PHYSICAL PROPERTIES OF LIQUID SCINTILLATORS

A THESIS SUBMITTED TO THE GRADUATE DIVISION OF THE UNIVERSITY OF HAWAI'I IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF

MASTER OF SCIENCE

IN

PHYSICS

DECEMBER 2006

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We certify that we have read this thesis and that, in our opinion, it is satisfactory in scope and quality as a thesis for the degree of Master of Science in Physics.
Acknowledgments

During the course of this project, I have benefited from the support of many individuals. I would like to express my appreciation to all, and in particular I would like to thank the following:

My advisor, John Learned provided me with an extraordinary opportunity; an opportunity to have ownership of a complete piece of a larger project. His decisive action, contacts, and “lets do the physics now” demeanor were significant in guiding my project forward. His patience and accommodation enabled me complete this research in spite of the “non-traditional” physics graduate student situation I am in.

Steve Dye’s questions were critical in the development of careful test system protocol and procedure. His questions further influenced the depth of analysis performed. Many extraneous variables were eliminated which may have otherwise adversely influenced the results I acquired.

Marc Rosen completed the finite element analysis on the stainless steel flange design, providing pertinent suggestions and direction for the final flange design moving it to production.

Ian Gordon saved me an inordinate amount of time rendering the stainless steel flange design in 3-D. His AutoCAD experience and work helped bring the flange from concept to integral test system component.

Troy Hix who measured the wavelength distribution for the LED’s.

Mark Olsson from DeepSea Power and Light provided excellent guidance in the development of the SS test assembly.

National Defense Center of Excellence for Research in Ocean Sciences (CEROS) whose financial support provided for this research under contract #53439. CEROS is a part of the Natural Energy Laboratory of Hawaii Authority (NELHA), an agency of the Department of Business, Economic Development and Tourism, State of Hawaii. CEROS is funded by the Defense Advanced Research Projects Agency (DARPA) through grants and agreements with NELHA. This work does not necessarily reflect the position or policy of the Government, and no official endorsement should be inferred.

Mahalo nui Loa.
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### Temperature Results

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Neutrino oscillation studies at KamLAND in Japan using a liquid scintillator detector concluded the “Solar Neutrino Problem” by observing that the electron anti-neutrinos from the reactors around Japan were oscillating. The parameters of the oscillation matched those deduced from solar neutrinos, so additionally demonstrated that the electron anti-neutrinos are not behaving differently from electron neutrinos in any significant manner. The data fits also eliminate some models competing with the oscillatory hypothesis [1, 2].

The secondary signal observed and presented by the collaboration involves the signal coming from the Earth itself, the “Geo Nu’s” or geo-neutrinos [3, 4].

The next step in studying the geological neutrinos is to characterize radioactivity within the layers of the earth using anti-electron neutrinos emanating from the Earth’s mantle and core. This characterization requires the ability to discriminate radiation coming from the mantle and core separately from the crust. This characterization requires that the detector is far removed from significant background signal contribution due to the crust and man-made nuclear reactors [5, 6].

Modeling the parameters of this type of neutrino experiment demonstrated the need for a 10 kilotonne liquid scintillator detector, ten or more times the size of the KamLAND detector. In order to avoid the neutrino background from regional man-made reactors and escape the contribution from the continental crust, we plan to place a detector over the thin mid-ocean crust. Submerging the detector in the deep ocean (\(>3000\) m) provides excellent shielding from the cosmic ray muons which otherwise induce background events. A key to the success of this detector is the liquid scintillator and its physical properties.

Any design for an underwater neutrino detector at a depth of a few thousand meters adds two new key parameters beyond land-based detectors such as KamLAND [2] and BOREXINO [14]. The first constraint on the scintillator to be employed is temperature; the temperature of seawater is approximately 4°C at depth. The second constraint is pressure; the pressure is approximately \(\sim6000\) psi (400 atmos.) at 4 km depth.

The goal is to find a liquid scintillator candidate that does not exhibit significant deterioration of the scintillators optical properties due to change in pressure and temperature. The concerns are mechanical and optical properties, about which there is little or no available relevant literature. Viable candidates must demonstrate little if any pressure dependence over a temperature between 0°C and 25°C. They must also
maintain a stable attenuation length at 0°C comparable to their attenuation length at ambient near surface conditions, between 20°C and 25°C.

An ideal liquid scintillator candidate would meet the following criteria:
- A very high light output (~ 10,000 photons/ MeV)
- An attenuation length on the order of 20 m at pressure and temperature.
- A high flash point; > 60°C to satisfy safety and deployment criteria.
- No chemical phase transitions within the constraint ranges.
- Optically stable near 4°C.
- Low Toxicity.
- Compatibility with Acrylic plastics.
- Cost Effective & available in industrial quantities.
- High Hydrogen Fraction (per unit weight).
- Low radioactive contamination.

Several candidate liquids have been identified and tested as will be discussed hereafter.
Chapter 2

Liquid Scintillator Background

2.1 LS Candidate Selection

The following four major liquid scintillator (LS) projects are operating or in proposal: KamLAND, SNO+, BOREXINO, and LENA. These projects have overall done a great deal of development work in locating acceptable materials and understanding how to make them radioactively extremely pure. These provide a starting point for evaluating possible LS candidates for a deep ocean detector.

KamLAND uses an 80/20 mix of Dodecane and Pseudocumene [28]. The SNO+ collaboration has proposed using Linear Alkyl benzene (LAB) and has explored its properties [9]. BOREXINO is evaluating the feasibility of using Pseudocumene and Phenyl-o-xylylethane (PXE) for their detector [10]. The LENA collaboration has proposed using PXE, or an 80/20 Dodecane and PXE mix [29]. Each of these LS candidates is an excellent scintillator at ambient temperature and pressure. Little or nothing has been studied about the pressure dependence, and only a little about temperature response (mostly just freezing temperature).

The following criteria were considered for each:

I. Safety: Flash Point, Toxicity
II. Feasibility: Cost, Availability, Acrylic Compatibility
III. Physical Properties: Density, Fluor Dissolution, Hydrogen Fraction
IV. Optical Properties: Light Yield, Attenuation Length
V. Contamination: Radioactive impurities, chemical impurities

See Table 1 which summarizes the properties of the materials considered.

2.1.1 Safety

Federal safety regulations would prompt the use of LS with a "Flash Point" beyond the range of working temperatures, including a safety margin. Surface temperatures for an ocean deployment vessel may reach above 30°C. As a result, the use of Pseudocumene (PC) containing solutions may not meet stated safety standards. Both PXE and LAB with flash points well above 100°C pose no risk for explosion.

Based on the MSDS sheet information for the LS mixes considered, all candidates other than pure PC do not pose a significant health risk for exposure in small quantities, or significant contamination risk to the environment. There would not appear to be a problem in obtaining relevant permits and approvals from interested government agencies at the federal or the state level. The environmental risks are comparable for PXE, LAB, and the KamLAND mix. Moreover, the use of a double
hull design for the vessel should satisfy USCG requirements, according to Makai Ocean Engineering.

2.1.2 Feasibility
Both PXE and LAB are industrial chemicals with high volume production and available from a multiplicity of sources. PXE is available from Dixie Chemical in Houston Texas in the quantities required, among a couple domestic producers. Of note, the VP for Research and Development at Dixie is very willing to work closely with us to meet the stringent criteria in quality, consistency, and process.

LAB is available from SASOL in Baltimore, Maryland in the quantities proposed, as well as from other companies in North America, Africa, and Europe. The total US capacity is some three times consumption, with most of it presently going to surfactant production [7]. LAB is the base for the familiar clear dish-soap with which almost everyone is familiar. Our contact at SASOL has been enthusiastic in working to develop the best LAB composition for use as a LS.

The structural framework for the proposed underwater detector utilizes a modular acrylic sphere. Any viable LS must be compatible with acrylic. The integrity of the structural and optical properties of the acrylic must not be degraded with time. Acrylic is incompatible with PC and its primary constituent Benzene according to acrylic vendor Ineos. The KamLAND mix (80/20) reduces the concentration of PC to an acceptable level, preventing polymer disassociation. PXE also needs to be diluted to at least (70/30), while (80/20) is recommended. Concentrated PC and PXE produce significant surface “crazing”, significantly reducing the optical properties of the acrylic [8].

LAB on the other hand has excellent compatibility with acrylic [9]. In summary; the KamLAND mix, an (80/20) mix of Dodecane and PXE, and LAB are sufficiently compatible with acrylic to be considered as LS candidates in the proposed underwater detector.

2.1.3 Physical properties
Density is a critical concern when considering the structural design for a 10 kilotonne LS detector. A LS density that is too high (~1.0) would require a design which included supplemental buoyancy. A density that is too low (~0.70) would prompt the need for significant extra ballast as well as a secure method of anchoring the detector to its ocean floor mooring. Each of the LS candidates’ density is within the design parameters of the proposed detector.

2.1.4 Contamination
Each LS candidate was initially selected meeting the minimum criterion for: Light Yield, Attenuation Length, Proton Target Ratio, and Fluor Dissolution. The production process produces highly pure material for each LS. Contamination is introduced during the transport and storage process as well as the introduction of
fluors. The primary contaminates are: $^{238}\text{U}$, $^{232}\text{Th}$, $^{226}\text{Ra}$, $^{222}\text{Rn}$, $^{85}\text{Kr}$, $^{39}\text{Ar}$, $^{40}\text{K}$, and $^7\text{Be}$.

Uranium, thorium, and radium are siliceous particulate contaminates. A solid column Silica gel filter has been shown to remove colloidal particles larger than 0.05 microns reducing $^{238}\text{U}$ and $^{232}\text{Th}$ from 10 ppm to $10^{-16}$ g/g [10]. Phase separation techniques are required to remove particles from 2 - 500 nm [11]. Measurements taken at Brookhaven on LAB showed that they were able to improve the attenuation length from the accepted value ~25 m to an attenuation that was too small to discriminate with a 10 cm cell, where the absorption at 430 nm is negligible [12].

Vacuum Distillation is used to remove low volatility components such as radioactive metals as well as chemical impurities that degrade the optical properties of the LS. It has been shown to reduce the $^{40}\text{K}$ measured in the fluor PPO at $\sim 10^{-6}$ g/g to $10^{-11}$ g- K/g-PPO [13]. It also improved the attenuation length of PC by a factor of three, from ~7 m to ~21 m according to the Brookhaven tests.

Radon, krypton, and argon are most likely to be introduced due to air exposure. The inert gases may be mitigated by maintaining a closed system and selecting materials for the structural frame which have a low permeability (to noble gases) and low radioactive content for the LS detector [14]. This may also require cooperation on the part of a vendor to upgrade their distillation and manufacturing plant to meet the stringent production requirements necessary to minimize radioactive contamination in the LS.

Tritium and carbon 14 are inherent contaminants and can not be removed (economically) with current technology and industrial processes [14]. The only suggestion found in the literature was to minimize cosmogenic exposure by minimizing the shipping time and maintaining tight control of logistics. $^{14}\text{C}$ has a long half life and is unaffected by filtering, therefore the only viable option is to minimize the amount generated cosmogenically [15].

The preferred method to remove polar and charged ionizable metals and salts such as uranium, thorium, potassium, and beryllium is water extraction. It is the method of choice since it allows for processing at ambient temperature ~20°C and avoids possible degradation of the LS [16]. It has the added safety benefit of avoiding temperatures near the flash point of the scintillator being filtered.

$^7\text{Be}$ is produced cosmogenically from $^{12}\text{C}$. Equilibrium exposure of the LS at the surface will produce a background of $\sim 10^5$ events/day which requires 1.5 years to decay to $\sim 10^0$ events/day [17]. Therefore it is advantageous to minimize the time from LS production to detector deployment to minimize the background from this decay chain.
Dry Nitrogen Stripping (Sparging) is employed after water extraction. This strips the water from the LS. Sparging also strips oxygen from the LS which if not removed quenches scintillation, which reduces the effective attenuation length. As an added benefit, nitrogen strips dissolved radioactive gasses such as $^{222}$Rn and $^{85}$Kr from the LS [18].
<table>
<thead>
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<th></th>
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<td>145°C</td>
<td>130°C</td>
<td>64°C</td>
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<td></td>
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</tr>
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<td>$$</td>
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<td>$</td>
<td>$</td>
</tr>
<tr>
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<td>Available</td>
<td>PC dependant</td>
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<td>Satisfactory</td>
<td>Excellent</td>
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<td>Fluor Dissolution</td>
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<td>Light Yield</td>
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<td>53.9% Anthracene</td>
<td>57% Anthracene</td>
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<td>Attenuation Length</td>
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<td>&gt; 25 m</td>
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Chapter 3

Measurement Criteria

The key measurement criteria this study reports upon are the effects on the optical properties of liquid scintillator candidates due to an increase in pressure of 400 atmos, and a decrease in temperature of 20°C to approximately 4°C. There is industry consensus that there is no inter-dependence between temperature and pressure on LS materials. It is expected that there is a linear relationship between pressure and volume as well as between temperature and volume in the range of interest.

The Benedict-Webb-Rubin-Starling (BWRS) Equation of State provides a series expansion for the relationship of liquids, such as liquid scintillators, in terms of $P = $ pressure, $R = $ Ryberg Constant, $T = $ temperature, and $\rho = $ density, and a number of constants:

$$P = \rho RT + \left( B_o RT - A_o - \frac{C_o}{T^2} + \frac{D_o}{T^3} - \frac{E_o}{T^4} \right) \rho^2 + \left( bRT - a - \frac{d}{T} \right) \rho^6 + \frac{c\rho^3}{T^2} \left( 1 + \gamma \rho^2 \right) \exp\left( -\gamma \rho^2 \right)$$

In “Fluid Properties for Light Petroleum Systems”, Starling provides mixing rules for compounds and procedures for calculating coefficients: $A_o, B_o, C_o$, etc. [18]. As may be inferred from this equation; density is linear with pressure and temperature to first order. Since $T$ is in Kelvin, the terms in $T^n$ fall off speedily. To first order there is also no inter-dependence between pressure and temperature for a liquid while maintaining a constant phase.

3.1 Pressure Dependence

To first order at the 95% level, it is expected that density of LS candidates will demonstrate a linear dependence on density due to pressure. The physical result of pressure at depth on Dodecane will produce a volume compression on the order of 3%; this volume differential must be accommodated in the design of an ocean detector at a depth of -4000 m, and a pressure of -400 atmos. [19].

Dodecane is the one component common to nearly all LS mixes in use currently or being proposed. As a result, its physical properties and measurements will be used throughout this report as a benchmark reference.

As may be observed in the Figure 3.1, in the range of 1 and 1,500 Torr there is a linear relationship between pressure and the density of n-Dodecane as measured by Caudwell [20]. This graph shows that a 10% increase in density corresponds to a pressure increase of three orders of magnitude.

The physical properties of pseudocumene may be inferred from Benzene, its dominant constituent. Based on the properties of Benzene and the volume
composition of the KamLAND mix it is expected that there will be no significant
differences due to pressure between the KamLAND mix and Dodecane [21].

![Pressure Dependence](image)

Figure 3.1: Dodecane density pressure dependence.

The physical properties of PXE and LAB have been insufficiently measured and
reported to provide a reference baseline with which to confidently compare laboratory
data measured in this experiment. In the case of PXE mixed with Dodecane at 1:4
with a resultant mix density of 0.82, it is reasonable to expect that there should be no
significant differential effects scaled off of the Dodecane results.

LAB may be compared only loosely with the Dodecane but it can be expected to
follow a similar scaling factor since the LAB estimate is based on only three data
points over a range of 100 psi. (Figure 3.2) Extrapolating the LAB data reflects a
change of only 0.41% in density over 1000 psi [22]. Using the more reliable data
from the Dodecane analysis provides a solid upper bound on the requisite amount of
make-up volume that needs to be engineered for in an underwater detector.

![Density as a function of Pressure](image)

Figure 3.2 shows the extrapolation and the increase in density with pressure at 293K, for LAB.
Based on experience working with LS materials, industry experts expect that there is no measurable degradation in the optical properties of liquid scintillators due to pressure. To date there has been no published work on the effect of pressure on LS optical properties. This study then will attempt to isolate and measure changes in the optical transmission of LS candidates due to pressure.

3.2 Temperature Dependence

The BWRS equation gives the general formula for the dependence of fluid density on temperature. This assumes that temperature is changing along one isotherm and there are no phase changes over the range of interest. As may be inferred from the equation, to first order at the 95% level, there is linear dependence due to a change in temperature on fluid density.

An analysis of Caudwell's data on Dodecane in the graph below (Figure 3.3) shows that as temperature drops the trend lines for different pressures converge [23]. As may quickly be observed, the data reflects a linear dependence on density due to temperature as predicted.

![Figure 3.3: Dodecane density temperature dependence.](image)

At ambient pressure, extrapolating on the graph to 0°C from 20°C represents a reduction of 2.8% due to the change in temperature. The extrapolated density of Dodecane is 767 kg/m³. Extrapolating along the 6100 psi fit line to 0°C from 20°C reflects a loss of 1.7% due to the change in temperature. Here the extrapolated density is 785 kg/m³.

Comparing the extrapolated Dodecane density reduction with data provided by SASOL for LAB 501 shown in Figure 3.4 below, the results due to temperature are comparable, reflecting a change in density of - 2.1% [24]. This value, as with that for pressure, represents a smaller variation than for Dodecane, but one that would not significantly change the proposed design for the Hanohano detector. As for the effects
of temperature on LS candidates' optical properties; researchers familiar with liquid scintillators suggest that there should be no change in optical properties due to a change in temperature over the proposed range. The prediction is that there will be no degradation in attenuation length or light yield.

The only study found which attempted to measure the effect of temperature on LS optical properties measured a decrease in light output of 1% per degree Celsius [25]. This study then will attempt to isolate and measure changes in the optical transmission of LS candidates due to temperature.

![Graph of N501 Alkylate Density vs. Temperature](image)

Figure 3.4 shows the correlation between the density of LAB 501 and temperature. The densities were measured using a digital density meter (Mettler-Paar) using water and n-Decane as calibration fluids. For lower temperatures, ethanol was used instead of water for calibration.

### 3.3 Combined Pressure/Temperature Dependence

Compared to Dodecane at STP, the combined effect of the conditions at depth on the Dodecane is a 5.2% volume reduction. The structural design for the LS detector must accommodate a 5% loss in volume. For a 10 kilotonne detector, this represents a makeup volume of 0.5 kt. The cost estimate on this makeup volume alone runs from approximately $100K - $1000K, depending on the scintillator mix chosen (but the material is not wasted, being employed in the detector when at depth, slightly increasing the target mass).

Interpolating between the data sets in Figure 3.3 reveals that increased pressure suppresses the effect due to temperature. As a result, it is expected from analyzing Figure 3.3 that there is a 2.0% change in volume due to a 25°C change in temperature, beyond the change due solely to increased pressure.
Reviewing the data on the melting points for all LS candidate constituents, it is expected that there are no phase transition points within a few degrees of the expected minimum ocean bottom temperature 4°C at any contemplated location. The above total estimate of 5% for Dodecane may be used as a benchmark then for the volumetric changes due to changes in pressure and temperature for each candidate.

The resulting estimates assume that pressure and temperature effects are linearly independent, consistent with standard industry practice. Using the data for Dodecane and LAB provide an educated prediction for the upper (5.2%) and lower (2.5%) bound for the volumetric change due to temperature and pressure.
Chapter 4

Test System Design

The need is to create a laboratory test setup in which samples of LS may be pressurized to deep ocean equivalent pressures and determine if any significant optical changes take place. A large volume is expensive and dangerous; moreover available materials without intolerable order times, necessitated designing a system with a small diameter, 1 m long assembly with windows.

4.1 Design Criteria

Chemical
- Foreign Contaminant Free
- Non-Reactive with LS Constituents
- Cleanable

Mechanical
- Closed System
- Flow Through Rate (1 liter per second – minimum)
- Working Pressure (≤6100 psi)
- Good Thermal Conductivity
- Modular
- Quick Assembly/Breakdown
- Window Ports at ends

Process
- Materials Procurement (< 30 days)
- Common Fittings

4.2 System Architecture

4.2.1 High Pressure Test System
A Swagelok ¾” tube and connector components was employed in building the high pressure stainless steel test assembly. (Figure 4.1) This was mated to a custom 316 SS flange designed to hold the view ports at a working pressure up to 6100 psi. (Figure 4.2) 2.50” diameter, 0.250” thickness, clear Sapphire windows (Swiss Jewel model W63.25) were mounted in the flanges as view ports.

The design of the seat for the windows is a very sensitive matter, where the flexing of the flange under pressure must not distort the windows in such a way as to lead to failure. The design for the flange was greatly aided by advice from DeepSea Power and Light. Casually reviewing the design, one may think that the flange is
Figure 4.1: Stainless Steel Test Assembly
greatly over-designed looking at the substantial size, but that is not the case due to the requirement for not edge loading the Sapphire windows.

Figure 4.2 Stainless Steel Flange Assembly

The SS system has a working length of one meter. As one of the system requirements was a modular architecture, the system may be configured for a 3 m length simply by changing the tube used. The one meter system has a working volume of 315 cm$^3$. A larger diameter is to be preferred but the order time and minimum volume for larger SS pipe was prohibitive.

An Enerpak model 11-100 single speed hand pump is used. It is multi-fluid compatible; a requirement for pressurizing liquid scintillator components such as: Dodecane, pseudocumene, PXE, and LAB. With the installation of the (Enerpak) fluid isolation cylinder it became unnecessary, as well as undesirable, to use LS materials in the pump. In order to maintain the longevity of the pump and prevent failure, hydraulic fluid is preferred for use with the pump.

Testing demonstrated that Enerpak’s standard high pressure hose connecting the pump with the isolation cylinder was incompatible with the polar molecules in the LS components. They were chemically reacting with the hose and degrading the physical integrity of the hose which would have led to catastrophic failure under high pressure, as well as contaminating the LS test samples. The hose was replaced with a thermo-plastic hose which solved the compatibility and safety issues.

An Enerpak model RD-46 double-acting cylinder is used to isolate the pump fluid from the test fluid contained in the SS system. The cylinder is attached to the system through the 3/8” NPTF ports. The inclusion of an isolation cylinder allowed for much greater sample quality control. No LS material is in contact with any pump parts, hydraulic fluid contaminants within the pump, nor with the hose.
4.2.2 Optical Components
Polished flat blue and green LED's from Linrose Electronics were used for baseline measurements. The blue LED (B4304H6-1) wavelength peak was at 430 nm, with a full width-half max value of 60 nm. (Figure 4.3) The green LED (B43004H95) wavelength peak was at 520 nm, with a full width-half max value of 30 nm. (Figure 4.4)

![Blue LED Wave Length Profile](image1)

![Green LED Wave Length Profile](image2)

A (CrystaLaser) 405 nm blue CW laser (BCL-025-405) was used as the primary source for evaluating the optical properties of each LS candidate and component. 405 nm is close to the emission peak and photomultiplier sensitivity maximum.

A United Detector Technology silicon photo-detector (J43053 053-1) connected to a Keithly pico-ammeter model #6517A provided the readout data.

4.2.3 Control Systems
A decanting system was built to provide for dry nitrogen purging of the SS system in preparation for an LS optical test. It also was designed to decant each chemical being tested from its source container into the test system using pressurized dry nitrogen. The third use of this system was to “sparge” test chemicals with the dry nitrogen before decanting in order to strip them of moisture and oxygen (moisture is known to be detrimental to the optical properties of liquid scintillators, particularly at low temperatures).

Common refrigeration Copper tubing and Brass fittings were used for this decanting system, available at any hardware store. No special care was taken to control radiopurity at this stage of the measurements, the focus being on water and oxygen.

4.3 Systematics

4.3.1 Cleaning Procedure
ASTM G-93 Level A requirement for cleaning methods and cleanliness levels for material and equipment was followed; in order to maintain the purity of each sample, and produce verifiable results free of contamination.
As part of this process, the SS system components were cleaned using an ultrasonic bath with an 80/20 bath of acetone and ethanol. Components were prepped using 100% ethanol. Cleaned components were promptly assembled and capped.

4.3.2 Fill System Procedure
- The test system was dry nitrogen purged for 15 minutes prior to each test fill.
- An overpressure of 3 psi was maintained in the SS system while decanting to maintain a nitrogen atmosphere.
- Prior to decanting, each test sample was sparged for 30 minutes to strip water and oxygen.

4.3.3 Component Reliability

405 nm Laser
According to the vendor certification from CrystaLaser for the 405 nm laser:
- Beam diameter = 1 mm.
- Beam divergence at 0.5 mrad (full angle)
- Noise (rms) at less than 0.5%.

Keithley Ammeter
The Keithley Electrometer specification sheet lists the peak-to-peak noise at 0.75 fA. The resulting signal-to-noise ratio was some six orders of magnitude lower than the signal measured with the United Detector Technology’s photo-diode (J43053 054-1).

Photodiode/ Output:
- *Photo-diode light sensitivity* is uniform over the photodiode surface (11 mm diameter) with an abrupt drop at the edge of the photodiode, consistent over two orthogonal axes using a surface pinhole test.
- Anticipated *photo-diode temperature dependence* was eliminated by removing the component from the system during cool-down. It was optimally positioned only when measurements were being performed and then set aside again.
- Dark current/noise was: 0.2 nA
- Drift was: +/-1.5 nA; consistent over all power settings and both LED’s.
- The zero point readout is: 0.04 nA

Power Source/ LED:
- *LED*: Input power was maintained by setting the power and either running the test with the system power/LED on or using the power button to turn off system and bring it back online. Any output voltage variation was below the sensitivity of the multi-meter (1 mV).
- *Laser*: Input power was set to 0.2 mW and consecutive tests were run using the key and switch to maintain a consistent output power.
4.3.4 Baseline Tests

There is no signal/light loss due to the SS system (including windows) being cooled from ambient to 0°C. Over the same path length, the SS system reduces the direct beam signal by 25%.

The SS system optical path includes two 0.25 in. sapphire windows and the SS tubing. The transmission curve provided by the manufacturer Swiss Jewel shows 85% transmission for synthetic Sapphire. (Figure 4.5) For two windows, the expected loss on the order of 30%, so the result is consistent with the data provided by the manufacturer.

![Transmission Curve](image)

Figure 4.5: Sapphire Transmission Graph provided by Swiss Jewel.

Mechanical compression testing using the SS test system reflected a 2.2% volume reduction in the Dodecane at 6000 psi, and a 1.9% volume reduction for LAB 501. With a measured volume differential of 0.3%, there was no realizable difference in compressibility or increase in density. Comparing this data with that reported earlier for Dodecane at 3%, the compression test is in agreement within 1%.

Based on the mechanical compression test, it would be expected that LAB 501 has the same approximate density gradient as Dodecane at 6000 psi. Compared to the data presented in chapter two at 0.41% for the density change in LAB, there should have been a factor of five smaller change in volume compared to the Dodecane. As this was not the case; it may be inferred that using the SS test system for a mechanical compression test only provides an order of magnitude comparison, with an overall variance of +/- 1.5%.
Chapter 5

Test System Protocol/Lab Procedure

5.1 Alignment
An aluminum optical-bench rail was used to support and align all optical path components. The rail was leveled and checked for level while loaded (including water bath). The SS system tube was then checked for level to confirm alignment in the horizontal, parallel to the optical rail.

The silicon photodiode was centered along three orthogonal axes on the sapphire window and then locked down. The photodiode was the only component to be physically moved while an optical test was being conducted. In order to eliminate the effect on the signal due to the temperature dependence of the detector it was only placed in position to take readings at specified system temperatures, as developed in section 5.3.2.

A lens was employed to produce a collimated beam from the LED. A second detector-side converging lens (f=26 cm) was at first employed to focus the light collected on the photo-detector. Experimentation showed though that once the light source was aligned, the detector side lens only degraded the signal received, making the signal more position sensitive. Changing the lens and the focal length did not eliminate the signal loss observed. Therefore the final optical arrangement used did not include the receiver side lens.

5.1.1 LED
The LED optical setup includes the LED and a 10 cm focal length converging lens. The lens was positioned at a distance equal to the focal length from the LED, and centered on axis with the sapphire window in the SS system. (Figure 5.1) Measurement of the lens's focal length was 9.6 cm, at which distance the light output is maximized. There was a factor of three increases in signal strength with the lens included in the optical path compared to the bare LED.

![Figure 5.1: Optical Bench Test Setup.](image)
The following alignment procedure was applied; where all distances were measured from the source side sapphire window and observations were made from the detector side:

1. Center the LED face (polished) (5 cm).
2. Center the LED again at (15 cm).
3. (Check) center on lens (3 cm).
4. (Check)/center LED through lens (30 cm).
5. (Check)/center LED through lens (15 cm).
6. (Check)/center LED through lens at lens focus.

In steps four and five, alignment could be verified by the observation of a central bright spot surrounded by what looked like a set of concentric Newton’s Rings when the LED was viewed from the detector side of the system on axis as illustrated here in Figure 5.2. Any asymmetry indicated that the LED was not in alignment.

![Figure 5.2: Correct alignment simulation for LED’s.](image)

Step six could be verified by the observation of an isotropic light as viewed from the detector side of the system. Secondly, the alignment could be checked by measuring the output signal from the detector at the end of the SS system and up to 8 cm away without a definitive drop in signal.

### 5.1.2 Laser

Experimentation demonstrated that the use of the source side converging lens with the laser reduced the output signal on center 5% when optimized (compatible with expectations from interface losses). Therefore the lens was removed from the optical arrangement for the 405 nm laser setup. The following alignment procedure was applied:

1. Center the laser on the sapphire window (5 cm). [horizontal, vertical]
2. Center the laser again at 30 cm on screen target through detector side window.
3. (Check)/center laser through detector side window (30 cm) on screen target along the three orthogonal axis.
4. Mark set positions, then verify position optimization with peak signal output in three dimensions with detector.

Alignment was performed after each test sample was prepared and the set up completed on the optical bench, including filling of the water bath and thermocouple placement.
5.2 Temperature control

5.2.1 Water Bath
It was determined by trial and error, that employing two bags (45 gallon plastic refuse bag) filled with water surrounding the SS tube and in contact with the inside face of each flange worked best in maintaining a constant temperature within the test assembly. Other water bath options considered were: using the 3 m tank, a single 45 gallon bag, and three or more 45 gallon bags.

The two bag system allowed for control of the temperature of each half of the test system. This arrangement produced the most consistent results, the smallest temperature gradients across the system, and resulted in the shortest time for thermal and optical stabilization.

Using an open bath (single bag) from end to end was shown to have greater variations in the temperature across the length of the tubing and end caps over time, even with systematic stirring. Subsequently, the system took longer to stabilize near 0°C.

Utilizing three or more bath bags made it increasingly difficult to produce the same temperature in each bag. The temperature variations were progressively worse for each bag added. As a result, the time for the test system to stabilize was the longest in the multiple bag arrangement.

5.2.2 End Caps
The inside section of the each end cap provided the contact end for the water bath. The exposed surfaces were insulated to minimize the thermal gradient across the end cap. Insulating the caps reduced the thermal gradient by 40% on each cap. The temperature of the end caps were measured on each face and at the sapphire window. In general the end caps stabilized before the fluid in the system reached equilibrium, in the roughly eight hours each test setup took to stabilize.

5.3 Test System Control

5.3.1 Pressure
From initial system pressurization at 6100 psi, there was a system "relaxation" of ~300 psi to 5800 psi (compatible with adiabatic heating upon pressurization and subsequent cooling). This required a wait time of approximately one minute, at which point the pressure could be raised to 6100 psi again where it would remain stable over the duration of the test.

After the leak test for each system test was completed no further pressure drop should be observed over the duration of the test (~ 30 min.). In one case there was a leak at the face of one of the windows, this required a rebuild of the entire test system.
Were the system not used for a few days, it was necessary to cycle the Enerpak piston 5-10 times to lubricate the internal seal on the piston. The piston as may be recalled, maintains a sealed barrier between the LS candidate being tested in the SS test assembly and the hydraulic pump fluid. Failure to cycle the piston would result in drops of hydraulic fluid leaking out of the piston, producing a subsequent drop in pressure at 6000 psi.

In order to achieve repeatable pressure test data, the temperature of the SS system needed to be equal that of the water bath. Therefore after pressurization, approximately five minutes was required for the system and bath to come to equilibrium at the bath temperature. Another 25 minutes was required on average for the LS candidate in the system to reach optical equilibrium.

5.3.2 Post-Test
In order to validate the results of each test, a benchmark of 5% was set for variation between the baseline signal measurement and the post-baseline measurement. Test runs with greater than 5% deviation were discarded, and the test rerun. Post tests results were taken after the system temperature and ambient room temperature were in agreement.

Movement of any of the optical path components in the horizontal or vertical quickly produced a significant change in the signal. Test runs where one or more of the optical path components were moved were invalidated. Notably, the detector diode once aligned allowed for removal from the optical bench and return without affecting the results. Test result variations were on the order of 0.1%.

Experimentation demonstrated that if the post test ambient temperature varied from the baseline by 3°C or more the post-baseline test signal diverged ~5%. The room temperature could be affected by having the adjoining door to the Anita Lab next door open. It could be (and was) radically affected by the occasional demise of the building air conditioning plant. Each test run that spanned a failure in the air conditioning system was scratched as the ambient temperature increased 5-8 degrees.
Chapter 6

Pressure Test

6.1 Sample Test Results

The data for each of the pressure and temperature tests may be found in appendix A. Sample tests that failed to produce a satisfactory post-baseline test for signal intensity were discarded and the sample retested. Recall that test designators in Figure 6.1 beginning with "B" reference the blue diode with the peak at 430 nm, and the full width-half max value of 60 nm, while those test designators beginning with "L" reference the 405 nm laser.

![Pressure Test Divergence](image)

Figure 6.1: Pressure test results for LS candidates. The estimated instrumental and systematic error for the tests presented is +/- 2%.

6.1.1 Dodecane

The pressure test on Dodecane (BDP2) produced a 3% loss in signal strength vs. both the baseline and the post-baseline measurements. The Dodecane test, being the first done did not have a water bath to maintain equilibrium, therefore the test temperature was 1.8°C above the ambient. There was an initial spike in temperature after pressurization of 4°C. In fact all of the test samples demonstrated an initial pressurization temperature increase of ~4°C before returning to equilibrium. It is of merit to note that although the post-baseline measurement was taken 15 hours later, it was equal to the baseline.

6.1.2 Pseudocumene

Test B1PP1 showed a -4% change in signal strength vs. the baseline and no change vs. the post-baseline measurements. Beginning with the pseudocumene, all pressure tests had the added benefit of the water bath. Approximately 30 minutes was required
to reach thermal equilibrium. This includes the return of the sample temperature to that of the ambient bath and the output optical signal remaining stable.

The temperature increase for each sample when pressurized was matched with a similar decrease in temperature when the pressure was released. Significant optical distortion was observed in the tube shortly after the system was pressured. As a result, this and each subsequent post-baseline measurement was taken the following day to insure stability. I included the signal change vs. the post-baseline measurement in Figure 6.1, and it is this data that is the most accurate.

6.1.3 KamLAND Mix
Both test B1KP1 and B1KP2 produced an interesting and anomalous result. All of the other test samples demonstrated no change or a small degradation in signal due to pressure. The KamLAND mix had an increase in light signal under pressure. There was an astounding increase in light of 40% vs. the baseline and 16% vs. the post-baseline. These results differ significantly from the mix’s constituents. It is expected that the KamLAND Mix follow closely with the results for Dodecane – its primary constituent. The optics were focused following the protocol developed, and the power reading for the LED remained constant.

Convinced that there was a mistake or tampering, test B1KP2 was set up from scratch and the pressure test run again on the same sample. Again there was a noticeable increase in light output under pressure, producing a 9% increase vs. the baseline and 5.5% vs. the post-baseline. Again there were no physical changes made to the system or optics.

There is no systematic explanation as to why the light transmission is greater for the pressurized test run than for the baseline measurements. Were there lensing or physical optical warping, one would expect that the results would be lower and not higher due to any change in the focal length from the optimized setup for the baseline test at ambient. In any event, if there was optical lensing due the flexing of the sapphire windows under pressure it would be identical for each material tested.

It is interesting to note and may merit further discussion pointing out that the post-baseline measurements for both tests are lower than the pressure test measurements. These were the only tests to show an increase in the post-baseline measurements over the baseline values.

Using a dental mirror to make observations showed a clear and isotropic sample under pressure. There was no evidence to suggest that there was any other optical process going on or that there was a change in the focal length. In fact moving the focal lens only reduced the signal during the pressure test as well as for the post baseline.
There were a number of suggestions as to what the possible cause of this anomaly is. They are as follows:

- Lensing (unaccounted for physically, but present none-the-less)
- A pressure hysteresis curve.
- A change in the index of refraction for the mix.
- A change in the chemical structure of the KamLAND mix.

Although interesting, no further study on the pressure dependence of the KamLAND mix is expected. The temperature test results effectively removed it from consideration for the proposed detector, ending further interest in it. Still, at a later time it would be interesting to explore this anomaly.

6.1.4 LAB
The pressure test on LAB 540L was consistent with other LS constituents. The test results produced a -3.7% change vs. the baseline and no change vs. the post-baseline measurements for the blue LED. The blue laser arrived in time for the test on the LAB samples. As a result, a pressure test was run with both the LED and the 405 nm laser for comparison. The laser test produced a ~ -7% change vs. the baseline and the post test. The laser setup, like the LED followed protocol developed for optimization of the focus along one axis parallel to the optical bench. In the subsequent LAB N501 pressure test there was an increase in signal output of 1.4% vs. the baseline and no difference with the post-baseline test.

6.1.5 PXE
The PXE pressure test produced results of -7.0% vs. baseline and -4.3% vs. the post-baseline value. Although these values are in line with the other samples, observation showed that the PXE has a longer relaxation time for reaching equilibrium. Each of the prior samples reached equilibrium with in 15-20 minutes – with the measurements taken at 30 minutes and one hour, the PXE on the other hand took nearly an hour and a half to stabilize. On the pressure down, the optical distortion effects took another 1.5 hours to dissipate.

The 70/30 Dodecane/PXE mix showed a change due to pressure of -2.3% vs. baseline and -1.6% vs. the post-baseline value. The time for this mix to come to equilibrium was in between that of Dodecane and the PXE, taking 40 minutes to stabilize. If PXE is chosen as a primary detector candidate, additional time must be built into the ocean test program for the scintillator to come to equilibrium. This issue of relaxation may become a significant factor in the reliability of the results for a detector with a PXE mix.

6.2 Pressure Analysis
The estimate for systematic error is +/-2%, which includes the error for the light sources and the detector electronics. Looking at the test data, and using the post-baseline values for comparison there is negligible pressure dependence for the LS candidates evaluated. Only pure PXE and the anomalous KamLAND mix fell
noticeably outside the error range. Since the Dodecane/PXE mix is effectively within the error range, there is no concern of pressure dependence for using this LS candidate.

Each sample had a peak temperature increase of ~4°C when pressurized to 6100 psi, as noted earlier. During an ocean test, the LS candidate would be pressurizing over approximately one hour according to simulations run by Makai Ocean Engineering, rather than the five seconds in this test. The relaxation time; the time the sample takes to reach equilibrium and maintain an isotropic optical path, is of more importance in future measurements.

For purposes of validation of the proposed Hanohano detector conceptual design one may can conclude that in the upper limit the absorption would increase by 3.7% over a 1 m path length. If the post-test were taken as a baseline, then this change would be less than 1%, and arguably the more accurate.

It is plausible that there was some coagulation of small chemical structures caused by the pressure. This would explain the hysteresis in the transmission measurement after return to ambient pressure. In future testing, this effect may be evaluated by heating the samples to redistribute the possibly coagulated material.
Chapter 7

Temperature Test

7.1 Temperature Results

Complete test results may be reviewed in appendix A. Below, figure 7.1 provides a snapshot of the optical transmission measured versus the ambient baseline. Samples’ test values are reported at 4°C unless otherwise noted. The path length is 1 m.

![Optical transmission thermal dependence](image)

Figure 7.1: LS candidate light transmission at 4°C. The estimated instrumental and systematic error for the tests presented is ±/−2%.

7.1.1 Dodecane

There was a light signal loss of ~50% as a measure of the photo-diode output current due to a change in temperature of 20°C, from ambient to 0.3-4.5°C. Visual observation did not provide any clues to the loss in signal, which is not surprising considering visual response has a huge dynamic range and is sensitive on a log power scale.

Developing the test protocol determined that approximately 8 hours would be required from the initial cool down before the test samples would stabilize at a chosen temperature. Optical "Schlieren" (see section 8.1) layers were visible for ~4 hours. Test measurements indicated that no further improvement in output current could be achieved by using a test time of longer than ~8 hours.
The loss observed is consistent for both the blue (430 nm) and green (520 nm) LED’s suggesting that there is no significant frequency dependence for Dodecane in the region the proposed study is interested in.

7.1.2 Pseudocumene

Below 4.5°C, Pseudocumene is basically opaque to visible light. There was a transition observed between 3°C and 4°C from opaque to “clear”, but there was extreme distortion in observing the LED’s through the one meter system. Repeated, carefully controlled testing produced similar results. Visual observations and optical test measurements demonstrated that below 12°C, Pseudocumene failed to produce non zero verifiable results.

7.1.3 KamLAND

Considering the results for the pseudocumene, the measurements for the 80/20 mix of Dodecane and pseudocumene comes as no surprise. The light transmission was only 10% at 4°C. Like the pseudocumene, the mix was opaque below 3.5°C. Above 4°C the mix looked much like the pseudocumene did at the same temperature. Again, careful repeat tests confirmed the results found.

Since the KamLAND mix was the initial “primary candidate”, extra caution was used after the initial observations were made of the signal loss due to the decrease in temperature. Even doubling the time of measurement produced no measurable change in light transmission.

7.1.4 LAB 540L

As in the case of the two prior test samples, the LAB 540L sample was opaque below 4°C. Above 4°C the sample looked “clear” with obvious schlieren effects creating distortion and lensing. Obvious gradient lines were visible.

In order to produce any transmission reference for this LAB sample; the temperature was slowly raised to 5.3°C, where the transmission was measured at 18%. This temperature was the first temperature where the signal remained stable for 5 minutes or more. As in the prior samples, the sample was cycled between 0°C and 7°C in order to confirm the optical quenching observed.

7.1.5 LAB N501

The N501 LAB sample was the first to appear as a viable candidate. At 0°C this LAB sample remained “clear” without the accompanying thermal gradient lines visible in the other test samples. The initial visual test showed the sample to be free of distortion/ lensing. The image of the laser beam morphology appeared consistent on a screen, and appeared as strong as during the baseline test.

At 4°C, 76% of the baseline transmission value was recorded. At 6°C there was a further 6% increase in transmission. Using the result at 4°C, a preliminary
attenuation length calculation yielded $\lambda = 4.5$ m. Although this value is smaller than that required for the proposed detector, this sample provides the first positive results for use as a liquid scintillator in the proposed detector.

An attenuation length of 4.5 m may conservatively be considered a lower limit for this LAB sample. In the LS purification study done at Brookhaven, electrophotometer results posted an order of magnitude improvement in transmission. The measured absorption was 0.011 at 410 nm for the unfiltered LAB sample and 0.001 for the silica column filtered sample. This result was accompanied by the claim that the resulting “attenuation length was too small to be measured by a 10 cm cell” [26].

7.1.6 PXE
The PXE sample produced 39% of the baseline transmission at 2°C. The PXE remained “clear”, yet there was obvious optical distortion even after 8 hours of carefully controlled cooling. The (laser) beam morphology appeared consistent on a screen, just weaker in intensity as observed on the detector end of the system.

7.1.7 Dodecane/PXE (70/30)
The Dodecane/PXE (70/30) mix appeared translucent at 4°C. Test LDPXETI produced a transmission reading of 13%. This was a somewhat surprising result considering that the mix appeared “milky” visually. If there were compounds precipitating out of solution, this output value may be due significantly to scattering and reflection off the walls of the tube. No beam spot was observed during the screen test.

7.2 Temperature Analysis
Contrary to expert predictions, a change in temperature from ~22°C to 4°C did have a measurable and significant effect on the transmissivity of the LS candidates tested and the corresponding attenuation length. The dependence of the attenuation length on temperature would disqualify LS candidates using pseudocumene or PXE.

The only LS candidate that shows promise is LAB (N501). As noted above, the calculated attenuation length is 4.5 m at a temperature of 4°C. This is below the requirements of the proposed detector with a target of 20 m. This LAB mix deserves further investigation.

It is expected that with column filtering alone, among many purification options available it should be reasonable to achieve similar results to those demonstrated by Minfang Yeh at Brookhaven on LAB as shown in Figure 7.2 below.

The criterion of a 20 m attenuation length in the proposed detector would require a 4X-5X improvement over the present value. Were purification to improve the attenuation in our LAB samples at half of what Minfeng demonstrated, our
attenuation length goal would be reached. Were his results achieved, we would have an attenuation length of 45 m, well beyond that required.

Figure 7.2: In this spectrophotometer plot of absorption vs. wavelength there is a marked improvement in the absorption after purification for wavelengths below 420 nm. As noted by the arrow; the absorption at 410 nm is 0.11 for the base sample – while that for the purified sample is ~ 0.001.

Experienced researchers familiar with filtering oil samples note that they are often somewhat of a mix of molecular types, and that some of these molecules may precipitate at low temperatures. In fact, string-like structures were observed in preliminary tests run on LS samples cooled in a freezer.

It is well understood in chemistry that precipitates form around particulate “seeds” at cloud point. By purification using silica or alumina gel columns these precipitate seeds can be removed.

The precipitation point for any compound is temperature dependent. Like moisture precipitating out of a cloud, long carbon chain molecules that may begin to change phase will remain in solution in liquid scintillators over a 20°C change in temperature with the “seeds” removed.

Filtering will produce a two fold benefit. First; removing particulate contamination will improve the attenuation length of the LS candidates at room temperature by as much as an order of magnitude. Secondly; by removing the “seeds” for precipitation, the temperature dependence of each LS may be mitigated and possibly eliminated.
Chapter 8

Application of findings

8.1 Optical properties stability
Schlieren “the German term for striations or inhomogeneities”; was applied by Toeper in 1864 to the refractive index inhomogeneities in a transparent fluid region. These index of refraction gradients are usually produced by density gradients, temperature gradients, or concentration of one species into the others within the sample [27].

Schlieren effects were evident in each sample tested until the sample reached optical thermal equilibrium. As the tests done have shown, in the extreme, Schlieren effects may be responsible for the total loss of light within the test system. In each of the other cases, Schlieren was a limiting agent in determining the amount of time required for each test sample to reach equilibrium.

At 4°C, even waiting an extended time for candidates such as pseudocumene, the KamLAND mix, and the mixed PXE to reach thermal equilibrium did not improve results. This result suggests changes in the sample density beyond those due solely to temperature gradients. These changes may be a product of disassociation between the molecules in solution in the liquid scintillator candidates. This would be consistent with the other noted causes of Schlieren.

8.2 LS selection
LAB N501 is the best candidate among those tested in meeting the all of the criterion given in the research impetus. The safety risks posed by a detector using LAB are nominal under the environmental conditions of deployment and during testing. LAB has excellent feasibility (see Table 1). The physical properties of LAB are adequate in meeting the required criteria. LAB N501 posted the only positive results for meeting the required optical properties. Finally, the contamination risk is among the lowest of all LS candidates tested. It is anticipated that the factory shipped N501 will be sufficient to produce positive test results in an ocean deployment test.

Purification will be a necessary next step in determining the true ability of LAB to perform as the LS in the proposed 20 m diameter detector. It is expected that purification of the LAB will improve the attenuation length of 405 nm light in the LAB from 4.5 m at 4°C to at least the detector criteria of 20 m.

8.3 Ocean deployment test
In an ocean deployment test any LS candidate used must be pre-chilled to at least 4°C. Cooling the scintillator will mitigate the time it will take for a one kilotonne test volume to come to equilibrium. For a one meter diameter LS test volume, “days”
would be required for the sample to come to optical equilibrium from an ambient
temperature (~25°C) to 4°C at 4 km depth.

One kilotonne of LS cooled to 4°C would require a method for cooling it as well as
an insulated container prior to deployment. The deployment process would require
minimizing the time in filling the test detector vessel and deployment time. Also any
process to transfer material must minimize the amount of contamination that may be
introduced. Contamination would reduce the light yield as well as the effective
attenuation length.

8.4 Recommended LS test system improvements

8.4.1 Test tube anodizing
Anodizing the inside of the SS test system tube black would reduce signal that may
be reflecting off of the inside of the tube walls. Any light that is scattered inside of
the test system may be reflected forward to the detector with the present surface.
Reflected light would skew the received output signal higher. The size of the detector
and the minimum required attenuation length predicate a definitive measurement for
light absorption.

8.4.2 LS sample purification
Purification of the LS candidates is of critical importance. Further testing is
incumbent on the outcome of any attenuation length measurements for LS’s at 4°C.
If attempts at improving the attenuation length of LAB 501 are unsuccessful at 4°C
for example, the Hanohano project’s feasibility becomes jeopardized. It is expected
that column filtering would produce immediate positive results in naming a LS
candidate for Hanohano.

8.4.3 10-cm Spectro-Photometer LS testing
Using a 10-cm spectro-photometer to test each sample would allow for verification of
the tests using the SS system. It would also allow for comparing filtered and
unfiltered samples for frequency dependence over the whole visible spectrum. Third,
these measurements could be compared with the results of Minfang Yeh on LAB and
pseudocumene, for example.

A 10-cm cell system is preferred over the 1-cm cell system due to the order of
magnitude difference in the verifiable attenuation length measurement. Minfang Yeh
noted that his recorded attenuation length was in fact limited by using a 10-cm cell.

8.4.4 Refrigeration System
Ideally, all test measurements could be performed in a test environment where there
were no temperature variations in any part of the test system. This would require the
use of a large temperature controlled room – a refrigerator large enough to contain the
whole SS test system apparatus including the optical bench. This suggestion must be
carefully considered for feasibility, benefit, and cost effectiveness.
### Appendix 1

**Scintillator Optical Test: SS System w/ Nitrogen fill**

<table>
<thead>
<tr>
<th>Test Run</th>
<th>Baseline</th>
<th>Ambient Temp</th>
<th>Test Temp</th>
<th>Test output</th>
<th>Post-Baseline</th>
<th>Post-Temp</th>
</tr>
</thead>
<tbody>
<tr>
<td>LN2T1</td>
<td>4.48 µA</td>
<td>21.9°C</td>
<td>0.5°C</td>
<td>4.47 µA</td>
<td>4.47 µA</td>
<td>21.8°C</td>
</tr>
</tbody>
</table>

**Scintillator Optical Test: DODECAN**

<table>
<thead>
<tr>
<th>Test Run</th>
<th>Baseline</th>
<th>Ambient Temp</th>
<th>Test Temp</th>
<th>Test output</th>
<th>Post-Baseline</th>
<th>Post-Temp</th>
</tr>
</thead>
<tbody>
<tr>
<td>GDT2</td>
<td>74.6 nA</td>
<td>21.6°C</td>
<td>0.3°C</td>
<td>35.8 nA</td>
<td>72.6 nA</td>
<td>21.4°C</td>
</tr>
<tr>
<td>GDT3</td>
<td>164.6 nA</td>
<td>22.1°C</td>
<td>0.3°C</td>
<td>83.3 nA</td>
<td>161.1 nA</td>
<td>22.2°C</td>
</tr>
<tr>
<td>BDT1</td>
<td>35.5 nA</td>
<td>21.4°C</td>
<td>0.3°C</td>
<td>26.0 nA</td>
<td>—</td>
<td>21.3°C</td>
</tr>
<tr>
<td>BDT2</td>
<td>149.1 nA</td>
<td>22.2°C</td>
<td>0.3°C</td>
<td>68.0 nA</td>
<td>144.6 nA</td>
<td>21.8°C</td>
</tr>
<tr>
<td>BDP1</td>
<td>168.6 nA</td>
<td>20.8°C</td>
<td>22.6°C</td>
<td>153.8 nA</td>
<td>167.1 nA</td>
<td>21.5°C</td>
</tr>
<tr>
<td>BDP2</td>
<td>156.1 nA</td>
<td>21.3°C</td>
<td>22.7°C</td>
<td>151.7 nA</td>
<td>156.0 nA</td>
<td>21.2°C</td>
</tr>
</tbody>
</table>

**Scintillator Optical Test: PSEUDOCUMENE**

<table>
<thead>
<tr>
<th>Test Run</th>
<th>Baseline</th>
<th>Ambient Temp</th>
<th>Test Temp</th>
<th>Test output</th>
<th>Post-Baseline</th>
<th>Post-Temp</th>
</tr>
</thead>
<tbody>
<tr>
<td>B1PP1</td>
<td>83.0 nA</td>
<td>21.6°C</td>
<td>21.6°C</td>
<td>79.6 nA</td>
<td>80.0 nA</td>
<td>21.4°C</td>
</tr>
<tr>
<td>B1PT1</td>
<td>73.7 nA</td>
<td>21.1°C</td>
<td>0.4°C</td>
<td>0.3 nA</td>
<td>49.0 nA</td>
<td>22.2°C</td>
</tr>
</tbody>
</table>
### Scintillator Optical Test: **KAMLAND MIX (80/20)**

<table>
<thead>
<tr>
<th>Test Run</th>
<th>Baseline</th>
<th>Ambient Temp</th>
<th>Test Temp</th>
<th>Test output</th>
<th>Post-Baseline</th>
<th>Post-Temp</th>
</tr>
</thead>
<tbody>
<tr>
<td>B1KP1</td>
<td>174.0 nA</td>
<td>22.3°C</td>
<td>22.4°C</td>
<td>245.0 nA</td>
<td>211.5 nA</td>
<td>21.4°C</td>
</tr>
<tr>
<td>B1KT1</td>
<td>235.5 nA</td>
<td>22.4°C</td>
<td>4.3°C</td>
<td>----</td>
<td>----</td>
<td>----</td>
</tr>
<tr>
<td>B1KT2</td>
<td>151.5 nA</td>
<td>20.5°C</td>
<td>4.0°C</td>
<td>15.8 nA</td>
<td>----</td>
<td>----</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>4.6°C</td>
<td>27.5 nA</td>
<td></td>
<td></td>
</tr>
<tr>
<td>B1KP2</td>
<td>265.5 nA</td>
<td>19.1°C</td>
<td>19.2°C</td>
<td>289.6 nA</td>
<td>274.5 nA</td>
<td>19.2°C</td>
</tr>
</tbody>
</table>

### Scintillator Optical Test: **LAB (540L)**

<table>
<thead>
<tr>
<th>Test Run</th>
<th>Baseline</th>
<th>Ambient Temp</th>
<th>Test Temp</th>
<th>Test output</th>
<th>Post-Baseline</th>
<th>Post-Temp</th>
</tr>
</thead>
<tbody>
<tr>
<td>B1540LP1</td>
<td>369.0 nA</td>
<td>24.6°C</td>
<td>24.6°C</td>
<td>356.0 nA</td>
<td>358.0 nA</td>
<td>24.5°C</td>
</tr>
<tr>
<td>L540LP2</td>
<td>2.79 µA</td>
<td>20.7°C</td>
<td>20.7°C</td>
<td>2.61 µA</td>
<td>2.78 µA</td>
<td>20.7°C</td>
</tr>
<tr>
<td>L540LT1</td>
<td>2.95 µA</td>
<td>20.4°C</td>
<td>5.3°C</td>
<td>0.54 µA</td>
<td>2.66 µA</td>
<td>20.6°C</td>
</tr>
</tbody>
</table>

### Scintillator Optical Test: **LAB (N501)**

<table>
<thead>
<tr>
<th>Test Run</th>
<th>Baseline</th>
<th>Ambient Temp</th>
<th>Test Temp</th>
<th>Test output</th>
<th>Post-Baseline</th>
<th>Post-Temp</th>
</tr>
</thead>
<tbody>
<tr>
<td>LN501P1</td>
<td>3.48 µA</td>
<td>21.4°C</td>
<td>21.4°C</td>
<td>3.53 µA</td>
<td>3.52 µA</td>
<td>21.4°C</td>
</tr>
<tr>
<td>LN501T1</td>
<td>3.52 µA</td>
<td>21.4°C</td>
<td>4.0°C</td>
<td>2.67 µA</td>
<td>3.55 µA</td>
<td>17.8°C</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>6.0°C</td>
<td>2.90 µA</td>
<td>3.55 µA</td>
<td>17.8°C</td>
</tr>
</tbody>
</table>
### Scintillator Optical Test: **PXE**

<table>
<thead>
<tr>
<th>Test Run</th>
<th>Baseline</th>
<th>Ambient Temp</th>
<th>Test Temp</th>
<th>Test output</th>
<th>Post-Baseline</th>
<th>Post-Temp</th>
</tr>
</thead>
<tbody>
<tr>
<td>LPXEP1</td>
<td>1.74 μA</td>
<td>23.2°C</td>
<td>23.2°C</td>
<td>1.62 μA</td>
<td>1.69 μA</td>
<td>23.1°C</td>
</tr>
<tr>
<td>LPXET1</td>
<td>1.69 μA</td>
<td>23.1°C</td>
<td>2.0°C</td>
<td>0.66 μA</td>
<td>1.67 μA</td>
<td>21.1°C</td>
</tr>
</tbody>
</table>

### Scintillator Optical Test: **DODECANE/PXE (70/30)**

<table>
<thead>
<tr>
<th>Test Run</th>
<th>Baseline</th>
<th>Ambient Temp</th>
<th>Test Temp</th>
<th>Test output</th>
<th>Post-Baseline</th>
<th>Post-Temp</th>
</tr>
</thead>
<tbody>
<tr>
<td>LPXEP1</td>
<td>1.74 μA</td>
<td>23.2°C</td>
<td>23.2°C</td>
<td>1.62 μA</td>
<td>1.69 μA</td>
<td>23.1°C</td>
</tr>
<tr>
<td>LPXET1</td>
<td>1.69 μA</td>
<td>23.1°C</td>
<td>2.0°C</td>
<td>0.66 μA</td>
<td>1.67 μA</td>
<td>21.1°C</td>
</tr>
</tbody>
</table>
Appendix 2

Bi-Ocular Scintillator Temperature Evaluation

All of the considered scintillator mixes passed the visual clarity test at zero degrees Celsius, and 1 atm. of pressure. Our working environment of 4°C thus will not eliminate any mix from consideration.

Proposed Mixes:
1. Dodecane, Pseudocumene (70/30)
2. Dodecane, Pseudocumene (80/20)
3. PXE
4. Dodecane, PXE (70/30)

Evaluation:

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Observed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dodecane:</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>clear</td>
</tr>
<tr>
<td>5</td>
<td>clear</td>
</tr>
<tr>
<td>0</td>
<td>clear</td>
</tr>
<tr>
<td>-10</td>
<td>clear, with a white/clear “fibrous weave” crystals along inside of beaker.</td>
</tr>
<tr>
<td>-20</td>
<td>white/clear fibrous crystals throughout beaker.</td>
</tr>
<tr>
<td>Dodecane, Pseudocumene (70/30)</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>clear</td>
</tr>
<tr>
<td>5</td>
<td>clear</td>
</tr>
<tr>
<td>0</td>
<td>clear</td>
</tr>
<tr>
<td>-3</td>
<td>clear</td>
</tr>
<tr>
<td>PXE</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>clear</td>
</tr>
<tr>
<td>0</td>
<td>clear</td>
</tr>
<tr>
<td>-5</td>
<td>clear</td>
</tr>
<tr>
<td>Dodecane, PXE (70/30)</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>clear</td>
</tr>
<tr>
<td>5</td>
<td>clear</td>
</tr>
<tr>
<td>0</td>
<td>clear</td>
</tr>
</tbody>
</table>

These tests were run in a freezer to eliminate water condensation from contaminating samples. When initial tests were run with an ice bath, each mix was observed to have temporary (~3 seconds) white strands in solution.
Appendix 3

Proposed LS Physical Parameters

Recipes of interest:
1. Dodecane, Pseudocumene (80, 20)
2. Dodecane, PXE (70, 30)

With these two primary recipes of interest the mechanical properties of Dodecane will dominate calculations for physical parameters and mechanical constraints.

Physical Properties:

<table>
<thead>
<tr>
<th></th>
<th>Pressure:</th>
<th>Temperature:</th>
<th>Density</th>
<th>Viscosity:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dodecane: (C(<em>{12})H(</em>{26}))</td>
<td>14.5 psi</td>
<td>298.15 K</td>
<td>0.7460</td>
<td>1.344 mPa</td>
</tr>
<tr>
<td></td>
<td>6091 psi</td>
<td>298.15 K</td>
<td>0.7715</td>
<td>2.139 mPa</td>
</tr>
<tr>
<td></td>
<td>14.5 psi</td>
<td>323.15 K</td>
<td>0.7274</td>
<td>0.911 mPa</td>
</tr>
<tr>
<td></td>
<td>6120 psi</td>
<td>323.15 K</td>
<td>0.7568</td>
<td>1.422 mPa</td>
</tr>
</tbody>
</table>

Where the Bulk Modulus is: B = 0.988 Gpa
Isothermal Compressibility: K = 9.9x10\(^{11}\) (sq.cms/dyne) @ 310.95 K, 1 atm.
Or K = 9.88x10\(^4\) (MPa\(^{-1}\))\(^{-1}\)

Cubic Thermal Expansion: Tc = 0.93x10\(^3\) K\(^{-1}\) @ 298 K

Pseudocumene: 14.5 psi 298.15 K 0.876 ----

Undocumented Pseudocumene properties may be reasonably inferred from Benzene which is the primary molecular component.

Benzene: (C\(_{6}\)H\(_{6}\))
Isothermal Compressibility: K = 8.09x10\(^{11}\) (sq.cms/dyne) @ 273 K, 1 atm.
K = 9.67x10\(^{11}\) (sq.cms/dyne) @ 298 K, 1 atm.
K = 5.07x10\(^{11}\) (sq.cms/dyne) @ 298 K, 1000 atm.
Or K = 9.66X10\(^4\) (MPa\(^{-1}\))\(^{-1}\) @ 298 K

Cubic Thermal Expansion: Tc = 1.14x10\(^3\) K\(^{-1}\) @ 298 K

Predicted Recipe Properties:

1. (80, 20) 14.5 psi 298 K 0.774
2. (70, 30) 14.5 psi 298 K 0.788

K = 8.0x10\(^{11}\) (sq.cms/dyne) @ 298 K, 6000 psi.
Bibliography


[20] DR Caudwell et al, *The Viscosity and Density of n-Dodecane and n-Octadecane at Pressures up to 200 MPa and Temperatures up to 473K,* Imperial College London, South Kensington Campus, (2003), 5

[21] DR Caudwell. *et al, The Viscosity and Density of n-Dodecane and n-Octadecane at Pressures up to 200 MPa and Temperatures up to 473K,* Imperial College London, South Kensington Campus, (2003), 6


[29] *LENA Homepage*, <http://www.e15.physik.tu-muenchen.de/research/lena>