

# Distillation and stripping pilot plants for the JUNO neutrino detector: design, operations and reliability

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## 49 **ABSTRACT**

50 This paper describes the design, construction principles and operations of the distillation and  
51 stripping pilot plants tested at the Daya Bay Neutrino Laboratory, with the perspective to adapt this  
52 processes, system cleanliness and leak-tightness to the final full scale plants that will be used for the  
53 purification of the liquid scintillator used in the JUNO neutrino detector. The main goal of these  
54 plants is to remove radio impurities from the liquid scintillator while increasing its optical attenuation  
55 length. Purification of liquid scintillator will be performed with a system combining alumina oxide,  
56 distillation, water extraction and steam (or N<sub>2</sub> gas) stripping. Such a combined system will aim at  
57 obtaining a total attenuation length greater than 20 m @430 nm, and a bulk radiopurity for <sup>238</sup>U and  
58 <sup>232</sup>Th in the 10<sup>-15</sup>÷10<sup>-17</sup> g/g range. The pilot plants commissioning and operation have also provided  
59 valuable information on the degree of reliability of their main components, which will be particularly  
60 useful for the design of the final full scale purification equipment for the JUNO liquid scintillator.  
61 This paper describe two of the five pilot plants since the Alumina Column, Flour mixing and the  
62 Water Extraction plants are in charge of the Chinese part of the collaboration.

63 *Keywords: LAB, radiopurity, liquid scintillator, attenuation length, scintillator transparency,*  
64 *light yield, nitrogen purging, large-scale experiments*

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## 67 **1 Scientific Motivations**

68 The extraordinary scientific results of the Borexino [1], Daya Bay [2], Double Chooz [3],  
69 KamLAND [4] and RENO [5] experiments pave the way for a new generation of multi-kiloton  
70 detectors that adopt the Liquid Scintillator (LS) detection technique (JUNO [6], RENO50[7], SNO+  
71 [8], ANDES [9], JINPING[10]).

72 The Jiangmen Underground Neutrino Observatory (JUNO) is a multi-purpose neutrino  
73 experiment, proposed mainly for neutrino mass ordering determination (mass hierarchy) by detecting  
74 reactor anti-neutrinos from two sets of nuclear power plants at a 53 km distance. JUNO, deployed in  
75 an underground laboratory (700 m overburden), consists in a central detector, a water Cherenkov  
76 detector and a top muon tracker. The central detector will be filled with 20 kton of LS and it will be  
77 submerged in a water pool, acting as a shield from the natural radioactivity of the surrounding rock.  
78 The water pool, in turn, will be instrumented with photomultipliers to act as a Cherenkov detector  
79 vetoing cosmic rays background. On top of the water pool, a muon tracker system will accurately  
80 measure incoming muons.

81 The JUNO Liquid Scintillator is a specific organic compound containing molecules featuring  
 82 benzene rings that can be excited by ionizing particles; it is designed to be composed by Linear Alkyl  
 83 Benzene (LAB) as solvent, doped with 2,5-Diphenyloxazole (PPO 2.5 g/l) as primary solute, and 1,4-  
 84 Bis(2-methylstyryl)benzene (bis-MSB 7 mg/l) as wavelength shifter.

85 Low-background conditions are crucial for the success of JUNO. From the point of view of  
 86 the LS, this means that the concentration of radioactive impurities inside the mixture should result in  
 87 an activity of the same level or below the rate of neutrino events. Radiopurity levels are usually  
 88 specified by the concentration of  $^{232}\text{Th}$ ,  $^{238}\text{U}$  and  $^{40}\text{K}$  in the LS and their typical concentration in the  
 89 environmental sources are listed in Table 1. The baseline scenario, which will be desirable for the  
 90 detection of reactor antineutrinos in JUNO, assumes a contamination in the range of  $10^{-15}$  g/g of U  
 91 and Th and  $10^{-15}$  g/g of  $^{40}\text{K}$  [11] in the LS. A more stringent regime, in the realm of  $10^{-17}$  g/g, would  
 92 instead be needed to accomplish the JUNO neutrino Astroparticle program.

93 **Table 1** List of the main radioisotopes solute in the organic liquid scintillators with their sources of contamination and  
 94 the typical concentration of the impurities in the sources [12,13]. In the last two columns are presented the removal  
 95 strategies used by the main neutrino experiment to reduce the radioimpurities contained in the LS and the JUNO  
 96 radiopurity requirements[6,11].

Radioisotope	Contamination source	Typical value	Removal strategy	JUNO requirement
$^{222}\text{Rn}$	Air and emanation from material	$<100 \text{ Bq/m}^3$	Stripping	-
$^{238}\text{U}$	Dust suspended in liquid	$\sim 10^{-6} \text{ g/g}$	Distillation and Water Extraction	$<10^{-15} \text{ g/g}$
$^{232}\text{Th}$	Dust suspended in liquid	$\sim 10^{-5} \text{ g/g}$	Distillation and Water Extraction	$<10^{-15} \text{ g/g}$
$^{40}\text{K}$	PPO used as doping material	$\sim 10^{-6} \text{ g/g}$	Distillation and Water Extraction	$<10^{-15} \text{ g/g}$
$^{39}\text{Ar}, ^{42}\text{Ar}$	Air	$\sim 1 \text{ Bq/m}^3$	Stripping	-
$^{85}\text{Kr}$	Air	$\sim 1 \text{ Bq/m}^3$	Stripping	$1 \mu\text{Bq/m}^3$

97 While members of the natural  $^{232}\text{Th}$  and  $^{238}\text{U}$  decay chains are the most common  
 98 contaminants, also other sources of radioactive impurities for the LS have to be taken into account.

99 Radioactive impurities can be divided in two main groups according to the process adopted  
 100 to remove them from the LS. Heavy impurities, such as  $^{238}\text{U}$ ,  $^{232}\text{Th}$  and  $^{40}\text{K}$ , can be discarded through  
 101 distillation and water extraction, while more volatile impurities, such as  $^{222}\text{Rn}$ ,  $^{39}\text{Ar}$ ,  $^{42}\text{Ar}$  and  $^{85}\text{Kr}$ ,  
 102 through steam or nitrogen stripping. Table 2 displays the concentrations of LS contaminants obtained,  
 103 after purification, by the main neutrino experiments. It is important to notice that only Borexino and  
 104 KamLAND achieved the radiopurity standard needed for JUNO.

105 The JUNO physics program requires reaching an energy resolution (3% at 1 MeV) never  
 106 achieved before in any large-mass liquid scintillator neutrino experiment. In order to reach the  
 107 required light collection, the attenuation length has to be comparable to the diameter of the LS acrylic  
 108 chamber ( A.L.> 20 m at 430 nm [6]). The 430 nm value has been selected since it is in the wavelength  
 109 region where the PMTs are more sensitive.

110 The optical performances of the LS are mainly affected by the solvent production methods,  
 111 and its method of transportation, but the LS attenuation length [14] is influenced also by the different

112 absorbance and cleanliness of each solute (see Table 3). The raw LAB attenuation length, from high  
 113 quality industrial production, is about 15 m [15], while it could become less than 10 m in standard  
 114 industrial quality production. For Daya Base pilot plants test a special LAB produced by SINOPEC  
 115 Jinling Petrochemical Company was selected. Its typical composition is reported in Table 4.

116 Moreover, any oxidation of the LAB worsens substantially its optical properties, so it is  
 117 mandatory to avoid any contact between oxygen and the LAB, by keeping any transportation and  
 118 storage vessel under a nitrogen blanket while removing any air leaks through the connections.

119 **Table 2** Purification efficacy for different radioisotope in the main LS neutrino experiment (Daya Bay [16], Borexino[17],  
 120 KamLAND [18] and Double Chooz [19]) in terms of concentrations of radioactive impurities in the LS or event rate.

Experiment	Radioisotope	Concentration
Daya Bay	$^{238}\text{U}$	$<10^{-12}$ g/g
	$^{232}\text{Th}$	$<10^{-12}$ g/g
Borexino	$^{238}\text{U}$	$(5.3 \pm 0.5) \cdot 10^{-18}$ g/g
	$^{232}\text{Th}$	$(3.8 \pm 0.8) \cdot 10^{-18}$ g/g
	$^{40}\text{K}$	$< 0.42$ cpd/100 ton-LS
	$^{222}\text{Rn}$	$(1.72 \pm 0.06)$ cpd/100 ton-LS
	$^{39}\text{Ar}$	$\sim 0.4$ cpd/100 ton-LS (95% C.L.)
	$^{210}\text{Bi}$	$(41.0 \pm 1.5(\text{stat}) \pm 2.3(\text{sis}))$ cpd/100 ton-LS
	$^{85}\text{Kr}$	$(30.4 \pm 5.3(\text{stat}) \pm 1.5(\text{sis}))$ cpd/100 ton-LS
KamLAND	$^{238}\text{U}$	$(1.87 \pm 0.10) \cdot 10^{-18}$ g/g
	$^{232}\text{Th}$	$(8.24 \pm 0.49) \cdot 10^{-17}$ g/g
	$^{40}\text{K}$	$(1.30 \pm 0.11) \cdot 10^{-16}$ g/g
	$^{39}\text{Ar}$	$<4.3 \cdot 10^{-21}$ g/g
	$^{210}\text{Pb}$	$(2.06 \pm 0.04) \cdot 10^{-20}$ g/g
	$^{85}\text{Kr}$	$(6.10 \pm 0.14) \cdot 10^{-20}$ g/g
Double Chooz	$^{238}\text{U}$	$<10^{-13}$ g/g
	$^{232}\text{Th}$	$<10^{-13}$ g/g

121  
 122 In order to test the purification efficiency of the purification process on a LAB based liquid  
 123 scintillator, it has been decided to build pilot plants with a maximum flow rate of 100 kg/h that will  
 124 process the LS needed for the filling of one Daya Bay detector in less than 10 days ( $23.5 \text{ m}^3$ ). In this  
 125 paper, we focus on the design and operations done during the commissioning phase of distillation and  
 126 stripping pilot plants, while  $\text{Al}_2\text{O}_3$  filtering system and Water Extraction plant will not be described  
 127 here since they are in charge of the Chinese part of the collaboration.

128 Nevertheless, just for comparison, it is worth to mention that one of the plants designed to  
 129 remove optical impurities and increase the attenuation length of LAB is the  $\text{Al}_2\text{O}_3$  (alumina oxide)  
 130 filtering system. Alumina is very effective in removing optical contaminants through absorption  
 131 mechanism. Optical impurities, in principle, could, be removed also through a distillation process by  
 132 retaining, in the lower part of the column, the high boiling point compounds (such as dust, metal  
 133 particle and usually oxides) that can affect the light transmittance of the LAB. The last purification  
 134 system is the Water Extraction plant that is based on the ‘‘Scheibel column’’ design and is intended to  
 135 remove radioactive contaminants like  $^{238}\text{U}$ ,  $^{232}\text{Th}$  and  $^{40}\text{K}$  [29].

136 In this paper, it is presented the achieved result, obtained with the distillation pilot plant, in  
 137 removing with high efficiency the optical contaminants.

138 The continuous many-months operation, implied by the JUNO detector filling, sets severe  
 139 constraints on the reliability of the final plants. Motivated by these requirements, in Sec. 3 we discuss  
 140 a reliability model for the distillation and stripping plant based on the data obtained from the operation  
 141 of the pilot plants during the commissioning and test phases.

142 **Table 3** Composition of the solvent and solute of the organic LS of the main neutrino experiments (Daya Bay [15, 16,  
 143 20], Borexino[13, 17, 24, 26], KamLAND [4, 18, 21,22], Double Chooz[3, 14, 19] and RENO [5, 7, 23]) together with  
 144 the attenuation length measured at a wavelength of 430 nm after the purification cycle. The attenuation length given for  
 145 KamLAND was measured at a wavelength of 436 nm.

Experiment	Solvent	Solute	Attenuation length (m)
Daya Bay	LAB	1 g/l Gd 3 g/l PPO 15 mg/l bis-MSB	$14 \pm 4$
Borexino	PC	1.45 g/l PPO	$\sim 10$
KamLAND	80% Dodecane 20 % PC	1.36 g/l PPO	$12.7 \pm 0.4$
Double Chooz	80% n-Dodecane 20 % o-PXE	4.5 g/l Gd-(thd) <sup>3</sup> 0.5% wt Oxolane 7 g/l PPO 20 mg/l bis-MSB	$7.8 \pm 0.5$
RENO	LAB	3 g/l PPO 30 mg/l bis-MSB 1 g/l Gd	$>10$

146 **Table 4** Composition of special LAB used for the commissioning of the distillation and stripping test at Daya Bay  
 147 Neutrino Laboratory produced by SINOPEC Jinling Petrochemical Company. LAB is a mixture of compound that can be  
 148 expressed in terms of n in the form of  $(C_6H_5)-C_nH_{2n+1}$ .

Components $C_6H_5C_nH_{2n+1}$	Concentration %
n = 9	0 %
n = 10	10 %
n = 11	35 %
n = 12	35 %
n = 13	20 %
n = 14	0 %

149

## 150 2 Distillation and stripping pilot plant overview

151 Distillation and stripping technologies are widely used for purification of Liquid Scintillators  
152 in large-scale neutrino experiments. In this respect, the JUNO LS purification system has a  
153 particularly difficult task since both excellent radiopurity and extraordinary optical quality have to be  
154 reached. In addition, a high production rate must be achieved together with compliance with Chinese  
155 and European safety regulations. In the following sections, we describe the main features of the  
156 distillation and stripping pilot plants, installed at the Daya Bay site. Pilot plants design, construction  
157 and operation has been a crucial step to understand and prove purification efficiency. All the  
158 knowledge and feedback acquired in this pilot test phase will be crucial to optimize and further  
159 upgrade the design of the full-scale plants of JUNO experiment.

### 160 2.1 Distillation plant

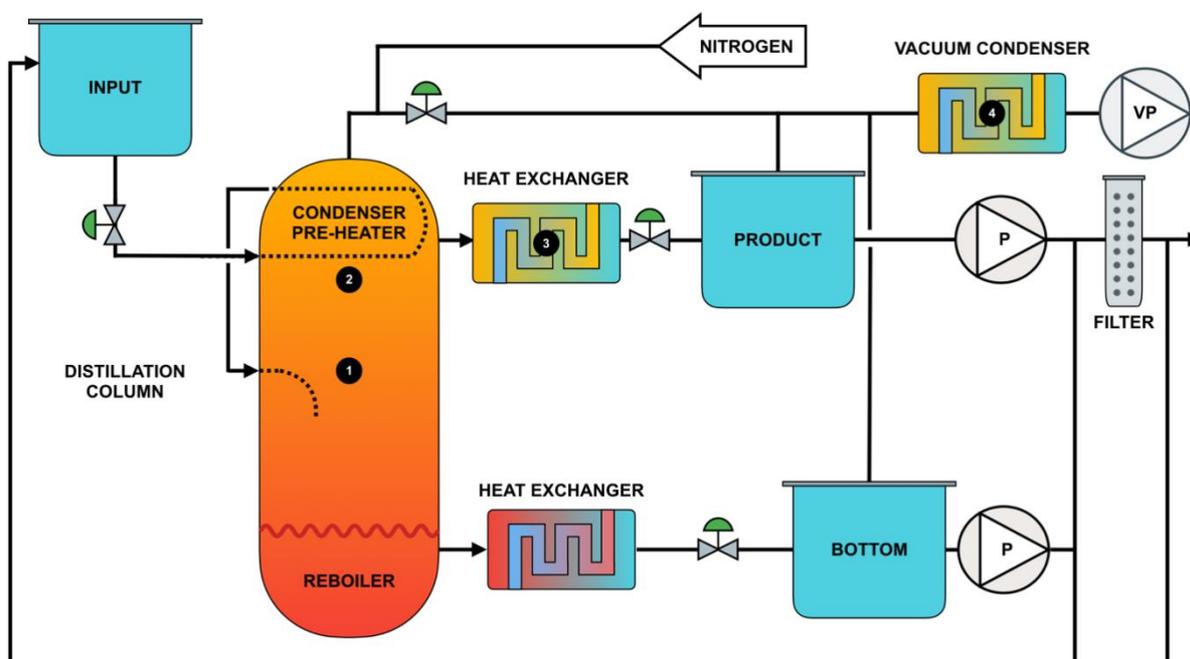
161 Distillation plant is used to remove from the raw LAB the heaviest impurities (mainly  $^{238}\text{U}$ ,  
162  $^{232}\text{Th}$  and  $^{40}\text{K}$ ) and to improve its optical property in terms of absorbance spectrum and attenuation  
163 length in the 350 nm – 550 nm wavelength region. This process is based on the heat and mass transfer  
164 between a liquid and a gas stream, due to the equilibrium conditions reached on each stage of a  
165 distillation column. These conditions depend on the difference of volatility between the constituents  
166 of the input stream and on the temperature and pressure in the column. The low volatility components  
167 are concentrated in the bottom of the system, while the high volatility ones on the top.

168 The distillation is carried out with counter-current flow of the liquid and gaseous LAB in a 7  
169 m high and 2000 mm wide column containing 6 sieve trays (see Fig. 1 and Table 5). In particular, the  
170 height of the column and trays number affect separation capability, while total flow rates determine  
171 the width of the column.

172 The three principal components of the distillation system are the column, the reboiler and the  
173 total condenser. Liquid LAB is fed to the column at a flow rate of about 100 l/h in the middle tray  
174 section (1 in Fig. 1), after being preheated ( $\sim 160\text{ }^\circ\text{C}$ ) in the vapour condenser (2 in Fig. 1) on the top  
175 of the column. The liquid stream, falling down by gravity through the sieve trays, reaches the reboiler,  
176 which evaporates the liquid with a  $15\text{ kW}_{\text{th}}$  electric heater (immersed resistors) generating the counter  
177 current flow of vapor. Temperature in the reboiler is around  $200\text{ }^\circ\text{C}$  depending on the column actual  
178 pressure and the LAB chain composition. The trays are designed in order to establish an intimate  
179 contact between the liquid stream and the gas stream for a sufficient period of time allowing the heat  
180 and mass transfer between the phases. This process enriches the liquid stream in the less volatile  
181 components (in particular  $^{238}\text{U}$  and  $^{232}\text{Th}$  and heaviest impurities) and decreases the temperature of  
182 the vapors. The liquid and vapor flows must be kept within a limited operating range to assure a good  
183 contact surface on the sieve trays.

184 The top of the distillation column features the total condenser (2 in Fig. 1), cooled by the LAB  
185 input flow, where the LAB vapors are liquefied. In this design, the total condenser has the function  
186 of energy recovery. The product liquid stream is then split by the condenser itself in two currents,  
187 one inserted back inside the column as a reflux flow (to increase the efficiency of the distillation  
188 process) and the other directed to the water based heat-exchanger (3 in Fig. 1) for the sub-cooling to  
189 ambient temperature and then sent to the product tank.

190 The distillation pilot plant is operated with a nominal reflux ratio of 25%, adjusted varying  
191 the product flow, and a 2% of the input flow discharge from the bottom of the column in order to get  
192 a good compromise between the product purity and a reasonable throughput [12].



193

194 **Fig. 1.** Distillation pilot plant sketch (not in scale). The raw LAB from the input tank falls by gravity through the top of  
195 the column where is pre-heated by the LAB vapour inside the total condenser installed right on top of the column (2). It  
196 is then, at a temperature of roughly 160 °C, sent to the column at the middle tray (1) where it falls down in the electric  
197 reboiler (~200 °C) integrated in the distillation column itself. The reboiler generates heat with submerged electric  
198 resistances. The LAB vapours are then condensed in the top of the column and split in the product stream and in the reflux  
199 stream (~ 25% of the product stream). The flow of the distilled LAB is then cooled down at ambient temperature (3) and  
200 collected in the product tank. The discharge flow (~ 2% of the input stream) from the reboiler and sent to its collecting  
201 tank after being cooled down at ambient temperature. The pressure inside the distillation column, the product tank and  
202 the bottom tank is kept constant at a value of 5 mbar<sub>a</sub> with a scroll vacuum pump (VP) and a continuous purge of nitrogen.  
203 The distilled LAB can be then pumped back by a diaphragm pump (P) to the input tank, so to distil it in internal loop  
204 mode, or sent to the next purification step passing through a 50 nm pore filter. In order to recover the LAB discharged  
205 from the bottom of the column it can be pumped back to the input tank.

206 The distilled LAB is then sent to the next purification process through a 50 nm pore filter in  
207 order to retain any dust or metal particles already present or introduced in the stream by the plant  
208 itself.

209 The entire plant is kept under a N<sub>2</sub> blanket provided by a continuous gas flow to avoid any  
210 oxidation inside the column, thus also reducing the risk of fire. The incondensable gas stream, if  
211 present, is then removed from the top of the column by a dry scroll vacuum pump, in order to keep a  
212 constant pressure of 5 mbar inside the column, passing through a vacuum condenser (4 in Fig. 1) to  
213 liquefy any possibly LAB vapor dragged by the nitrogen flow.

214 The plant can be operated in two different ways: the internal loop mode, where the LAB from  
215 the product tank and the filter, is sent back to the feed tank, and the continuous mode where the feed  
216 tank (1 m<sup>3</sup>) is constantly filled with raw LAB and the distilled LAB is sent from the product tank (0.5  
217 m<sup>3</sup>) to the next purification step continuously. The first configuration is used only in the start-up  
218 phase of the plants or if a stop of the detector filling occur, while the second is the production mode.

219 **Table 5** Main operational parameters for the different features of the distillation pilot plant tested at Daya Bay.

Feature	Value
Height	7 m
Diameter	200 mm
Number of trays	6
Pressure	5 mbar <sub>a</sub>
Temperature in the reboiler	200 °C
Temperature in the top of the column	160 °C
Input flow	100 l/h
Reflux flow	25 l/h
Discharge flow	2 l/h
Nitrogen flow	2 kg/h
Electrical Power for the heater	20 kW <sub>th</sub>
Cooling Power	14 kW <sub>th</sub>
Feed tank Volume	1 m <sup>3</sup>
Product tank Volume	0.5 m <sup>3</sup>
Bottom Tank Volum	0.5 m <sup>3</sup>

220 The solutions listed below are adopted in order to achieve better performances in terms of  
 221 removal of the radioactive impurities, energy saving and cleanliness.

- 222 • Sieve Trays: they have the simplest design among various tray types and feature neither  
 223 mechanical moving parts nor welding, which permits an easy and effective cleaning. The trays  
 224 have 55 holes with a diameter of 12 mm to allow a good contact surface between the vapor  
 225 and the liquid phase and no down-comer in order to avoid any parts that could be difficult to  
 226 clean. The size and number of the holes in trays are based on nominal flow rates of vapor  
 227 rising up and liquid falling down the column. If the flows are too high or too low, bypassing  
 228 occurs, reducing the contact surface and the stage efficiency.
- 229 • Total Condenser: the condenser is positioned directly on the top of the column in order to  
 230 reduce the size of the plant. Moreover, the LAB vapor is cooled down by the LAB liquid input  
 231 stream. The pre-heating of the LAB input stream permits an energy recovery of the order of  
 232 10 kW<sub>th</sub>, while also avoiding the destabilization of the column temperature profile, due to the  
 233 insertion of cool fluid in the middle.
- 234 • Vacuum distillation column: in order to achieve better purification performances, the  
 235 distillation process pressure is kept below 5 mbar<sub>a</sub>, increasing the difference between the  
 236 vapor pressure of the LAB and that of heaviest impurities. A low pressure inside the column  
 237 reduces the LAB boiling temperature (less than 200 °C), decreasing effectively the risk of  
 238 thermal degradation of LAB.
- 239 • At the design conditions of 100 l/h feed and reflux ratio 1, the six-tray column was predicted  
 240 to have four theoretical stages based on design correlations.

241

## 242 2.2 Stripping plant

243 After LAB purification through Alumina and Distillation plants, liquid scintillator is prepared  
244 by online mixing of purified LAB with the right percent of a Master Solution mixture (MS). MS is a  
245 concentrated solution of LAB + 100 g/l PPO and 280 mg/l bisMSB, pre-purified in a dedicated plant  
246 (water extraction in batch mode). Liquid scintillator stream is finally processed through Water  
247 Extraction and Stripping plants.

248 The gas stripping is a separation process in which, one or more dissolved gases are removed  
249 from the liquid phase and transferred to the gas phase by the desorption mechanism. For example,  
250 radioactive gases (mainly  $^{85}\text{Kr}$ ,  $^{39}\text{Ar}$  and  $^{222}\text{Rn}$ ) and oxygen (which potentially decreases the light  
251 yield due to photon quenching) can be removed from the scintillator mixture by stripping it with a  
252 variable mixture of superheated steam and nitrogen in counter current mode. The stripping pilot plant  
253 was designed to measure the process efficiency with superheated steam,  $\text{N}_2$  or a combination of the  
254 two in order to identify the best configuration for the future full size plants.

255 In this paragraph

256 The pre-heated liquid stream (2 in Fig. 2) enters the stripping column (1 in Fig. 2) from the  
257 top and falls down by gravity through an unstructured packing (Pall rings) that permits a high contact  
258 surface between the liquid and the gas coming from the bottom of the column (Fig.2 and Table 5).

259 The concentrations of dissolved gases in the two streams ( $y_i$  for the liquid phase and  $x_i$  for the  
260 gas mixture) vary in each stages of the column, depending on the equilibrium conditions between  
261 liquid and gaseous flows, as governed by the Henry law:

$$262 \quad \square \quad y_i \cdot p_t = H_i \cdot x_i$$

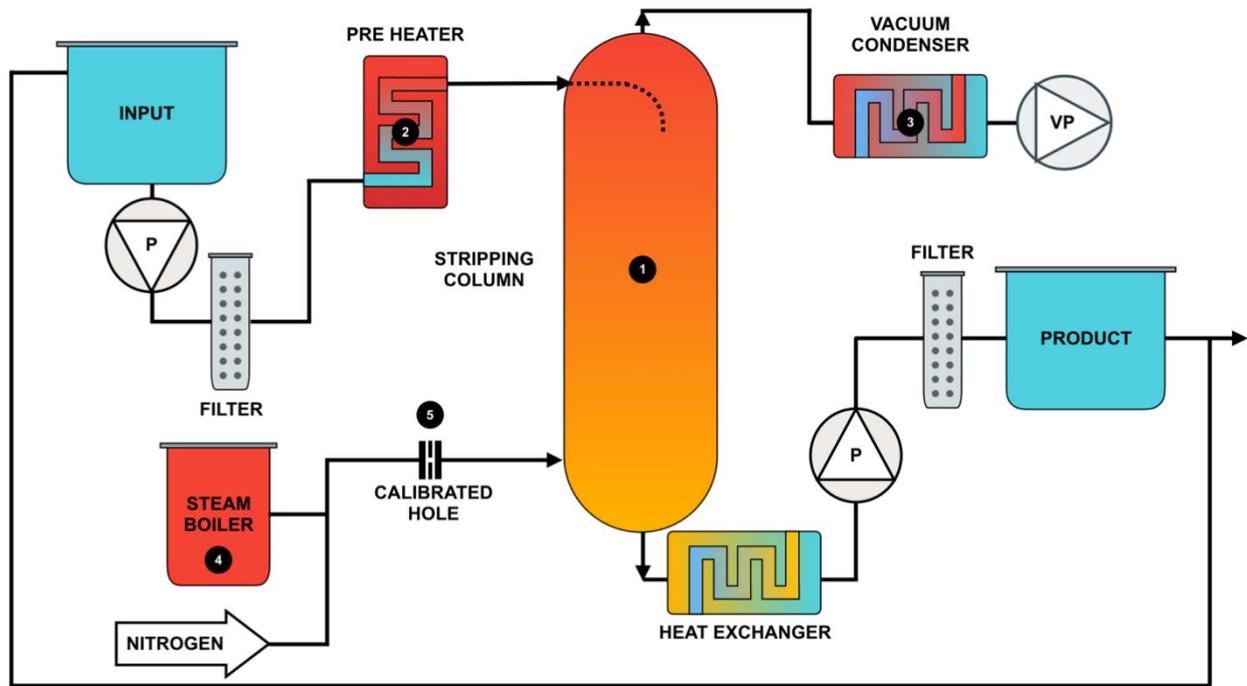
263 where  $p_i$  is the process pressure and  $H_i$  the Henry's law constant that depends on temperature,  
264 pressure and the composition of the streams at the  $i$ -th theoretical stage. In order to keep the pressure  
265 gradient constant inside the stripping column, the steam is condensed in vacuum condensers, while  
266 the incondensable constituents of the gas stream are discharged by a scroll vacuum pump (3 in Fig.  
267 2).

268 The Henry constant, in combination with the molar fraction, determines the maximum ratio  
269 between liquid flow  $L$  and gas flow  $G$ . By applying the mass balance to the column:

$$270 \quad \frac{L}{G} \Big|_{\max} = \frac{x_2 - x_1}{y_1 - y_2}$$

271 The optimal liquid-gas ratio is higher than 70% of the maximum L/G ratio, to avoid large gas  
272 flow and high pressure loss inside the column, and lower than 85% of L/G max, not to increase too  
273 much the height of the column due to a minor driving force between liquid and gas.

274 The stripped liquid, collected in the bottom of the column, is sent to the product tank ( $0.5 \text{ m}^3$ )  
275 by a pump through a water based heat exchanger to lower its temperature, and through a 50 nm filter  
276 used to retain the dust and the particulate that can be released by the plant itself.



277

278 **Fig. 2.** Stripping pilot plant sketch (not in scale). The LAB, collected in the input tank from the previous purification  
 279 steps, is pumped by a diaphragm pump (P) to the top of the stripping column after being filtered through a 50 nm pore  
 280 filter and preheated at 80 °C in the oil based heater (1) in order to avoid the condensation of steam inside the liquid stream.  
 281 The gas flow is an adjustable mix of nitrogen and steam produced inside the electrical steam boiler (2) at a pressure >  
 282 150 mbar<sub>a</sub> kept constant by the continuous flow of the steam through a calibrated orifice (5) to the stripping column (1).  
 283 The stripping column is filled with Pall rings in order to maximize the contact surface between the liquid and the gas  
 284 stream. The stripped LAB is then collected in the bottom of the column and sent to the product tank after being cooled  
 285 down in a water based heat exchanger and filtered. The liquid can be then sent back to the input tank or pumped out to  
 286 the filling station of the detector. The gas flow is discharged by a scroll vacuum pump (VP) after being cooled down in  
 287 the vacuum condenser (3) in order to condense the steam remove the water from the stream.

288 The nitrogen used is carefully purified with active carbons at cryogenic temperature to reach  
 289 low concentration of radio-contaminants, because they set a lower limit for the radiopurity that can  
 290 be achieved by gas stripping.

291 The steam flow is produced in a 50 l volume steam boiler (4 in Fig. 2), at a temperature around  
 292 70 °C (pressure around 300 mbar<sub>a</sub>) using ultrapure water from the high purity water plant of Daya  
 293 Bay [16]. Its flow is controlled by a calibrated orifice hole with a diameter of 0.3 mm (5 in Fig. 2)  
 294 located between the heater and the needle valve installed on the superheated steam line before the  
 295 column. Possible condensation of steam in the column is avoided by operative solution. The LS, and  
 296 the entire column as a consequence, is pre-heated at 90 °C. This temperature is 20 °C more than the  
 297 production temperature of the steam at even higher pressure of the column (300 mbar vs 250 mbar).  
 298 These precautions bring the steam a superheated steam at soon as it enter the column. The superheated  
 299 steam could therefore be treated like a gas with no phase separation.

300

301 This plant can be operated in the internal loop mode (during the start-up operations and self-  
 302 cleaning procedures) and in continuous mode where the purified LAB is sent, after stripping, from  
 303 the product tank (0.5 m<sup>3</sup>) to the filling station of the Daya Bay detector.

304 In order to reach the purity and optical standards needed for JUNO, the following design  
 305 options have been adopted.

- 306 • Unstructured Packing: the column is filled with AISI316 Pall rings to increase the contact  
 307 area between the liquid and gas stream. They have been electro polished and effectively  
 308 cleaned before the installation inside the column with an ultrasonic bath.
- 309 • Stripping under vacuum: the reduced pressure can improve the efficiency per theoretical stage  
 310 of gas stripping. On the other hand, the inter-facial mass transport rate is substantially reduced  
 311 in the absence of gas flow. In a stripping column of fixed size, there is an optimal pressure for  
 312 gas stripping: reducing pressure increases the efficiency per theoretical stage, but also  
 313 decreases the number of theoretical stages. The optimal pressure for our stripping operations  
 314 is between 150 and 250 mbar<sub>a</sub>.
- 315 • Steam: the use of steam instead of Nitrogen (the Borexino choice [13]), has two advantages.  
 316 Firstly, it is generally easier to produce ultrapure water than N<sub>2</sub> with a low content of  
 317 radioactive contaminant reaching a concentration of  $^{222}\text{Rn} < 3.4 \cdot 10^{-6}$  Bq/kg and a very low  
 318 concentration in  $^{39}\text{Ar}$  and  $^{85}\text{Kr}$ . [24]. Moreover, using Nitrogen as a stripping gas requires  
 319 adopting an exhaust system to displace it in a sufficiently well ventilated place. The amount  
 320 of dissolved water in LAB at 100% saturation at atmospheric pressure and room temperature  
 321 is ~200 ppm. Stripping at ~250 mbar<sub>a</sub> (even if at a temperature around 90 °C) reduce the  
 322 amount of water dissolved in the LS after the cooling heat exchanger. The measured content  
 323 of water in LS after steam stripping was ~50 ppm and it does not represent an issue for JUNO  
 324 experiment.
- 325 • LS pre-heater: as already mentioned, in order to avoid any condensation of steam in the LS  
 326 stream, the LS is heated at a temperature of 90 °C. Increasing the temperature give also the  
 327 advantage to enhance the stripping efficiency.
- 328 • At the design conditions the 4 m, unstructured packed column was predicted to have three  
 329 theoretical stages.

330 **Table 6** Main operational parameters for the different features of the stripping pilot plant tested at Daya Bay.

Feature	Value
Height	7 m (4 m of unstructured Packing)
Diameter	75 mm
Packing Material	AISI 316 Pall rings
Pressure	150 – 250 mbar <sub>a</sub>
Input LAB Flow temperature	90 °C
Steam temperature	70 °C
Input LAB flow	100 l/h
Steam flow	100 g/h
Nitrogen flow	1 Nm <sup>3</sup> /h
Electrical Power for the heater	10 kW <sub>th</sub>
Cooling Power	5 kW <sub>th</sub>
Feed tank Volume	0.5 m <sup>3</sup>
Product tank Volume	0.5 m <sup>3</sup>

331

332

### 333 **2.3 Common Features**

334 In order to avoid any contamination due to the dust, dirt and oxide particles which could be  
335 released into the detector or liquid handling systems, it is mandatory to use electro-polished 316L  
336 stainless steel and special cleaning process. Following we describe the cleaning procedures adopted  
337 to treat all the parts of the distillation and stripping pilot plants such as pipes, tanks, valves, pumps  
338 and sensors.

339 The desired cleanliness standard for the plant is MIL STD 1246 Level 50 [25], which defines  
340 limits on the residual particulate size distribution. This goal assumes the scintillator causes particulate  
341 wash-off similar to water, and that Class 50 is the acceptable level for the scintillator, assuming the  
342 remaining particulate has a radioactivity similar to dust. Hopefully, the second assumption is not true,  
343 and the remaining particulate is mostly metallic (i.e. less radioactive than dust), resulting in very  
344 conservative specifications for the lines.

345 The procedure has followed these steps [26]:

- 346 • detergent cycle, to remove oil, grease and residuals with Alconox Detergent 8 or equivalent  
347 (concentration 3% at 60 °C);
- 348 • Ultra-Pure Water (UPW) cycle for rinsing (Until resistivity > 4 MΩ cm)
- 349 • pickling and passivation;
- 350 • UPW cycle for final rinsing (Until resistivity > 14 MΩ cm.)

351 Small parts have been cleaned in ultrasonic baths, while bigger parts with appropriate  
352 methods, like spray balls or immersion.

353 Moreover, at the end of each plant we decided to install a (pre-wetted) ultra-filter with the  
354 nominal pore diameter of 50 nm, to retain any kind of particles that can be released by the plant itself.

355 Specific attention is given to avoid leaks through the connections. In particular, all large  
356 flanges and the ones withstanding ambient temperature are sealed with Ansiflex gaskets or Viton  
357 Teflon coated gaskets, while in the high temperature parts of the plant the tightness is assured by  
358 using metal loaded TUF-STEEL gaskets. All process line connections are orbital-welded or TIG-  
359 welded using low thorium content electrodes. Where welding is not possible, metal gasket VCR  
360 fittings are used. Moreover, all instrument probes are connected to the plant with vacuum tight fittings  
361 for high seal, and stainless steel diaphragm sealed valves are used throughout the system. (The overall  
362 integral leak rate of each plant was proved to be less than  $10^{-8}$  mbar-l/s by means of a He leak  
363 detector).

364 The skids have to meet safety European and Chinese requirements in terms of certification of  
365 seismic safety. A Hazop procedure was used to identify potential problems during operations and led  
366 to modifications for the sensing and alarming parts of the system. In order to avoid the prescription  
367 of the PED directive, rupture disks are installed to assure in every tank a local pressure lower than  
368 0.49 bar<sub>g</sub>. In particular, rupture disks are designed to be operative between full vacuum up to the  
369 trigger point of 0.45 bar<sub>g</sub>.

370 All the electric equipment are under ATEX specification [27], in Class 1 Zone 2 T2, to prevent  
371 any fire risk since the LAB temperature is above its flash point in the distillation plant.

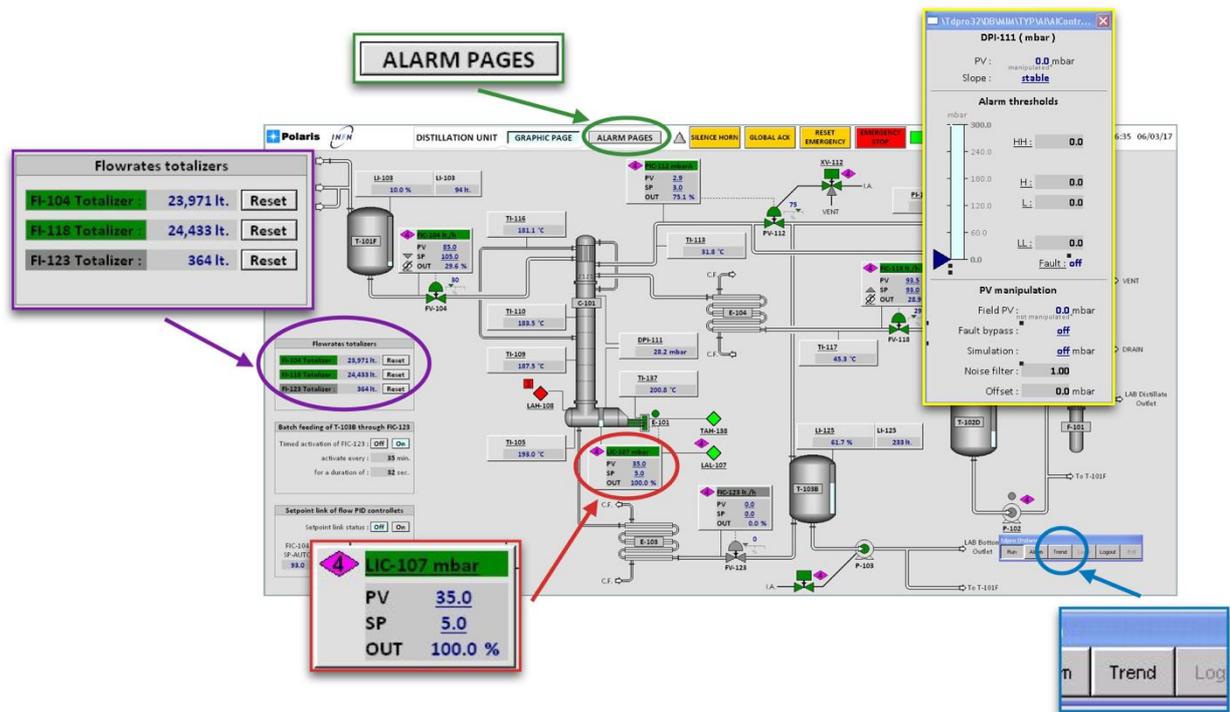
372 All the process pumps used are volumetric diaphragm pumps with Teflon membranes,  
373 installed in the lower part of the plants in order to help the pump priming and to avoid the cavitation  
374 in compliance with instrument NPSH. The pumps used to move liquid from a low-pressure tank to  
375 an ambient pressure tank are compressed air driven DEBEM pump, while in all the other cases we  
376 use motor driven PROMINENT pump.

377 These purification plants need a very stable and reliable Distributed Control System (DCS) to  
378 adjust the purification parameters and to assure the safety of both the plants and the operators,  
379 considering the elevated temperatures that exist in the plant (in distillation mode) and the enclosed  
380 environment in which the plants are located. The purification system has to be under the control of a  
381 master system that provides, for 24-h/day operation, alarm notification, and automated shutdown in  
382 case of problems.

383 It has been decided to adopt a Siemens system for distributed automation because it guarantees  
384 good performances in terms of reliability and a modular and safety oriented design. Moreover, it can  
385 be used in hazardous areas (ATEX Zone 2). The CPU module chosen is the 1512SP-1P. It assures  
386 different communication options between the PLC and the PC with the possibility to integrate a  
387 channel specific diagnostic.

388 The DCS can be controlled and monitored via a SCADA application, designed integrating an  
389 operator friendly User Interface (UI), with the purpose to permit a quick learning of the plant  
390 operations and to understand and solve easily the cause of any alarms generated by the DCS. This  
391 application runs on a Local PC, where it saves all the processes parameter values every minute. It is  
392 linked to the PLC via an Ethernet connection.

393 The general UI is divided in three tabs: an overview of the plant (see Fig. 3), an alarm panel  
394 and a trend panel.



395

396 **Fig. 3.** The slow control User Interface (UI) is designed in order to guarantee a fast identification of the values of the  
 397 process parameter. It is possible to set each instrument alarm thresholds (HighHigh, High, Low and LowLow) and to  
 398 adjust the process parameters with the instrument panel. In the Alarm Pages tab are collected all the previous and active  
 399 alarms and it is possible to examine the progress of each instrument value with the trend graph. The slow control User  
 400 Interface (UI) shows also the flowrates totalizer keeping always under control the amount of processed LS.

401 In the first tab, the core of the UI, it is possible to set the process parameters and the alarm  
 402 thresholds, open and close the automatic valves and turn the pumps on and off. Here the measured  
 403 values of each instrument connected to the DCS are also displayed.

404 The second panel collects all the alarms that are active or were active, but not acknowledged,  
 405 while in the last it is possible to monitor the trend over time of the process values, which are also  
 406 saved on the PC.

407 The DCS manages also part of the safety rules that prevent any damage to the plant and to the  
 408 operators. In particular, it prevents the switch-on of the equipment if the proper conditions are not  
 409 satisfied: for example if the LAB level in the distillation reboiler is not high enough the heaters cannot  
 410 be turned on.

411 It is foreseen also an account based system in order to establish a hierarchy between users of  
 412 the DCS and to give the privileges of change the settings only to expert operators and just monitoring  
 413 capabilities to the guests.

### 414 3 Reliability

415 The JUNO purification plants will have to face the highly demanding challenge of assuring a  
 416 constant delivery of purified LS for the entire filling period. A further hurdle arises from the fact that  
 417 the last stages of the purification process will take place in the underground laboratory with the aim  
 418 of minimizing the length of the pipe from the stripping plant to the filling stations and of reducing  
 419 the risk of contaminating the purified LS. In this scenario, the replacement of LS in case of failure of  
 420 the purification process will be almost unfeasible. For these reasons, a reliability assessment is

421 mandatory in order to identify the less resilient components and possibly maximize the robustness  
 422 and safety of the whole purification system. Essentially It has been decided to use the experience  
 423 gained by the design and operations done on the pilot systems in order to develop a reliability study  
 424 of the future JUNO purification plants. In the following the calculations done for pilot plants are  
 425 given. The collected statistic after 2 years of pilot plants operations is in good agreement with the  
 426 expectations.

427 Reliability is generally defined as the probability  $R(t)$  of successful performance under  
 428 specified conditions of time and use and it is related with the failure rate  $\lambda(t)$  of every single  
 429 component of the system [28]:

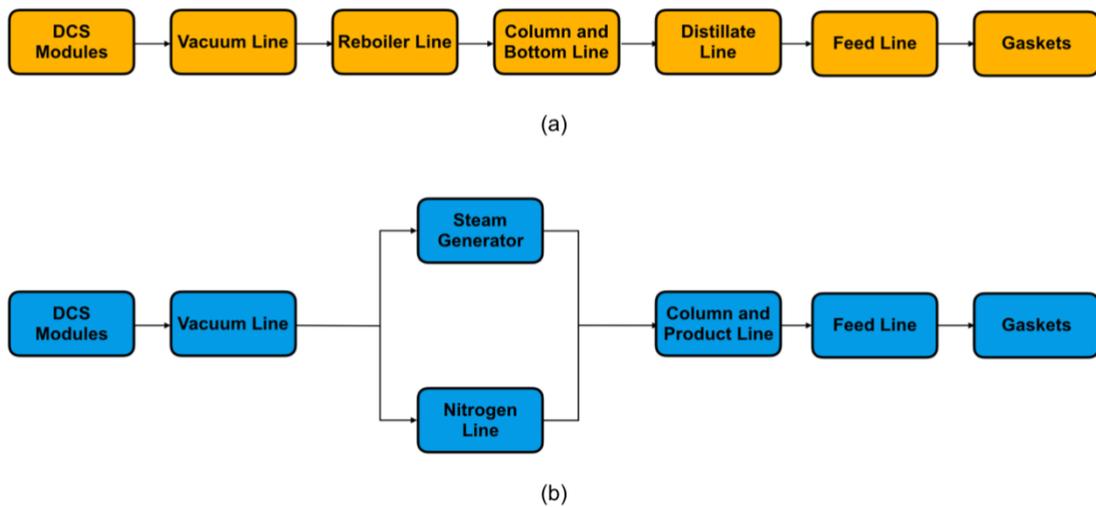
430 
$$R(t) = e^{-\int_0^t \lambda(t) dt} \quad (1)$$

431 The lifetime of a component can be divided in three stages: the infant mortality period when  
 432 the failure rate is not constant and decreases rapidly with time, the life period when the failure rate is  
 433 considered constant and the wear out period where the failure rate increases rapidly due to ageing of  
 434 the component.

435 In our case, the infant mortality period is considered finished after the commissioning of the  
 436 plants, so we consider the components inside the constant failure rate period and it is possible to use  
 437 failure rates from literature or from similar plants.

438 The total reliability of a complex structure can be calculated using the probability theory  
 439 breaking down the entire system in simpler modules or subsystem arranged in series or in parallel  
 440 [28].

441



442

443 **Fig. 4.** Subsystem of the distillation pilot plant (a) and stripping pilot plant (b). The distillation pilot plant total reliability  
 444 can be calculated as the product of the reliability of the single subsystem because all the plant works in series one to each  
 445 other. While the stripping plant reliability can be evaluated as the product of all the other subsystem with the reliability  
 446 of the subsystem composed by the Steam Generator and the Nitrogen.

447 In the distillation plant all the subsystems are arranged in series (see Fig. 4a), implying that  
 448 the total reliability can be estimated using equation (2). In the stripping pilot plant one stage involves

449 a parallel between the Steam Generator and the Nitrogen Line (see Fig. 4b): therefore the total  
 450 reliability  $R_{tot}$  can be evaluated by combining the reliability of the Steam Generator plus Nitrogen  
 451 Line subsystems in parallel using equation (3) with the reliabilities of the remaining components:

$$452 \quad \square R_{tot} = \prod_i \tilde{R}_i \quad (2)$$

$$453 \quad \square R_{tot} = 1 - \prod_i (1 - R_i) \quad (3)$$

454 The failure rate of each components, listed in Table 7, are combined with the previous  
 455 equations to get the final reliability and the Mean Time Between Failure (MTBF) (see Table 8) in  
 456 order to estimate the number of stops for the plants, considering the reliability of the external utilities,  
 457 provided by the lab (i.e. chiller, water supply, nitrogen supply). The reliability of the hand-operated  
 458 valves is set to 1. The MTBF (measured in hours) is correlated with the failure rate through the  
 459 following equation, when  $\lambda(t)$  is considered constant:

$$460 \quad \square MTBF = \frac{1}{\lambda}$$

461 **Table 7** List of the main components of the distillation and stripping pilot plant used and their failure rate given by the  
 462 production company and from Borexino experience.

Component	Failure Rate $\lambda$ (fail/10 <sup>6</sup> h)
Pressure sensor	1.7
Regulating valve	30
Heat exchanger	20
Vacuum pump	15
Level sensor	12
Thermocouple	10.1
Level switch	4.5
On/Off valve	20
Rupture disk	13.5
Centrifugal pump	20
Flow meter	5
Filter	1
Gaskets	0.2
DCS module	1
Filter	1
Steam generator	50
Pressure reducer	0.3

463 Due to a less complex system and less physical objects inside the plant, the stripping system  
 464 has a lower failure probability than the distillation plant. Therefore, it has a longer MTBF meaning a  
 465 longer continuous activity between two stops for maintenance. Finally, considering 6 months of  
 466 continuous working time to fill the JUNO detector, we will have 2 stops in 6 month of continuous  
 467 operation for each plant (stripping and distillation) with a mean down time estimated of 36 h/failure,  
 468 with a total of 3 days of stops for each plant.

469

470 **Table 8** Probability of successful performances (R) and Mean Time Before Failure (MTBF) in months calculated for  
 471 each subsystem composing the distillation and stripping pilot plant and for the entire plants. The model used for the  
 472 calculation is shown in Fig. 4 and the failure rate for each component of the subsystem are listed in

	Line description	R	MTBF (10 <sup>3</sup> h)
Distillation	Vacuum line	0.637	30.9
	Reboiler line	0.797	23.8
	Column + bottom	0.576	7.9
	Distillate line	0.665	7.9
	Feed line	0.722	15.8
	Gaskets (200)	0.916	14.4
	DCS modules	0.961	98.6
	Total	0.124	2.2
Stripping	Vacuum Line	0.835	36.7
	GV	0.698	12.2
	Column + product	0.524	5.8
	Feed line	0.613	8.6
	Nitrogen line	0.978	98.6
	Gaskets (150)	0.936	19.4
	DCS modules	0.961	98.6
	Total	0.235	2.9

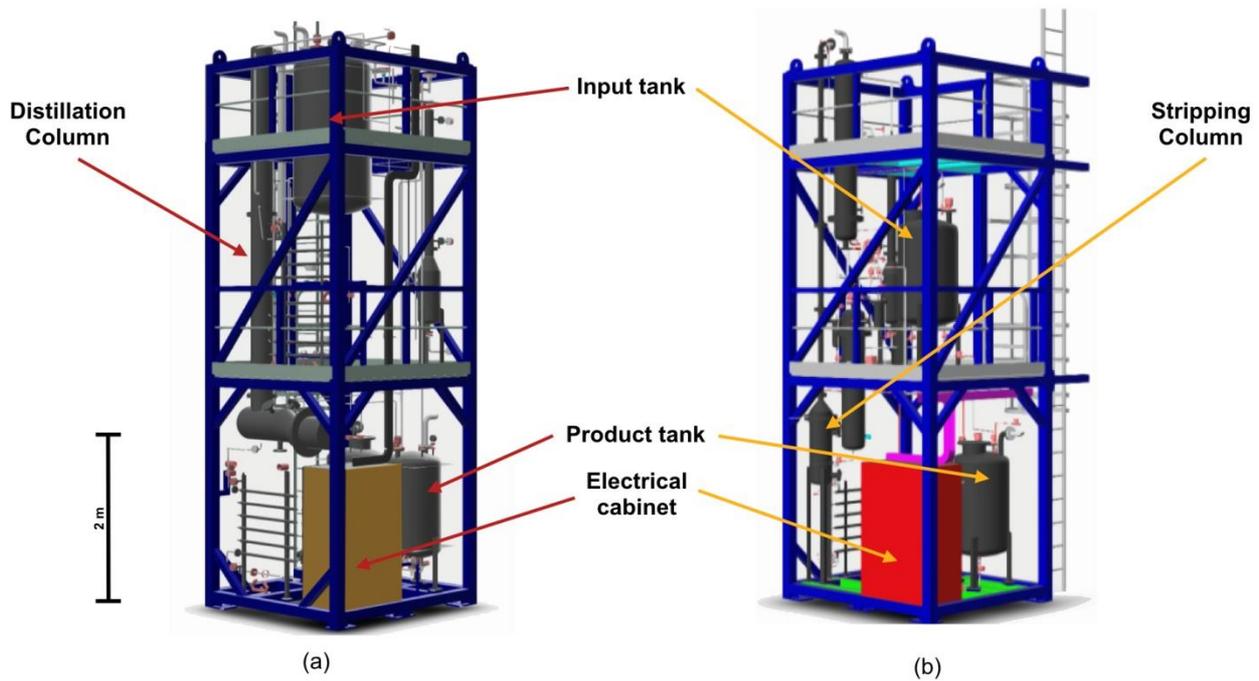
473

474 **4 From designing to commissioning**

475 In 2014-2015 the design and the construction of the JUNO purification pilot plants was  
476 started, with the aim to test them in the Daya Bay Laboratory and to find the optimal process  
477 parameters for the design of the final full scale plants.

478 During the period between 2015-2016, the construction work for the distillation and stripping  
479 plants was carried out in conjunction with Polaris Engineering (MB, Italy) under the supervision of  
480 the Istituto Nazionale di Fisica Nucleare (INFN) crew.

481 The plants were designed and built as a skid-mounted system (see Fig. 5) for transportation  
482 flexibility in China (they fit into two 2.15m x 2.4m x 7m skids). INFN reviewed and approved all  
483 materials, equipment selections and fabrication methods to ensure that the system was leak tight and  
484 had the possibility to be completely cleaned.



485 **Fig. 5.** 3D drawing of the distillation plants skid (a) and stripping plant skid (b). The plants are mounted inside a blue  
486 skid that can fit a standard ISO container for transportation. They are divided in three floors: in the top floor are mounted  
487 the vacuum pumps and the input tanks while in the bottom the product tanks in order to minimize the usage of pumps.  
488 The distillation column and the stripping column are placed on a side of the skids and they run from the top floor to the  
489 bottom floor to minimize the space required for the installation. In the bottom floor, it is enlightened the electrical cabinet  
490 containing the connection for the heaters and pumps power supply and for the CPU of the slow control system receiving  
491 the signals from the instruments.  
492

493 Between February 2016 and March 2016, distillation and stripping pilot plants, under nitrogen  
494 atmosphere, were crated in a container and shipped to Shenzhen, China, by sea. One month later, they  
495 arrived at the Daya Bay laboratory. After the skids were mounted, all the final connections were  
496 made, including the connections to the process lines in Hall 5 of Daya Bay Underground Laboratory.

497 Before the detector filling each plant has been operated in internal loop mode (described in  
498 sec. 2.1 and 2.2) to ensure that they work properly and to adjust the process parameters. During these

499 steps, some problems on the level sensors were identified and solved with a re-calibration of the  
500 instruments via HART communicator.

501 The main features investigated during the commissioning phase were the discharge process  
502 of the LAB from the bottom of the distillation column and the thermodynamic parameters that insure  
503 a stable and efficient functioning of the stripping column. In particular, regarding the first item it was  
504 decided to avoid a continuous discharge of liquid from the bottom of the distillation column because  
505 the magnitude of the flow would have been lower than the minimum value measurable by the flow  
506 meter.

507 Regarding the distillation plant, it was decided to further decrease the pressure inside the  
508 column in order to reduce the temperature of the LAB and avoid any degradation of the organic  
509 compound. In total, around 4000 l of LAB has been distilled and stripped for plant commissioning  
510 and final self-cleaning.

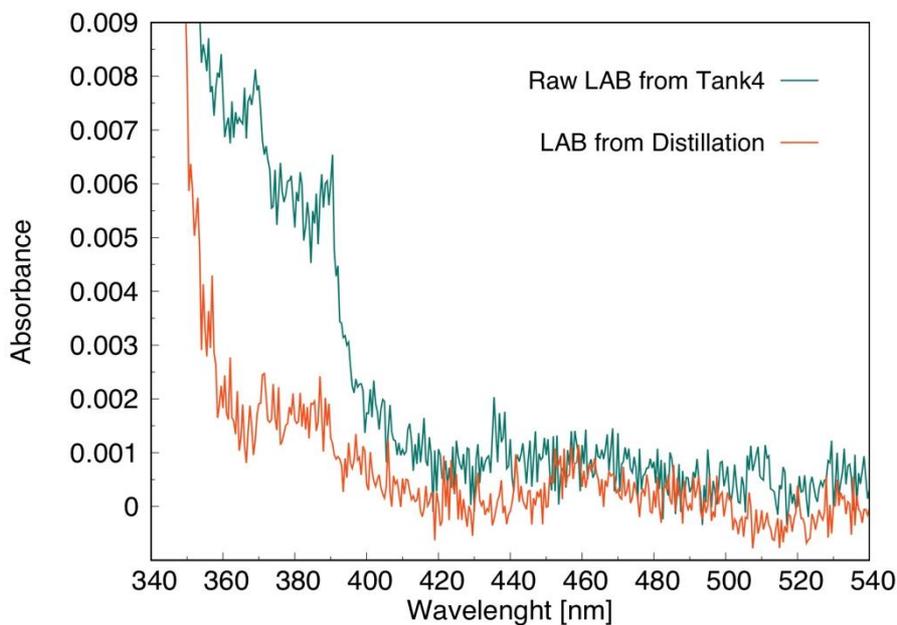
511 After these tests, the plants were connected with Alumina oxide and Water Extraction  
512 purification systems through the interconnection system, to the goal of testing the complete  
513 purification chain. By reference, Alumina Column plant is based on absorption technique on high  
514 quality alumina powder to remove optical impurities and increase the attenuation length of LAB [29]  
515 while Water Extraction column is based on the “Scheibel column” design and is intended to remove  
516 radioactive contaminants like  $^{238}\text{U}$ ,  $^{232}\text{Th}$  and  $^{40}\text{K}$  [29]. These plants are in charge of the Chinese part  
517 of the collaboration and they are not described in this paper.

518

519 **5 Results**

520 The performances of the commissioning phase of the distillation and stripping pilot plants are  
521 assessed by measuring the remaining content of radio impurities in the LAB and its absorption spectra  
522 evaluated after each purification process. The effectiveness of these purification methods in removing  
523 the radio impurities cannot be measured by laboratory tests, giving only generic hints on their  
524 efficacy. The Daya Bay detector, instead, enables the quantitative evaluation of the residual  
525 background in the LAB, which will be reported in the paper describing the full procedure of tests and  
526 measurements performed on the whole sets of pilot plants at Daya Bay.

527 However, meaningful preliminary indications of the effectiveness of the plants can be  
528 gathered indirectly through the inspections of the absorption spectra. Indeed, the LAB attenuation  
529 length and the absorption spectra were measured before filling the detector and after each purification  
530 step [29].



531

532 **Fig. 6.** Comparison of the absorption spectra of raw and distilled LAB (modified from [29]). It is important to notice that  
533 even if the most reduction of the optical impurities is carried out by the alumina plant, the distillation has a small effect  
534 on reducing the attenuation length in the wavelength region around 365 nm.

535 In Fig. 6 the absorption spectrum is reported as a function of the wavelength (where on  
536 abscissa there is the wavelength in nm and on y-axis the absorbance in arbitrary unit). By comparing  
537 the spectrum of the raw LAB with the one after distillation, we can infer the very high effectiveness  
538 of the distillation plant to remove optical impurities over the whole region of interest.

539 Moreover, from [29], it is possible to conclude that the stripping procedure, intended to  
540 remove gaseous compound and hence not expected to affect the absorption spectrum, is clean enough  
541 not to spoil the optical quality as obtained from the previous distillation step.

542

## 543 6 Conclusion

544 This paper described the features and the commissioning phase of a distillation and a stripping  
545 pilot plant designed to test the purification efficiency of this processes for a LAB based liquid  
546 scintillator in terms of removal of radio and optical impurities. Moreover, the study permitted to  
547 evaluate the model built for the calculation of the total reliability of the two pilot plants. For the first  
548 time, well-established technologies are integrated for the purification of a LAB based LS. The  
549 purification effectiveness, the safety of the plants and of the operators are guaranteed adopting the  
550 peculiar features summarized below:

551

- 552 • Using the distillation column input feed (LAB) as a cooling fluid in the total condenser (Fig.  
553 1) leads to a substantial reduction of the energy consumed for the liquefaction of the LAB  
554 vapor and for the warm-up of the input feed. Moreover, positioning the condenser (pre-heater)  
555 on the top of the column implies a substantial reduction of the plant size.
- 556 • The installation inside the distillation column of sieve trays allows to maximize the contact  
557 surface between the liquid and vapor phase keeping a high cleanliness level and in turn to get  
558 a greater efficiency of the distillation.
- 559 • The LAB thermal degradation is reduced by performing the distillation under vacuum with  
560 lower boiling temperature.
- 561 • Using a variable mixture of steam and nitrogen as gas stream in the stripping column leads to  
562 better results on purification efficiency due to the lower  $^{222}\text{Rn}$  content in ultra-pure water, as  
563 compared to regular nitrogen. Moreover, since the steam is completely liquefied in the  
564 vacuum line condenser and the water disposed properly, a dedicated exhaust system is not  
565 necessary.
- 566 • While the stripping process has no effect on the optical property of the LAB, the distillation  
567 increases the attenuation length in the wavelength region of interest (Fig. 6). The attenuation  
568 length measured on scintillator (LAB + 2.5 g/l PPO and 7 mg/l bisMSB) after all the  
569 purification process reaches a value of 20 m @ 430 nm, greater than typical values obtained  
570 in previous neutrino experiments (Table 3). The attenuation length of pure LAB reaches 25  
571 m @ 430 nm after distillation.
- 572 • Adopting the data from the pilot plants, the reliability study for the future JUNO purification  
573 plants shows an average of greater than 3 months of MTBF (Table 6). The JUNO distillation  
574 plant will be more subject to failure due to its greater complexity and number of components.  
575 This model will give also an indication on hierarchy of the most fragile parts of the system  
576 that will need a prompt back-up solution in case of failure.

577 In the perspective of the realization of JUNO, as well as for future massive neutrino  
578 experiments, the distillation and stripping processes are expected to play a key role in reducing the  
579 radio background contamination and in increasing the attenuation length of the LS.

580

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587 operation of the distillation and stripping plants.  
588

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