



The liquid handling systems for the Borexino solar neutrino detector

G. Alimonti^{c,d}, C. Arpesella^{c,d}, M.B. Avanzini^c, H. Back^f, M. Balata^e, D. Bartolomei^g, A. de Bellefon^h, G. Bellini^{c,d}, J. Benziger^{i,*}, A. Bevilacqua^{a,b}, D. Bondi^{a,b}, S. Bonetti^{c,d}, A. Brigatti^{b,c,d}, B. Caccianiga^{c,d}, L. Cadonati^g, F. Calaprice^g, C. Carraro^{a,b}, G. Cecchet^{j,k}, R. Cereseto^{a,b}, A. Chavarria^g, M. Chen^g, A. Chepurinov^l, A. Cubaiu^e, W. Czechⁿ, D. D'Angelo^{c,d,n}, F. Dalnoki-Veress^g, S. Davini^a, A. De Bari^{j,k}, E. De Haas^g, A. Derbin^o, M. Deutsch^o, A. Di Credico^e, A. Di Ludovico^g, G. Di Pietro^e, R. Eisenstein^g, F. Elisei^g, A. Etenko^p, F. von Feilitzsch^p, R. Fernholz^g, K. Fomenko^q, R. Ford^{c,d,e,g}, D. Franco^{c,d,r}, B. Freudiger^{c,d,r}, N. Gaertnerⁿ, C. Galbiati^{c,d,g}, F. Gatti^{a,b}, S. Gazzana^e, V. Gehman^f, M. Giammarchi^{c,d}, D. Giugni^{c,d}, M. Goeger-Neffⁿ, T. Goldbrunnerⁿ, A. Golubchikov^{c,d,p}, A. Goretti^{c,d,e,g}, C. Grieb^{f,n}, E. Guardincerri^a, C. Hagnerⁿ, T. Hagnerⁿ, W. Hampel^r, E. Harding^g, S. Hardy^f, F.X. Hartmann^r, R. von Hentigⁿ, T. Hertrichⁿ, G. Heusser^r, M. Hult^m, A. Ianni^e, An. Ianni^g, L. Ioannucci^e, K. Jaenner^r, M. Joyce^f, H. de Kerret^h, S. Kidner^f, J. Kiko^r, T. Kirsten^r, V. Kobychew^u, G. Korga^e, G. Korschinekⁿ, Y. Kozlov^p, D. Kryn^h, P. La Marche^{e,g}, V. Lagomarsino^{a,b}, M. Laubenstein^e, C. Lendvaiⁿ, M. Leung^g, T. Lewkeⁿ, E. Litvinovich^p, B. Loer^g, F. Loeser^g, P. Lombardi^{c,d}, L. Ludhova^{c,d}, I. Machulin^p, S. Malvezzi^{c,d}, A. Manco^{a,b}, J. Maneira^{c,d}, W. Maneschg^r, I. Manno^{c,d,s}, D. Manuzio^{a,b}, G. Manuzio^{a,b}, M. Marchelli^{a,b}, A. Marternianov^{a,b}, F. Masetti^{v,w}, U. Mazzucato^{v,w}, K. McCarty^g, D. McKinsey^g, Q. Meindlⁿ, E. Meroni^{c,d}, L. Miramonti^{c,d}, M. Misiasek^t, D. Montanari^e, M.E. Monzani^{c,d}, V. Muratova^o, P. Musico^{a,b}, H. Neder^r, A. Nelson^g, L. Niedermeierⁿ, S. Nisi^e, L. Oberauerⁿ, M. Obolensky^h, M. Orsini^e, F. Ortica^{v,w}, M. Pallavicini^{a,b}, L. Papp^e, R. Parsells^g, S. Parmeggiano^{c,d}, M. Parodi^a, N. Pelliccia^{v,w}, L. Perasso^{a,b}, S. Perasso^a, A. Pocar^g, R. Raghavan^f, G. Ranucci^{c,d}, W. Rau^{e,r}, A. Razeto^{a,b,e}, E. Resconi^{a,b,c,d}, P. Risso^{a,b}, A. Romani^{v,w}, D. Rountree^f, A. Sabelnikov^p, P. Saggese^{c,d}, R. Saldanha^g, C. Salvo^{a,b}, R. Scardaoni^{c,d}, D. Schimizziⁱ, S. Schonert^r, K.H. Schubeckⁿ, T. Shutt^g, F. Siccardi^{a,b}, H. Simgen^r, M. Skorokhvatov^p, O. Smirnov^q, A. Sonnenschein^g, F. Soricelli^e, A. Sotnikov^q, S. Sukhotin^p, C. Sule^g, Y. Suvorov^{c,d,p}, V. Tarasenkov^p, R. Tartaglia^e, G. Testera^{a,b}, D. Vignaud^h, S. Vitale^h, R.B. Vogelaar^{f,g}, V. Vyrodov^p, B. Williams^f, M. Wojcik^t, R. Wordel^m, M. Wurmⁿ, O. Zaimidoroga^q, S. Zavatarelli^{a,b}, G. Zuzel^{r,t}, Borexino Collaboration

^a University Genoa, Dipartimento Fis, I-16146 Genoa, Italy

^b Ist Nazl Fis Nucl, I-16146 Genoa, Italy

^c University Milan, Dipartimento Fis, I-20133 Milan, Italy

^d Ist Nazl Fis Nucl, I-20133 Milan, Italy

^e Lab Nazl Gran Sasso, I-67010 Assergi, AQ, Italy

^f Virginia Polytechnic Institute and State University, Department of Physics, Blacksburg, VA 24061, USA

^g Princeton University, Department of Physics, Princeton, NJ 08544, USA

^h Astroparticule and Cosmol APC, F-75205 Paris 13, France

ⁱ Princeton University, Department of Chemical Engineering, Princeton, Engineering Quadrangle, NJ 08544, USA

^j University Pavia, Dipartimento Fis, I-27100 Pavia, Italy

^k Ist Nazl Fis Nucl, I-27100 Pavia, Italy

^l Moscow MV Lomonosov State University, Moscow, Russia

^m EC JRC IRMM, B-2440 Geel, Belgium

ⁿ Technical University Munich, D-85747 Garching, Germany

^o St. Petersburg Nuclear Physics Institute, Gatchina, Russia

^p RRC Kurchatov Institute, Moscow 123182, Russia

^q Joint Institute of Nuclear Research, Dubna 141980, Moscow Region, Russia

^r Max Planck Inst Kernphys, D-69029 Heidelberg, Germany

^s KFKI RMKI, H-1121 Budapest, Hungary

^t Jagiellonian University, Institute of Physics, PL-30059 Krakow, Poland

^u MSP, Institute of Nuclear Research, UA-03680 Kiev, Ukraine

^v University Perugia, Dipartimento Chim, I-06123 Perugia, Italy

^w Ist Nazl Fis Nucl, I-06123 Perugia, Italy

* Corresponding author. Tel.: +1 609 258 5416; fax: +1 609 258 0211.

E-mail address: benziger@princeton.edu (J. Benziger).

ARTICLE INFO

Article history:

Received 10 July 2009

Accepted 11 July 2009

Available online 28 July 2009

Keywords:

Liquid scintillator

Radiopurity

Solar neutrino

Low background detectors

ABSTRACT

The successful deployment of the Borexino solar neutrino detector required assorted physical and chemical operations to produce exceptional pure fluids and fill multiple detector zones. The composition and flow rates of high purity gases and liquids had to be precisely controlled to maintain liquid levels and pressures. The system was required to meet exceptional requirements for cleanliness and leak-tightness. A large scale modular system connecting fluid receiving, purification and fluid delivery processes was developed for Borexino. At the core is a flow control system that delivers scintillator components to plants for purification, and then fills the Borexino detector volumes with ultrahigh purity buffer or ultrahigh purity scintillator. The liquid handling system maintains precise control over the liquid levels and differential pressures between the different volumes of the detectors that are separated by flexible nylon vessels. The preparation, commissioning and operation of the system for filling the Borexino detector with scintillator is described.

© 2009 Elsevier B.V. All rights reserved.

1. Overview of Borexino detector and purity requirements

The Borexino detector is a low background calorimetric liquid scintillation detector [1–4]. The extraordinary low backgrounds are achieved by graded shielding to reduce external backgrounds and high purity materials to reduce the internal backgrounds. The detector is located in Hall C of the underground laboratory at the LNGS. The mountain provides equivalent shielding of 3500 m water from cosmic radiation. Inside Hall C, the detector has four layers of shielding. The entire detector is contained in a water shielding tank (diameter 18 m, height 16.9 m) filled with ultra-clean water. Inside the water tank is a 13.7 m diameter stainless steel sphere (SSS). The central volume within the SSS houses the active scintillator and it is surrounded by two layers of high purity organic solutions that provide additional shielding. The scintillation light is viewed by 2212 8" PMTs (ETL 9351) uniformly distributed on the inner surface of the SSS [5,6]. The PMTs are mounted with light cones to collect the light emanating from a central fiducial volume of the active scintillator.

A unique aspect to the Borexino detector is a nested vessel system that shields the active scintillator with ultrahigh purity organic liquids. The volume inside the stainless steel sphere is divided into three regions separated by two thin nylon spherical vessels. The inner vessel (IV) defines a central zone 8.5 m in diameter (321 m^3) and containing 278 tons of the active scintillator solution comprised of 1.5 g PPO (2,5 diphenyl oxazole) per liter of pseudocumene (PC) (1,2,4 trimethyl benzene) [3,7]. Ionizing radiation excites the PC, which scintillates at 266 nm. PPO is added to absorb the PC scintillation light and re-emit it at longer wavelengths ($\sim 380 \text{ nm}$). The wavelength shifter reduces self-attenuation of radiation below 300 nm, increasing the optical absorption length of the scintillation light to $>7 \text{ m}$. The longer wavelength also better matches the peak sensitivity of the phototubes improving the photoelectron yield.

The region between the scintillator volume and the SSS is divided into two zones by an 11 m diameter nylon vessel, the outer vessel (OV). These two zones, referred to as the inner buffer (IB) and outer buffer (OB), surround the active scintillator with a solution of 5.0 g/L DMP (dimethylphthalate) in pseudocumene that acts as a passive shield to external radiation. The DMP suppresses the scintillation caused by γ -ray emission from the photomultiplier tubes; DMP quenches the scintillation process but does not attenuate light at $>375 \text{ nm}$ propagating from the scintillator zone through the buffer region. The buffer is separated into two separate zones separated by a nylon vessel. The outer nylon vessel keeps impurities, such as radon and dust from the phototubes and light collectors, from diffusing to the interface between the buffer and active scintillator volume.

The total liquid passive shielding of the active scintillator from external radiation in Hall C (such as the rock) amounts to 5.5 m of water equivalent. Even though PC is slightly less dense than water

the purity of PC provides a superior low background buffer shield. We have previously established that the U and Th backgrounds in freshly distilled pseudocumene (PC, 1,2,4 trimethyl benzene) are the lowest of all known materials ($\sim 10^{-16} \text{ g/g}$), more than an order of magnitude greater purity than has been achieved with water [8]. The use of PC for both the scintillator and buffer volumes provided for superior reduction in the internal backgrounds of the detector. Employing the same solvent in the both the scintillator and buffer regions also matched the densities to a few parts per thousand; by matching the densities between zones thin nylon vessels could be employed separating the zones minimizing the radioactive background from the vessels. The thickness of the nylon films was determined from a balance between minimizing the background contribution from the nylon vessels and the mechanical integrity of the vessels to withstand stresses that occurred during detector filling. Thin ropes made of ultra-high density polyethylene hold the nylon vessels in place while minimizing contributions to the radioactive background. The materials for construction were all tested and chosen to keep the background activity to a minimum. The details of the vessel construction have been reported in our previous paper [7]. Based on independent testing of the materials of construction the background due to external γ -rays in the fiducial volume is predicted to be less than 0.5 counts/day-100 ton) in the neutrino window (250–800 keV) [9].

The thin nylon vessels introduced a challenge to filling the Borexino detector. They were flexible and easily deformed. A special filling system was necessary to maintain balance levels between the scintillator and two buffer volumes during filling. A detailed description of the filling system and principles of operation is published elsewhere [10].

To minimize the backgrounds from internal contributions associated with impurities in the scintillator the scintillator components were purified by distillation, water extraction and gas stripping to achieve the high purity necessary for Borexino; these purification system and the methods have been described elsewhere [11]. It was also necessary to purify the gases used for vessel inflation and gas blanketing operations. High purity nitrogen that had been specially purified to be low in Ar, Kr and Rn was essential to creating a low background detector [12,13].

Equally important to the purification of the scintillator was the cleaning of all the systems employed in the filling of the Borexino detector. The Borexino collaboration went to great extremes to design and build the detector vessels and all the piping and equipment that contacted the scintillator so it could be cleaned to high standards prior to filling the detector. The cleaning procedures were directed at micron sized particulates that could shed from surfaces and be carried into detector. A special cleaning system was developed to establish a high quality control over particulates in all parts contacting the scintillator fluids.

The Borexino detector involved a complex set of engineering operations that required a variety of ancillary plants and complex engineering operations to prepare it and subsequently fill it with scintillator. This paper will provide an overview of all the equipment and processes associated with liquid and gas handling operations that have been required for the successful deployment of the Borexino detector. The Borexino collaboration has published an overview of the detector [1,2]. Several of the most important subsystems, including the nested vessels, the purification system, the fluid filling system and the high purity gas system have been described in detail in separate reports. The integration of all the different liquid handling operation and facilities is emphasized in this paper.

2. System requirements

2.1. Radiopurity requirements

To achieve low backgrounds in the Borexino detector it has been necessary to identify the different means by which radioactive impurities can enter the detector, and seek methods to ameliorate them. We do not know all the different sources of radioactive impurities that may be encountered but we developed hypotheses based on chemical heuristics for cleanliness from the electronics and pharmaceutical industries. Table 1 summarizes several background impurities their suspected sources and strategies for reduction.

There are several different routes by which radioactive impurities may enter the Borexino detector. The estimates of these impurities from different sources and the requirements for Borexino were discussed in detail in our previous papers describing the CTF and Borexino purification systems. We summarize the purification strategies here.

Inherent impurities such as ^{14}C in the scintillator cannot be reduced using “simple affordable schemes”. It was necessary to chose scintillator components that were derived from petroleum. The ^{14}C in the petroleum had decayed over millions of years reducing its level by many orders of magnitude. We tested scintillator made from a couple of different petroleum sources with the CTF and found $^{14}\text{C}/^{12}\text{C} < 1 \times 10^{-17}$ for all the samples tests (cf. $^{14}\text{C}/^{12}\text{C} = 10^{-12}$ in biological samples at the earth's surface) [14].

Nobel gas impurities, ^{39}Ar , ^{85}Kr and ^{222}Rn are in air; scintillator that comes in contact with the air or nitrogen will absorb noble gas impurities. ^{222}Rn may also enter the scintillator as a decay product of ^{238}U and its daughters. ^{238}U is found in materials of construction, like stainless steel and nylon and also occurs in dust particles in Hall C. Emanation is minimized by choice of materials and using as little material as possible. Nobel gas concentration in the air or nitrogen can be reduced by eliminating air leaks and reducing the Ar and Kr content in the nitrogen used for gas blankets in the experiment. ^{85}Kr occurs at a level of $\sim 1 \text{ Bq/m}^3$ air, ^{39}Ar occurs at a level of $\sim 10 \text{ mBq/m}^3$ air. Borexino sought to achieve a background of $< 1 \text{ count/day/100 tons}$ of scintillator from Ar or Kr, which required an overall system leak rate of $10^{-8} \text{ m}^3 \text{ air/h}$ [11]. There are over a thousand valves and fittings associated with the Borexino fluid handling system, which required a leak rate of $10^{-11} \text{ m}^3 \text{ air/h}$ ($\sim 10^{-8} \text{ mbarL/s}$ at a differential pressure of 1 bar) for each component. Low Ar/Kr nitrogen was required for the gas filling and blanketing applications in Borexino; nitrogen with $< 300 \times 10^{-9}$ Ar and $< 10^{-14}$ Kr was required by Borexino [13]. SOL was one of two industrial gas suppliers who were able to supply nitrogen meeting those purity specifications.

A major source of contamination to the experiment is from dust particles containing U and Th. In addition, ^{210}Pb is deposited

Table 1
Radiopurity requirements for Borexino

Radioisotope	Source	Typical concentration	Tolerable level	Strategy for reduction
^{14}C	Cosmogenic bombardment of ^{14}N	$\frac{^{14}\text{C}}{^{12}\text{C}} \leq 10^{-12}$	$\frac{^{14}\text{C}}{^{12}\text{C}} \leq 10^{-18}$	Petroleum derivative (old carbon)
^7Be	Cosmogenic bombardment of ^{12}C	$3 \times 10^{-2} \text{ Bq/ton-carbon}$	$< 10^{-6} \text{ Bq/ton-carbon}$	(i) Distillation (ii) Underground storage of scintillator
^{222}Rn	Air and emanation from materials	100 radon atom/cm ³ -air	1 radon atom/ton-scintillator	(i) Nitrogen stripping from liquids (ii) selective adsorption from gases (iii) leak-tightness of fluid system
^{210}Pb	Surface contamination			(i) Positional discrimination (ii) cleaning and limiting exposure to air with ^{222}Rn
^{210}Po	Product of ^{210}Pb decay			Surface cleaning
^{210}Bi	Product of ^{210}Pb decay			Surface cleaning
^{238}U ^{232}Th	Suspended dust, organometallic	$2 \times 10^{-5} \text{ g/g-dust}$	$< 10^{-16} \text{ g/g-scintillator}$	(i) Water extraction/distillation (ii) surface cleaning
^{40}K	Dust or contaminant in fluor	$2 \times 10^{-6} \text{ g/g-dust}$	$< 10^{-13} \text{ g/g}$ in scintillator $< 10^{-11} \text{ g/g}$ in fluor	(i) Water extraction of fluor solution (ii) distillation of fluor solution
^{39}Ar , ^{42}Ar	Air	1 Bq/m ³ -air		(i) Nitrogen stripping (ii) high purity, low Ar/Kr nitrogen (iii) leak-tightness of fluid system
^{85}Kr	Air	1 Bq/m ³ -air		(i) Nitrogen stripping (ii) high purity, low Ar/Kr nitrogen (iii) leak-tightness of fluid system

on vessel surfaces that have been exposed to air with ^{222}Rn . ^{210}Pb decay can emanate ^{210}Po and ^{210}Bi into the scintillator. The U, Th, ^{210}Pb daughters and other impurities (e.g. ^{40}K) were removed from the PC by distillation. These impurities were removed from the concentrated fluor master solution by filtration and water extraction and distillation. To further reduce the effects of U daughters a rigorous and thorough cleaning procedure was employed to reduce the concentration of surface impurities and adsorbed dust particles. Surfaces were cleaned with detergent, rinsed, treated with glycolic formic acid and citric acid, rinsed with EDTA solution and lastly rinsed with high purity water until the level of suspended particles in the rinse water in the 10–50 μm range was <50 particles/L (corresponding to level 50 by Mil Spec 1246C) [15–17]; after cleaning the systems were flushed with nitrogen and left sealed filled with nitrogen.

2.2. Fluid handling systems and operations

The success of Borexino as a low background detector required filling the detector with the cleanest scintillator materials possible. This required (i) choosing materials with the lowest impurity levels possible for construction of the detector, (ii) purifying the scintillator materials to remove potential radioactive impurities and (iii) making everything that contacted the scintillator as clean as possible. The Borexino detector requires many different systems to purify and deliver high purity nitrogen, high purity water and high purity scintillator. This required connecting together a complex set of chemical plants with precise controls over temperature, pressure, flow rate and composition, and the chemical plants had to be built and operated to extraordinary levels of cleanliness. Borexino required a clean system, controlled gas and liquid flows for filling the detector and systems to prepare high purity fluids.

Before beginning any operations with scintillator components all the systems that contacted the fluids (nitrogen, water, scintillator, buffer) entering the Borexino detector were thoroughly cleaned in place, filled with nitrogen and sealed until use. After everything was clean the nylon vessels were inflated with high purity synthetic air, low in radioactive impurities, ^{39}Ar , ^{85}Kr and ^{222}Rn . SF_6 was added to the synthetic air for leak checking the nylon vessels. Filling the vessels with synthetic air permitted leak checking the detector with minimal safety risk, while minimizing impurity levels. After the nylon vessels were certified to be leak free the air was replaced with high purity nitrogen; thorough purging of oxygen was required because oxygen impurities quench the scintillation in the detector. Gaseous nitrogen for the detector is delivered by vaporizing the high purity liquid nitrogen supplied by SOL group.

The nested nylon vessels were next filled with high purity water produced in Hall C by a deionized water plant [18,19]; the water was stripped of oxygen and radon by sparging with high purity nitrogen. Water was introduced into the three zones of the detector in a cyclic fashion maintaining uniform liquid levels. The details of this procedure are described in a companion paper [10].

Water was displaced from the three zones by addition of scintillator and buffer solutions. Scintillator components were purified and mixed on-line in Hall C to deliver the highest purity materials to the Borexino detector. Pseudocumene used in the scintillator and buffer solutions was delivered by specialty tanker to Gran Sasso during production campaigns at a Polimeri Europa Plant on Sardinia. The tanker was built to the collaborations specifications and used only for the pseudocumene delivery. The pseudocumene is unloaded from the tanker and placed into one Storage Vessel in Hall C, where it was stripped with high purity nitrogen. The pseudocumene was then redistilled in a vacuum distillation column, stripped with low Ar/Kr nitrogen and humidified to 70% RH with ultrapure water. The freshly distilled PC is mixed in-line with pre-purified concentrated fluor solution for the scintillator or DMP for the buffer and put directly into the Borexino detector.

The systems for all these operations had to be done in a highly controlled manner for the success of Borexino. Fig. 1 is a floor plan of Hall C at LNGS that shows the location of the detector and the different physical and chemical plants associated with preparation, purification, filling, and maintenance of the Borexino detector. The plants were grouped as scintillator handling (the plants that store and move the pseudocumene), filling stations (the plants that put the fluids into the Borexino detector), purification (the plants that purified scintillator, water or nitrogen), and auxiliary plants that provide utilities for the various processes. A simplified process flow diagram is shown in Fig. 2 to indicate how material flows between these different plants. The cleaning, purification, filling and detector maintenance all involved complex fluid operations that required specialized fluid handling equipment and control systems. We will first provide a brief introduction to the systems and plants required for the fluid operations and then we review the fluid handling operations.

3. Fluid handling systems

3.1. Gas supply systems

Regular nitrogen: This is a convention liquid nitrogen storage tank with a heater to provide boil off nitrogen for pneumatic instrumentation, routine purging of gas lines. Regular nitrogen is standard grade liquid nitrogen from Linde.

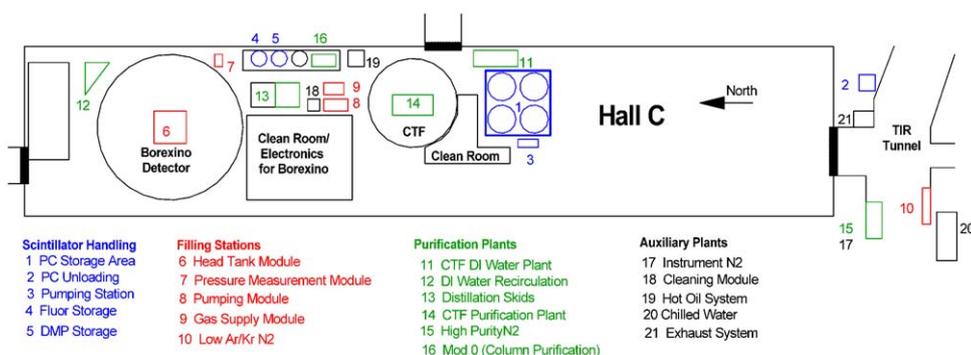


Fig. 1. Layout of Hall C in LNGS and the location of the subsystems associated with the Borexino detector. Hall C is 100 m long and 20 m wide and 20 m high. Borexino occupies approximately 60% of the area in Hall C. The receiving and unloading are done in the entryway (TIR tunnel) to Hall C. All the other operations, storage, purification, cleaning, etc. are done in Hall C.

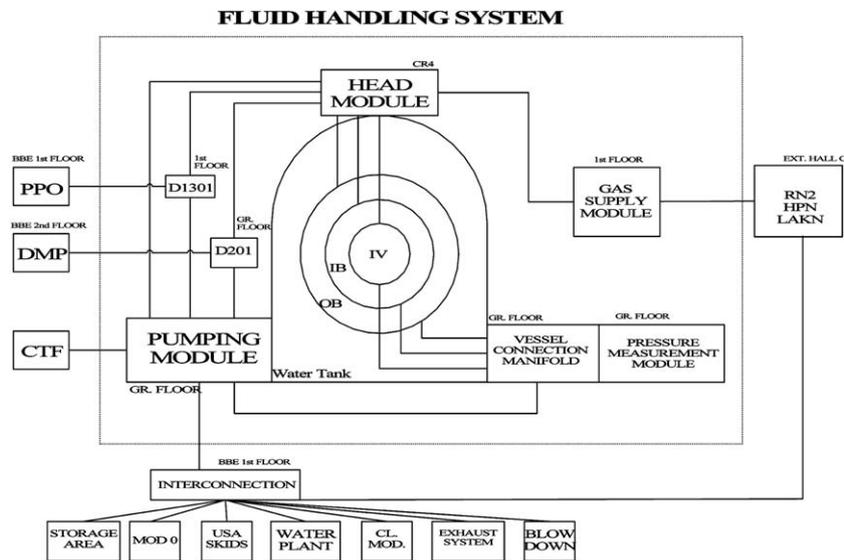


Fig. 2. Process flow diagram showing the interconnections between the different plants and subsystems. The interconnection system was the control and command center for moving the different liquids and gases between the modular subsystems performing different functions.

High purity nitrogen: This is employed for the gas blankets in the pseudocumene storage area, and in the gas stripping operations of the water and for gas inflation of the nested nylon vessels. Nitrogen with low ^{222}Rn levels is required to keep the background low. Radon has been removed from the regular nitrogen by adsorption onto high purity activated carbon adsorbent at 77 K to reduce the ^{222}Rn concentration in the nitrogen by approximately a factor of 100. The radon adsorption facility is installed in the entryway to Hall C; it was designed and built by the Heidelberg group of the Borexino collaboration and is described in detail in Ref. [20].

Low Ar/Kr (LAK) nitrogen: This is specialty nitrogen supplied by the SOL group that was tested to have much lower levels of Ar and Kr than found in standard commercial liquid nitrogen [12,13,20]. This nitrogen is used for final gas purging of the nested nylon vessels, blanketing of the scintillator and buffer volumes during filling with water and scintillator and for gas stripping of pseudocumene during purification. The low Ar/Kr nitrogen reduced the ^{39}Ar and ^{85}Kr to keep their levels low in both the water and pseudocumene filling the Borexino detector.

Synthetic air: This is a system that mixed high purity oxygen from gas cylinders with the high purity Nitrogen for the initial gas inflation of the nested nylon vessels.

Gas humidification system: Synthetic air and LAK nitrogen were humidified with a membrane humidifier. The membrane humidifier was a commercial shell and tube membrane unit from PermaPure LLC. It was incorporated into a system that employed strict temperature control to humidify the gases to 80% RH at 15 °C at flow rates of 1–40 m³/h. (The humidity is necessary to keep the nylon vessels flexible during their inflation [21].) High purity DI water that had been stripped of Radon was employed in the humidifier.

The gas supply module (GSM) for the gas inflation of the nested nylon vessels provides for both mixing and humidifying gases. The GSM was one of the modules of the filling station systems employed to inflate and fill the Borexino detector [10]. High purity nitrogen is mixed with bottled oxygen to produce synthetic air. The oxygen content of the synthetic air is monitored with an oxygen sensor. Flow rates of all components are monitored with Coriolis type mass flow meters (Kurtz Instruments) and controlled by feedback control to pneumatically driven diaphragm valves (Swagelok) by the digital control system (DCS described below).

The gas supply module was equipped with a manually controlled input for SF₆ gas, and a circulating blower system to circulate air through the different detector zones. The SF₆ was added to the synthetic air of the inner buffer after inflation to test the nylon vessels for leak-tightness. Mass spectrometry was employed to check for the concentration of SF₆ in the inner vessel and in the outer buffer. Measurements of concentration of SF₆ as a function of time permitted the determination of the leak rate through the nylon vessels (Table 2).

3.2. High purity water systems

CTF water system: A high purity DI water system with a combination of water softening, reverse osmosis, ion exchange beds and nitrogen stripping capable of producing ~2 m³/h of high purity water was installed as part of the counting test facility [18,19]. This system was used to produce feed water used to fill the inner three zones of the Borexino detector and the Water shield tank. This high purity DI water was also used for cleaning and rinsing the components of the Borexino fluid handling system and detector.

Borexino high purity water system: This was a combined RO/ ion exchange system for the recirculation of the high purity water in the water shield tank of the Borexino detector (Table 3).

3.3. Scintillator purification plants

CTF purification system: This system originally combined either vacuum distillation or water extraction with nitrogen stripping of a PPO/PC scintillator for the counting test facility [22,23]. The CTF purification system was designed to process 20–50 L/h. The CTF system was too small to process the quantities of water and pseudocumene used in Borexino. However, the CTF system was the proper scale to purify the 5 m³ of concentration fluor solution employed in Borexino. Piping changes were made to the CTF purification system to distill a concentrated PPO/PC master solution for Borexino [11].

Module zero: This system was built to test an alternative scintillator and selective adsorption purification for the counting test facility [24]. The system was scaled for Borexino but the

Table 2
Borexino gas supply systems.

Use	P (bar)	Q (m ³ /h)	Special requirements	²²² Rn (μBq/m ³)	³⁹ Ar, ⁸⁵ Kr (μBq/m ³)
<i>Gas supply systems</i>					
REGULAR Pumps, sealing purging, boxes purging, water Tank purging	8	200		51	–
HPN Water plant stripping, DMP blanketing, cleaning drying	8	100		0.78	³⁹ Ar < 18.1 ⁸⁵ Kr < 18.5
LAKN Purification system stripping, purging, PPO blanketing, vessels blanketing	8	100		6.8	³⁹ Ar < 0.5 ⁸⁵ Kr < 0.2
Synthetic air Inflation of the nylon vessels		40	80% RH @15 C	6.8	³⁹ Ar < 0.5 ⁸⁵ Kr < 0.2

Table 3
High purity water system.

Use	ρ (MΩ/cm)	Outlet Q (m ³ /h)	Stripping with HPN (kg/h)	Filtration (μm)
<i>Water plant</i>				
Cleaning, filling with water	> 18	2	30	0.02-PTFE

process was never implemented because the other purification techniques were successful.

Borexino purification skids: The purification skids employed either distillation or water extraction followed by nitrogen gas stripping or vacuum steam stripping to process 1000 L/h of scintillator or scintillator components. A detailed description of the purification skids and the principles of their operation are published in a previous paper [11]. Minor modifications were implemented to permit distillation and gas stripping of the water as well as pseudocumene [25]. The skids were used to strip the high purity water with low Ar/Kr nitrogen for water filling of the nylon vessels. The primary function of the purification skids was to distill PC and strip PC of noble gases (Ar, Kr and Rn); the purified PC was then used to fill the scintillator and buffer zones of the Borexino detector.

3.4. Delivery and storage systems

PC storage area: The PC storage area was designed receive deliveries of pseudocumene, provide storage capacity of 400 m³ of pseudocumene (capable of holding the inventory of scintillator in the Borexino detector) and pump pseudocumene into the interconnection system for delivery to different plants of Borexino. A schematic of the storage area is given in Fig. 3. The storage area contains 4–100 m³ stainless steel storage tanks, 3.5 m in diameter × 10 m tall. The tanks are in a walled enclosure that serves as safety containment against spills. The tanks are connected together by upper and lower liquid manifolds and a gas manifold at the top. There are level transducers in each tank. A pressure transducer is installed in the gas manifold. One tank was equipped with a gas sparger at the bottom; this tank had a separate pressure transducer and valve arrangement for independent control of the pressure. A small gas pressure on the top of the tanks is maintained by a PID control loop between the pressure transducer and two regulating valves. Each storage tank is connected through separate valves to the lower liquid manifold. The lower manifolds are connected via a pumping station to the interconnection system. The Storage area was provided with four manifolds to have independent lines for the buffer fluid and

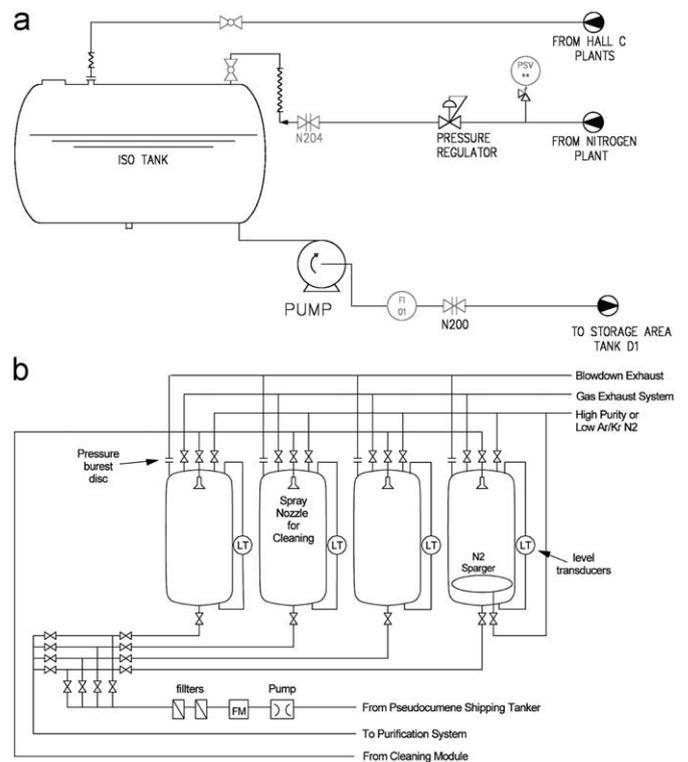


Fig. 3. Process and instrumentation diagrams for pseudocumene storage area and unloading station for Borexino. Pseudocumene is delivered by tanker from Polimeri Europa in Sardinia to the unloading station in the entryway to Hall C in Gran Sasso. The schematic at the top shows pumping PC from the ISO tank on the truck to the storage area. Piping is also provided for the eventual draining of the Borexino detector and removal of PC from Hall C. The schematic at the bottom shows the storage tanks in Hall C. The PC is unloaded into one of the 100 m³ tanks in the storage area. One of the storage tanks is equipped with a nitrogen sparger system. The storage area can accept and deliver pseudocumene to the various purification and fluid handling systems in Hall C.

the scintillator; this also made it possible to purify from one vessel to another. Fluid from any tank can be supplied to the interconnection system which can deliver the PC to the

purification skids or other locations. Pseudocumene can also be delivered through the manifold to fill any one of the tanks. The liquid manifold at the top of the storage tanks was provided to assist in cleaning the tanks. This manifold connected to spray nozzles inside each tank that permitted detergent sprays followed by DI water rinsing. The storage tanks were thoroughly cleaned prior to their use for pseudocumene storage. When the storage tanks are filled with pseudocumene the gas manifold at the top of the tanks keeps all the tanks under a high purity nitrogen blanket. A small nitrogen flow is maintained through the manifold exiting the manifold to the exhaust system. Each tank is also equipped with a pressure burst disc for safety.

There are two pumping stations connected to the storage area. One pumping station is for unloading tanker trucks delivering pseudocumene to Hall C. The second pumping station is for pumping the pseudocumene from the storage area to other locations, e.g. the purification system. The unloading pumping station employs a centrifugal magnetic driven pump while the storage area pumping stations employ White Knight shuttle pumps. The shuttle pumps are all Teflon nitrogen gas driven so they are safe from fire safety and are also very clean. The valves in the storage area and the pumping stations are all electropolished stainless steel valves from Swagelok or Carlen. The valves are all either manually operated or pneumatically operated so the pumping stations are intrinsically safe for fire protection.

Unloading system: A pumping system located in the entryway to Hall C pumped PC from 20 m³ delivery trucks into the sparged tank in the storage area. After nitrogen stripping, the PC was moved into the other tanks using the Storage Area pumping station. The unloading system is connected to the nitrogen system for blanketing and the exhaust and blow down systems for safety. The unloading system is also shown in the Process and Instrument Diagram for the storage area (Fig. 3).

PPO master solution storage: A concentrated solution of ~100 g PPO/L-PC was prepared and stored in a 2 m³ stainless steel vessel (HT1). The vessel has a funnel with load lock for addition of the PPO powder with minimal addition of air and an impeller mixer with a sealed shaft to mix the PPO/PC solutions. This tank was mounted on a load cell to measure the weight changes from addition or removal of the concentrated fluor solution. HT1 is connected to the water plant. The concentrated PPO/PC solutions were water extracted in HT1. After mixing concentrated fluor solution in HT1 water was added and the mixer was used to agitate the water and organic phases for several hours. After mixing the liquids were allowed to phase separated and settle circa 12 h. Water was removed from the bottom of the tank and drained into a waste tank. A glass window on the drain line was used to see the breakthrough of the organic phase. HT1 was also connected to the CTF purification system. The CTF purification system was retrofitted to distill the concentrated fluor solution. The final purified concentrated PPO solution was stored in a 1.6 m³ pressure tank (rated at 4 bar); the pressure head was employed to feed the concentrated PPO master solution to the filling station pumping module where the concentrated solution was mixed in-line with pseudocumene to make scintillator solution for Borexino. The in-line mixing is controlled by a PID loop between a regulating valve and flow meter. The flows were regulated to achieve the desired scintillator composition of 1.5 g PPO/L-PC.

DMP storage: A 5.5 m³ electropolished stainless steel tank held dimethyl phthalate for the Borexino buffer region. The DMP storage tank was pressure rated to 3 bar. It was located next to the PPO master solution storage tank 7 m above floor level. DMP flow was controlled by the combination of hydrostatic and applied gas pressure to force the DMP flow to the filling station pumping module where it was mixed in-line with purified pseudocumene to fill the inner and outer buffer of the Borexino detector.

Concentration is controlled by regulating the valve from the DMP tank to match the set point on the flow meter to match the pseudocumene flow; DMP flow is controlled to deliver buffer solution of 5 g DMP/L-PC.

PC delivery trucks: Four specially fabricated tanker trucks with 20 m³ electropolished stainless steel tanks were built to obtain PC from the Polimeri Europa Production Plant on Sardinia and deliver it the underground lab in Hall C at Gran Sasso. The tanks were built to ISO standards for transportation of flammable liquids on the highways. These tanker trucks minimized the transit time thereby reducing the effects to cosmogenic radiation.

3.5. Piping and fluid flow systems

Cleaning system: The cleaning module provided high flows of heated detergent solutions for flow through pipes and vessels to clean all the subsystems in advance of introducing fluids that would be employed in the Borexino detector. The system also could provide flows of formic, citric and glycolic acid solutions to pickle and passivate welds on the stainless steel piping. The cleaning system was also connected to the high purity water system and nitrogen system so the piping and vessels were thoroughly rinsed and filled with nitrogen to dry before closing up a section of pipe or vessel.

A schematic of the cleaning module is shown in Fig. 4. It consisted of a 1 m³ tank containing detergent, acid or chelating agent solution. Solutions were changed corresponding to the stage in the cleaning process. The fluid from these tanks was pumped by a centrifugal pump through an electrically heated heat exchanger into flexible Teflon lines adapted to connect to Swagelok fittings on pipe sections or vessels. A second flexible line connected to another end of the pipe section to return the detergent solution to the cleaning module. The return line had a 0.1 µm cartridge filter (Millipore Model no. GDCZ 01 TPE) to remove particulates from the circulating detergent solution. A high purity DI water supply was piped into the cleaning module from the CTF water purification plant. The DI water was used to rinse the piping and vessels after cleaning with detergent. The rinse water was filtered and checked for particulate count. Waste acids were collected in 3 × 10 m³ polyethylene tanks and trucked out for water treatment and disposal.

Several different detergent solutions were tested and the detergent solution chosen was Detergent 8 from ALCONOX; Detergent 8 was effective and had low potassium content. Flow rates from the cleaning module were typically ~2–3 m³/h, depending on the distance from the cleaning module, the size of the piping, length of the piping section and the number and type of valves and fittings in the isolated section.

Interconnect system: All the different plants and systems were connected through a series of trunk lines with valving to arterial lines for the individual plants. The interconnect system was laid out to accommodate all the different purification and filling operations. Valving was included to facilitate clean in place of all the fluid lines for filling the Borexino detector. The interconnect system was an impressive network of 1/2", 1", 1-1/2" and 2" electropolished stainless steel tubing and electropolished stainless steel fittings and valves. All the piping was put together with orbital welding with non-thoriated electrodes and inert (Ar) gas purge. Valves and fitting were electropolished stainless steel installed with either orbital welding or SS gasketed VCR fittings.

A process and instrumentation diagram of the interconnect system is shown in Fig. 5. Fig. 5 is included to show the concept of the trunk lines and arterial lines; detailed process and instrumentation diagrams can be obtained by contacting Andrea Ianni (andrea.ianni@lngs.infn.it). Individual trunk lines were

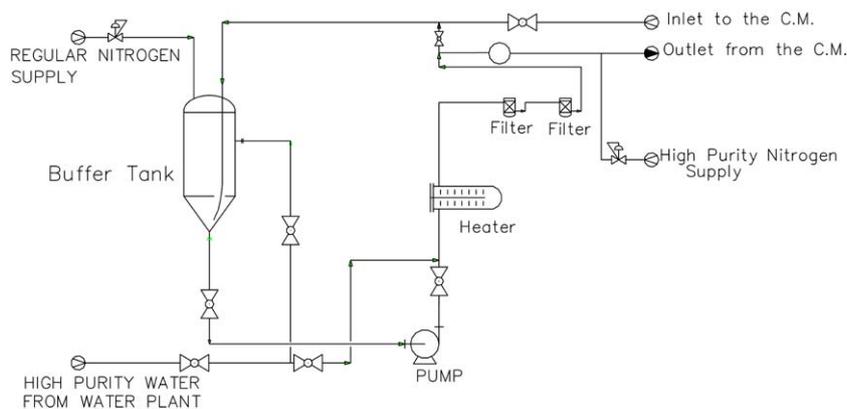


Fig. 4. Schematic of the cleaning module. Flexible piping systems were employed to connect piping sections and/or system vessels to the cleaning module.

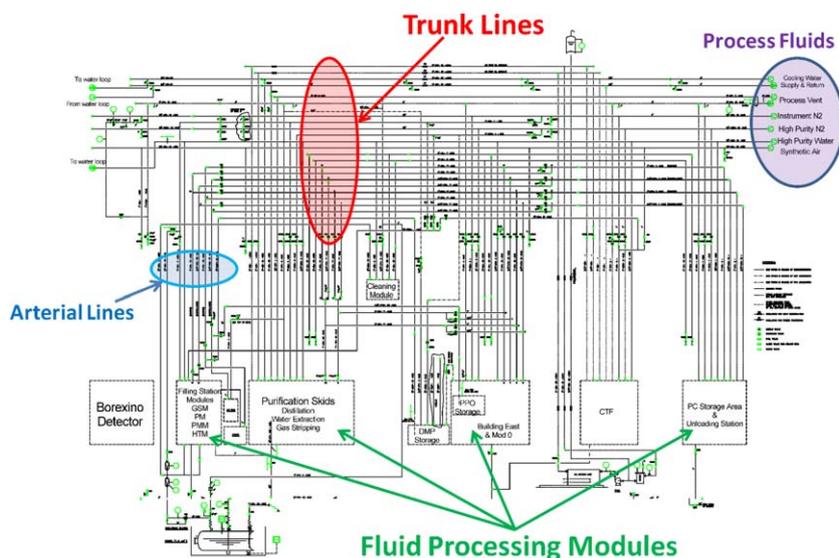


Fig. 5. Process and instrumentation diagram of the interconnection system. This drawing is to illustrate the concept of the trunk line connecting to the different modular systems for moving all the different liquid and gas streams in Borexino. Several of the major systems, i.e. the filling stations, purification systems, CTF and storage area are readily identified. These are all connected with both feeds and returns to the trunk lines for the scintillator components and process fluids. Most of the interconnect system trunk lines were run along the walls in Hall C at LNGS. All the utilities, such as the different nitrogen supplies and the chilled water were piped from outside Hall C in pipe racks along the walls at elevations of 3–4 m above floor level.

provided for regular and high purity nitrogen, DI water, scintillator and buffer solutions, process chilled water, and hot oil. Arterial lines connected the different plants to the trunk lines. All the process and utility fluids were available via these trunk lines. The lines carrying process fluids, high purity nitrogen, high purity DI water, pseudocumene, scintillator and buffer solutions, were all cleaned by detergent wash and DI water rinse prior to use.

Filling stations: This is a set of modules employed to inflate with gas, fill with liquid and maintain levels and pressures in the central three zones of the Borexino detector. The filling station had three modules: (1) *Pumping module* moved fluids (synthetic air, LAK nitrogen, high purity water, PC, master fluor solution and DMP quencher) into the nested nylon vessels of the Borexino detector; (2) *Pressure Measurement Module* monitored the pressures at the bottoms of the scintillator vessel and the inner and outer buffers. It also measured the liquid levels in the three central zones of the Borexino detector and (3) *Head Tank Module* monitored the pressures at the top of the three central zones of the Borexino detector. It also provided for liquid level and pressure control and maintenance in three central zones employing a spillover weir

system. The detailed design and operating principles of the filling stations is provided in a companion publication [10].

3.6. General lab utilities

Chilled water: A chilled water loop ran through Hall C. Chilled water was available from underground reservoirs at 8 °C and was circulated through the lab. The chilled water was employed in condensers for the distillation system and heat exchangers for temperature control of the fluids entering the Borexino detector.

Hot oil: An electric hot oil heater was used to heat oil and circulate the hot oil to the purification plants and heat exchangers. The hot oil was used as the source of heat for distillation. Hot oil was available at temperatures up to 250 °C.

Gas exhaust and blow down system: The nitrogen gas blankets and all gas exhausts were connected to a negative pressure exhaust drawing all the exhaust gases through a carbon filter and purified before release to the environment. All the vessels in the

Table 4
Auxiliary system specification.

Use	P (bar)	Q (m ³ /h)	Inlet T (°C)	
<i>Chilled water</i>				
Purification systems cooling	4	200	10	–
Use	P (bar)	Q (m ³ /h)	T (°C)	Oil
<i>Hot oil</i>				
Purification systems reboilers	4	1500	150	ALARIA3
Use	P (bar)	Q (m ³ /h)	PC content (ppm)	
<i>Exhaust system</i>				
Nitrogen treatment before discharge	1	150	<3	–

storage system, purification system, a filling stations had pressure relief valves or rupture discs connected to a main collector of a blow down system. The blow down system consists of a main collector entering at the bottom of a packed vessel in nitrogen atmosphere, partially filled with water; in case of fire and subsequent rupture of a burst disc, it cools down the gas–liquid stream and it keeps the liquid in the vessel exhausting the gas from a line at the top (Table 4).

3.7. Digital control system

All the plants and systems were interfaced to a digital control system dedicated to the fluid operations. A Delta V programmable logic controller by Emerson Process Management forms the heart of the control system. It provided automated process control capabilities, parameter monitoring and alarming. Temperatures, pressures, fluid levels in the detector and all the storage and process tanks are continuously monitored and logged. PLC functionality included automated control of temperatures, pressures and fluid levels (important for operation of the purification systems and for the detector filling). A reliable system was essential, considering the elevated temperatures that exist in the plant (in distillation mode), the flammability of the scintillator, and the enclosed environment in which the plant was located. The controller allowed for 24 h/day operation, alarm notification and automated shutdown in case of problems.

The status of all the controlled variables was continuously monitored and logged with the DCS. Alarms and automatic shut offs were implement as part of DCS to meet the safety requirements of the lab. Each plant or subsystem had its separate monitoring and control page as part of the larger overall system. The control systems were tuned and tested prior to the filling operations. PID and ON–OFF controllers were instituted for over 100 pumps and valves in the storage, purification and filling systems. The parameters of the PID controller were determined during the commissioning phase using Ziegler Nichols closed loop tuning to determine proportional gain, integral time and derivative time for each feedback loop [26]. A fine tuning of the parameter was done during the operations. Critical control parameters are interlocked and alarmed. The history of the process variables is stored and available for analysis. Fig. 6 illustrates the information flow of the DCS system. Each system and plant was set up with individual control to handle specific tasks.

Fig. 7 is a snapshot of the main page of the control of the purification system, providing an overview of the purification system. From the main page, it is possible to zoom in with greater detail on the individual elements. There is a master page of the control system from which an operator can navigate to other

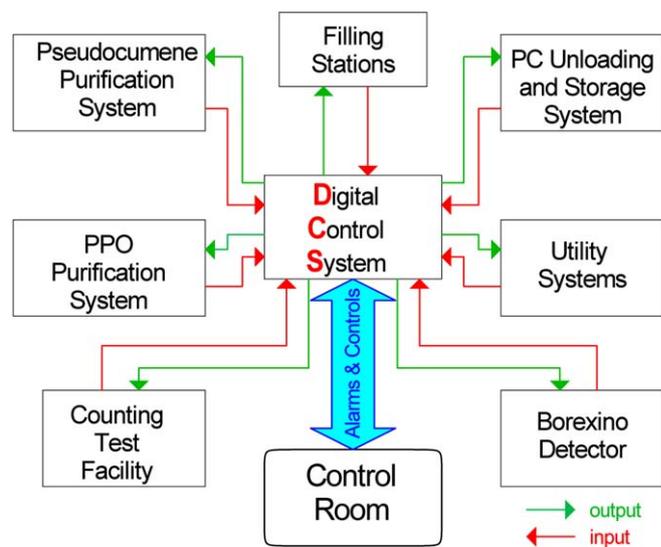


Fig. 6. Purification system overview of the DCS.

pages and obtain detailed views of a single portion of the plant such as that shown in Fig. 7.

4. Fluid handling operations

The fluid operations were broken down into a series of specific tasks that can be categorized as cleaning, receiving, pre-purification and testing, detector filling, on-line purification, and materials recovery and shutdown. Detailed procedures were written for each of these individual tasks by members of the Borexino collaboration. These procedures contained specific conditions to be satisfied prior to commencing with the procedure including specifications for manpower and other resources required for the tasks. A critical part of the procedure was an assessment of risks and safety requirements associated with the procedure, detailed steps for all operations and emergency response procedures. The procedures were written so they could be followed by technicians with supervisory personnel on call. These procedures were reviewed and approved by the technical board for the collaboration. Once the collaboration approved the specific project the procedure had to be approved by the Borexino project or operation manager, the safety officer for the experiment, the technical manager for the LNGS laboratory and the director of the LNGS laboratory. The equipment and procedures all were reviewed for hazardous operations (a HAZOP review) for

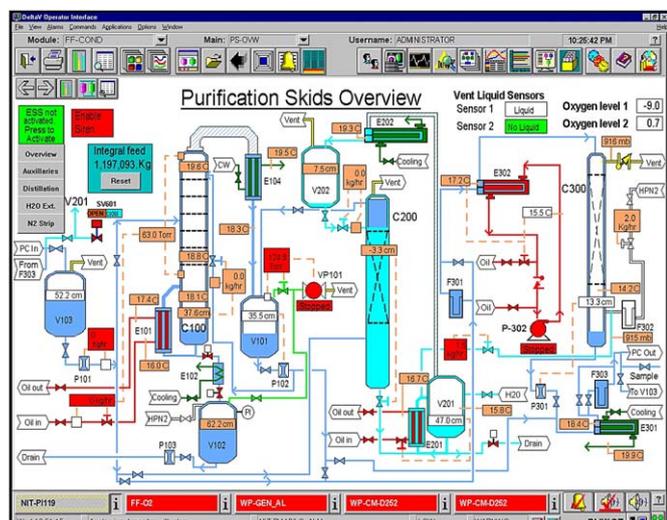


Fig. 7. The main page of the purification system control. This page provides an overview of the distillation, water extraction and gas stripping systems. The temperatures and pressures at the various locations throughout the system are displayed for the operators. The status of key variables and the alarm systems can all be viewed. Greater detail on particular equipment items can be obtained by clicking on the unit, opening up a details page for that unit.

personal safety of the workers as well as the environmental safety. Requests to view specific operating procedures should be directed to the Chairman of the technical board (contact Marco Pallavicini, Marco.Pallavicini@ge.infn.it); the detailed operations may be released at the discretion of the technical board.

Listed below are the fluid handling operations for which procedures have been developed. For each subset of fluid operations, we provide a general description of the principles of the operations.

I. Cleaning processes:

1. Cleaning storage vessels for both the PC, fluor master solution and DMP.
2. Cleaning trunk lines and interconnection system.
3. Cleaning the CTF purification skid.
4. Cleaning the vessels and piping in the Borexino purification skids.
5. Cleaning unloading station.
6. Cleaning the filling station modules.
7. Cleaning the tankers for PC delivery from Sardinia to Gran Sasso.

II. Receiving:

8. Loading tanker truck with PC at Polimeri Europa.
9. Receiving scintillator solvent to storage in Gran Sasso.
10. Receiving of solvent quencher to quencher storage.

III. Pre-purification of scintillator components:

11. Pre-purification of solvent by distillation and nitrogen stripping.
12. Concentrated fluor solution preparation.
13. Concentrated fluor solution pre-purification by water extraction.
14. Pre-purification of solvent by distillation.

IV. Testing of scintillator:

15. Scintillator preparation for testing in CTFII.
16. Filling of CTFII with scintillator for testing.
17. Disposal of tested scintillator from CTFII.

V. Borexino detector filling:

18. Gas inflation of vessel with synthetic air.
19. Leak testing of nylon vessels.
20. Closing of stainless steel sphere and displacement of synthetic air with nitrogen.
21. Filling of the nylon vessels with high purity water.
22. Just-in-time delivery of pseudocumene.
23. On-line mixing of scintillator.
24. On-line mixing of buffer with quencher.
25. Filling buffer and scintillator volumes by displacing water.

VI. On-line fluid control and purification processes:

26. Purification of mixed scintillator with water extraction/nitrogen stripping.
27. Purification of mixed scintillator with adsorption column/nitrogen stripping.
28. Re-purification of buffer.
29. Level and pressure control in the scintillator and buffer regions.

VII. Material recovery and shutdown:

30. Separation of mixed scintillator into solvent and fluor by distillation.
31. Re-purification of buffer to recover pure solvent.
32. Empty buffer to shipping tankers.
33. Empty scintillator to storage.
34. Empty buffer to CTF tank.
35. Re-purification of buffer for sale.

4.1. Cleaning processes

A systematic cleaning of all fluid lines and vessels was done before purification or filling operations commenced. A great deal of time and effort went into the cleaning processes. Many of the ideas for cleaning were based on procedures developed in the electronics and pharmaceutical industries [15]. The cleaning process started with the isolation of a section of processing piping or a process vessel through which detergent solution and water would flow continuously. Piping sections were isolated by specific valves. Many of the valves employed were Swagelok electropolished ELD type valves with drain ports. The drain ports had either a VCR cap or a 1/4" valve attached, which permitted the piping section to be cleaned and rinsed without requiring any disassembly of the piping system. The isolated piping sections were connected to the cleaning module with flexible 1" Teflon tubing and Swagelok or VCR fittings.

The pipe cleaning operation began with circulating the hot detergent solution (heated to 80 °C) through the piping section for several hours. After the detergent cleaning the piping section was rinsed with DI water until the resistivity of the rinse water was 4–5 MΩ cm. Then the line was rinsed with filtered 1% EDTA solution (ethylenediaminetetraacetic acid). The EDTA is a chelating agent to remove metal ions from the water to keep them from redepositing on the surfaces of the pipes and entering the pseudocumene later. After the EDTA rinse there was there was a DI water rinse; at the completion of the DI water rinse the water was tested for particulates. A fixed amount of rinse water was filtered through 0.2 μm filter paper; the number of particles and their size distribution was determined by optical microscopy. When the particle size distribution was below level 50 by Mil Spec 1246C (< 1 × 10–50 μm particles per liter) the piping section was

deemed to be clean; additional rinsing and washing was carried out if level 50 cleanliness was not achieved [17].

Once the section of piping was clean it was dried by flowing nitrogen through the section for 2–3 h and finally capped off. The piping section was left in the clean dry state until use.

Piping sections that had parts with new weld joints were passivated after cleaning. After the initial rinsing, nitrogen was employed to push most of the water from the pipe. A pickling solution of 1% formic acid and 2% glycolic acid was pumped into the pipe section and allowed to react for 3 h. The pickling solution was flushed out with DI water and replaced with a passivation solution of 4% of citric acid. Passivation caused iron oxide scale to be displaced as small particles into the solution. After passivation the piping lines were rinsed with DI water, 1% of EDTA solution and a final DI water rinse. After they met level 50 cleanliness standards they were flushed with nitrogen to dry them before capping.

The detergent flow rate in the piping sections had sufficient velocity to be turbulent with good mixing to remove impurities from the surfaces of the pipes. Process vessels in the purification systems and the filling stations were too large to permit a significant fluid flow velocity for cleaning. Spray balls or nozzles were employed to clean process vessels. Typically a flange on the vessel was removed and a temporary replacement was inserted that connected the liquid lines to a spray ball inside the vessel. The spray balls were chosen to provide a high velocity jet spray onto the inner surfaces of the vessel. The detergent solution was sprayed on the inside of the vessel for a period of several hours, with the liquid allowed to run down to the bottom of the vessel where a drain valve was opened to permit the liquid to be removed by gravity. With large vessels we sometime connected a temporary pump to the vessel drain back to assist moving the detergent solution back to cleaning module. After the detergent wash DI water was sprayed via the spray balls to rinse the vessels; samples of the rinse water were tested for particle content. Once a vessel met level 50 cleanliness standards the temporary flange with the spray ball was removed and the vessel was flushed with nitrogen and then sealed off.

The rinse water had to be collected in large waste tanks throughout most of the cleaning operations. The water from the waste tanks were periodically removed and the water taken for industrial water waste processing. The nitrogen gas used to flush and dry the pipes and vessels was exhausted through Borexino exhaust gas treatment where it was filtered by carbon adsorbents and then exhausted through a 5 km pipe to the atmosphere above ground. A great deal of caution was exercised during the cleaning process to minimize the environmental impact of Borexino.

4.2. Shipping and receiving scintillator components

The highest purity scintillator components are essential to achieve low backgrounds in Borexino. Pseudocumene makes up over 99% of the scintillator and buffer so we sought to obtain PC of the highest quality for Borexino. Pseudocumene is a commodity chemical used as a solvent for various resins and also in some chemical syntheses. The demand for pseudocumene is limited and it is produced intermittently at various petrochemical plants around the world. The nearest supplier in Europe is the Polimeri Europa refinery on Sardinia where they have 2–3-month campaigns once or twice a year to produce pseudocumene. Borexino negotiated an agreement with Polimeri Europa to obtain pseudocumene directly from the final purification stage during production campaigns. Our operations were required to be flexible to match the Polimeri Europa production schedule. A special stainless steel delivery line was installed at the refinery to permit Borexino to directly fill Borexino tankers trucks with pseudocumene during a production campaign. The collaboration has four

tankers that were 20 m³ electropolished stainless steel tanks built to all the highway transportation standards. After filling a tanker on Sardinia, it traveled by highway and ferry to Gran Sasso arriving within 1 day of filling.

At Gran Sasso the tanker was driven directly into Hall C to an unloading station. Fig. 8 shows a photo of one of the tanker trucks hooked up to the unloading station in the access way to Hall C. The PC was pumped from the tanker into one of the 100 m³ storage vessels in Hall C. The storage area provided a buffer to even out the supply of PC for filling the Borexino detector. In addition, the storage area provided an inventory of PC for testing and preparing concentrated master solution of fluor. From the storage are the PC was pumped to the Borexino purification system and then through the filling stations of the Borexino detector to its final destination. All the PC transfer operations, both at Polimeri Europa and LNGS were done under nitrogen atmospheres. This was done for both safety considerations and to minimize potential radioactive contamination from noble gases in air.

There were two primary motivations for the elaborate shipping plans for the pseudocumene. The major concern is that the carbon in organic solvents stored at the surface of the earth is bombarded with cosmic radiation that results in ⁷Be contamination by the ¹²C(*p,x*)⁷Be reaction. ⁷Be decays with a half life of 53 days with a 10% branch by electron capture giving a 478 keV gamma-ray line which is in a background in the region of the ⁷Be neutrino scintillation line. By limiting the exposure time of the scintillator to 1 day the potential background to cosmogenically derived ⁷Be is reduced by a factor of 50.

The ubiquitous problem of particulate contamination from storage vessels or general piping in the refinery was the second major concern. The Borexino collaboration built our own transfer lines at both the Polimeri Europa refinery and in Hall C and cleaned those lines in advance of using them for PC. We also had our own shipping tankers built which we cleaned ourselves. Through these measures Borexino was able to limit the potential contamination by dust particles that could have introduced additional background.

These procedures also limited the potential exposure of the pseudocumene to air. Through a detailed set of studies the Borexino collaboration showed that exposure of pseudocumene to air and



Fig. 8. Tanker truck unloading pseudocumene into the storage area in Hall C at Gran Sasso. The tanker made trips back and forth to Sardinia to bring freshly distilled pseudocumene from Polimeri Europa for the Borexino detector. The tanker is connected to the pump of the unloading station and the pseudocumene is pumped into the 100 m³ storage tanks in Hall C.

stainless steel surfaces can produce small amounts of dimethyl benzaldehyde and dimethyl benzoic acid, and these impurities severely reduce the optical transparency of the pseudocumene [27]. Since optical transparency is essential in Borexino the collaboration felt it was essential to exert quality control over the handling of the pseudocumene during its shipment.

4.3. Scintillator testing

The scintillator components were all tested in a renovated counting test facility (CTF) for radiopurity prior to use in Borexino. The renovated counting test facility, CTFII, was rebuilt with new phototubes and a shrouded detector vessel in 2000–2002. A new improved sealing system for the electrical connections to the phototubes was introduced for phototubes in water. This new phototube sealing methods proved to be vastly superior to the original CTF design and only 15% of the phototubes have failed over a period of 7 years of CTFII operation. The new shrouded detector vessel used a nested nylon vessel design. The 2 m diameter central nylon vessel was filled with scintillator just as in the original CTF; the scintillator vessel was a leak tight seal. A 4 m diameter nylon shroud was fabricated that surrounded the scintillator with a stagnant water layer. The shroud allowed for a slow leak of water to go out of the shroud into the water tank; it served as a diffusion barrier that reduced diffusion of radon to the region near the active scintillator. The phototubes, light collectors, cabling and steel water tank all emanated Radon into the water. In the original CTF, this created a high count rate at the outer surface of the scintillator vessel. By reducing the Radon diffusion to the surface of the scintillator vessel the event rate was reduced improving the event reconstruction and sensitivity of CTFII. Fig. 9 shows a photo taken inside the water tank of CTFII that shows the scintillator vessel and shroud.

Scintillator prepared by the same procedure employed in Borexino was tested in CTFII. Concentrated master solution of scintillator of 100 g PPO/L-PC was prepared. 100 kg of PPO powder was loaded into a 2.5 m³ storage tank through a vacuum lock hopper. 1 m³ pseudocumene was distilled and added to the storage tank. The PPO and PC were mixed by combined action of nitrogen bubbling into the solution and a stirring impeller. The concentrated master solution was subsequently pre-purified by water extraction, nitrogen stripping and vacuum distillation.

Concentrated fluor solution and pseudocumene were independently metered and mixed in-line to the desired concentration of the scintillator solution, 1.5 g-PPO/L-PC. The solution was added into the scintillator vessel of the CTF by displacement of water. Before any test high purity water was pumped into the bottom of the scintillator vessel of CTFII displacing scintillator out the top. After the old scintillator had been displaced fresh scintillator was added in through the top of the scintillator vessel while draining water from the bottom of the scintillator vessel.

The processing and flow paths of the scintillator components for the testing were nearly identical to those being employed in Borexino. Purification was identical and the in-line mixing and pumping were done in the pumping module of the filling stations so it also employed the identical path to that used for Borexino. This testing procedure verified the purity of the scintillator components and the cleanliness of the purification and piping system.

The scintillation of the solution in the CTF was measured to determine the levels of ¹⁴C, and background levels of ²²²Rn, ²¹⁴Bi–²¹⁴Po–²¹⁰Po. These were all determined to be consistent with the low levels achieved previously with the CTF verifying the scintillator components and their processing were all adequate for filling the Borexino detector.

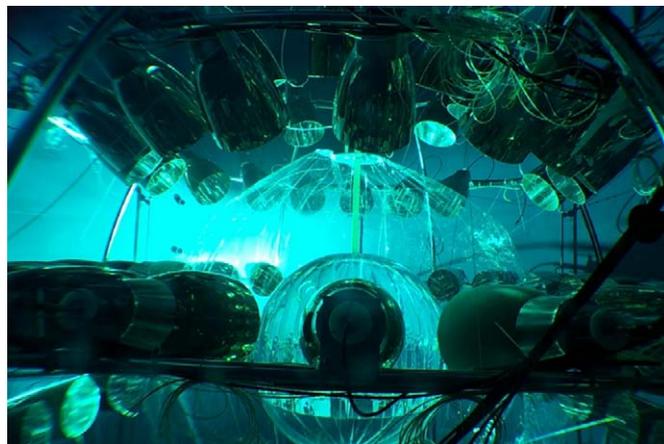


Fig. 9. A photo of the scintillator vessel and shroud inside CTFII. Scintillator is contained inside the central 2 m diameter nylon sphere. The 4 m diameter shroud is slightly misshapen. It contains water and acts as a diffusion barrier keeping radium and radon emanating from the phototubes and tank away from the surface of the scintillator vessel.

4.4. Borexino detector filling

The detailed filling procedure employing the filling stations for the Borexino detector is published elsewhere [10]. In summary, the process involved inflation of the nested nylon vessels with synthetic air, leak checking, displacement of air by high purity low Ar/Kr nitrogen, displacement of the nitrogen by high purity DI water and finally displacement of water by scintillator and buffer solutions. The gas inflation and the three fluid displacement operations were all done with tight control over the pressures and liquid levels in the inner zone containing scintillator, and the inner and outer buffer zones. Pressures, temperatures, gas and liquid flow rates, relative humidity, liquid levels, forces on the constraining ropes around the nylon vessels were frequently monitored during the filling operations. All these process variables were recorded by the digital control system and flow paths and flow rates were controlled by pneumatic valves controlled by the DCS. The most critical times during the filling operations were the crossing of the poles of the nylon vessels. Flow had to be initiated or terminated for a zone at a pole crossing, and the liquid levels changed rapidly with volume at the poles. Frequently switching of the flows between volumes was required at the pole crossings; the flow rates were reduced and liquid levels and differential pressures closely monitored to minimize the stresses on the nylon vessels.

The process variables recorded during the filling operations and the controlled parameter are listed in Table 5.

Pressures were recorded from the gas pressures in the head tanks at the top of the three zones and either gas or liquid pressure was recorded at the bottom of the three zones. The absolute liquid level was monitored in the out buffer, and the liquid levels in the inner buffer and scintillator volume were determined by the differential pressures between adjacent zones. The total mass of gas flow, along with temperature and relative humidity to the three head tanks was logged at a frequency of 0.1–1 Hz. The differential gas pressures were maintained at 1 cm H₂O between sequential zones with the highest gas pressure being in the inner zone. When the vessels were filled with water the three zones were filled together maintaining the differential water levels to ± 1 mm, always maintaining a small positive gradient from the inner zone to the outer buffer zone (the liquid level gradient was inverse to the gas pressure gradient). The tension in the ropes constraining the nylon vessels was monitored

Table 5
Process variables monitored or controlled during filling of Borexino.

Process variable	Range, sensitivity	Function
Gas pressure in outer buffer head tank	0–80 mbar g	Measure gas pressure during gas inflation Absolute pressure obtained by adding Hall C pressure
Absolute pressure at bottom of stainless steel sphere	0–18 m H ₂ O	Measure liquid level in SSS by pressure relative to the gas pressure in the water tank
Differential pressure between bottom of SSS and bottom of inner buffer	–100 to +100 mm H ₂ O	Measure differential pressure between outer buffer and inner buffer. Measurement by electronic gage and site gage
Differential pressure between bottom of inner buffer and bottom of scintillator volume	–100 to +100 mm H ₂ O	Measure differential pressure between inner buffer and scintillator zone. Measurement by electronic gage and site gage
Liquid level in stainless steel sphere	0–18 m	Liquid level site gage for the stainless steel sphere
Mass flow to head tanks	0–50 S m ³ /h	Mass flow rate, density, temperature and integrated mass flow from the gas module
Relative humidity of gases to head tanks	0–100% RH	Relative humidity of synthetic air or nitrogen after humidification
Mass flow of liquids to scintillator volume	0–3000 kg/h, 0–100 kg/L, 0–100 °C	Mass flow rate, density and temperature into the scintillator zone (inner vessel)
Mass flow of liquids to buffer volumes	0–3000 kg/h, 0–100 kg/L, 0–100 °C	Mass flow rate, density and temperature into the inner buffer and outer buffer
Force on constraining ropes to top of inner vessel	18 @ 500 kg _f /cell	Load cell measurements of the force on the ropes surrounding the inner vessel
Force of constraining ropes to bottom of inner vessel	18 @ 500 kg _f /cell	Load cell measurements of the force on the ropes surrounding the inner vessel
Force on constraining ropes to top of outer vessel	20 @ 1000 kg _f /cell	Load cell measurements of the force on the ropes surrounding the outer vessel
Force on constraining ropes to bottom of outer vessel	20 @ 1000 kg _f /cell	Load cell measurements of the force on the ropes surrounding the outer vessel
Temperature measurement inside the detector	8 locations, 0–100 °C	Pt RTD sensors inside filling pipes (4 in the IB, 4 in the OB)
Liquid level in outer buffer head tank	0–1000 mm H ₂ O	Measure of liquid flow in and out of detector during filling

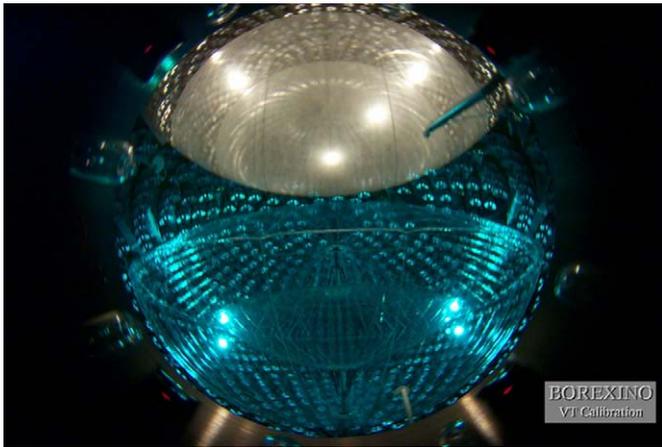


Fig. 10. Photo during the water filling of the Borexino detector. The top sections of the vessels are filled with nitrogen while the bottoms (blue color) are filled with water. A small differential nitrogen gas pressure (10–20 Pa) keeps the vessels spherical during the filling.

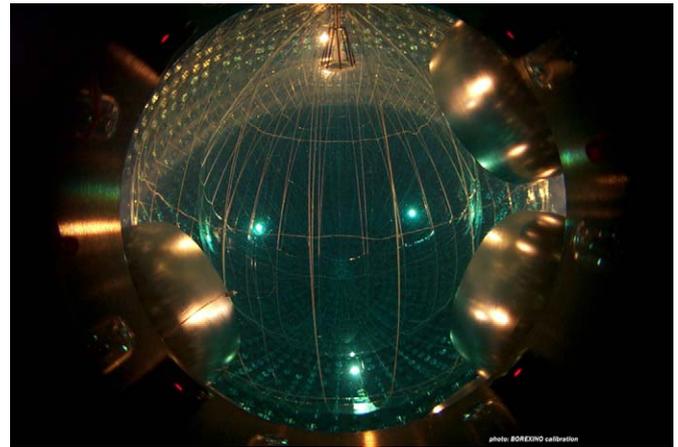


Fig. 11. Photo during the scintillator displacement of water in the Borexino detector. The photo is taken from a camera mounted on the stainless steel sphere and looks out between light cones. Both nylon vessels can be seen to be half full of water at the bottom and scintillator at the top. The support and guide ropes can be seen surrounding both the inner and outer nylon vessels. At the top of the inner nylon vessel is the pipe which feeds the scintillator into the inner vessel.

with load cells. These measurements were checked to make sure they were consistent with the filling schedule. The forces were maintained within a range of ± 2000 N corresponding to a liquid level differential of 2 mm.

Fig. 10 is a photo taken during the water filling process. The top sections of the vessels are inflated with nitrogen while the lower parts of the nylon vessels are filled with water. The gas pressure is maintained at 30 Pa to keep the shape of the vessels spherical. The water levels in each zone are kept the same to within 0.1 cm. The two nylon vessels can be seen in the gas filled region. Fig. 12 is a photo taken during the scintillator displacement of water. The nested nylon vessels can be seen with the water at the same level in the three zones. The pipe at the top of the inner nylon vessel where the scintillator is fed can be clearly seen. The polyethylene ropes that guide and constrain the nylon vessels are also evident in Fig. 11. There are load cells at the top and bottom attachment points for these ropes to measure the upward and downward forces on the vessels due to differences in liquid levels.

4.5. Online pressure maintenance and re-purification

Experience with the CTF had proven that the scintillator could be purified to the background levels that were necessary for Borexino. However, Borexino also requires that data be taken for extended periods of time to look for annual variations in the neutrino flux. The fluid handling system was designed to maintain the liquid levels and pressures in the scintillator and buffer zones of the detector to compensate for pressure and temperature fluctuations in the Hall C laboratory. In addition, the system was design to permit period circulation of scintillator and buffer fluids and permit on-line re-purification of the detector fluids. Both of these capabilities proved essential for the Borexino detector.

After all three detector zones have been filled to the tops of the vessels the final topping operation is done to set a small differential pressure between successive zones. A positive differential pressure from inside to outside is necessary to make the flexible nylon vessels assume the proper shape. The pressure differentials are set by adjusting the liquid level in standpipes at

the top of each vessel. This standpipe system can be seen in Fig. 12 as the pipes that go from the top of each vessel to a liquid head tank. The liquid head tanks are located in a clean room on the top of the external water tank located approximately 3.5 m above the stainless steel sphere. Each of the standpipes terminates inside a tank with an adjustable overflow weir. The tanks are connected to a common nitrogen blanket so the gas pressure is equal in each tank. By filling a volume until it overflows the hydrostatic pressure is set by the height of the weir. The difference in the heights of the weirs can be adjusted to fix the pressure differential between adjacent zones of the detector. If the pressure is higher in an outer zone than the inner zone a negative differential pressure causes the nylon vessels to fold; this is an intrinsically safe condition, but makes event reconstruction more difficult because the boundaries of the zones are not well defined. In contrast by positioning the weir for the inner zone higher than the outer zone creates a positive pressure differential that will cause the flexible nylon vessel to fully inflate and assume its spherical shape. However, if the pressure differential becomes too large the stresses on the nylon vessels and can cause the nylon to stretch and even break. The nylon vessels were designed to withstand pressure differentials of 500 Pa (5 cm H₂O) without irreversible deformation [21], and the typical operating conditions sought to limit the pressure differential to <50 Pa (5 mm H₂O).

An overall PID (process and instrumental drawing) of the three central zones of the Borexino detector (Scintillator, inner buffer and outer buffer) and the associated fluid handling systems is shown in Fig. 12. The topping operation begins by filling the outer buffer with solution from the bottom of the stainless steel sphere until liquid overflow the weir in the head tank for the outer buffer. The weir for the inner buffer is then set to a desired differential height of 2 mm above that for the outer buffer and buffer solution is flowed into the bottom of the nylon vessel that defines the inner buffer. Fluid is added until it overflows the weir in the head tank for the inner buffer. Lastly the weir height for the scintillator zone is set 2 mm above that for the inner buffer. Scintillator solution is flowed into the bottom of the inner vessel until fluid flows over the weir of the scintillator head tank. After this procedure, all three zones are filled and the vessels are fully inflated with well defined spherical shapes.

Temperature fluctuations and temperature gradients in Hall C disrupt the levels and pressures in the three zones of the Borexino detector. There are seasonal temperature fluctuations where the average temperature in Hall C changes by 3–5 °C. In addition, there can be additional fluctuations in Hall C of 3–4 °C resulting from heat dissipation from equipment operating in Hall C. (There are other experiments with equipment such as electromagnets that dissipate substantial heat when they are turned on.) Lastly, there is a temperature gradient where the temperature at the bottom of the water tank is typically 3–4 °C lower than water temperature at the top of the tank. The combination of the temperature gradient and temperature fluctuations cause the liquid levels in the three zones to change and disrupt the pressures.

When the temperature increases in Hall C the volumes of the fluids changes by ~0.1%/°C; a change in temperature of 1 °C will increase the volume of fluid in each zone by approximately 0.5 m³, which will overflow the weirs in the head tanks. But the volumes of the three zones are not the same so the volume of fluid from each zone is different. During positive temperature excursions the weir system in the head tanks maintains the proper liquid pressure head differential and the nylon vessels are protected against overpressure.

When the temperature decreases the liquid levels in the standpipes will fall. Because the different volumes and different diameters of the standpipes the height of liquid falls differently in the three zones. The differential pressures between zones are determined by the liquid level in the standpipes. It is necessary to repeat the topping operation to restore the liquid levels in all three zones to the levels of the weirs in the head tanks. The topping operation is done on a routine basis of once every 1–2 weeks to maintain the proper differential pressure.

Normally the temperature fluctuations in Hall C are slow and change < 1 °C/week, so pressure topping operations need to be done only once per week. Data logged by the control system showed that a large temperature swing of 4 °C occurred within hours on a single day in February 2008. This rapid temperature swing appears to have created a differential pressure that exceeded the design specifications, stressing the nylon vessels excessively creating a small leak to open up between the scintillator and inner buffer. The evidence of

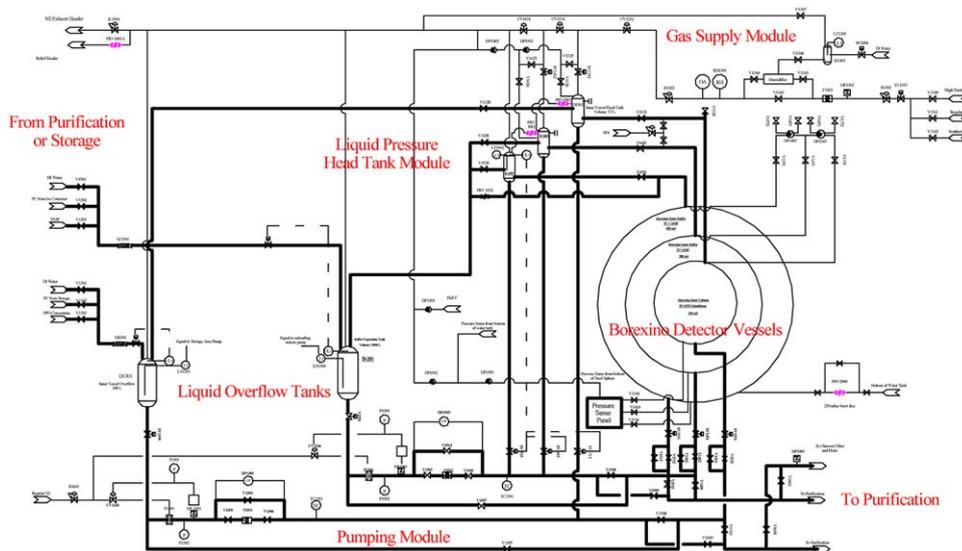


Fig. 12. Overall process and instrumentation drawing of the active scintillator and buffer zones of the Borexino detector vessels. The stainless steel sphere and two nested nylon vessels are the scintillator zone, the inner buffer and the outer buffer. The liquid pressure head tanks and nitrogen pressure maintenance combine to maintain the total pressure (hydrostatic and gas) in the nested nylon vessels. The Head Tank Module of the filling stations is located in a clean room on the top of the Borexino water shield tank. The surge tanks and assorted pumps and valves at the lower left of the drawing are part of the pumping module of the filling stations. The Pressure Sensing Module of the filling stations is connected to the lines from the bottom of the three detector zones.

the leak was not detected until August 2008 when it was noticed that there was an asymmetry in background scintillation events in the buffer. After careful analysis of the scintillation events and the fluid inventories of the scintillator and the buffer and testing samples from the buffer it was determined that approximately 1 m^3 of scintillator per month had been leaking into the buffer from the scintillator driven by the small differential pressure between the two zones. In January 2009, the collaboration decided to repurify the solutions in the buffer to remove the fluor from the buffer that had leaked in from the scintillator. The re-purification of the buffer commenced in February 2009.

There are multiple ways to operate the scintillator or buffer re-purification. They all consist of an in-line loop where fluid is removed from a zone, run through the purification plant and then purified material is returned to the zone. Re-purification of the buffer zones carried out in February and March 2009 can be followed in Fig. 12. Since the leak in Borexino was detected near the top of the vessel the collaboration chose to remove fluid from the top of the buffer zones and return cold purified pseudocumene to the bottom of the buffers.

The re-purification began by purifying $4\text{--}5\text{ m}^3$ of pseudocumene from the storage area and pumping it into the buffer surge tank, D201. From D201, purified PC was pumped into the bottom of the detector alternating between the inner and outer buffer. Automatic control valves alternately directed PC into the two buffer volumes. Purified PC entering the bottom of the buffer volumes pushed contaminated buffer material out the top of the inner and outer buffers into the pressure head tanks for the zones. The contaminated PC overflowed the weirs in the head tanks and was directed to the feed tank for the purification skids. The buffer solution was purified by distillation in the purification skids to remove the DMP and other heavy impurities. Purified PC was pumped into D201 to be fed into the bottom of the buffer zones. This process was run for a period of 3 months to purify the contents of the buffer zones one time assuming no mixing within the buffer zones.

4.6. Detector draining and shut down

The liquid handling system was designed for the safe draining and recovery of the scintillator and buffer components from the Borexino detector. This follows a similar path for the filling. The scintillator or buffer is drained from the bottoms of the three zones and is directed to the distillation system in the purification skids. The scintillator and buffer are distilled to remove the PPO and DMP and purified PC is recovered and sent to the storage tanks, from which the PC is loaded into tanker trucks and can be delivered to a long term storage facility, oil refinery, or a commercial user of pseudocumene.

5. Construction of the fluid handling system

Because of the complexity of the fluid operations and the need to share responsibility for building and installing the system a modular approach to construction and installation was adopted. All the different modules and their functions were described above. The modules of the fluid handling systems were designed and built to specifications of cleanliness and leak-tightness to avoid contamination and minimize the radioactive impurities. These criteria are described in detail in a previous paper [11]. Different parts of the overall fluid handling system were built by different contractors in the US, Italy and Germany, but all were built to the same criteria. The distillation/gas stripping purification system was built in the US by Koch Modular Processes (Paramus, NJ, USA) [11]. The filling station modules were built by Vacuum Processes, Inc (Everett, PA, USA) [10]. The adsorption column purification system was built partly in Munich and

complete in Gran Sasso by the Physics Department at Technical University of Munich and CMG Ltd. The high purity nitrogen system for radon adsorption was built in at the Max Planck Institute in Heidelberg. The interconnection system, cleaning module, exhaust system, and upgrades and local changes in Italy were done by CMG Ltd. The large systems were all shipped to Gran Sasso and placed in position in Hall C. All the local piping connections to the interconnection system were carried out by CMG.

Piping is electropolished 316L stainless steel (SS) throughout. Fittings are electropolished SS VCR fittings from Swagelok. Most valves are bellows sealed high vacuum electropolished SS from Swagelok and Carten. In places where use of stainless steel was not possible Teflon and quartz were acceptable alternatives. The welding procedures were specified to be orbital welding with inert Ar gas throughout to minimize any scale and corrosion. Flanges on larger vessels and tanks were double sealed; Helicoflex metal gaskets on the inside with Viton o-rings on the outside and a vacuum pumpout port between.

A special feature to the fluid handling systems was clean-in-place technology. We provided valves for draining so the piping sections could be isolated for cleaning. Dead-legs and traps in the piping were avoided. After making the connections in Hall C each module was cleaned in place with detergents, chelating agents and finally copiously rinsed with DI water until the particulate count in the rinse water was below level 50. Particulate filters were installed near the point of final use for gas and liquid lines. During the cleaning the filter cartridges were removed from their housing. After the final rinsing, the submicron filter cartridges from either Millipore or Pall were installed.

All sections of the fluid handling system were vacuum leak checked with Helium. Each component (valve, sensor, flow meters, fitting, etc.) was required to be leak tight to a maximum leak rate of $10^{-8}\text{ cm}^3/\text{s}$. The combination of the clean-in-place design, the rigorous cleaning procedure and leak testing were keys to achieving low backgrounds in the Borexino detector.

All the system modules were examined by a safety committee for hazardous operations. The systems had to have at least two levels of safety against hazards to people or the environment. Safety requirements included the following:

1. Earthquake safety. All the modules containing an inventory of more than 100 L of pseudocumene had to meet structural requirements of the supporting structures to withstand a specified seismic load. Independent engineering analyses were carried out for all major structures including the Borexino purification skids, the pseudocumene storage vessels and storage area, the buildings that the DMP and concentrated fluor solution, the stainless steel sphere and the external water tank.
2. Containment safety. All modules and systems that had an inventory of more than 100 L of pseudocumene was required to be surrounded by a containment basin that could hold the liquid inventory in case of a vessel or piping failure. The containment basins around the purification skids and storage areas were cinder block walls painted on the inside with epoxy to hold pseudocumene.
3. Fire safety. All areas were protected by either spray foam systems or inergen gas systems for fire. Confined systems such as the purification skids had separate sensors and an inergen system to protect against fire. Systems in open areas, such as the pseudocumene storage area had spray nozzles for fire extinguishing foam.
4. Explosion safety. All vessels that could contain pseudocumene were required to have pressure relief. Most of the pressure relief systems were burst discs, sized to match the volume of

the vessel and with a pressure rating below the pressure rating on the vessel. The relief systems piped directed into a blow down system with a water tank where any hot gases would be quenched and liquids would be trapped. All vessels also had pressure sensors and the pressures in all tanks were monitored by the Digital Control System. Alarm systems were set to stop all flows, heating, etc. in case of an alarm. The alarms were set to terminate any operation before the pressure burst discs would release. This was typical for two levels of safety.

5. Exhaust gas safety. To maintain control over pressure in most vessels containing pseudocumene the vessels had a nitrogen gas blanket above the liquid. A small gas flow was provided that permitted any gas impurities such as Radon to be continuously removed, while also permitting active control over the pressure. The nitrogen gas flows were all exhausted to an exhaust system. The exhaust system was a blower that maintained a small negative pressure in the exhaust lines. The blower forced the exhaust gases to flow through an activated carbon filter before allowing the gases to be vented to the atmosphere.
6. Gas sensors. Hydrocarbon and oxygen sensors were placed in every contained module including the clean rooms, the stainless steel sphere, the purification skids, and the containment building for the interconnection system and storage of DMP and concentrated fluor. These sensors were monitored by the digital control system which activated an alarm if the atmosphere was unsafe for people.
7. Inventory control. Strict inventory control on pseudocumene was practiced. The volumes of pseudocumene contained in the storage area, the purification skids, the three zones of the Borexino detector, and in the waste tank. Strict environmental regulations adopted by the labs after 2002 made it essential to have precise inventory control and assure the local governments there were no releases of pseudocumene to the environment. Through use of level sensors, load cells and mass flow meters the inventory and flows of pseudocumene were carefully regulated and documented.

The design, construction, installation, and commissioning of the different parts of the fluid handling system for Borexino extended over a 10-year period of 1997–2007. The start date corresponds to the successful demonstration of a low background scintillator ($<10^{-15}$ g-U/g-scintillator) with the counting test facility. That success permitted the members of the collaboration to obtain funding from the INFN, the NSF and (German agencies) to construct Borexino. The design of the fluid handling system and operations was based on scaling of the systems demonstrated with the CTF. Mostly this required scaling equipment to process $1\text{ m}^3/\text{h}$ of scintillator components in Borexino compared to $0.025\text{ m}^3/\text{h}$ with the CTF. There were two significant differences between Borexino and the CTF were (1) Borexino employed a buffer of pseudocumene rather than water and (2) Borexino could not provide for on-site storage for all the pseudocumene employed in both the scintillator and the buffer for Borexino. These differences required a system that could couple multiple processes – shipping and receiving pseudocumene, purification, mixing of scintillator and buffer, and filling of the three detector zones simultaneously. This required careful coordination among the groups designing and building the different systems.

The major milestones for the design, construction and commissioning is outlined in Table 6.

After each different module (purification system, storage tanks, unloading system, filling stations) was installed with its safety systems in place at LNGS it was connected to the interconnection system in Hall C. Connections were also made to the auxiliary plants, the chilled water system for the condenser, the hot oil

Table 6
Milestones for Borexino fluid handling systems.

Dates	Task
Design phase April 1996–January 2000	<ol style="list-style-type: none"> 1. Design of Purification Plants 2. Design of filling stations 3. Design of PC unloading station and storage area 4. Design of Radon adsorption system for high purity N_2 5. Design of Interconnection system 6. Design of cleaning module 7. Design of blowdown and exhaust systems 8. Design and CTF testing of silica gel adsorption system 9. Review of safety systems
Construction phase April 1999–January 2002	<ol style="list-style-type: none"> 1. Fabrication of distillation Purification plant at engineered mechanical systems (Paramus, NJ, USA) 2. Fabrication of filling stations at Vacuum Processes Inc. (Everett, PA, USA) 3. Fabrication of high purity nitrogen system Heidelberg, Germany 4. Fabrication of Interconnection system CMG in Hall C LNGS 5. Fabrication of Storage vessels at W. Tosto (Chieti, Italy) 6. Fabrication of cleaning module by CMG in Hall C LNGS Fabrication of nylon vessels (Princeton, NJ, USA)
Shipping and installation of Borexino June 2000–August 2002	<ol style="list-style-type: none"> 1. Installation of water shielding tank and stainless steel sphere for Borexino detector 2. Precision cleaning and reassembly of purification system 3. Shipping distillation purification system from US to LNGS 4. Installation of PC storage tanks in Hall C 5. Installation of high purity N_2 system in Hall C
Fluid handling system installation	<ol style="list-style-type: none"> 1. Installation of purification skids in Hall C of LNGS 2. Installation of hot oil system in Hall C 3. Rebuilding of counting test facility (CTFII) 4. Installation of clean air system and fire and spill safety systems for purification system 5. Installation of containment system and fire safety for PC storage area 6. Connection of process lines 7. Installation of DMP storage and concentrated fluor systems 8. Installation of digital control system and connection to process instrumentation 9. CTFII installation
Commissioning August 2001–August 2002	<ol style="list-style-type: none"> 1. Installation of cleaning module 2. Purification system testing with water 3. Purification system testing and controller tuning with PC 4. Scintillator testing with CTFII
Shutdown August 2002–June 2005	<ol style="list-style-type: none"> 1. Shutdown period 2. Lab testing of cleaning methods 3. Evaluation of safety systems
Recommissioning June 2005–December 2006	<ol style="list-style-type: none"> 1. Installation of nested vessels for Borexino detector 2. Drain pseudocumene and water from all piping and vessels 3. Restart operations with in-situ cleaning of purification plants, filling stations and interconnection system 4. Restart purification plants 5. Retest purification system with water 6. Retest purification system with PC – test purification results with CTF

Table 6 (continued)

Dates	Task
Detector filling February 2006–May 2007	<ol style="list-style-type: none"> 1. Gas inflation of Borexino detector 2. Leak testing of nested nylon vessels 3. Gas stripping of DI water for water filling 4. Water filling of Borexino detector 5. Distillation, gas stripping and PPO master solution preparation 6. Receiving pseudocumene from Polimeri Europa 7. Pseudocumene distillation and on-line mixing of scintillator and buffer 8. Water displacement by PC based scintillator and buffer
Detector operation May 2007 to present	<ol style="list-style-type: none"> 1. Pressure and level maintenance in the nested nylon vessels by topping operations
Repurification of buffer December 2008–April 2009	

system for the reboiler in the distillation system and preheat for the gas stripping column, the nitrogen system for the instruments, pneumatic controls and pumps, the high purity nitrogen system for the gas stripping operations, the high purity water for water filling of the Borexino detector, and the cleaning system for cleaning the system. The gas exhausts from vacuum pumps and nitrogen blanketing flows were all connected to a central system with carbon filters to safely exhaust the gas to the environment without contamination by organic vapors. All the connections in Hall C followed the same protocols as specified for the module construction. Orbital welding with inert gas was done in place to keep the system leak tight and clean. This work was done principally by CMG. Some of the smaller modifications were done by the technical staff at LNGS.

All the instruments and sensors required power and signal wiring to the Digital Control System. This work was all performed by the technical staff from LNGS and collaboration members. They also tested and calibrated all the instruments after installation and connection to the DCS.

6. Commissioning

After installation at Gran Sasso every system had to go through a series of tests to commission it for operation. Frequently through the testing it was necessary to make some minor modification to the systems. We illustrate the commissioning process using the purification system as an example. The commissioning involved the sequence of steps listed below:

1. Testing all process instruments (e.g. flow meters, pressure transducers, thermocouples). Calibration of the instruments, readout verification and the interface to the digital control system were tested for all test fluids, nitrogen, water and pseudocumene. This frequently relied on pumping fluids through different vessels and other parts of the system to verify instrument operation.
2. Testing functionality of auxiliary plants (chilled water, hot oil, nitrogen systems and exhaust system).
3. Leak testing and final cleaning of all equipment.
4. HazOp reviews of all equipment and proposed process. Approval of the safety of the system by independent safety consultant and safety representative of LNGS.

5. Development of written procedures for all operations. Procedures were reviewed by a committee and submitted to the director of LNGS and the safety representative for approval.
6. Test operation of all purification systems (distillation, extraction, gas stripping) with DI water and nitrogen.
7. Preliminary controller tuning based on operations with DI water. Standard tuning of single loop and cascade loop controller were implemented by the digital control system. Standard open and closed loop tuning methods were implemented for the controllers, followed by adjustments to improve system responses. Process stability and minimal overshoot after a disturbance was generally the most important criteria for controller tuning.
8. Test operations of purification systems with PC in a loop mode. A total of $\sim 2\text{--}3\text{ m}^3$ of PC was circulated through the purification system. The controller tuning was fine tuned to be consistent with PC purification.
9. Scintillator purification tested in CTFII.

The initial commissioning of Borexino was nearly completed in August 2002 and testing of the purification system efficacy with the CTF was underway. During the final testing an accidental spill of 30–40 L of PC occurred. The spill was the result of operator error – a multi-position valve was turned the wrong way. The operators notice the error when the control system showed no change in mass in a receiving vessel. The system was immediately shut down by the operators. Upon investigation of the incident it was discovered that several of the laboratories permits for use of water and wasting materials were inadequate for scale of the laboratory and Borexino. Borexino and most other activities at the LNGS were put on hiatus for 3 years while proper systems were put in place for the experiments at LNGS.

During the hiatus period all the fluid handling operations for Borexino were carefully scrutinized. A number of modifications were made to improve process safety. There were also a number of large scale improvements made throughout Hall C associated with the use of water for both processing (in the DI water plant) and for cooling (a new circulation system of cold water for process chilling was installed). The fluid handling system was also carefully reviewed and a number of changes were made to facilitate clean-in-place procedures.

The Borexino fluid handling systems and purification system were put back in operation in the second half of 2006. The system was all re-cleaned and leak checked. Many of the gaskets on flanges were replaced. The cleaning was done with great care and all the rinse water and other waste product fluids were drained into waste tanks and periodically removed from Hall C for waste treatment. When the labs were permitted to restart operations the CTF purification system was modified to carry out the distillation of the PPO master solution.

During the planning for filling the Borexino detector it was decided that the nested nylon vessels that separated the buffer regions and the active detector volume would be filled with water and then the water would be displaced by scintillator and buffer solutions. There was concern that R_n in the water would be transferred into the scintillator during the displacement operation. Hence, a decision was made by the collaboration to improve the quality of the water used to fill the scintillator and buffer volumes by gas stripping of the DI water in the purification system. Gas stripping of the water followed the same procedure as gas stripping of pseudocumene.

While the Borexino detector was filled with water the concentration PPO master solution was prepared and purified as described in Section 7.2. After the water filling was completed the purification system was switched over and distilled PC for filling

the Borexino detector by displacing the water with scintillator and buffer.

6.1. Cleaning and leak testing

The purification system and all the equipment involved to the process (pumps, sensors, rupture discs, etc.) were cleaned in place and vacuum leak checked at the end of the cleaning campaign, in accordance to the leak-tightness requirements reported elsewhere [11]. The subsystem was pumped down and helium leak checked. The pump down time and dynamic response time to He exposure were both measured to quantify the leak rate.

The purification system passed all the leak tests at the level of 10^{-8} mbar L/s. However, between the initial commissioning and the re-commissioning we observed that leak rate at several of the large gasketed seals increased over time. In order to protect against such leaks two safety precautions were introduced:

1. Construction of aluminum enclosures with a continuous nitrogen purge over fittings and sensitive joints (sensors, rupture discs, flanges) that contact the scintillator.
2. Purging of all the double sealing gaskets with nitrogen.

6.2. Loop mode and CTF testing

The CTF detector was extremely important during the commissioning and the operations of the fluid handling system and purification system; before filling Borexino a complete filling of the CTF detector with the same procedures was performed and the scintillator purity was assessed.

Scintillator testing was done by displacing the existing scintillator in the CTF with water and then displacing the water with fresh scintillator, following the same procedures and piping that was employed for the Borexino filling path; only few valves and few meters of lines were different between the CTF testing and the Borexino filling. During the Borexino filling, the CTF was also used periodically to test the quality of the scintillator adding 0.5 tons of fresh scintillator in each test.

7. Operation of the fluid handling system during filling of Borexino

The nested nylon vessels of the Borexino detector were filled sequentially with synthetic air, nitrogen gas, DI water and finally scintillator and buffer. The vessels were inflated with nitrogen during the period January–March 2006. The vessels were maintained inflated during both water filling and filling with scintillator. Details of the filling operations are reported in a companion paper [10,21].

Detailed procedures were developed and reviewed before the operations. The procedures specified the number of personnel and training requirements for the personnel to carry out the operations. The filling operations were done with shift operations 24 h a day for 5 days a week. There were three shifts a day with 3 persons per shift. The shift workers included one expert who was knowledgeable of the fundamental operation of all the fluid handling subsystems. The shift expert was also expected to be knowledgeable with the safety conditions necessary to fill the nested nylon vessels without causing damage. That shift leader was capable of making changes to the operations in response to small excursions away from the expected performance. There were two shift workers who monitored all the process variables for the purification system, the filling stations and the nested

nylon vessels during the filling of Borexino. Additional personnel were required during receiving and unloading of pseudocumene shipments. These deliveries were always done after 18:00 to avoid interfering with normal activities in the LNGS underground laboratory and to minimize the personnel present during this potentially hazardous operation.

7.1. Water filling

Borexino was filled with DI water during the period August 2006–November 2006. DI water was obtained from the Borexino high purity water system [18,19]. The water was pumped to the Borexino scintillator purification system and stripped with nitrogen. The stripping has been performed under a partial vacuum (400 mbar) at the flow rate of 900 L/h of water and 20 S m³/h of LAKN. The final measured radon content in the water has been less than 1 mBq/m³ with feed water into the purification system at the level of 300 mBq/m³.

After nitrogen stripping, the water was directed into the three detector volumes: 315.8 tons into the inner vessel (IV), 367.5 into the inner buffer (IB) and 643 into the outer buffer (OB). The filling was performed cyclically sending the water to the three zones. The flexible nylon vessels were all kept fully inflated with a nitrogen gas pressure above and water filling from below. The water levels were kept the same in all three volumes to <0.5 cm. The control of the water flow to the three volumes during filling, as well as all the control of the gas and liquid flows in the purification system was implemented by the Borexino DCS. The DCS provided a trouble free automated system operating 24 h/day with a minimum of operator attention.

7.2. Scintillator filling

Borexino was filled with scintillator (PC+PPO 1.5 g/L) and with liquid buffer (PC+DMP 5 g/L) during the period December 2006–May 2007. The purification system distilled and nitrogen stripped all 1326.3 m³ of PC needed to fill the scintillator and buffer volumes of the detector.

Prior to filling the master solution of PPO in PC was purified by water extraction, filtration, distillation and nitrogen stripping. The master solution preparation followed the procedure below:

1. 120 kg of PPO powder was placed in a 2 m³ stainless steel tank through a load-lock funnel.
2. The tank holding the PPO was purged with nitrogen and then 860 L of PC was added to the 2 m³ tank. The purging was necessary to remove oxygen, which has a detrimental effect on the scintillator.
3. The solution was thoroughly mixed by an impeller for ~1 h.
4. 1000 L of high purity DI water was distilled and added to the tank.
5. The water and concentration master solution were thoroughly mixed with the impeller for 6–8 h.
6. After mixing the solution was allowed to settle for 10 h with the PC/PPO master solution separating to the top of the tank; after settling the water was drained from the bottom of the tank.
7. The water extraction was repeated 4 times for each batch of PPO master solution.
8. After water extraction the PPO master solution was sparged with nitrogen.
9. The water extracted master solution was metered at 20 L/h through a 0.05 μ m filter and fed to a single stage evaporator for distillation.

10. The PPO/PC master solution was distilled at 30 mbar; the distillation was done for ~8 h. The temperature in the system crept up from 190 to 205 °C over the 8 h period.
11. After 8 h, the bottoms from the evaporator were drained. Approximately 1 L of bottoms was collected.
12. The overhead vapor from the evaporator were condensed and cooled to ~ 15 °C. The distilled PPO master solution was pumped to the top of a 1.5 m tall packed column. Low argon krypton nitrogen was flowed upward through the column at 1.2 kg/h.
13. The purified master solution was pumped into a buffer tank for temporary storage for the filling.

Pseudocumene was delivered by tanker truck from Sardinia to LNGS. The PC was loaded directly into an electropolished tanker from the final distillation of PC at the production plant. It was shipped directly to LNGS in 1 day. This rapid delivery limited the exposure of the freshly produced PC to cosmic radiation, thereby minimizing the production of cosmogenic ^7Be in the PC [28]. At LNGS the PC was offloaded into one of the 100 m³, electropolished stainless steel storage tanks located in Hall C of LNGS. The PC was pumped from the storage tanks to the input of the distillation column of the purification system. At steady state, the purification system was fed 850 L/h.

A detailed description of the Borexino purification system is published. During the filling process the distillation column was operated at a partial vacuum of 80 mbar at the temperature of 92 °C, with a reflux ratio of 0.25. Nitrogen stripping was performed under a partial vacuum (300 mbar) operated at the flow rate of 850 L/h of PC and 9 kg/h of LAKN at the temperature of 35 °C. To control the flexibility of nylon containment vessels the relative humidity of the PC must be maintained at 60–80% RH [7]. During the stripping operation with Low Ar/Kr N₂ 30 g/h of DI water was injected into the stripping column to control of the relative humidity of the PC. The solubility of water in PC at 15 °C is ~0.045 g/L, so the added water is sufficient to produce PC with a dissolved water content corresponding to 75% RH.

The buffer and scintillator volumes of the Borexino detector were filled following a calculated table in order to replace 0.5 cm of water with PC+PPO in the IV and PC+DMP in the IB, OB. The PPO and the DMP were added to the PC at the exit of the purification system, through an automatic regulated mixing in line. Samples were drawn periodically and tested for the PPO concentration and the humidity of the product; measurements were performed on samples taken from different locations. The filling operations were run for 24 h a day, 5 days a week. The purification plants were shut down for the weekends. In addition five CTF campaigns were run

during the filling to check the radioactive properties of the scintillator, in particular the levels of ^{222}Rn , ^{210}Po and ^{14}C .

8. Efficacy of the Borexino fluid handling system

Laboratory tests have been able to give guidance about the effectiveness of different purification methods, but the laboratory tests cannot measure the level of impurities that are necessary for Borexino. Furthermore, the ability to ship and move the large quantities of pseudocumene necessary for Borexino through a complex plant without introducing contamination cannot be effectively tested in the laboratory. The Borexino detector is the ultimate test of the fluid handling and purification system. The background levels of ^{14}C , ^7Be , ^{40}K , ^{238}U , ^{232}Th , ^{210}Bi , ^{210}Po , ^{39}Ar and ^{85}Kr have all been determined after the initial filling of Borexino [4]. To highlight the effectiveness of the materials selections and the fluid handling and purification system we compare the impurity levels with no purification to those achieved with purification. The radioactive impurity levels with no purification make the assumption that the organic scintillator is equilibrated with air or has some minimal amount of dust contamination. Those standard impurity concentrations are summarized in Table 7.

The design specifications for radioactive impurities in Borexino along with the levels achieved are compared to the typical levels without purification. The impurities have been reduced by four orders of magnitude or more by the purification processes employed by Borexino. These results are impressive and show that well known chemical purification methods can be employed to achieve extraordinary levels of radiopurity in liquid organic scintillators. Although the effectiveness of each specific purification method to remove radioactive impurities has not been identified we have demonstrated that a multi-pronged approach extrapolated from well known processes applied in the petrochemical industry has proved successful (Table 8).

9. Summary and conclusions

Borexino is a large scale low background scintillation detector. Borexino has demonstrated extraordinarily low background permitting the low energy solar neutrino spectrum to be obtained with unprecedented sensitivity. The implementation of a novel flexible vessel containment system for scintillator and shielding and the systems for processing and handling more than 1000 tons of organic liquid scintillator components for the Borexino Detector was a major engineering challenge. Fluid handling strategies were at the core of Borexino's success. Borexino relied on careful

Table 7
Typical concentrations of radioactive impurities in organic scintillators.

Radioisotope	Typical concentration in equilibrated organic scintillator
^{14}C	$^{14}\text{C}/^{12}\text{C} = 10^{-12}$ level corresponding to equilibrium from cosmic radiation at the earth's surface
^7Be	2.7×10^3 cpd/ton corresponding to equilibrium from cosmogenic production of ^7Be from ^{12}C at the earth's surface [28]
^{222}Rn	1.3×10^7 cpd/ton. This corresponds to equilibrium Rn absorption into PC from air with 10–100 Bq/m ³ -air
^{210}Pb	2×10^4 cpd/ton for ^{210}Bi and ^{210}Po . This corresponds to exposing the surface of the scintillator containment vessel to air with 10 Bq/m ³ Rn for 1 year and assuming all the ^{210}Pb is deposited on the vessel surface
^{238}U , ^{232}Th	10^5 cpd/ton. This corresponds to 1 mg dust suspended in 1 ton of scintillator. The dust is assumed to have $\sim 10^{-5}$ g-U(Th)/g-dust, this value is approximately the natural abundance of U and Th in the earth's crust [29,30]
^{40}K	2700 cpd/ton. This is based on a level of 10^{-6} g-K/g-PPO, and 1.5 g-PPO/L-PC. The 1 ppm level of K in PPO was determined previously by neutron activation measurements [23]
^{39}Ar	200 cpd/ton. This corresponds to equilibrium Ar absorption into PC from air with 13 Bq/m ³ -air [31]
^{85}Kr	4.3×10^4 cpd/ton. This corresponds to equilibrium Kr absorption into PC from air with 1 Bq/m ³ -air [32]

Table 8
Purification efficacy in Borexino.

Radioisotope	Typical level in equilibrated scintillator	Design level for Borexino	Level achieved in Borexino [4]
^{14}C	10^{10} cpd/ton	10^4 cpd/ton	3.5×10^4 cpd/ton
^7Be	2.7×10^3 cpd/ton	<0.1 cpd/ton	Below threshold
^{222}Rn	10 Bq/m ³ in air	<0.1 cpd/ton	1 cpd/100 ton
^{210}Pb	1.3×10^7 cpd/ton		
	2×10^4 cpd/ton	<0.1 cpd/ton	15 cpd $^{210}\text{Bi}/100$ ton
$^{238}\text{U}, ^{232}\text{Th}$	10^{-5} g-U(Th)/g-dust	^{210}Bi and ^{210}Po	6000 cpd of $^{210}\text{Po}/100$ ton
	10^5 cpd/ton	$<10^{-16}$ g-U(Th)/g-scintillator	$<10^{-17}$ g/g
	10^5 cpd/ton	<0.1 cpd/ton	<1 cpd/100 ton
^{40}K	10^{-6} g-K/g-PPO	$<10^{-14}$ g-K/g-scintillator	$<10^{-14}$ g-K/g-scintillator
	2700 cpd/ton	<0.027 cpd/ton	<3 cpd/100 ton
^{39}Ar	13 mBq/m ³ -air	<500 nBq/m-N ₂	<30 cpd/100 ton
	200 cpd/ton	<0.01 cpd/ton	
^{85}Kr	1 Bq/m ³ -air	<100 nBq/m ³ -N ₂	<30 cpd/100 ton
	4.3×10^4 cpd/ton	<0.01 cpd/ton	

strategies to minimize the radioactivity. The key considerations were as follows:

1. Choice of materials with low levels of radioactivity impurities.
2. Implementation of design strategies that allowed all fluid handling equipment to be cleaned-in-place to minimize dust and other contamination that might dissolve or be suspended in the scintillator.
3. Construction of a system to high purity and leak tight standards. This included: the use of electropolished stainless steel piping, vessels and fitting throughout that were non-particulating; use of high vacuum fittings and double gasketing or purge systems to avoid infiltration of radioactive impurities from the atmosphere.
4. Implementation of a purification system that effectively removed radioactive impurities and maintained high optical quality of the scintillator and shielding buffer fluids.

The strategies and procedures to build and clean the fluid handling system for delivery, storage and preparation of scintillator materials have been outlined in this paper. Ideally the entire system would have been built within a clean room with high efficiency particulate filters, but the size and location of Borexino made that impossible. Instead we developed a modular fluid handling system that could be cleaned in place with detergent washing and DI rinsing cycles to reduce the suspended particles to below Level 50 (by Mil Spec 1246C). The results with the Borexino detector show this strategy was probably as effective as building everything in a clean room.

On-site purification of pseudocumene and a concentrated PPO/PC fluor solution was described. This permitted a just-in-time delivery system for receiving the pseudocumene and directly filling the Borexino detector with high purity scintillator. These processes were done achieving unprecedented low radioactivity in a large scale scintillator.

The fluid handling strategies and purification methods employed in Borexino can be adapted as a foundation to developing new low background detectors.

Acknowledgements

We sincerely thank the funding agencies: INFN (Italy), NSF (USA), BMBF, DFG and MPG (Germany), Rosnauka (Russia) and we acknowledge the generous support of the Laboratori Nazionali del Gran Sasso.

References

- [1] C. Arpesella, et al., Physical Review Letters 101 (9) (2008).
- [2] C. Arpesella, et al., Physics Letters B 658 (4) (2008) 101.
- [3] Borexino_Collaboration, et al., Astroparticle Physics 16 (3) (2002) 205.
- [4] Borexino_Collaboration, et al., Physics Letters B (2007) PLB 24344.
- [5] A. Ianni, et al., Nuclear Instruments and Methods in Physics Research Section A – Accelerators Spectrometers Detectors and Associated Equipment 537 (3) (2005) 683.
- [6] A. Brigatti, et al., Nuclear Instruments and Methods in Physics Research Section A – Accelerators Spectrometers Detectors and Associated Equipment 537 (3) (2005) 521.
- [7] J. Benziger, F.C.L. Cadonati, E. de Haas, R. Fernholz, R. Ford, C. Galbiati, A. Goretti, E. Harding, An. Ianni, S. Kidner, M. Leung, F. Loeser, K. McCarty, A. Nelson, R. Parsells, A. Pocar, T. Shutt, A. Sonnenschein, R.B. Vogelaar, Nuclear Instruments and Methods in Physics Research Section A – Accelerators Spectrometers Detectors and Associated Equipment 582 (2007) 509.
- [8] Borexino_Collaboration, et al., Nuclear Instruments and Methods in Physics Research Section A – Accelerators Spectrometers Detectors and Associated Equipment 406 (3) (1998) 411.
- [9] C. Arpesella, et al., Astroparticle Physics 18 (1) (2002) 1.
- [10] J. Benziger, et al., Nuclear Instruments and Methods in Physics Research Section A – Accelerators Spectrometers Detectors and Associated Equipment (2009), submitted for publication.
- [11] J. Benziger, et al., Nuclear Instruments and Methods in Physics Research Section A – Accelerators Spectrometers Detectors and Associated Equipment 587 (2–3) (2008) 277.
- [12] H. Simgen, G. Zuzel, Topical Workshop on Low Radioactivity Techniques: LRT 2006, 2006, vol. 897, pp. 45–50.
- [13] G. Zuzel, H. Simgen, G. Heusser, Applied Radiation and Isotopes 61 (2–3) (2004) 197.
- [14] Borexino_Collaboration, et al., Physics Letters B 422 (1–4) (1998) 349.
- [15] B. Kanegsberg, E. Kanegsberg, Handbook for Critical Cleaning, CRC Press, Boca Raton, FL, 2001, p. 658.
- [16] ASTM_International, Standard Specification for Chemical Passivation Treatments for Stainless Steel Parts, 2005, p. 6.
- [17] US_Army, Product cleanliness levels and contamination control, Mil Std. 1246C, US_Army, Editor, 1994, p. 26.
- [18] M. Balata, et al., Nuclear Instruments and Methods in Physics Research Section A – Accelerators Spectrometers Detectors and Associated Equipment 370 (1996) 605.
- [19] M. Giammarchi, M. Balata, R. Scardaoni, Ultrapure Water Journal 13 (1995) 95.
- [20] H. Simgen, G. Heusser, G. Zuzel, Applied Radiation and Isotopes 61 (2–3) (2004) 213.
- [21] J. Benziger, et al., Nuclear Instruments and Methods in Physics Research Section A – Accelerators Spectrometers Detectors and Associated Equipment 582 (2) (2007) 509.
- [22] J.B. Benziger, Nuclear Physics B – Proceedings Supplements 78 (1999) 105.
- [23] J.B. Benziger, et al., Nuclear Instruments and Methods in Physics Research Section A – Accelerators Spectrometers Detectors and Associated Equipment 417 (2–3) (1998) 278.
- [24] H.O. Back, et al., Nuclear Instruments and Methods in Physics Research Section A – Accelerators Spectrometers Detectors and Associated Equipment 585 (1–2) (2008) 48.
- [25] G. Schlowsky, Stripping calculation Gran Sasso Module, Personal communication, Editor, 2002.
- [26] D.E. Seborg, T.F. Edgar, D.A. Mellichamp, Process Dynamics and Control, second ed., Wiley, New York, 2004, p. 713.
- [27] J. Benziger, et al., Environmental effect on the optical properties of pseudocumene, 1998.

- [28] R. Vogelaar, et al., Nuclear Instruments and Methods in Physics Research Section A – Accelerators Spectrometers Detectors and Associated Equipment 372 (1996) 59.
- [29] K. Barbalace, Periodic Table of the Elements, Environmental Chemistry.com. 2007.
- [30] Abundance of Elements in Earth's Crust, 2007 [cited]. Available from: <http://en.wikipedia.org/wiki/Abundance_of_elements_in_Earth's_crusti>.
- [31] H.H. Loosli, Earth and Planetary Science Letters 63 (1983) 51.
- [32] S. Cimbak, et al., Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms 17 (5–6) (1986) 560.