Report on the Borexino Storage Vessels

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1 Introduction

The design of Borexino requires some storage volume for the liquid scintillator solvent used in the experiment: $\approx 320m^3$ of Pseudocumene (PC, 1,2,4-trimethyl benzene) at room temperature.

For this purpose 4 cylindrical stainless steel tanks have been built, and have been installed in august and september 1999 in the Gran Sasso Hall-C. Three tanks will be used to store the solvent before mixing it with PPO and put it into the Borexino Inner Vessel; the fourth tank will be used for N_2 stripping and, if necessary, to store the buffer PC.

Borexino very peculiar requirement is an extreme radioactive purity of all the materials used in the detector construction and in particular of the liquid solvent. Since the PC could stay in these storage tanks for 2-3 months, very special care was necessary during and after their construction.

I report here some information about their characteristics and the procedures followed and the tests done during their construction at the WTS Co. (Walter Tosto Serbatoi, Chieti Scalo, Italy) in the period october 1998 - august 1999.

2 Technical Specifications

Each container is a standing, cylindrical shaped, single-walled 304L stainless steel tank. The top and bottom are torospherical shell plates and each tank stand on a iron (430B) conic skirt. One tank (Tank1) has been equipped with a Nitrogen sparging system to be used to remove Radon and Oxigen from the solvent via Nitrogen stripping.

Each tank has 5 flanged nozzles on top and 2 (3 for Tank1) on bottom for the connections with the general fluid piping and one flanged man-hole (800 cm diameter) to allow any eventual work inside. The tank technical characteristics are listed in table 1.

Total volume (1 tank)	113.79 m^3
Total surface (1 tank)	138.19 m^2
Internal Diameter	3.688 m
Height cylindrical part	9.430 m
Total height	11.8 m
Top and Bottom Thickness	10. mm
Cylinder thickness	6. mm
Weight (empty)	6.509 $10^3 Kg$
Weight (full of water)	120.3 $10^3 Kg$
Assumed Density	7.85 gr/cm^3

Table 1: Technical characteristics of each tank.

The technical design had to take into account the limited Hall-C available surface and the quite stringent eathquake safety requirements: $0.3g$ had to be considered both for vertical and horizontal

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acceleration, based on a study done by Ing. Ezio Faccioli [1] according to Eurocodice 8 (EC8).

The structural calculations have been made by Finzi Associati - Milano, and the technical designs have been made by INFN - Milan and by WTS Co.

In order to prevent any uplift the four tanks stand on a unique concrete foundation and each tank is tight to it throught 32 M20 Fe 510B girts.

Since the PC is a flammable liquid, for safety reasons the tanks have to be sorrounded by a 4m height containment concrete wall.

3 Quality Requirements

The main requirement for these storage tanks is that they do not contaminate the PC which will be stored in them for some months. This implies:

• low intrinsic radioactivity of all the components, which required an intrinsic Stainless Steel U content lower than $10^{-9} g/g$ before and after machining and to use Th free welding electrodes;

• low induced radioactivity, due for example to implantation of radioactive daughters of the Rn chain on the internal surface or to poor tank air tightness; this required to keep the contact between the internal surface and any cleaning liquid and air to a minimum and to require, for each tank, a total leaking rate below $10^{-5} atm\ cm^3/s^2$ for a 90% N 10% He mixture;

• very clean internal surface, which means very smooth internal surface, with R_a[3] $< 0.6\mu$m, to avoid, or minimize, any possible particulate deposit; in order to acheive this value the internal surface had to be electropolished, with the additional advantage of removing eventual surface steel contamination occurred during construction, by removing a $\approx$ 20-30 μm thin layer.

4 Construction and Cleaning Procedures

The stainless steel was bought from AVESTA, Sweden, in coils of 6 and 10 mm thickness for the shell course and the top and bottoms respectively. The coils were hot rolled sheet, with a 1D finish, wich corresponds to a surface roughness of R_a= 2.5-4 μm.

Since we required R_a to be less than 0.6 μm, and this value could not be reached via electropolishing starting from the coils as they were, they needed to be grinded to reach an intermediate R_a value of $\approx$ 0.8 μm. The 6mm coils were then sent to a Company [4] to be grinded, before reaching WTS. Since the first grinding did not satisfy our requirements, it had to be repeated.

This procedure could not be followed for the 10mm coils since any previous gringing would have been subsenquetly destroyed by their machining. These 10mm plates were then first welded together then mechanically molded in the top and bottom torospherical shape. They were then manually grinded at WTS and were then electropolished[5].

The 6mm coils were then welded together to form the shell and then they were welded to the top and bottom parts.

To check the mechanical robustness X-ray, dye penetrant and hydraulic tests have been performed at WTS, as will be briefly explained below.

The lateral internal surface of each tank was then electropolished[5], having the tank rotating around its vertical axis. A surface layer of $\approx$20-30 μm has been removed with this process.

Before the elctropolishing all the surfaces have been de-greased using C_2HCl_3. After electropolishing they were cleaned with ECOCLEAN 2000 [6] and thoroughly washed with high pressure ($\approx$

[1] Professor of Earthquake Engineering, Politecnico di Milano, December 1997

[2] this was the original requirement

[3] where R_a is defined as the arithmetic average of the absolute values of the surface distances (along the surface vertical) from the average surface line, calculated with the least square method

[4] CLA, Sulbiate (Milano), Italy

[5] at WTS, by DELMET co., Gorgonzola, Italy

[6] a DELMET product, a detergent made with phosforic acid and nitric acid

100-120 Atm, using a hand lance, hold by a man inside the tank) tap water first then with demineralized water till there were no visible (by my eyes) particulate on a white cloth, and the *ph* of the water was the same at the input and output of the tank.

Each tank was then dried by blowing N_2 inside it, and was then filled with a N_2 and He mixture (90% and 10%) to perform the He leak test (see below). The tanks were then flushed again with N_2 then stored at WTS, shipped and installed in Gran Sasso with N_2 at $40 mbar$ inside.

5 Quality Tests

5.1 Stainless Steel Tests

The tests on the Stainless Steel concerned the verification of its chemical composition and its contamination by radioactive components.

• The chemical composition was verified at the University of Genova, Chemistry Department[7], for the Fe, Cr and Ni content and by the Centro Sviluppo Materiali S.P.A. (Rome) for the C content[8]. The results are in good agreement with the AVESTA values, and with the Stainless Steel 304L official content values: they are reported in table 2.

	Cr	Ni	C	Mn	Si	Fe
6mm AVESTA values	18.66	9.16	0.019	1.46	0.45	70.01
6 mm measured	19.27	10.05	0.021			70.68
10mm AVESTA values	18.76	9.14	0.024	1.48	0.49	69.93
10 mm measured	19.66	10.06	0.023			70.29
304L off.values	19.0	10.0	< 0.03	< 2.0	< 1.0	> 68

Table 2: Chemical composition of the stainless steel (in %) as stated by AVESTA and measured by us; the official 304L values are reported in the last line for reference.

• The intrinsic radioactivity of samples taken from these coils has been measured using solid state Ge detectors in Gran Sasso and in Heidelberg[9].

Samples have been measured as they arrived at WTS (A), after the grinding (G) and after the electropolishing (GE) : the sample names, masses and measurement time interval are listed in table 3, the results of these tests are summarized in table 4 and 5.

name	mass $[g]$	measurem. time $[h]$
10A	393.1	271
10B	344.6	168
6A1	237.1	304
6G2	91	140
6GE2	8.1	169
6G3	91.1	170
6GE3	90.7	141

Table 3: Radioactive contaminant test: sample names, their mass and measurement time interval; 10 or 6 refere to the sample thickness (in mm); A = raw sample from AVESTA; B = Bombato (for top and bottoms); G = Grinded sample; GE = Grinded then Electropolished; 1, 2 and 3 refere to three different samples.

[7] by Prof. M. R. Pinasco

[8] according to ASTM E 1019-94

[9] many thanks to Cristina Arpesella, Matthias Leubenstein, Aldo Ianni in Gran Sasso; Gerd Heusser and Hermino Neder in Heidelberg

name	Th-232	U-238	K-40	Co-60	Cs-137
10A	$< 3\ mBq/kg$ $< 7.4\ 10^{-10} g/g$	4.2 ± 1.9 $3.4 \pm 1.5\ 10^{-10}$	< 18.6 $< 7.21\ 10^{-11}$	9 ± 1 $2.16 \pm 0.24\ 10^{-19}$	< 1 $3.13\ 10^{-19}$
10B	< 6.75		< 42.95	48.98	< 2.14
6A1	3.8 ± 2.6 $9.4 \pm 6.4\ 10^{-10}$	17.0 ± 2.5 $1.4 \pm 0.2\ 10^{-9}$	< 18.6	11.9 ± 1.8 $2.86 \pm 0.43\ 10^{-19}$	< 0.6 $1.88\ 10^{-19}$
6G2	260 ± 17 $6.43 \pm 0.44\ 10^{-8}$			24.28 ± 6.34 $5.84 \pm 1.52\ 10^{-19}$	
6GE2	< 126 $< 3.12\ 10^{-8}$			< 60.8 $1.46\ 10^{-18}$	< 68.51 $2.14\ 10^{-17}$
6G3	250 ± 35 $6.18 \pm 0.87\ 10^{-8}$	29 ± 12 $2.34 \pm 0.96\ 10^{-9}$	< 52 $< 2\ 10^{-10}$	25 ± 6 $6 \pm 1.4\ 10^{-19}$	
6GE3	< 15 $< 3.7\ 10^{-9}$	< 9 $< 7.26\ 10^{-10}$	< 14 $< 5.4\ 10^{-11}$	28 ± 6 $6.7 \pm 1.4\ 10^{-19}$	

Table 4: Radioactive contaminant test results: units are in mBq/kg, as measured, in first (or single) lines; in g/g in second lines.

As can be seen, the final values of the used material (samples 6GE2 and 6GE3) are within the accepted limits.

Some Be^7 contamination was present in the 6A sample, that is in one of the raw (from AVESTA) 6mm coil, but went away after grinding: this means that it was a very superficial contamination.

On the contrary, some "new" Th contamination was found after the grinding (see samples 6G2 and 6G3) caused by a Th contaminated sand paper used for the grinding: we analized the used sand paper and we found $33.3 \pm 0.3 Bq/kg$ for $^{232}\ Th$ and $47.7\ 10^2 \pm 2.7 Bq/kg$ for $^{238}\ U$. As can be seen, this contamination has been completely removed by the electropolishing.

name	Others
10B	$^{214}Bi :< 4.47$
6A1	$^{7}Be : 486 \pm 45$
6G2	$^{192}Ir : 110 \pm 16$ $^{134}Cs :\approx 10$ $^{137}Cs :\approx 5$
6GE2	$^{192}Ir : 110 \pm 30$ $^{214}Bi :< 176.6$
6G3	$^{192}Ir : 114 \pm 13$ $^{134}Cs :< 2$ $^{132}Cs :< 3$
6GE3	$^{192}Ir : 114 \pm 12$ $^{134}Cs :< 2$ $^{132}Cs :< 3$

Table 5: Radioactive contaminant test results: units are in mBq/kg; naming is as in previous tables.

5.2 Helium Leak Test

Since the intrinsic radioactivity of the used material was satisfactory, care had to be taken to avoid any possible contamination from the outside, once the tanks were constructed. We required a single leaking rate $< 10^{-6} atm\ cm^3/s$ (equivalent to $< 10^{-6} mbar\ l/s$) and a total leaking rate below $10^{-5} atm\ cm^3/s$ for a 90% N and 10% He gas mixture inside each tank, at 1.2 *bar* absolute pressure.

For the nozzle and the man-hole flanges we decided to use metalic Helicoflex[10] O-rings; these are flat metalic O-ring with a double seal: nichel seal on the inside and Viton seal on the outside. The He tightness has been measured for both seals.

For the He leaking test only the final man-hole Helicoflex has been put in place [11], while temporary regular rubber O-ring were placed on the other flanges.

To guarantee perfect air-tightness of the rings, a flange surface roughness $< 1.2\mu$m was required; all flanges surfaces have been tested and whenever necessary the flanges were smoothed again.

Two leak tests have been done for each tank: for the full tank and for the man-hole Helicoflex, in sequence, using for both tests the same 90% N and 10% He gas mixture.

• Leak test of the full tank: weldings and flanges.

This measurements were done by OMECO S.R.L.[12] at WTS[13].

Each tank has 6 horizontal weldings around it (each welding length= circumference), 5 vertical weldings (total length = tank height) and 2 weldings on the top and 2 on the bottom, for a total of 15 welding lines. The so called "accumulation" method was used to test the welding lines: it consists in sealing each of them by a plastic foil ($\approx$ 30 cm wide) and, after a proper "accumulation" time, determined with a calibration leak, which was of 2 hours for us, the inside He value was measured. This measure was taken every 40cm (value, choosen after measurements with the calibration leak) and was quite uniform along each entire line. The maximum values, for each tank, among the 15 values are listed in table 6 (after background subtraction and proper calibration):

Tank	max value $[mbar\ l/s]$
TK1	$5\ 10^{-8}$
TK2	$2.5\ 10^{-8}$
TK3	$2.5\ 10^{-8}$
TK4	$7.6\ 10^{-8}$

Table 6: Maximum values obtained for each tank, after background subraction and proper calibration.

• Leak test of the man-hole Helicoflex.

These measurements were done by Richard Ford using the Princeton Helium leak detector [14], in parallel with the full tank Helium leak test. The values found were quite similar for Tanks 2, 3 and 4 and were between 2 10^{-6} and 7 10^{-7} $mbar\ l/s$. For Tank1 the Helicoflex ring had to be replaced and found to have a small (few mm long) iron filament on the Viton seal. After the replacement a low vacuum value was still obtained and even applying the maximum allowed torque value with the wrench we could not go below 4 10^{-6} $mbar\ l/s$.

6 Mechanical Construction Tests

Other more standard tests have been performed to check the tank mechanics, (like the X-ray test and the hydraulic and penetrant dye tests) and the external shell passivation. Only this last test was done by us, while WTS did the other ones.

6.1 Surface roughness measurements

It is mandatory to have a very smooth internal surface to easily remove any dust and particulate contamination. Since the original coil surface was not smooth enough, it was necessary, as said

[10] by CEFILAC, Saint Etienne, France
[11] by WTS people, following the procedures given by Cefilac
[12] Monza, Italy
[13] using a LEYBOLD UL 200 helium sniffer
[14] by BALZER

before, to grind it and electropolish it to reach the wanted value ($R_a < 0.6\mu$m), with

$$R_a = \frac{1}{N}\sum_{i=1}^{N} |P_i - A_{verage}|$$

P_i being the measured vertical surface measured values and A is the average line, calculated from the measured N values, filtered using a low-frequency cutoff, using the least square fit.

All the internal surfaces have been measured using a profilometer[15]. Since the R_a values after grinding were not within our limits, all the raw coils had to be reprocessed, as had to be re-polished the Tank1 bottom.

Finally, the wanted R_a values were reached almost everywhere, with few exceptions here and there where isolated scratches were found, as is probably unavoidable for such big tanks, with the measured maximum values listed in table 7 and values up to $1.5\mu m$ maximum on welding lines.

tank	internal surface max R_a $[\mu m]$
TK1	< 1.1
TK2	< 0.6
TK3	< 1.1
TK4	< 1.4

Table 7: Maximum R_a values for each tank.

6.2 Flange roughness measurements

The Helicoflex O-ring, used to have the maximum air tightness, required the flange flat surface to have $R_a < 1.2\mu$m: these values were checked, the flanges were worked again when the requirement was not fulfilled (roughly 1/3 of the times) and finally all the flanges were within the limit. So far the Helicoflex O-rings are placed only on the man-holes, while temporary rubber O-rings are on the other flanges, and they will be replaced during the Tank connection with the general piping system.

6.3 X-ray tests

We required an X-ray examination of 100% of the welding joints. Acceptance criteria were the Class B of the European Standard EN25817. These tests were done using an X-ray tube of 290-300 KV[16] put in the inside of each tank, which irradiates at 360^o on a plane perpendicular to the the tank vertical axis. In this way all the films are exposed at the same time.

The films were placed on the outside surface of the Tanks: thirty on each of the 6 horizontal circunferential welds, seven on top of each of the 5 longitudinal welds and 23 (+23) on the torospherical top (and botom) welds. Two films were put on top of the man-hole weld.

The pictures were then carefully examined and the faulty weldings were repaired.

6.4 Penetrant Dye and Hydraulic Tests

The penetrant dye test was done before the X-ray test while the Hydraulic test was done just before the electropolishing. The water pressure read on the tank bottom was $1.5 Kg/cm^2$.

[15] model SJ-201 by Mitutoyo; the instrument settings, used also for the instrument calibration, were: cutoff length = 0.8mm, sample legth = (0.8mm X 5), number of data (P) taken in a sample length=(1600 X 5).
[16] by Gilardoni S.p.A., Mandello del Lario, Lecco, Italy

6.5 Passivation of the External Surface Test

To be sure no superficial inclusion of iron particles remained on the external wall, C.Salvo[17] did this test according to ASTM A 380-88 parts F-21: it consists in spraying the greenish liquid solution ($HNO_3 + FE(cn)_6FE_3$) on the external surface and see if it turns blue within few minutes. The external surface had been already passivated by WTS, and the results were negative, exept for few millimeter wide spots here and there and some tank surface patch which was not passivated at all, areas which were then passivated.

7 Open Problems

• Radon daughters implantation (from air):

As can be seen from table 8 only for Tank1 and Tank4 the proper final cleaning and N filling was done right after the electropolishing, with the minimum possible exposition to air; for Tank2 and Tank3 there was a time interval of 15 and 25 days respectively. The tops and bottoms time interval was of three months but $\approx 1/3$ of their surface was electropolished again during the electropolishing of the lateral tank surface.

Many calculations have been done and already circulated by e-mail about the real danger of this contamination, but no real measurement has been done yet and no clear conclusion has been reached: we plan to do some measurement early next year and decide then which eventual further cleaning is necessary.

tank	top and bottom electropolishing	shell course electropolishing	final cleaning and N filling
TK1	march 2	june 28	june 28
TK2	march 2	june 11	june 22
TK3	march 2	june 4	june 29
TK4	march 2	june 23	june 24

Table 8: Dates when the some processes have been done (in 1999).

• dust and particulate deposit:

Because of the tank internal surface exposure, after the electropolishing, in the dirty air of WTS, Tank2 and Tank3 were found very dirty[18] before the final cleaning.

They were then first washed again with high pressure water (at 100-120 Atm, using a hand lance), then checked again. Tank2 passed our visual examination (the usual "white cloth test") while Tank3 was still dirty, with scaling on the internal surface not completely removed by the high pressure water.

Another 2 hour ECOCLEAN cleaning was then necessary to descale it completely. According to the ECOCLEAN etching rate measurements [19], this 2 hour time should have etched $\approx 0.5\ \mu m$. Of course my "visual" check is limited (I can reach 80-100μm), and more quantitative tests on the inside tank dust contamination are planned for the beginning of next year, whenever the final ladder and catwalk will be in place.

[17] with the help of L.Perasso

[18] on the contrary I found a note of mine saying "TK1 is very clean!"

[19] done by C.Salvo and L.Perasso in Prof. Vigo Lab in the Chemist Department of the University of Genova