Materials Testing and Certification for the KamLAND 4π Full-Volume Calibration System

WORK IN PROGRESS
Updated – February 1, 2005

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Introduction

According to the general guidelines outlined by the calibration committee and the calibration coordinator [1,2] the 4π system needs to be certified for compatibility and cleanliness before it is installed, commissioned, and used as a calibration source deployment system. The relevant documents of the KamLAND calibration group describing the existing certification requirements for the KamLAND 4π system as well as other KamLAND calibration systems and sources are attached in the Appendix of this note.

The quantitative question of what constitutes acceptable background levels and sufficiently clean parts is not addressed in this note and will require further discussion between the calibration committee and the collaboration. This docoument outlines the procedure and steps taken to date to certify the materials compatibility and cleanliness of the

Status of Materials Testing and Certification

1. Completed transparency measurements on 4pi cable samples.
2. Completed radioactivity measurements on soak samples of 4pi bulk fabrication materials.
3. Completed dry-counting of various 4pi components.

Outstanding Certification

1. Analysis of counting results of 4pi cable soak samples. *(Mauger, Keefer)*
2. Transparency measurement of 4pi cable with and with out red tracer. *(Nakamura)*
3. LS soak test of final 4pi parts *(Heeger)*
4. Weak nitric acid soak of final 4pi parts *(Heeger)*
5. Low-background count of items #3 and #4.

II. KamLAND 4pi System Hardware

The KamLAND 4π system consists of an extension to the existing glovebox including a new deployment system and the calibration pole that will be immersed into the inner liquid scintillator of the detector. The most stringent cleanliness criteria apply to the parts that will come in contact with the liquid scintillator in the inner volume of the detector. These are the control cables and the calibration pole.

Table 1: Components of the glovebox extension and the 4pi deployment system.

Glovebox and Deployment System	*Quantity*	*Material*
Glovebox extension frame	1	Stainless
Extension windows	4	Lucite
Glove ports and gloves	1	Stainless and viton
Motor plate	1	Stainless
Cable spools	2	Stainless
Encoders	2	Stainless vapor-tight enclosures
Slip rings	2	Stainless vapor-tight enclosures
Pin block lock system	1	Stainless
Cable guide for pin block	1	Teflon
Pulley system	1	Stainless
Limit switch	1	Stainless with ??? enclosure
Pole rack	1	Stainless
Torque wrench	1	Stainless
Motor and gears	2	Stainless vapor-tight enclosures
Cabling and connectors	7	Teflon, Connectors with approved materials See Ref. [5]
Emergency lockout clamp	1	Nylon and stainless screws

Table 2: Components of the calibration pole that will be immersed in the liquid scintillator.

Part #	*In-Situ Calibration Pole*	*Quantity*	*Material*	*Total Mass*	*Surface Area*
1	Control cable 1 (~18m?)	1	Nylon, Teflon, Stainless	22.7 g per ft	
2	Cable markings	?	Nylon?		
3	Cable attachment for cable 1	1	Titanium, Stainless	170.1 g	
4	Instrumentation unit at cable end	1	Lucite enclosure Nylon cap Stainless electrical pins Stainless pressure sensor Acrylic cement		
5	Source mount	1	Titanium	129.2 g	
6	Control cable 2 (~18m?)	1	Nylon,	22.7 g per	

			Teflon, Stainless	ft	
7	Cable attachment for cable 2	1	Titanium, Stainless	192.6 g	
8	Standard pole segments	8	Titanium, Stainless	626.8 g	
9	Weighted pole segment	1	Titanium, stainless		
10	Pivot block	1	Stainless, nylon		
11	Source protection cage	1	Stainless		
12	Instrumentation unit at pivot block and in pole	2	Lucite enclosure Nylon cap Stainless electrical pins Stainless pressure sensor Acrylic cement		
13	Calibration sources in pole	5	Stainless		
14	Calibration source at far end	1	Stainless		

The materials testing and certification plan outlined in this note is designed to address the issue of materials compatibility and cleanliness for these items. All hardware in the glovebox and its extension is designed and built according to the general guidelines outlined in the Appendix of this document.

III. Exposure of 4pi System to KamLAND

We expect the calibration pole and cables of the 4pi system to be exposed to the liquid scintillator for a maximum of 1 week continuous deployment. This is equivalent to about 168hrs or 604800 sec.

Once the system is installed one of the 4pi cables will be used to perform the regular z-axis calibration, with a deployment period of ~ 1hr every 2 weeks or 6-12 hrs during every global calibration every 3-6 months.

Table 3: Typical exposure parameters for calibrations with the new 4pi system.

Type of Calibration	*Max Deployment Period*	*Parts to be Deployed*
4pi calibration	1 week or 168hrs	All parts listed in Table 2.
z-axis calibration	1-12 hrs	Only parts #1-5 in Table 2

IV. Materials Compatibility and Cleanliness Test Program

All materials used in the construction of the 4pi deployment system and the calibration pole were chosen to be compatible with the liquid scintillator. The construction material for the 4pi system and the calibration pole are listed in Tables 1 and 2. The materials compatibility and testing program ensures that these items and the materials used to build them

A. do not affect the optical property of the liquid scintillator
B. do not leach any radioactive contaminants into the liquid scintillator
C. do not emanate an unacceptable amount of Radon into the liquid scintillator

Issues A-C will be addressed through

- spectrometer measurements of liquid scintillator soak samples of the materials
- low-background counting of weak nitric acid soak samples
- low-background counting of liquid scintillator soak samples

In addition some of the parts will be counted dry to provide an early verification and cross-check of the cleanliness of these parts. The dry-count will also give us information on the event rate and activity we expect to see from each part of the system when it is deployed in the KamLAND detector.

General guidelines followed in the 4pi material certification program are:

1. All parts in the glovebox are made out of LS-compatible materials, or shielded appropriately against the LS vapor The individual parts in the glove do not have to be soak tested as they do not come into contact with the scintillator fluid.

2. All parts that will be introduced into KamLAND and immersed into the liquid scintillator will undergo a soak test in liquid scintillator and in weak nitric acid. The soak samples will be counted in one of the low-background counting facilities (Mozumi, University of Alabama, or LBNL) for traces of residual activity.

3. Additional transparency measurements will be performed on soak samples of the 4pi cable. It is made out of materials that are known to be compatible with LS (nylon, stainless, teflon) but its complexity and novel construction make it desirable to perform additional testing.

The following materials are considered “approved materials” for use inside the glovebox:

1. Stainless Steel
2. Teflon
3. Lucite
4. Nylon

The materials testing and certification will make use of the various counting facilities. At LBNL we will (in the following order)

1. Perform the dry count of selected UHV-cleaned parts for an early evaluation.
2. Build a container for a LS and weak nitric acid soak of all parts that will be immersed in the liquid scintillator and perform subsequent counting of a liquid soak sample to check for leaching effects. The parts will be soaked for about 1 week. This test samples all finished parts that will be immersed into the inner detector. After this test the parts will be UHV cleaned according to the procedures in [4].

At Mozumi the following tests can be performed (depending on the availability of personnel):

1. Transparency tests of long-term liquid soak samplesto check for materials compatibility with liquid scintillator.
2. Low-background counting of liquid scintillator soak samples.

Before any of the 4π parts are tested for LS compatibility and counted they will undergo the UHV cleaning procedure described in Ref. [4]. This procedure will also be used for the final cleaning of the 4π parts before they are shipped to KamLAND.

IV. Materials Testing & Results

This section summarizes the preparation of the 4pi soak samples and the result obtained to date.

Preparation of Long-Term Soak Samples of 4pi Construction Materials
(M. Galloway, E. Yakoushev)

The following soak samples were produced by M. Galloway and E. Yakoushev in early 2004. These samples are the basis of the counting analysis performed by G. Keefer. See below.

Table: Soak samples of 4pi fabrication materials.

Material	*Part Description/Potential Use*	*Start Date of Soak Test*
delrin	detector misc	January 2004
Titanium from coupling	4pi pole	January 2004
Autocoat epoxy	detector misc	January 2004
PTFE 30 AWG wire, black	4pi cable-specific sample	January 2004
Nylon monofilament	4pi cable-specific sample	January 2004
302/304 SS cable	4pi cable-specific sample	January 2004
Connectors	4pi	January 2004

Preparation of Long-Term Soak Samples of the 4pi Cable (B. Berger, K. Nakamura)

Kengo and Bruce started some soak samples on August 16, 2004. We soaked the two shorter (~6") cable samples, one with and one without the red wire. We did not soak the longer (12"?) cable because it is too large to fit into the containers we have. We started three different soaks of the black wires, each with three wires apiece. This way we can stop them at different times. We also started a "blank", just a container full of LS, for reference.

Outline of procedure:

For each sample we
1) Weigh empty container
2) Put some LS into container to rinse
3) Rinse container by closing and shaking
4) Pour out rinse LS
5) Fill container with LS
6) Weigh container
7) Add sample
8) Weigh container with sample and LS
9) Note date, time, results of weighing, sample description on container label

Details:

We filled a clean container of LS from the reserve tank. This is the same procedure used to extract LS for all other soaks. Picture of Kengo filling the LS container:
http://www.awa.tohoku.ac.jp/~beberger/mine-camera-040816/P1010201.JPG

The available soak containers were small "PE" (Kengo thinks this is polyethalene) containers. They are smaller than those used for the previous 4pi soak samples. They are identical to the containers Tommy used for counting the 4pi soak liquid. We transferred LS from the large container into the soak bottles using a plastic squeeze bulb. Here's a picture of Kengo doing it:
http://www.awa.tohoku.ac.jp/~beberger/mine-camera-040816/P1010204.JPG
The bulb is *not* LS compatible, but the standard procedure is to use it anyways because the exposure time to LS is so short. (Kengo did not know the exact bulb material, but says Ogawa will know if we need to find out.)

We had a little trouble getting accurate readings from the scale. It reads to 0.0001g, but it tends to drift quickly, so the measurement errors are on the 0.1-0.2g level We measured two other empty containers as well to get a sense of the mass variation, as we didn't weigh the first two containers before filling them. Those masses were 22.8 and 23.0 g.

More pictures: Soak samples on the shelf:
http://www.awa.tohoku.ac.jp/~beberger/mine-camera-040816/P1010205.JPG

Bottles from the previous soaks. Large bottles are those used forsoaking. Small bottles in plastic wrap were the soak liquid counted in the GE detector:
http://www.awa.tohoku.ac.jp/~beberger/mine-camera-040816/P1010203.JPG

Samples previously soaked:
http://www.awa.tohoku.ac.jp/~beberger/mine-camera-040816/P1010202.JPG

Table: 4pi cable soak samples

Sample No.	Description	Date/Time	Weight Of Container	Weight w/ LS	Weight w/LS+Sample
1	4pi cable w/ red wire	8/16/2004 ~12:10 pm	(not measured)	88.5 g	99.8 g
2	4pi cable w/o red wire	8/16/2004 ~12:25 pm	(not measured)	84.1 g	93.9 g
3	3 black teflon wires	8/16/2004 ~12:35 pm	23.6 g	77.6 g	79.1 g 101.9 g (extra LS added as sample was not fully submerged)
4	3 black Teflon wires	8/16/2004 ~12:45 pm	21.8 g	81.8 g	83.3 g
5	3 black Teflon wires	8/16/2004 ~12:50 pm	23.2 g	95.3 g	96.8 g
6	(no sample)	8/16/2004 ~12:55 pm	23.5 g	90.6 g	N/A

Radioactivity Measurements of Soak Samples at KamLAND (G. Keefer)

Samples

The samples used in this test were prepared by M. Galloway and E. Yakoushev. They represent bulk materials and sample parts of materials used for the construction of the 4pi system. See Section 1.

Outline of Analysis Method

In the absence of any visible peaks in the majority of the spectra the following analysis procedure was adopted:

1. Summed over channels and subtracted background.
2. Peak width was calculated by using source calibration free fit gauss +/-1σ.
3. All errors are statistical.

Procedure for Calculating the Rates

The results shown here were calculated using the following method. Let N be the number of counts in the channels corresponding to 3 sigma around the mean energy of the gamma line. Then we take a region to the left and to the right of this region corresponding to (3 sigma)/2 channels respectively. The number of events corresponding to the area under the peak is then N' = N - (NL+NR).

Due to the number of visible peaks being small in each of the spectra, regions of interest were defined using the energy calibration of the runs and the gamma peaks corresponding to the isotopes of interest. The following gamma lines were used in the evaluation of the associated activities

Table: Gamama lines used in the evaluation of activities.

Isotope	*Energies*		
^{210}Pb	46.539 keV		
^{40}K	1460.83 keV		
^{208}Tl	2614.53 keV	583.191 keV	
^{212}Pb	238.63 keV		
^{214}Pb	351.932 keV	295.224 keV	
^{226}Ra	186.211 keV		
^{214}Bi	609.312 keV	1120.287 keV	1764.494 keV
^{228}Ac	911.204 keV	968.971 keV	338.3220 keV

The energy calibration provided by Christopher was fit for the log E and thus is not sufficient for actual activity calculations on energies below 200 keV. The 238.63 keV peak could be above this threshold.

One may also question the use of the 238.63 keV line in the manner described above for analysis due to there being a gamma line in the 214Pb spectrum at 241.981 keV. These two peaks would be resolvable on the ge detector in the mine. The branching ratio for these two lines is 43.3 % and 7.5 % respectively. The sigma used at this energy is 1 keV and thus a 3 sigma region around the mean ends at 240.13 keV. The background interval on this side of the peak is 1.5 keV and ends at 241.63 keV. This does encompass part of the peak region for the 214Pb region. However, the difference in branching ratios makes the total number of events that would contribute to the

background in this region negligible at the rates observed in the higher branching gamma lines for 214Pb.

Note on ^{214}Pb 351.932 keV peak in Stainless Steel Sample

To determine the count rate in the 214Pb peak in stainless steel sample we performed three calculations of the rate. The procedure and results are shown in the table below

.

Table: Procedure for determining counts in the 214Pb peak of the stainless steel sample.

Method	*Result*
Free fit of gauss + linear background.	2.37 +/- 2.02 counts/day
Free fit of Gauss + linear background after subtracting scaled blank spectrum	4.61 +/- 1.26 counts/day
Sum channels and subtracted background	1.32 +/- 1.68 counts/day

Results & Discussion

In evaluating all of the data taken to date there are a few things that still need to be performed.

1. Measure the mass of the blank LS sample
2. Redo the delrin sample and a background run
3. Efficiency measurement of low energy region

The reason why the current runs for the delrin and background are invalid is due to power outages. The runs were performed after power shutdowns in the mine or unexpected outages in which the HV had to be shut down and then taken back up. These procedures more than not cause a shift in the spectrum do to fluctuations in the HV. Since there are no observable peaks in the spectrum there is no way to calibrate the individual spectrums. The procedure used in evaluating the activity relies heavily on a known calibration of the ge detector and thus one can not assume that the spectrums are equivalent. A few keV shift in the spectrum would result in larger background spectrums being subtracted from the shifted peak region, resulting in lower and often times more negative rate results.

If it seems desirable to quantify the rates in terms of an absolute activity for energies below 200keV then a calibration of the detector efficiency for this geometry should be performed. There is also no systematic error known and this could be calculated fairly accurately with a calibration source

A tabulated summary of all results is given below.

The mass off the blank soak sample needs to be determined.

It may not be necessary to re-do the Delrin measurement as this material is no longer used in any of the 4pi system parts.

Table: Summary of radioactivity measurements performed by University of Alabama at Mozumi.

Sample	**LS mass[g]**	**Sample mass[g]**	**^{210}Pb [cpd]**	**^{226}Ra [cpd]**	**^{40}K [mBq]**	**^{208}Tl [mBq]**	**^{212}Pb [mBq]**	**^{214}Pb [mBq]**	**^{214}Bi [mBq]**	**^{228}Ac [mBq]**
Nylon mono-filament cable part	71.129	0.0722	3.23±1.55	0.934±1.36	2.14±1.13	0.21±0.17	0.19±0.13	0.47±0.39	0.39±0.10	0.37±0.90
Titanium	77.869	5.415	4.90±1.13	2.12±1.05	1.40±0.73	0.10±0.11	0.08±0.10	0.30±0.27	0.29±0.75	0.44±0.67
Stainless Steel Cable Part	76.4	3.7167	1.57±1.50	-0.50±1.43	4.10±1.26	0.11±0.16	0.18±0.14	0.88±0.43	0.29±1.06	0.08±0.94
Teflon Conductor	71.679	0.8051	1.55±1.56	0.95±1.61	2.39±1.08	0.21±0.18	0.11±0.13	0.50±0.46	1.34±1.32	0.61±0.93
Connector (motors, possible transducers	79.889	25.3037	0.89±1.47	2.10±1.47	1.12±1.03	0.23±0.20	0.22±0.14	0.47±0.39	0.56±1.15	0.31±0.87
Delrin	73.202	4.059	5.65±1.61	-0.53±1.45	1.92±1.07	0.24±0.17	0.03±0.14	0.44±0.21	0.63±1.19	0.19±0.87
Blank	------	------								
Background	------	------								

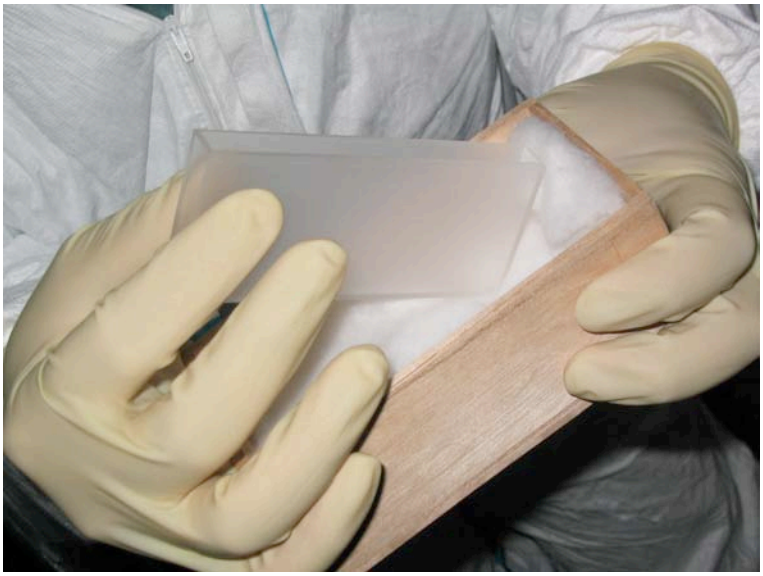
Transparency Measurement of 4pi Cable Soak Samples (K. Nakamura)

Apparatus & Method

The transparency measurements use a commercial spectrometer in the purification area. In each measurement the light absorption is compared between a blank sample and the test sample. The liquid samples are contained in quartz cells with an overall length of about 10cm. Each quarz cell has a volume of ~ 50ml. See picture. Before each measurement the quartz cells are thoroughly cleaned with acetone and alcohol and blow dried. The liquids are then transferred into the cells. Prior to the actual measurement a baseline measurement is taken. This measures the light absorption of both quartz cells filled with air. The "transparency" as obtained in this measurement is the ratio of absorptions measured in the cells compared to the baseline run. It defines the double ratio:

$$Transparency = \frac{T_{sample}/T_{blank}}{T_{cell1}/T_{cell2}}$$

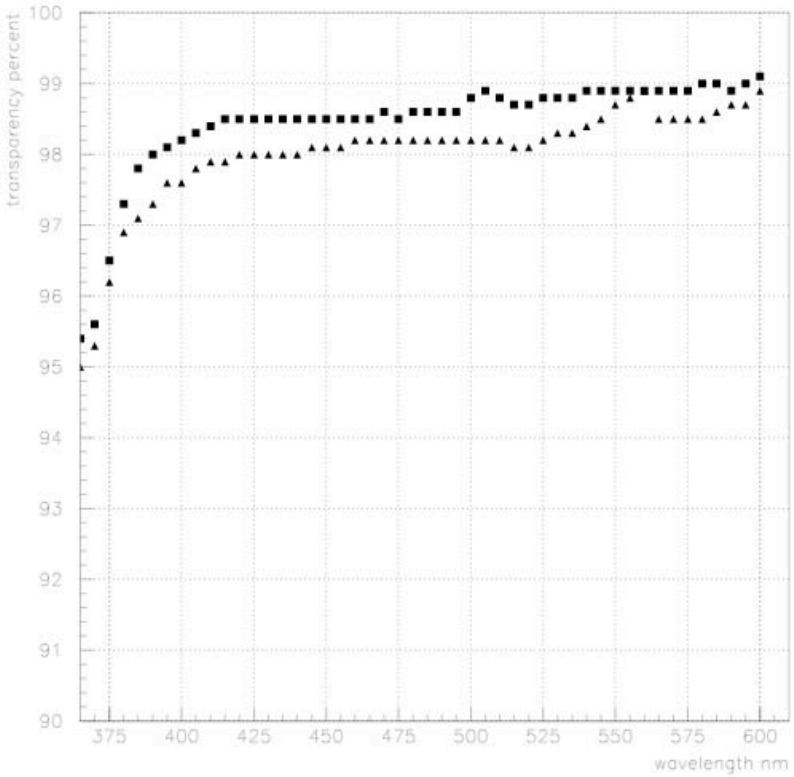
Figure: Quartz cell used in the transparency measurements with the on-site spectrometer.



Measurement 1- November 26, 2004

Measured one of the soak samples with the 4pi cable. The sample was made on August 16, 2004 with Bruce. The blank sample in this measurement is clean liquid scintillator in a teflon jar. This blank sample was probably probably prepared a few months ago, too. The measurement of the soak sample including the baseline measurement was performed twice. The time interval between the two measurements is about 30 min. The results are shown in the figure below.

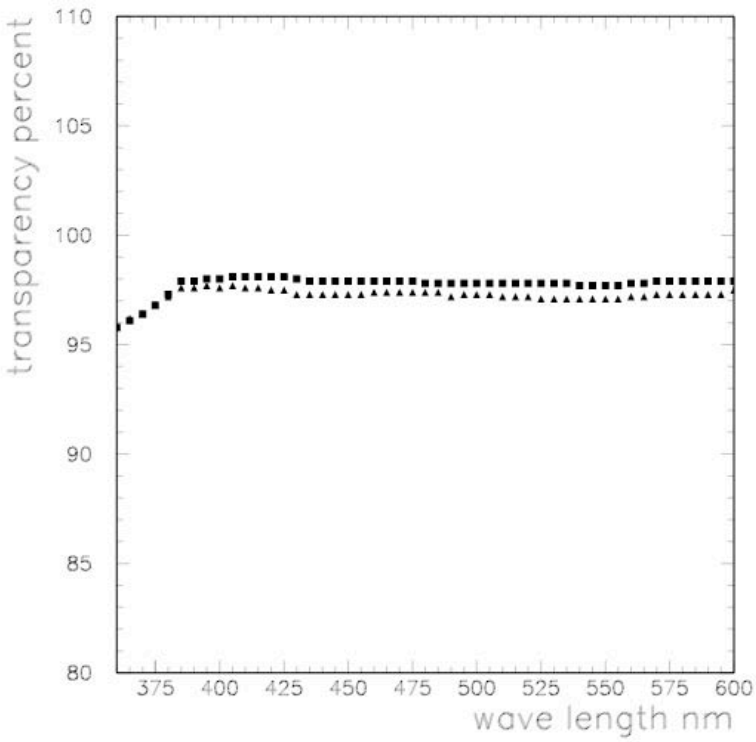
Figure: Two transparency measurements of one of the 4pi cable soak samples. The measurements were taken 30min apart. Their difference is a measure of the reproducibility of these measurements. There is an overall systematic uncertainty of ~2% on the absolute normalization of these curves, i.e. a transparency of 98% is considered to be consistent with 100% or no change in transparency.



Measurement 2 – December 24, 2004

Measurement of one of the 4pi cable soak samples without the red tracer. The soak sample of the cable with the red tracer still needs to be tested. During the measurements for this test it was noticed that the transparency can be more than 100% at some wave length. This is due to the fact that the double ratio of the sample measurement versus the baseline measurement is calculated. It is possible that the one blank sample changes its properties as it is opened for every measurement of any materials samples. Oxygen may react with the LS and cause a long-term change of properties. Both the blank sample and the cable soak sample used in this test were prepared on August 16, 2004.

Figure: Two transparency measurements of one of the 4pi cable soak samples. The measurements were taken 30min apart. Their difference is a measure of the reproducibility of these measurements. There is an overall systematic uncertainty of ~2% on the absolute normalization of these curves, i.e. a transparency of 98% is considered to be consistent with 100% or no change in transparency.



Results&Discussion

The absorption peak of the PPO in LS is at ~370 nm. The absolute value of the transparency for the wave length above 400 nm is less than 100 %. Taking into account the systematic uncertainty this measurement is consistent with 100% transparency. We find that the reproducibility of two measurements taken ~30 min apart is better than 1%. We make a conservative estimate of the effect of the cable in the soak sample from the transparency measurement at the absorption peak. It is found to be 96% at 360 nm. More precisely, the transparency at 360 nm of the soaked sample compared with the blank sample is 96 ± 2% (syst.) for a light path of 10 cm.

To understand the impact of the 4pi cable on the attenuation length in KamLAND we can construct a scaling argument with the following constants:

- Duration of soak: ~4 months.
- Duration of 4pi calibration: ~7 days.
- Surface of sample: 6" length x 1" width x 2 sides = 38.7 cm^2
- Surface of cable: 15 m x 1" width x 2 sides = 7620 cm^2
- LS volume of sample: 60g
- LS volume of KamLAND: 880 ton

The contamination effect of the 4pi cable during 4pi calibration in KamLAND will be

$$7/122 \times 7620/38.7 \times 0.060/ 880000 = 0.77 \times 10^{-6}$$

of the effect seen in the soak sample.

Low-Background Counting of 4pi Parts and Materials at LBNL (K. Heeger)

Al Smith from LBNL counted various parts of the 4pi system both bulk fabrication materials as well as finished 4pi parts. The following measurements were performed at LBNL or the low-background counting facility at Oroville.

Part	*Limit*	*Location*	*Date*
Instrumentation unit	Th <10 ppb. U < 7 ppb. weak evidence for K at 0.025 pct.	LBNL	Jan 30, 2005
Control cable	U-series <30 ppb Th-series <15 ppb Potassium <50 ppm	Oroville	May 10, 2004
Control cable without tracer	Flexible Cable, 81 grams File CI-24 694857 sec count U(early) ND U(late) 2.7(10) ppb Th(early) ND(6) ppb Th(late) ND(3) ppb K 25(4) ppb	Oroville	June 11, 2004
Titanium BTC parts	Tube/fittings, 129 grams File CI-25 345258 sec count U(early) 46(10) ppb U(late) ND(1) ppb Th(early) 16(6) ppb Th(late) 16(3) ppb K ND(3) ppb	Oroville	June 11, 2004
Source mount + cable attachments	Misc fittings, 450 grams File CI-27 689443 sec U(early) 19(3) ppb U(late) 1.0(1) ppb Th(early) 3.9(13) ppb Th(late) 5.7(8) ppb K ND(6) ppm	Oroville	June 11, 2004

Note:
- These limits are calculated from one-sigma SD's on the BKG peaks, where one-sigma is on the order of +-10%.
- Values in parentheses are single standard deviations on the listed values.
- In the Titanium samples, Uranium is present, but the major gamma-emitters (Ra-226 daughters) are essentially absent. There is no evidence for disequilibrium in the Th-series.

Radioassay Results from Titanium Tube and Coupling Samples

All three samples were counted together on
the largest detector (MERLIN) at the LBNL Facility.
The two samples of round stock were positioned on the
flat face of the detector endcap, while the pieces of
split tube (12 pc) were arranged around the cylindrical
sides of the endcap.

Sample weights:	Total	=	1074 grams
	Rnd #HC12362	=	254 grams
	Rnd #8607528	=	324 grams
	Tube pieces	=	496 grams

Data File: 17378
Count time = 176601 sec

U(early)	=	19.(1) ppb
U(late)	=	0.5(4) ppb
Th(early)	=	ND
Th(late)	=	8.(1) ppb
K	=	2.7(6) ppm

U(early) signifies the equivalent U-content of nuclides
earlier in the decay chain than Ra-226. U(late) signifies
the equivalent U-content of nuclides from Ra-226 through
the end of the chain.

Th(early) signifies the equivalent Th-content of nuclides
earlier in the decay chain than Th-228, with the following
qualifier. If refining of the metal occurred within about
6 months of the measurement, the late members would not
have grown in to a detectable level. Th(late) signifies
the equivalent Th-content of nuclides from Th-228 through
the end of the chain.

ND signifies no gamma-rays from these nuclides could be
detected. Numbers in parentheses() indicate the magnitude
of one-sigma uncertainty in the listed values. The one-
sigma numbers are derived from counting statistics alone.

V. Material Handling Procedures for 4pi Shipment and Installation

References

[1] J. Busenitz, H. Steiner
[2] E. Yakoushev
[3] A. Piepke, *private communication*, April 2004
[4] LBNL UHV cleaning procedure, January 2004
http://kmheeger.lbl.gov/kamland/4pi/materials/UHV_CLEANING_PROCEDURES.pdf
[5] A. Franck and F. Gray, "Cabling in the 4pi System", 2004

<u>Appendix A1</u> – Kamland Materials Certification Procedures

Kamland 4π ystem - Materials Certification Protocol
(HS Draft of 1/31/03 as modified by JB&HS on 2/6/03)

Policy

In order to avoid compromising the performance or operation of the KamLAND detector the following policy will govern all deployment-related tasks:

- No materials shall be submerged in the scintillator or put into the glove box without prior approval by the designated on-site representatives of the KamLAND collaboration. Until further notice these persons are G. Guillian and M. Koga.

- All materials used in the detector and in the glove box must be properly certified. There will be two classes of certification: A and B. Class A pertains to all materials to be submerged in the scintillator. Class B pertains to materials that will normally remain in the glove box**.** These certifications will be described in the next section of this document.

- A responsible person must be associated with every item to be deployed, and that person must enter a record of the certification procedure into the KamLAND e-log.
  Until further notice M. Galloway will be the 4π-system certification coordinator, and will be responsible for all 4π-system certifications.

- All certified materials must be re-certified if they are exposed to an unclean environment.

- Only certified operators are authorized to put or manipulate materials in the detector and the glove box.

- Only approved deployment procedures are to be used.

- The fire marshal must approve all procedures and operations involving potential fire or explosion hazards.

<u>Certification</u>

The purpose of the certification procedure is to insure that insertion of materials does not introduce lasting radioactive or chemical contaminants into the experiment, nor to endanger the mechanical integrity of the detector. The certification requirements are intended to be stricter for materials in direct contact with the scintillator (class A) than those exposed only to dilute vapors (class B). No hard and fast rule will cover every case. Common sense and safety consciousness should always be prime considerations.

<u>Class A Certification</u>

- Only materials on the pre-approved materials list should be submerged in the scintillator.

- Class A certification must include (but is not limited to) the following procedures:

(1) Approved cleaning. Whenever possible this should include a weak acid soak (at least one day), unless there is a danger that the material would be damaged by the acid.

(2) Scintillator soak for period of time comparable to or greater than time the material is expected to be submerged in the detector. Care must be taken to provide controls for counting and optical measurements.

(3) No detectable increase in post-soak scintillator/weak acid radioactivity

(4) No detectable changes in post-soak scintillator optical properties

(5) No significant increase in detectable chemical contaminants This step may not be necessary if material is already certified to be compatible with scintillator.

(6) All pressure sensitive items must pass a standard pressure test of at least 2 atmospheres before they are deployed. All seals must be free of leaks.

(7) All certified items shall be carefully packed, preferably hermetically, and assembled in a clean environment

(8) A full record of the certification procedure shall be entered into the e-log. This record shall include a statement by the responsible person that in his/her best judgement the certified item is safe for insertion.

- Every item to be inserted into the liquid must be receive class A certification except for identical copies of certain mechanical components (e.g., mast and boom sections, couplers, other hardware, etc). For these pieces a preparation procedure will be defined based on assay of samples (filings, swipes, etc.). All objects to be submerged must have undergone an approved cleaning procedure and have been checked for gross radioactivity prior to deployment.

- All individual parts should be marked permanently with an appropriate serial number (or other identifying label). That label should be used to identify items in the certification document.

Class B Certification

- Whenever possible only pre-approved materials should be used inside the glove box. Exceptions to this policy will be considered as needed, but only if the associated risk is deemed to be acceptable by the collaboration. Except in emergency situations a lead time of one week is required for all such requests. The heads of the calibration group, in close consultation with other members of the collaboration and especially relevant experts, will make the required decision(s).

- Except for radioactive calibration sources all materials to be put in the glove box (and associated environment) must be certified to be clean and free of gross radioactivity.

- In general no soak tests or low level radioactivity measurements need to be made for glovebox items that are not submerged.

- Glovebox items must be certified not to introduce light or gas leaks, nor to introduce a potential fire hazard.

- Class B certification of all glovebox items shall be recorded in the e-log.

The attached spread sheet, prepared by M. Galloway, spells out the various certification tasks in more detail.

Appendix A2 – General KamLAND Source Certification Requirements

KamLAND Certification Requirements

The following information must be provided about all materials to be inserted into the detector and glove box. (For a radioactive source, this information must be supplied and approval granted before the source can be brought into the dome area.) The method of deployment should be clearly specified. For each item the test procedure should be described in sufficient detail that judgements can be made about its effectiveness. In all cases you are asked to provide the nature of the test, the names of those making the test, time and place of the test, the test procedure(s), any references, and the results of the tests. Please indicate if others are responsible for certain parts of this procedure. You should point out any potential problems of which you are aware. Approval to deploy will not be considered without a written statement from the requester that he/she certifies that to the best of his/her knowledge it is safe to do so. This statement should be submitted (e.g. e-mailed) to the entire collaboration at least one week prior to the requested deployment. No deployment is to be made without prior approval by the designated representatives of the calibration group, at present G. Guilliam and M. Koga. They are authorized to deny or delay approval if in their opinion the procedures and certifications are not acceptable. They are encouraged to consult with other members of the collaboration (and elsewhere if necessary) to clarify any unresolved issues.

The written certification should contain:

(1) A brief description of the source: What is it? What is its purpose? How will it be used?

(2) What is the material composition and geometry? How was it fabricated? When?

(3) What is known about radiopurity? Describe all tests that have been done.

(4) What is known about compatibility with the scintillator? Describe all tests that have been done.

(5) Mechanical integrity and stability? Pressure tests?

(6) How will the source be deployed? Use of deployment systems other than the “standard” phase 0/1 systems must be described in detail. Describe what has been done to insure that the source will not fail in the detector

Comments:

Loose or untethered componenets inside the glove box should be avoided. It is important to remind users that there may be only restricted direct visual information available when the HVs to the PMTs are on, and the gate valves are open. A wide-angle CCD camera that can be used with IR light is foreseen for future installation inside the glove box. Another important requirement is that only persons who are "licensed" by Gene and/or Koga-san will be allowed to use the glove box, and then only for predefined operations.

<u>Appendix A3</u> - Email by on the Preparation of New Radioactive Sources

Date: Fri, 21 Nov 2003 19:34:43 -0600 (CST)
From: "Evgueni A. Iakouchev" <yakushev@bama.ua.edu>
Subject: Procedure of preparation of new radioactive sources to deployment will be changed
Sender: owner-kamland@awa.tohoku.ac.jp

Dear collaborators,
As you know now we have low background counting facilities on the site.

From now we will use it for additional checking of surface contamination of ANY new radioactive sources arriving on the site. Only sources already completely certified and approved to be deployed into KamLAND will be subject for the checking. So, this check is part of preparation to deployment, not certification.

Minimal procedure will be as follows:
1. Source should be soaked for 24 h (min) in a liquid known as a good solvent for radioactive material(s) used for source preparation. We expect to have recommendation from source producer about what liquid to use. Liquid for soak should be compatible with material of encapsulation. If solubility of used radioactive materials is under questions then KamLAND ls should be used for soak. For example both new AmBe and composite sources which will come on the site soon will be soaked in 0.1M solution of nitric acid. (we know it as a good solvent for radioactive materials used and encapsulation (sst) is compatible with it)
2. Soak sample will be counted on low background HpGe detector for a reasonable time.(that is not certification, just checking!!!)
3. If results are negative source will be deployed into KamLAND after any additional test and preparation procedures need for deployment. If any indication of a surface contamination will be found any other works for preparation of such a source to deployment will be stopped immediately, source will be send outside of the KamLAND area for future investigations.

Please allow 1-2 week additional delay time for deployment of any new sources. We hope that source producers will be cooperated in that final step before deployment.

Responsibility for above procedure is on the person on the site currently in charge for KamLAND calibration with radioactive sources.

Regards
Evgueni