central detector (CD), which will contain about 20 kt of liquid scintillator (LS). The scintillation light produced inside the CD will be detected by 17612 20-inch and 25600 3-inch photomultipliers, providing a total coverage of $\sim$78% with an unprecedented energy resolution of 3% at 1 MeV. The CD, which is supported by an outer stainless-steel structure, is immersed into a pure water pool to shield the active region from natural radioactivity of surrounding rocks and air. The water pool acts also as a muon veto detector, together with the muon veto top tracker [3], installed on the top of the pool, to exclude muons from the signal contribution.

2. – JUNO liquid scintillator

The JUNO target is based on an organic liquid scintillator mixture which is composed of Linear Alkyl Benzene (LAB) as solvent, 2,5 g/l PPO as fluor and 3 mg/l bis-MSB as wavelength shifter [4]. The light yield, transparency, absorption and radio-impurities content are the key parameters of the LS. Given the huge dimensions of the CD, the attenuation length of the LS must be >20 m at 430 nm. Since JUNO is a low background experiment, also stringent radiopurity requirements must be satisfied: for ^{238}U and ^{232}Th, the maximum content must be $<10^{-15}$ g/g. All these optical [5] and radiopurity features are crucial to achieve the experimental goals of JUNO. For this purpose, the LS will be purified by a sequence of purification processes: alumina filtration plant, distillation under vacuum, mixing plant for master soution (LAB + concentrated PPO and bis-MSB) production and washing, water extraction and gas stripping. Each process is performed in a dedicated plant at JUNO site. In the following, the details and results of the Distillation and Stripping plants are presented, which have been designed and optimized by JUNO INFN-Milano group and Polaris s.r.l. company, after a successful test campaign done in 2018 at Daya Bay laboratories, in China, using small scale pilot plants [6].

2·1. *Distillation plant.* – The distillation process is used to remove from LAB the heaviest and high-boiling radioactive metals, such as ^{238}U, ^{232}Th and ^{40}K, and to further improve its optical properties, mainly in terms of transparency, attenuation length and absorption spectrum in the region of interest of 300 nm-500 nm wavelengths. The distillation consists in boiling the LAB inside a 7 m-high distillation column: the high-boiling and low volatile contaminants remain in the bottom part of the column, which is discarded regularly every 30 minutes, while the lightest ones can reach the top part, where they are condensed and collected into the product tank. A reflux ratio up to 50% of the nominal flow rate (7 m^3/h) of distilled LAB can be immediately sent back the column to enhance the purification efficiency. The column is equipped with 6 sieve trays with holes, where a layer of liquid can build-up, ensuring an intimate contact with the upward stream of LAB vapours. Each tray corresponds to a stage of equilibrium condition, thus forming multiple gas-liquid equilibrium stages along the distillation column. The plant is designed to operate in partial vacuum (60 mbar$_a$) at about 220°C, thus reducing the LAB boiling temperature, also to avoid thermal degradation, and increasing the purification efficiency of the process. The boiling temperature can be set controlling the pressure at the bottom (60 mbar$_a$) and top (5 mbar$_a$) parts of the column: the pressure difference is given by the total layer of liquid on the trays ($\sim$78 cm with 720 kg/m^3 average LAB density at 200°C, $\sim$13 cm on each tray).

2·2. *Stripping plant.* – The gas stripping is a separation process used to remove gaseous impurities naturally dissolved into the LS, mainly ^{222}Rn, ^{85}Kr,^{39}Ar, which could generate undesired signals, and O_2, that causes oxidation and photon quenching in LS. The liquid phase is purified by desorption mechanisms, flushing a stream of N_2 and/or purified water steam in counter current mode. The process is performed inside a 9 m-high stripping column, where the LS (nominal flow rate: 7 m^3/h) is fed from the top and driven down by gravity, while the gas is supplied from the bottom. The column is internally filled with an unstructured packing, consisting of metallic Pall rings, to ensure a high contact surface between the gas and the liquid phases. The column has been studied to perform the stripping process both with high-purity N_2 (max 40 Nm^3/h) or ultra-pure water (max 40 kg/h), which is evaporated and overheated before entering the column, or a variable mixture of them. The plant is designed to operate at around 300 mbar$_a$ and 90°C, thus reducing the LS viscosity and increasing the purification efficiency.

2.3. *Common features.* – Several precautions have been taken to do not spoil the quality of the LS. Dedicated cleaning procedures have been adopted to treat all internal surfaces of the plants and reach the cleanliness requirements of JUNO (MIL STD 1246 Level 50). 50 nm-pore size filters have been installed to prevent dust and particles to circulate into the plant.

The sealing of each flange and component has been certified with a He leak detector to be $<10^{-8}$ mbar·l/s ($<10^{-6}$ mbar·l/s as integral leak rate). A vacuum pumping and nitrogen purging procedure has been carried out several times to remove O_2 and Rn. The tanks and the column are kept under N_2 overpressure blanketing, while interconnection points and flanges are continuously purged with N_2.

3. – Results of the first commissioning

The first round of internal commissioning has been successfully concluded at JUNO site both for Distillation and Stripping plants. They were run continuously for more than 10 hours for several times, in internal loop mode, recirculating back the purified LS to the input feed tank. The operating parameters were optimized to maintain nominal flow rates while ensuring suitable purification efficiencies. Both plants ran very stably and safely.

For the Distillation plant, the most critical issue was the fine adjustment of the LAB condenser together with temperatures and flow rates of the distilled LAB and the reflux ratio: the goal is to have the condensed LAB outlet at ∼120°C, in order to do not subcool the reflux going back to the column and have an optimal energy and heat recovery in the following heat exchanger. The cooling power of the condenser should be adjusted step-by-step, by carefully regulating the valves while keeping the temperatures and flow rates monitored.

For the Stripping plant, the stripping process was tested both with N_2 and water steam. We found some issues while using water steam, because of different solubility levels of water into LAB at different temperatures: when cooling the LS down to room temperature after stripping, the dissolved water condensates and creates microscopic suspended droplets, turning the LS milky and temporarily spoiling its transparency. After suitable recovery time (>24h), water and LS separate and the quality of the latter is restored. Anyway, it is safer to strip only with N_2, to avoid having too much water in the scintillator. All further commissioning of the plant has been done using only HP-N_2.

During the commissioning phase, several samples have been collected and analysed to evaluate the purification efficiencies of the two plants. Some preliminary results of the absorption spectra of raw LAB, distilled LAB and stripped LAB+PPO are shown in fig. 1. As you can see, both the distilled and stripped samples are significantly lowered in the absorption peaks at 370 and 390 nm compared to raw LAB, thus meaning that the optical impurities were successfully removed by distillation (green, solid line) and the quality not spoiled after stripping plant (blue, dash-dot line). For the latter, the

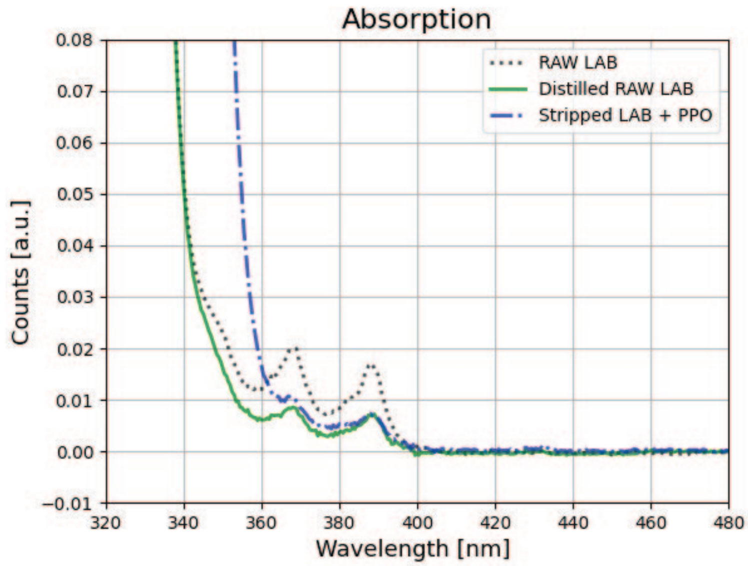

Fig. 1. – Absorption spectra of raw LAB, distilled LAB and stripped LAB+PPO.

absorption edge starts to raise at around 360 nm because of the presence of PPO, which shifts the absorption towards higher wavelengths.

Further tests, such as particle counting, attenuation length measurements, ICP-MS, NAA, are ongoing to verify fully compliance with JUNO standards.

4. – Conclusions and perspectives

This work summarizes the features and the progress status of the Distillation and Stripping plants, which have been designed and built for the purification of the liquid scintillator of JUNO. The plants are installed at JUNO site and the internal commissioning phase has been successfully completed, tuning the operating parameters of the plants and verifying the purification efficiency. Some preliminary results on absorption spectra have been presented.

The joint commissioning with all other purification plants is foreseen in the nearby future, to test the LS production mode to be adopted during the 6-months filling of JUNO detector, scheduled to start in 2024.

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