10 Liquid Scintillator

10.1 Introduction

This chapter describes the details of the liquid scintillator, the active detector medium for the NOνA experiment. In this chapter we demonstrate that the composition and technical specifications for the scintillator have been determined, suppliers for the components have been identified, QC methods to test that the components and the blended scintillator meet technical specifications have been developed, a model for blending the scintillator components has been established, and a transportation model for delivery of the scintillator to the NOνA detector in Ash River is understood.

10.1.1 Liquid Scintillator Composition and Light Spectrum

Liquid scintillator is typically made up of four components: a primary scintillant that produces light in the UV when ionizing particles traverse it, waveshifters that downshift the UV photons to longer wavelength to facilitate absorption by the wavelength shifting (WLS) fibers, and a solvent to blend the components into a stable solution. The light production mechanism is shown below in Figure 10.1 with data from [1]. Pseudocumene [1,2,4-Trimethylbenzene], a benzene derivative with many uses in the plastics and paint industries, is the primary scintillant. Pseudocumene is excited by traversing ionizing particles and the photons that are given off as it de-excites are in the UV, as shown in Figure 10.1(a). Figures 10.1(b) and 10.1(c) show how PPO [2,5-diphenyloxazole] and bis-MSB [1,4-di(methylstyryl)benzene] shift the UV photons from the pseudocumene to the wavelength region where they can be absorbed by the dyes in the WLS fiber.

The normalized output spectrum of two commercial scintillator families is shown in Figure 10.2. The BC517 family is manufactured by Saint-Gobain (Bicron); the EJ-321 family is manufactured by Eljen Technologies. The NOνA baseline scintillator was engineered to have properties similar to the commercial liquid scintillator BC517P.

BC-517P [2] is made up of approximately 5% pseudocumene by weight as the primary scintillant in a 95% mineral oil base with small amounts of UV waveshifters and small amounts of anti-oxidants. Most of these components have been known since the 1950s [1]. These scintillators have a moderate light output, 28% of anthracene when fresh and 21% of anthracene when fully oxygenated [2,3]. The advantages of this mixture include stability, low cost, availability in large quantities, low toxicity, high flashpoint and low potential as an environmental hazard. Previous work has shown that this scintillator attacks neither wavelength shifting fiber nor PVC over lifetimes exceeding this experiment (see Chapter 11, Section 11.4).

To study the performance of liquid scintillator in the NOνA detector, a Monte Carlo program was written that simulates a prototype NOνA PVC extrusion cell with a loop of WLS fiber running through it. In this simulation, the composition and concentration of the TiO_2 in the PVC and the diameter of the fiber could be varied. Figure 10.3 shows a Monte Carlo calculation of the output spectrum of the NOνA baseline liquid scintillator in a prototype NOνA PVC cell in which the plastic is loaded with 18% anatase TiO_2 and the light is collected by a 0.7mm WLS fiber. This figure shows the output spectrum of the photons that are captured by the fiber and transmitted to the photodetector. For the baseline scintillator, the output spectrum peaks in the wavelength range 410 – 440 nm.

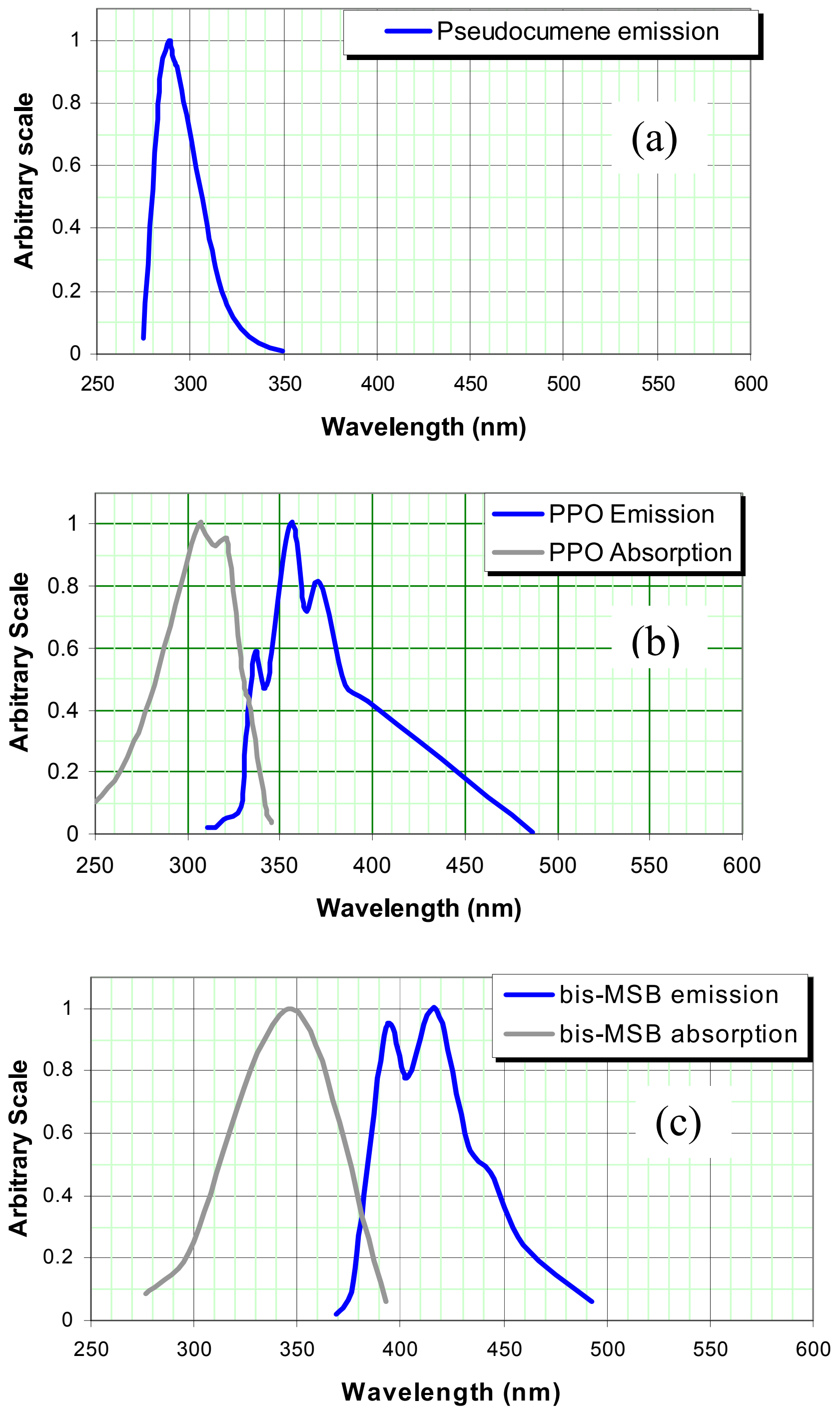

Fig. 10.1: Light production by liquid scintillator. The emission spectrum of the primary scintillant psueudocmene when traversed by an ionizing particle is shown in (a); the absorption and emission spectrum of the first waveshifter PPO is shown in (b); the absorption and emission spectrum of the second waveshifter bis-MSB is shown in (c).

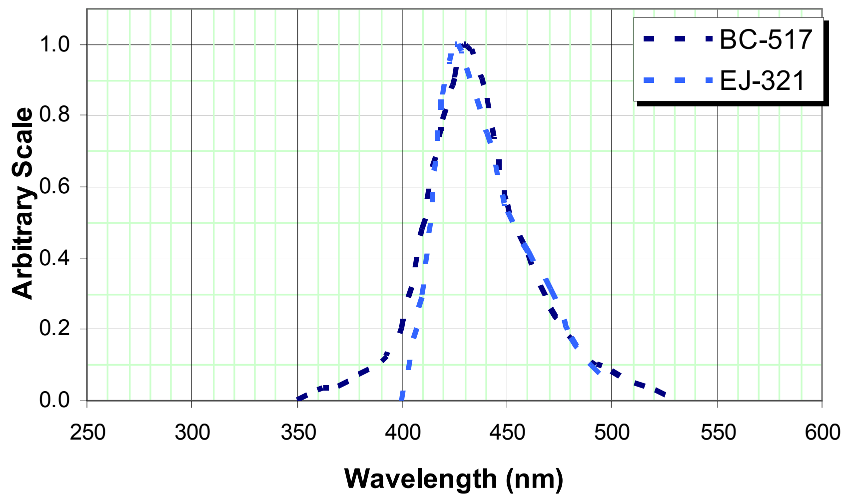

Fig. 10.2: The output spectrum of two commercial scintillators, Saint-Gobain (Bicron) BC-517 and Eljen Technologies EJ-321.

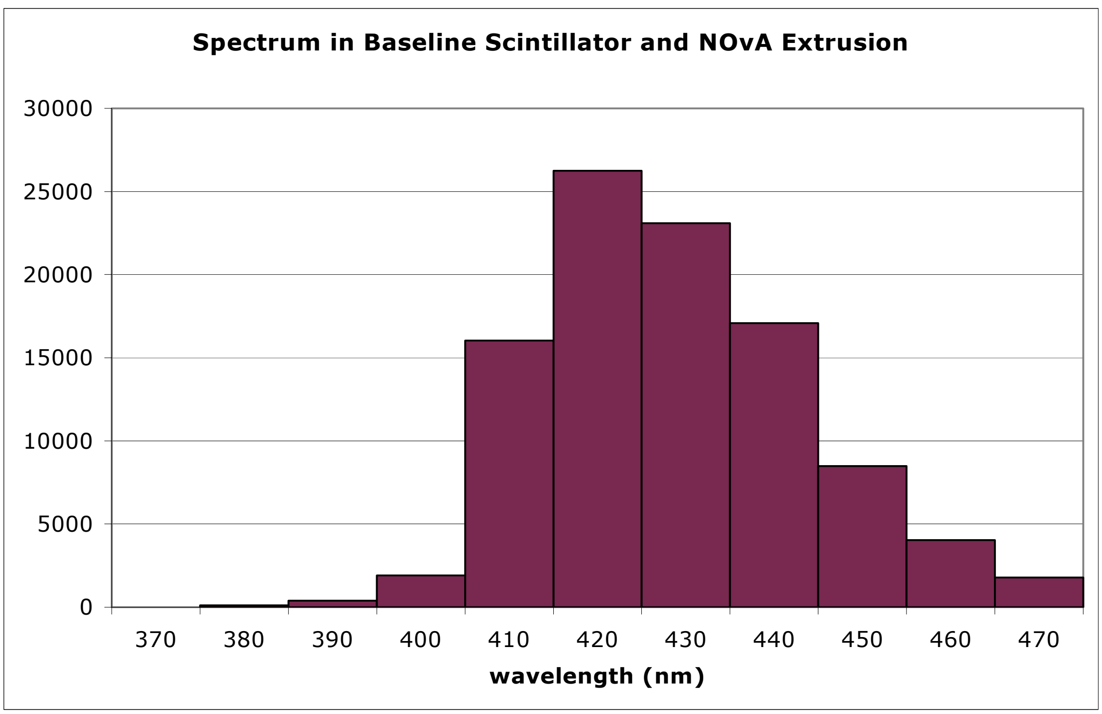

Fig. 10.3: A Monte Carlo calculation of the spectrum of photons absorbed by the fiber for the baseline NOνA liquid scintillator in a prototype NOνA PVC cell in which the plastic is loaded with anatase TiO_2 and the light is collected by a 0.7mm WLS fiber.

Unlike previous experiments that require photons to travel long distances to the photodetector, the NOνA design requires a scintillator with only a modest attenuation length for the relevant wavelengths of scintillator light. This requirement can be demonstrated by Monte Carlo simulations that compute the mean distance traveled by a photon before its absorption by the WLS fiber. Figure 10.4 shows the results of such a simulation for the prototype NOνA PVC extrusion loaded with anatase TiO2, and 0.7mm WLS fiber running through it. The figure shows that the mean distance traveled by a photon before absorption is only about 0.5 m, although there are tails extending out to much longer path lengths. In other high energy physics experiments, photons more typically must travel tens of meters before detection.

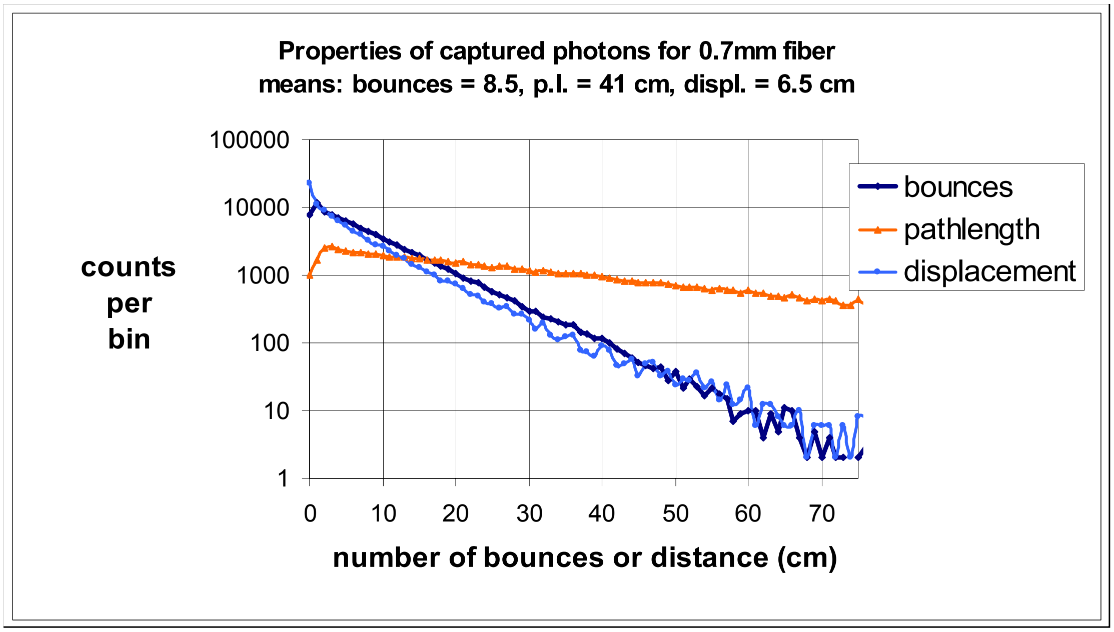

Figure 10.4. Distributions of the number of bounces seen by a photon before capture, the path length (p.l.) traveled before capture, and the displacement relative to its starting position computed by Monte Carlo. The Monte Carlo simulation uses the properties of baseline scintillator, a prototype NOνA PVC extrusion cell, and 0.7 mm WLS fiber.

10.2 Technical Design Requirements for the NOνA Liquid Scintillator

As discussed in Chapter 6, based on our current understanding of the performance of the PVC, WLS fiber and APDs, the technical performance specification can be achieved using liquid scintillator with a light output equivalent to 80% of Saint-Goban (Bicron) BC-517P [2]. The Technical Design Requirement on the light yield for the NOνA Liquid Scintillator is therefore 80% of the light yield of BC-517P.

The Technical Design Requirement on the attenuation length for the NOνA Liquid Scintillator was determined by the following considerations. R&D studies at Indiana University over several years have shown that liquid scintillator blended with 75% of the fluor concentration found in BC-517P is adequate to yield a light output equivalent 80% of BC-517P if the solvent for the fluors, mineral oil, has a long enough attenuation length. We therefore set about testing the light output of scintillators loaded with 75% of the fluors in BC-517P and blended with mineral oils having a broad range of attenuation lengths. Figure

10.5 shows the results of these studies. The methods used to measure light output and attenuation length are described in later sections of this chapter.

From Figure 10.5, we determined that mineral oil with an attenuation length of ≥ 5m when blended with 75% of the fluors in BC-517P meets the Technical Design Requirement on NOνA Liquid Scintillator light yield. We therefore adopt 5 m as the Technical Design Requirement on NOνA mineral oil.

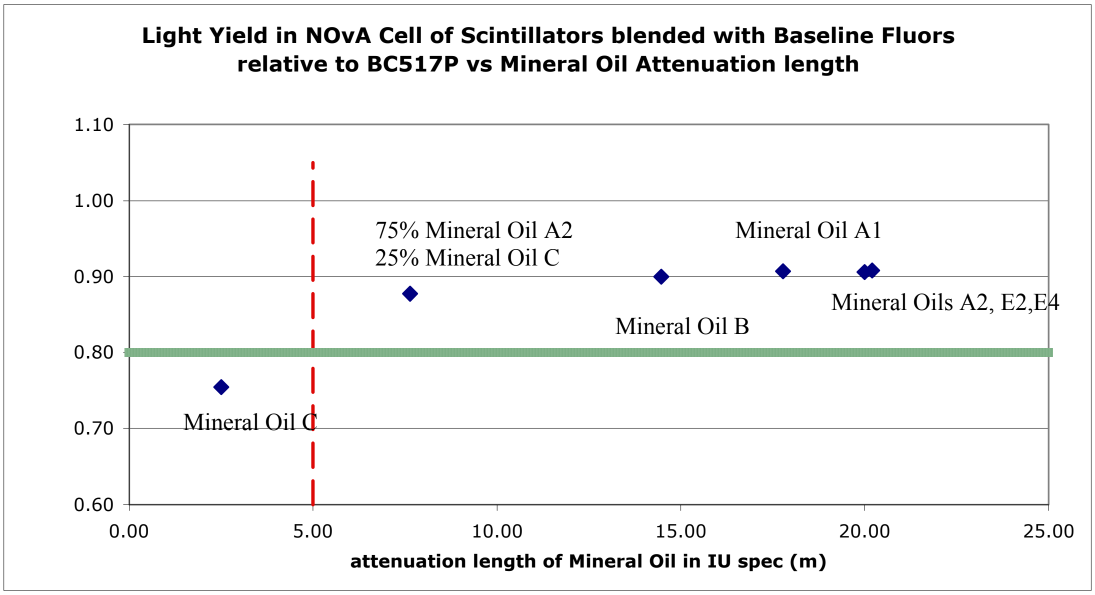

Fig. 10.5: Liquid scintillators blended with 75% of the fluors found in BC-517P and mineral oil solvents of varying attenuation lengths. The dashed line indicates the 5m Technical Design Requirement on the attenuation length of mineral oil.

Liquid scintillator is extremely non-conductive. Non-conductive fluids develop a net charge through the triboelectric effect during flow, which can under certain circumstances lead to spark discharge between the liquid and container or between non-bonded plumbing components. To mitigate potential sparking problems, the baseline scintillator has as an added component the anti-static agent Stadis-425 [4], which brings the scintillator conductivity up to safe levels. The technical requirement for conductivity has been taken from the recommendations given by the National Fire Protection Association (NFPA) [4]. NFPA safe practices dictate that the scintillator be made "semi-conducting", which is defined as "possessing a conductivity at least 100 picosiemens/meter".

The Technical Design Requirements for the NOνA Liquid Scintillator (NLS) are given in Table 10.1.

Scintillator Property	**Technical Design Requirements**
Liquid Scintillator Light Yield	NOνA Light Yield / Light Yield (BC-517P) ≥ 0.80
Attenuation Length of Mineral Oil Solvent	AttenuationLength ≥ 5 m @ 420 nm
Conductivity	≥ 100 picosiemens/meter

Table 10.1. Technical Design Requirements for the NOνA Liquid Scintillator (NLS)

10.3 Scintillator Composition

The NOνA detector liquid scintillator volume is 3,210,584 gallons. The components of the scintillator are given in Table 10.2 The anti-oxidant agent tocopherol (Vitamin E) is added in order to minimize the degradation in light due to oxygenation.

component	purpose	mass fraction	volume (gal)	tot mass (kg)
mineral oil	solvent	95.8%	3,082,145	9,917,109
pseudocumene	scintillant	4.1%	128,439	425,908
PPO	waveshifter #1	0.091%		9,373
bis-MSB	waveshifter #2	0.0013%		131
Stadis-425	antistatic agent	0.0003%		46.6
tocopherol (Vit.E)	antioxidant	0.0010%		104
Total		**100.0%**	**3,210,584**	**10,352,551**

Table 10.2. The composition of NOνA Liquid Scintillator

The component masses given in Table 10.2 are based on the nominal component densities given in the MSDS sheets.

During the R&D studies at Indiana University, many batches of liquid scintillator with the composition given in Table 10.2 were blended and tested. Of particular concern in these studies was the effect of various mineral oils on the liquid scintillator performance, mainly because liquid scintillator is mostly mineral oil. Below we describe the effects of several technical grade mineral oils purchased from multiple producers. These oils have been labeled A through E. Mineral oil from producer A has been adopted as the “Baseline Mineral Oil”. Three different shipments of this baseline mineral oil were studied and these oils have been labeled A1, A2, and A3. We also tested six different grades of mineral oil from producer E and these have been labeled E1-E6. Finally, we tested several blends of mineral oils made up of Mineral Oil A and Mineral Oil C. Our tests include both studies of the properties of the mineral oils themselves and the scintillators blended from them.

Pseudocumene for these studies was obtained from two suppliers and these have been labeled Pseudocumene A and Pseudocumene B. Both pseudocumenes have the same purity and chemical composition as measured at Indiana University using Gas Chromatography Mass spectroscopy (GCMS).

There is a sole US supplier for the waveshifters, Curtiss Laboratories in Pennsylvania. This company has worked successfully with other high energy physics experiments (MINOS, MINERνA) .

In this chapter scintillators blended with the fluor composition (pseudocumene + PPO + bis-MSB) given in Table 10.2 are called “Baseline Scintillator”; the fluor concentration is called “Baseline Fluors”.

10.3.1 Light Yield of NOνA Liquid Scintillator

Light yield tests were made in the Indiana University "NOνA cell". This is a 60 cm NOνA PVC cell that has walls loaded with 15% anatase TiO_2. The cell has two 0.8 mm diameter, 1.2 m long WLS fibers that are read out with a green extended phototube. NOνA cell measurements are made with cosmic ray muons.

The light yield of Mineral Oil A2 + Baseline Fluors is shown in Figure 10.6. As expected from Figure 10.5, these measurements demonstrate that the scintillator blended with Baseline Mineral Oil + Baseline Fluors meets the technical design requirement for light yield given in Table 10.1.

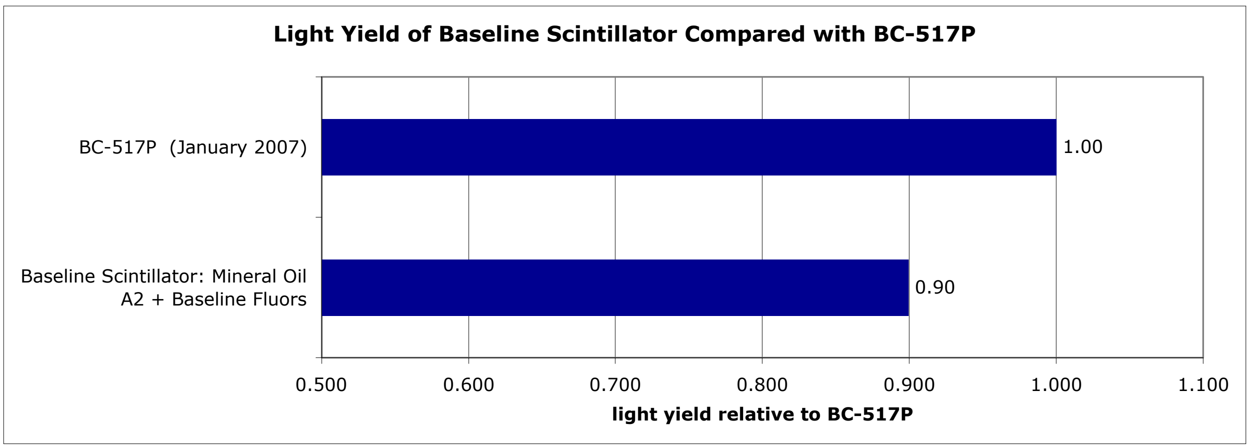

Fig. 10.6: Light yield of Mineral Oil A2 + Baseline Fluors compared with the light yield of commercial BC-517P in the NOνA cell. The measurements for BC-517P were made with scintillator received from Bicron in January 2007.

10.3.2 Conductivity

Conductivity measurements were made with a "Digital Conductivity Meter" (EMC Electronics Corp., Venice, FL). See Section 10.4.3 for a discussion of how the conductivity is measured. The conductivity of Mineral Oil A + Baseline Fluors is shown in Figure 10.7 as a function of Stadis-425 concentration.

The addition of 2 ppm of the anti-static agent Stadis-425 makes the scintillator semi-conducting. We choose 3ppm (instead of 2 ppm) because in large scale production, we want to guarantee that small errors in measuring out small quantities of Stadis-425 do not compromise the anti-static technical requirement in Table 10.1 for a large batch of scintillator. The addition of 3 ppM of Stadis-425 does not affect the light yield of the scintillator when measured in a NOνA cell as shown in Figure 10.8.

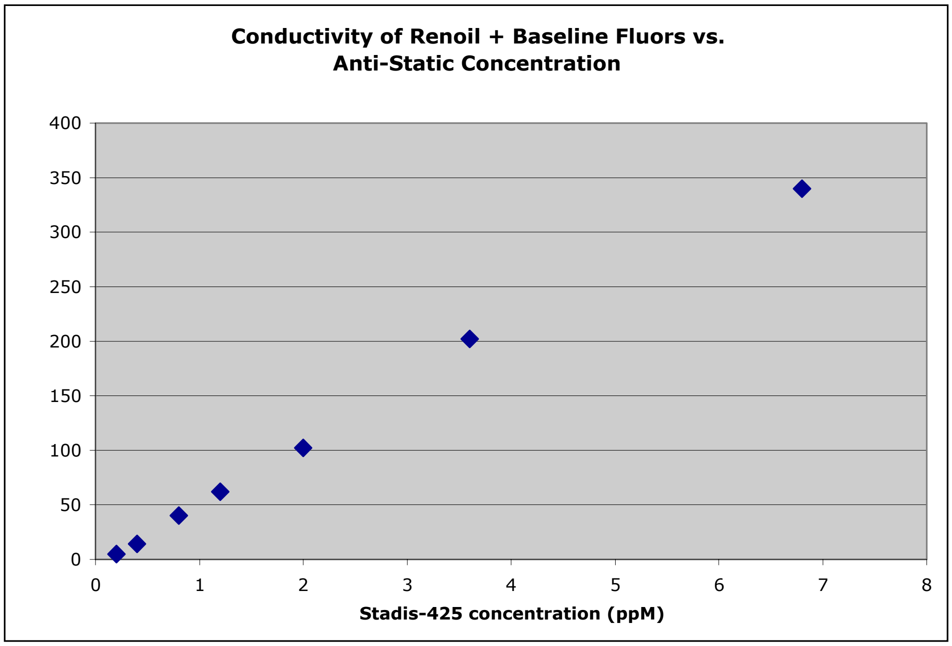

Figure 10.7. Conductivity of Mineral Oil A + Baseline Fluors as a function of the concentration of the anti-static agent Stadis-425 measured with the Digital Conductivity meter.

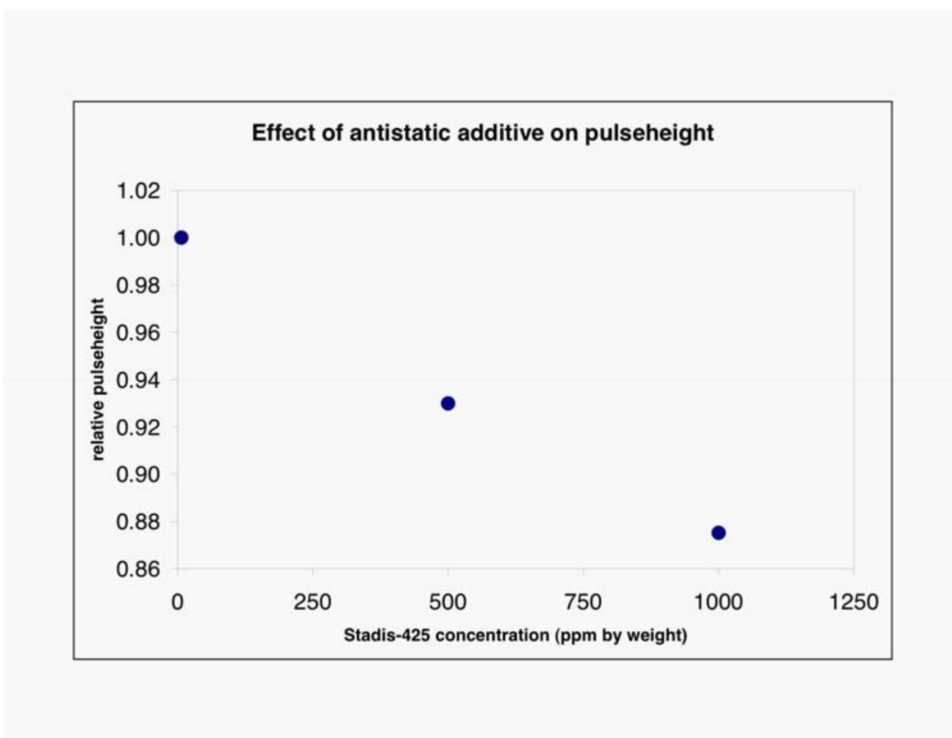

Figure 10.8. The effect of anti-static agent Stadis-425 on the light yield of baseline liquid scintillator. At the level recommended by the NFPA, 3ppM, there is no adverse effect on the light yield.

10.3.3 Anti-Oxidant

The anti-oxidant requirement for the scintillator was set by the Vitamin E concentration in the liquid scintillator used in the MACRO experiment [5].

10.3.4 Technical Specifications for Mineral Oil

The NOνA liquid scintillator constitutes 69% of the NOνA detector mass and is itself 96% mineral oil by weight. The mineral oil is inert and acts as a solvent for the scintillant and waveshifters. The primary performance feature of the mineral oil for NOνA is its attenuation length for light near 420 nm (see Figure 10.3). Mineral oil is a cost driver and the attenuation length of different grades and qualities of mineral oil must be compared to the costs of those different grades.

Experiments like MiniBooNE and MACRO used large quantities of mineral oil and mineral oil based liquid scintillator, respectively. But these experiments required mineral oil with very long attenuation lengths because of their detector technologies and geometries employed. The NOνA geometry is very different from those detectors since the light is collected locally by the wave shifting fiber. Since typical light path lengths in the NOνA scintillator are approximately 1 meter (Figure 10.4), long attenuation lengths are not required. This allows NOνA to relax the mineral oil requirements.

The most highly refined mineral oils are classified as food grade and meet the FDA requirements for consumption by humans. US Pharmacopoeia (USP) mineral oil is considered a heavy food grade mineral oil and has a large viscosity. National Formulary(NF) mineral oil is considered light food grade mineral oil and has a smaller viscosity. Higher viscosity means higher price. The step from USP to NF is about a 5 - 15% reduction in price. MiniBooNE used an NF mineral oil for their detector since an attenuation length of 20 meters was required.

Technical grade mineral oils are the next grade down from food grade. Technical grade mineral oils are not as highly refined as food grade mineral oils, but are approved by the FDA for indirect food contact and are typically used as lubricants for food processing machinery. The cost of technical grade mineral oil is less than NF mineral oils by about another 10 - 40%, depending on the viscosity.

The petroleum industry is not familiar with attenuation length as a specification and instead uses the Saybolt scale to characterize the color range of petroleum products including aviation fuels, kerosene, white mineral oils, hydrocarbon solvents and petroleum waxes. The Saybolt color index scale runs from -16 (darkest) to +30 (lightest) and, unfortunately, attenuation lengths above a few meters all lie at +30 on this scale. NOνA has blended scintillators using Saybolt +28 Technical Grade Mineral Oil and found these scintillator to be unacceptable for the experiment. Consequently, NOνA requires the development of a reliable and efficient method for determining the transmission properties of mineral oil in a way that does not demand a detailed understanding of attenuation length by the mineral oil producers.

Mineral oils are derived from petroleum feedstocks called Paraffinic Group II Base Oils. The American Petroleum Institute has defined the broad Base Oil group categories to create guidelines for licensing engine oils. All the groups cover a wide range of viscosities, but solvent-refined base oils typically fall into Group I , while hydroprocessed base oils fall into Group II. Hydroprocessing [6] is a way of adding hydrogen to the base oil at elevated temperatures in the presence of a catalyst to stabilize the most reactive components in the oil, improve the color, and increase the working life of the oil. Several hydroprocessing steps have been introduced in recent years to advance this industry: Hydrocracking was introduced in 1969 and adds hydrogen at high temperatures and pressures to crack feedstock molecules into smaller molecules. Catalytic dewaxing was added in 1984 and catalytically removes n-paraffins and other molecules with waxy side chains by cracking into smaller molecules. In 1993 hydroisomerization was added to this string of processing steps to reshape the n-pariffins and other molecules with waxy side chains into desirable compounds instead of cracking them completely away. This third step has resulted in distinctive Paraffinic Group II base oils which typically have no color.

An increasing fraction of the base oil manufacturers use this full range of technology, but as recently as 2003 less than half used the full three step package. This technology is proprietary and several competing ones now exist, each using different catalysts and different temperatures and pressures for the various steps. NOνA has obtained identically classified (and similarly priced) mineral oils with attenuation lengths for 420 nm light in the range 2m to 10m. While our current state of knowledge is incomplete, we suspect that the range is due to the technologies being applied.

From our R&D studies, it is clear that a Technical Grade (cheaper) oil can be found that will meet NOνA's needs.

The technical design requirements for the NOνA mineral oil are given in Table 10.3. These requirements can be met by Technical Grade mineral oils.

	Technical Design Requirement
attenuation length @420 nm	≥ 5 m
specific gravity @ 60/60F	0.850 < specific gravity < 0.865
viscosity @ 40 C	< 15 cSt
anti-oxidants	10 ppM
Water	< 30 ppM

Table 10.3. Technical Requirements on the NOνA Mineral Oil

The attenuation length requirement for the NOνA mineral oil @420 nm was discussed in Section 10.2 and shown on Figure 10.. The data for Figure 10.5 were obtained by blending the baseline fluor mix (c.f., Table 10.2) with mineral oils of differing attenuation lengths. The attenuation lengths of the mineral oils and blended scintillators were measured in the "Indiana University Spectrophotometer" (IU Spec) [5]. The narrow band measurements were made by passing the scintillator light through narrow band interference filters before reaching the PMT.

Figure 10.9 shows the attenuation lengths at 420 nm of the mineral oils used in the IU R&D studies. Scintillators were blended with several of the mineral oils shown in Figure 10.9 and Baseline Fluors. These scintillators were tested for light yield in the NOνA cell and the results of these tests are shown in Figure 10.10. These are the data used in Figure 10.5. The attenuation lengths of these blended scintillators were measured in the IU Spec and the results are shown in Figure 10.11.

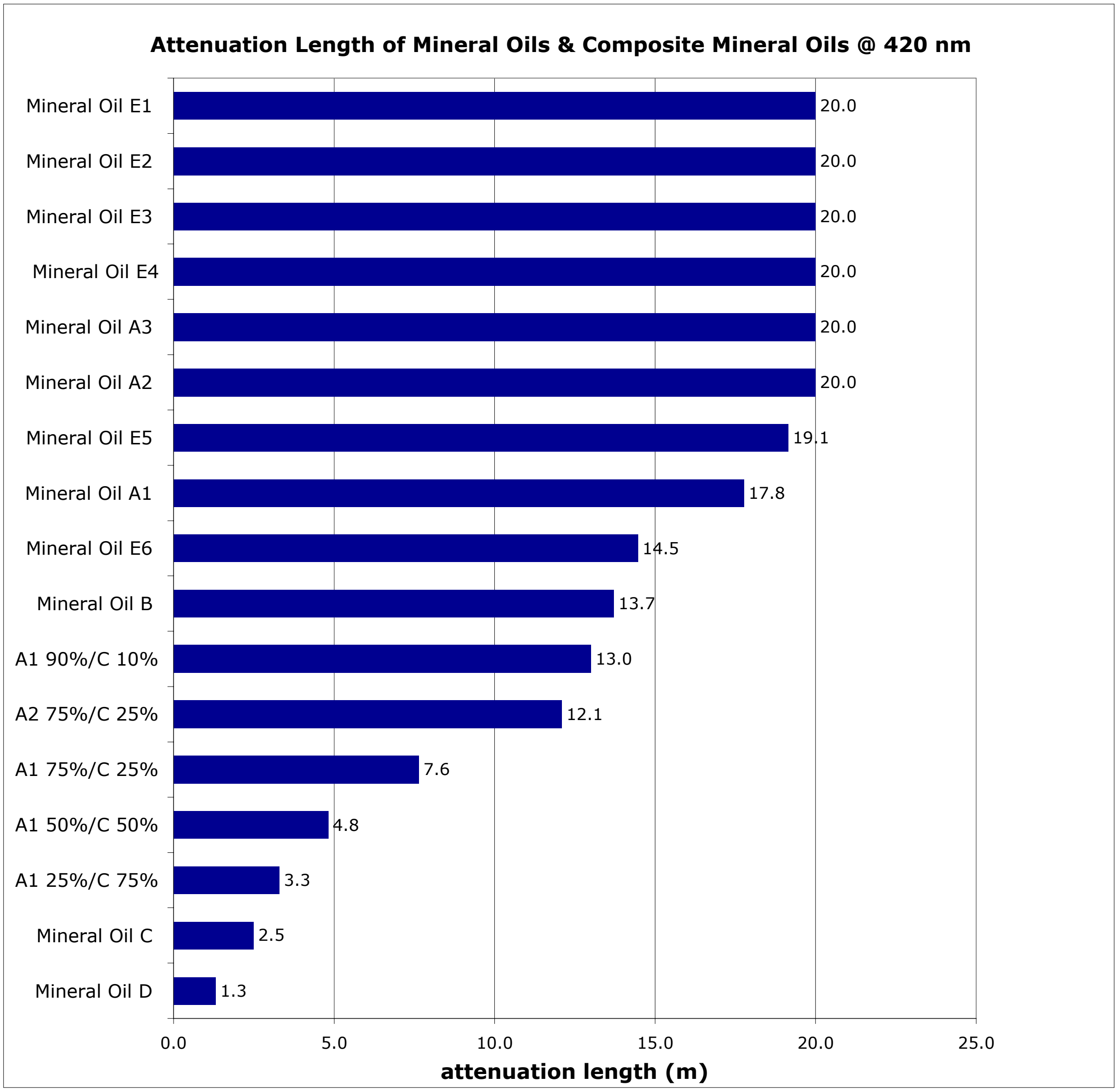

Fig. 10.9: Attenuation of mineral oils at 420 nm as measured in the IU Spec. Since the IU Spec does not accurately measure attenuation lengths > 20m, all measured attenuation lengths > 20m are shown as 20m.

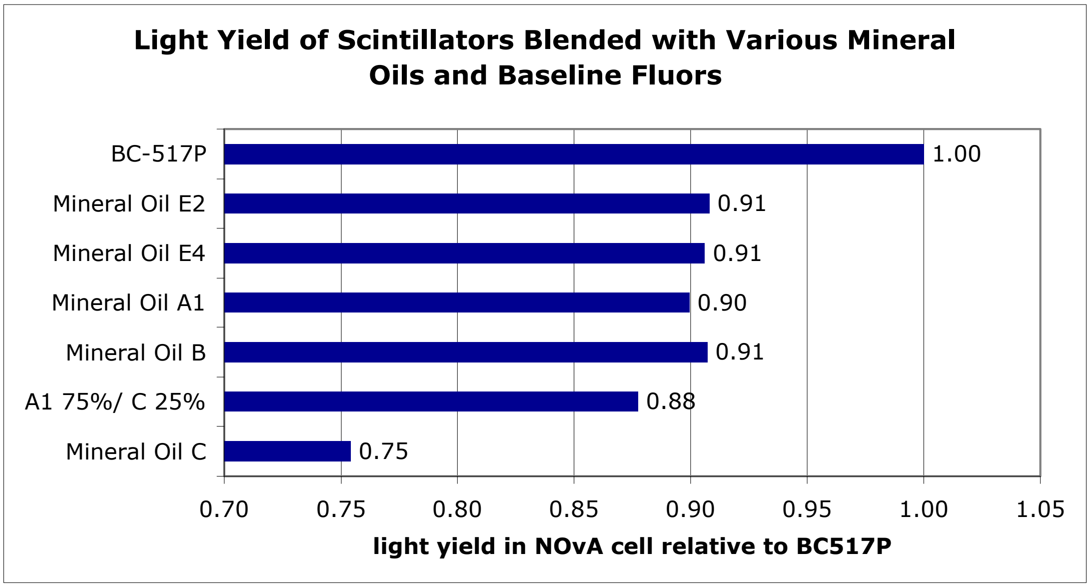

Fig. 10.10: Light yield of the scintillators blended with mineral oils shown in Figure 10.9 and Baseline Fluors. The light yield measurements were made in the NOνA cell.

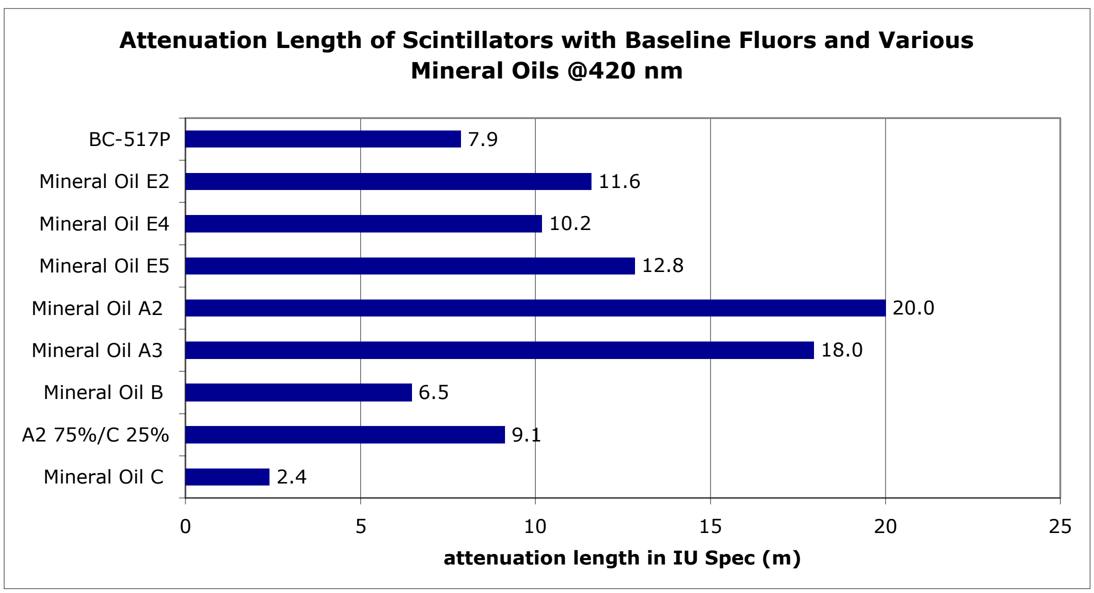

Fig. 10.11: Attenuation length of scintillators blended with mineral oils shown in Figure 10.9 and Baseline Fluors.

Additional mineral oil Technical Requirements:

- specific gravity – a narrow range of density distributes the scintillator uniformly throughout the detector, which in turn leads to a better understanding of fiducial mass. Producers routinely manufacture mineral oil to this standard.
- viscosity – higher viscosity mineral oil is more expensive and high viscosity mineral oil does not add value to this detector since it can be more difficult to pump.
- anti-oxidant – the anti-oxidant requirement for the scintillator was set by the Vitamin E concentration in the liquid scintillator used in the MACRO experiment [5]. Antioxidants are common additives in mineral oil and mineral-oil based liquid scintillators. They are used to stop the slow degradation occurring in long hydrocarbon chains and the radicals formed in this process react with oxygen dissolved in the liquid. The resulting product becomes discolored (yellowing) and is therefore detrimental to the transmittance of the liquid scintillator at 420 nm. The antioxidant essentially acts as a radical scavenger.
- water – technical specification taken from MiniBoone; industry standard; water settles out but takes up detector mass

10.3.5 Technical Specifications for Pseudocumene

Table 10.4 lists the technical specifications for the Pseudocumene.

	Technical Design Requirement
Purity	≥ 98%
specific gravity @ 60/60F	0.875 < specific gravity < 0.882
Clarity	< +25 Color Units measured on Pt-Co scale
Total Sulfur content	< 2.0 ppM

Table 10.4. Technical Requirements on the NOνA Pseudocumene

Source of the requirements in Table 10.4:

- purity – this requirement is the industry standard for pseudocumene production. When delivered to the toll blender, samples of pseudocumene are drawn and then sent to Indiana University where their purity will be measured using GCMS by the Indiana University Chemistry Department.
- specific gravity – this requirement is the industry standard for specific gravity.
- clarity – this requirement is the industry standard for clarity, as measured on the Pt-Co scale. The Pt-Co scale is measured by the Lovibond Tintometer;
- sulfur content – The Certificate of Analysis from the vendors lists the sulfur content in the pseudocumene. The presence of sulfur in the original distillate (fraction) is fairly common. NOνA plans to monitor the sulfur level reported by the manufacturer since it may potentially lead to light yield quenching and acidity variations.

Pseudocumene has been obtained from two suppliers. A GCMS analysis of the pseudocumene from the two suppliers shows that they both meet the Technical Design Requirements for purity. Figure 10.12 shows that scintillators blended with pseudocumene from the two suppliers meet the Technical Requirement on light yield. In this figure, the scintillators were blended with 100% of the fluor concentration found in BC-517P.

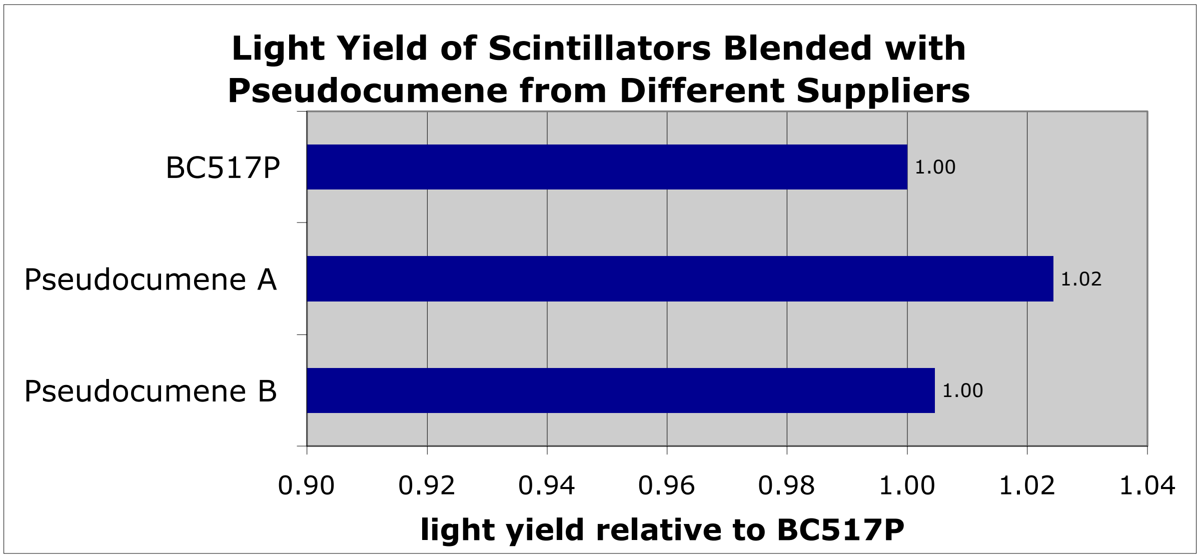

Fig. 10.12: Light yield of the scintillators blended with pseudocumene from two suppliers as measured in the NOvA cell. The scintillators blended with Mineral Oil A1 and 100% of the fluors found in BC-517P.

10.3.6 Technical Specifications for Scintillator Waveshifters

The technical specifications for the wavelength shifters have been presented to the vendors and are listed in Table 10.5. The Certificate of Analysis prepared by the manufacturer, Curtiss Laboratories, will include the results of these tests with the exception of the transmittance in toluene (1 g wavelength shifter in 100 mL toluene, measured in optical cells with 1-cm and 10-cm path lengths) which will be performed at Fermilab. The manufacturer will carry out a parallel test as a check. The NOvA experiment will repeat the check for melting point, appearance and odor at Fermilab. We will also perform new tests using ultraviolet (UV), Infrared (IR) and nuclear magnetic resonance (NMR) to better define potential impurities. Most of the impurities in the wavelength shifters are related to unreacted materials and solvents. The wavshifters are susceptible to the presence of impurities so that testing must ensure that the wavelength shifters used are over 99.6% pure. PPO was used as the primary wavelength shifter in the MINOS experiment and the manufacturer met the same technical specifications over a two year delivery schedule.

	Technical Design Requirement	
	PPO	bis-MSB
Melting point	71-73 °C	179-181 °C
Appearance	white powder	light yellow powder
Odor	Odorless	Odorless
Loss in drying	≤ 0.2%	≤ 0.2%
Residue	none	none
Purity	≥ 99.6%	≥ 99.6%
Tansmittance in Toluene	≥ 85% @ 370 nm	≥ 90% @ 420 nm

Table 10.5. Technical Requirements on the NOvA Waveshifters

10.4 Quality Assurance and Quality Control

The liquid scintillator is the heart of the NOνA experiment. In combination with the wavelength shifting fiber and PVC cells, the scintillator is crucial to the performance of the detector. We must be sure that the scintillator is free of impurities and properly blended. Quality Control (QC) and Quality Assurance (QA) are fundamental to the successful construction of this detector.

The problem with petroleum based products like mineral oil and pseudocumene is that they come out of oil fields from a variety of wells, so the base material is variable. In addition, mineral oil and pseudocumene are often distilled by many successive vendors via proprietary processes in a long supply chain. These products are blended by the vendors to meet specifications having little to do with our applications in high energy physics.

10.4.1 QA/QC Measurements of Attenuation Length using a Tintometer

NOνA needs a reliable and efficient method of measuring the attenuation length of mineral oil and blended scintillator. Currently, production plans call for monitoring the mineral oil attenuation length before shipping at the producer, on arrival at the toll blender, and before it is blended into scintillator. Once blended, the scintillator attenuation length will be monitored to assure that the blended scintillator meets the Technical Design Requirements.

Given the large volume of mineral oil required by NOνA and the necessity for a reliable and efficient method of measurement, our work has focused on the Lovibond PFX880 Tintometer shown in Figure 10.14 as the attenuation QC/QA device. The Lovibond tintometer makes transmission measurements from 400-700nm through a 6" glass cell. As will be shown below, the transmission measurements at 420 nm correlate well with the attenuation length at 420 nm in the range of interest to NOνA. Equally important, tintometer measurements on an individual sample take less than a minute to make.

Fig. 10.14: The Lovibond PFX880 Tintometer in the Indiana University lab. In the foreground are 6" optical glass cells.

10.4.2 QA Attenuation Length of Incoming Mineral Oil

In the production plan for the NOνA Liquid Scintillator, there are three stages at which the attenuation length of the mineral oil is tested: (1) at the production facility before shipping to guard against shipping the mineral oil from the producer that will be rejected at the blender, (2) at the blender to guard against accepting mineral oil that does not meet the technical requirements, and (3) at the blending tank, before adding the pseudocumene and waveshifters. These attenuation length measurements will be made with the tintometer and the results will be kept in the NOνA database.

Figure 10.5 shows that scintillators blended with the mineral oil having an attenuation length ≥ 5 m meet the Technical Design Requirements. In Figure 10.15 we plot the attenuation lengths of several of the mineral oils shown in Figure 10.9 vs. their transmission at 420 nm as measured in the tintometer. There is a clear separation (dashed line) between oils that have an acceptable attenuation length and those with an unacceptable attenuation length when measured in the tintometer. Figure 10.15 demonstrates that the tintometer is capable of making the QC mineral oil measurements required by the NOνA Liquid Scintillator production plan. In addition, QC procedures based on the tintometer are quick and cost effective.

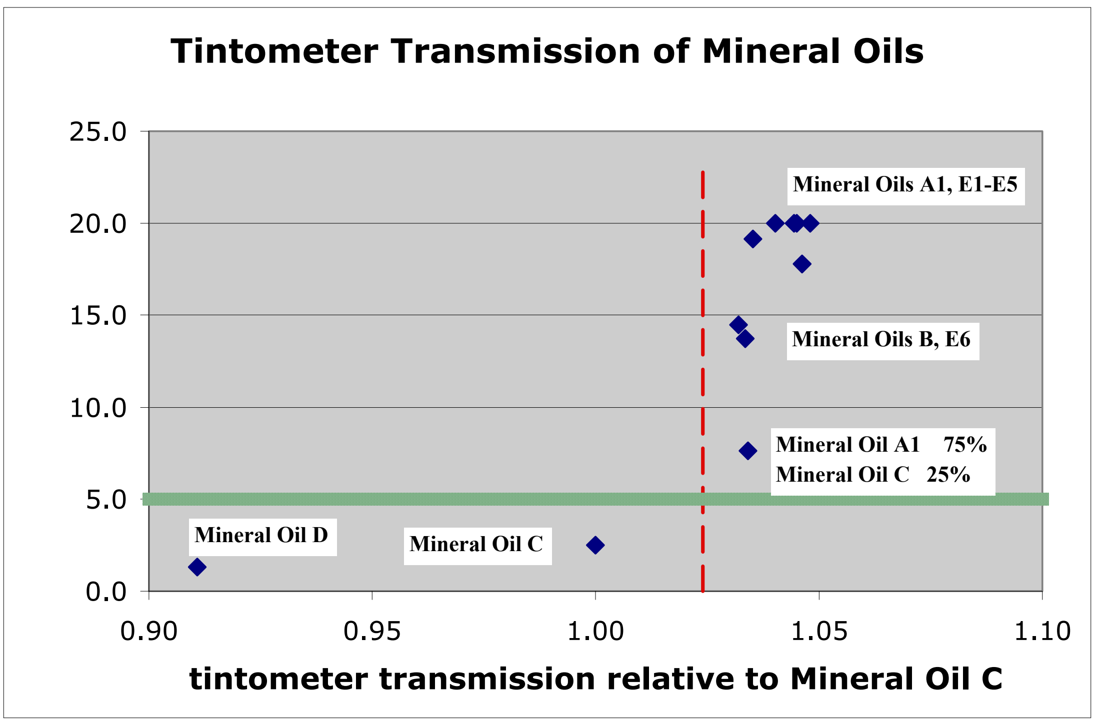

Fig. 10.15: The attenuation length (meters) of several of the mineral oils in Figure 10.9 as measured in the IU Spec vs. transmission measurements in the tintometer at 420 nm. The dashed line represents the approximate acceptance criterion for the attenuation length of mineral oil.

10.4.3 Alternative Method to QA/QC Mineral Oil Attenuation Length

In parallel with the proposed QA/QC methods that use the tintometer, NOνA is developing a fully automated instrument that makes an absolute measurement of attenuation length, without the use of reference standards, for both mineral oil and liquid scintillator. A prototype of this device, incorporating some features of the existing IU Spec is being evaluated to optimize measurement time, readout electronics performance, fluid column length and attenuation length resolution.

10.4.4 QC Attenuation Length of Outgoing Blended Scintillator

In the production plan for the NOνA Liquid Scintillator, there are two stages at which the attenuation length of the scintillator is tested: (1) at the blending facility before being shipped to Ash River and (2) at Ash River before being unloaded into the detector. The results of these tests will be kept in the NOνA database. For these QC measurements we again plan to use tintometer transmission measurements at 420 nm. In Figure 10.16 we plot the attenuation lengths of several of the blended scintillators shown in Figure 10.11 vs. their transmission at 420 nm as measured in the tintometer. Again there is a clear separation (dashed line) between oils that have an acceptable attenuation length and those with an unacceptable attenuation length when measured in the tintometer. As for mineral oil, we adopt the tintometer as the efficient and cost effective QC measurement apparatus to qualify the attenuation length of NOνA Liquid Scintillator.

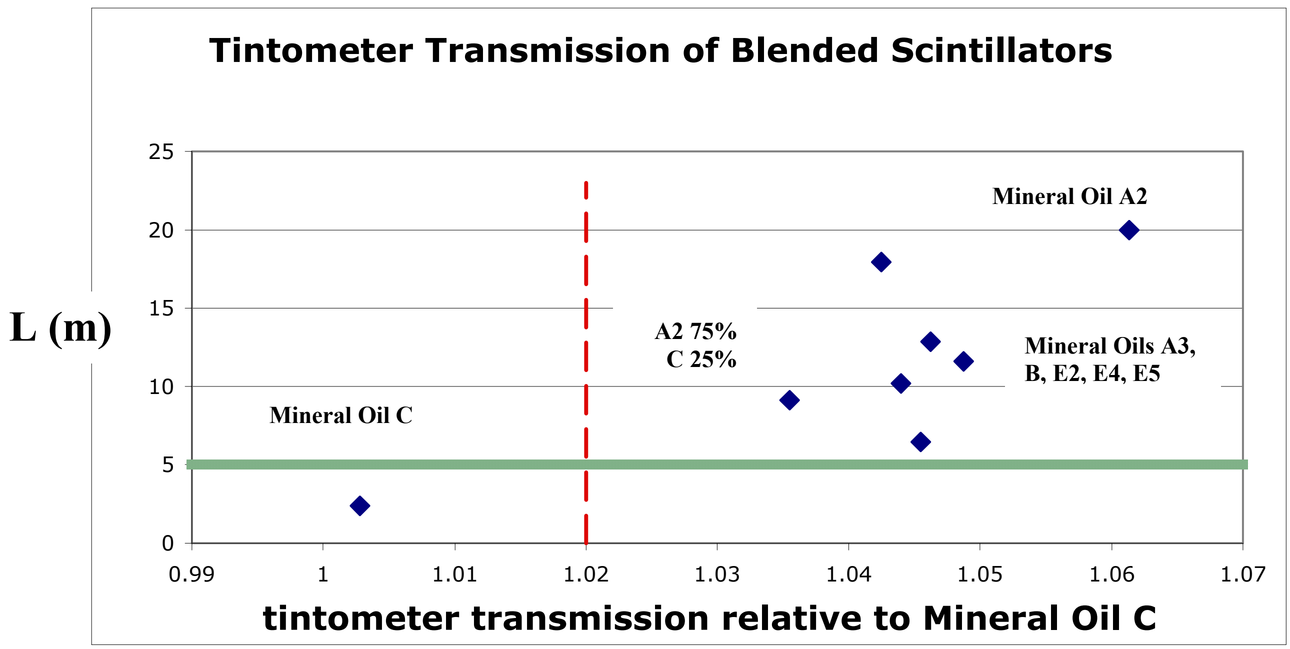

Figure 10.16. The attenuation length L (meters) of several of the scintillators in Figure 10.11 as measured in the IU Spec vs. transmission measurements in the tintometer at 420 nm. The dashed line represents the approximate acceptance criterion.

10.4.5 QA Incoming Pseudocumene

Samples of all incoming batches of pseudocumene will be drawn and shipped to Indiana University where they will be analyzed by gas chromatography-mass spectroscopy

(GCMS) to determine for compliance with the Technical Requirements on composition, purity and sulfur content in Table 10.4.

In addition, the pseudocumene will be tested with the tintometer for compliance with the Technical Requirement on clarity in Table 10.4. In the production plan for the NOνA Liquid Scintillator, there are two stages at which the clarity of the pseudocumene is tested: (1) at the blender to guard against accepting pseudocumene that does not meet the technical requirements and (2) at the blending tank before being blended with the waveshifters. The results of these tests will be kept in the NOνA database.

10.4.6 QA Incoming Waveshifters

The vendor will send a sample of each wavelength shifter (PPO and bis-MSB) from each batch manufactured to Fermilab. The vendor will provide a Certificate of Analysis for each lot. A series of tests will be performed at Fermilab. These tests will aim to verify some of the parameters measured by the manufacturer such as melting point, appearance and odor; and to check the spectral characteristics of each component such as UV transmittance under specific conditions, infrared (IR) and nuclear magnetic resonance (NMR) data. These tests can be completed in 1-2 days. The vendor will then be notified to proceed with the shipment to the location where the blending of the wavelength shifters and pseudocumene will take place. A similar procedure was used for the wavelength shifters used in the MINOS experiment. The results of these tests will be kept in the NOνA database.

10.4.7 QC Outgoing Scintillator Composition with an Alpha Source Test

A 1 μCi Am-241 α-source will be used to measure the light yield of liquid scintillator. Light yield in the "α-test" is correlated with scintillator composition. In the production plan for the NLS, there are two stages at which the light yield of the scintillator is tested: (1) at the blending facility before being shipped to Ash River and (2) at Ash River before being unloaded into the detector. The results of these tests will be kept in the NOνA database.

The test is performed as follows: A 7.5 cm diameter photomultiplier (PMT) is mounted vertically (window up) in a darkbox. Two commerical filters are placed on the PMT to block all but the useful spectrum wavelengths. Uniform size/shape bottles (inner volume ~3 cm diameter X ~8 cm high) are filled approximately half full with scintillator. One bottle contains a standard scintillator (BC-517P) and a second bottle contains the sample whose relative light output is to be determined. A special bottle lid has been manufactured that was fitted with a 1 μCi Am-241 α-source attached and this is placed on the bottle (standard or sample) to be measured. The bottle is placed on the PMT and the light detected by the PMT is recorded. The procedure requires that for each sample measurement, a standard measurement is made before and after to track drift.

The PMT output is then fed through an electronics chain: charge sensing preamplifier, linear (voltage) shaping amplifier, and MultiChannel Analyzer (MCA). The MCA spectrum shows a clear peak for the α energy deposited in the scintillator. Determining the peak (with peak-fitting software) gives a measurement of the light output. A direct comparison (ratio) of peak locations of standard and sample indicates the relative light yield of the sample.

Because the range of alphas in liquid scintillator is very short (order of 50-60 um), the α-source must be cleaned in soap and water, rinsed, and dried between measurements. The high voltage driving the PMT must also be kept within a very tight range (+- 0.5 V for a ~1000 V divider voltage) in order to minimize drift. For the above setup, the event rate is sufficient to collect a spectrum in 90 sec.

We have studied the results of the α-test during the R&D period and find that the light output measured is primarily correlated with the composition of liquid scintillator. To test this hypothesis further, we tested two samples of BC517P purchased from Bicron, one purchased in 2005 and another purchased in 2007, in which the composition is advertised to be equivalent. The chemical analysis of these two commercial scintillators in Table 10.6 shows that the two scintillators have virtually the same composition.

	"2005" BC517P	**"2007" BC517P**
component	**mass fraction**	**mass fraction**
mineral oil	94.4%	94.7%
pseudocumene	5.5%	5.2%
PPO	0.12%	0.12%
bis-MSB	0.0016%	0.0014%

Table 10.6. Composition of Two Batches of Commercial BC517P

On the other hand, as shown in Figure 10.17, the attenuation length of these two scintillators differ significantly.

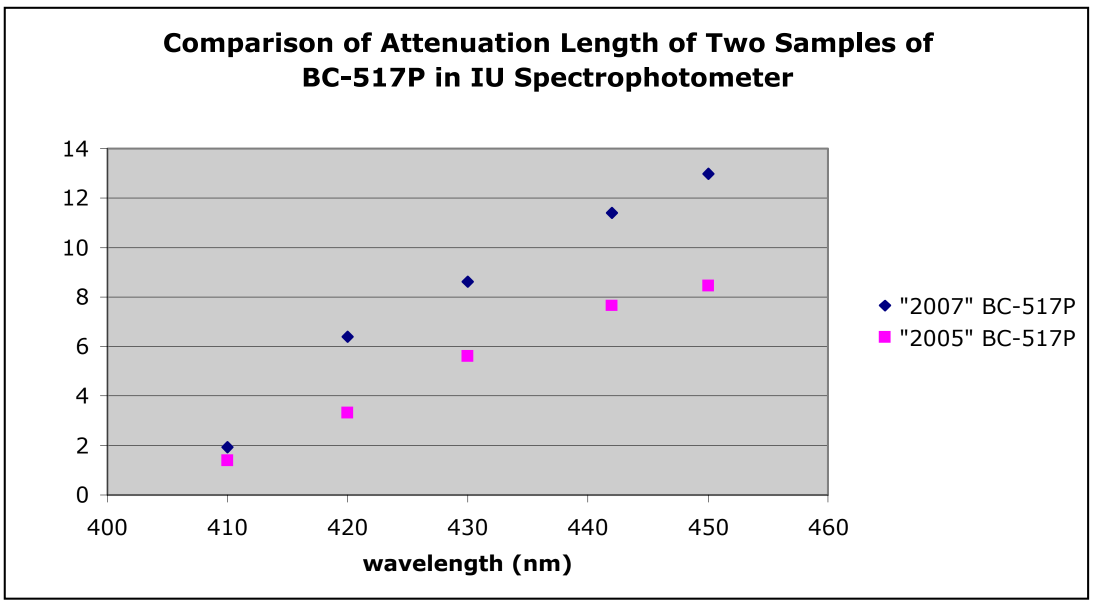

Fig. 10.17: Attenuation length of the two BC517P as measured in the IU Spec.

The α-test, if it is primarily a measure of composition, should give similar results for both scintillators. Figure 10.18 shows this expectation is met. These results are consistent with the α-test being primarily a measure of composition.

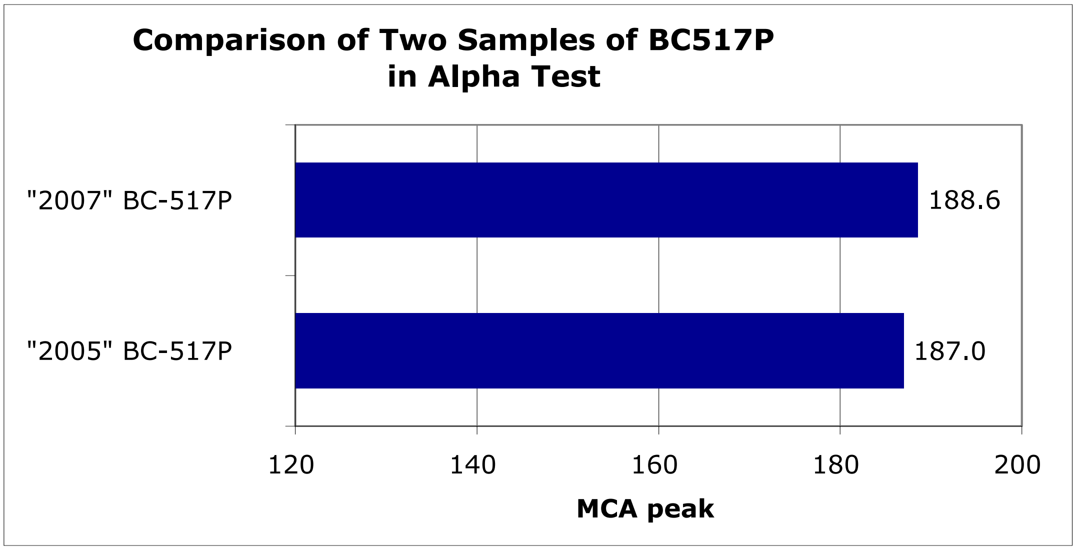

Fig. 10.18: α-test of the two BC517P scintillators produced at different times.

Finally, in Figure 10.19, we show the light yield of the two BC517P scintillators in the NOνA cell. Not surprisingly, scintillators with similar compositions but differing attenuation lengths will have different light yields in the detector.

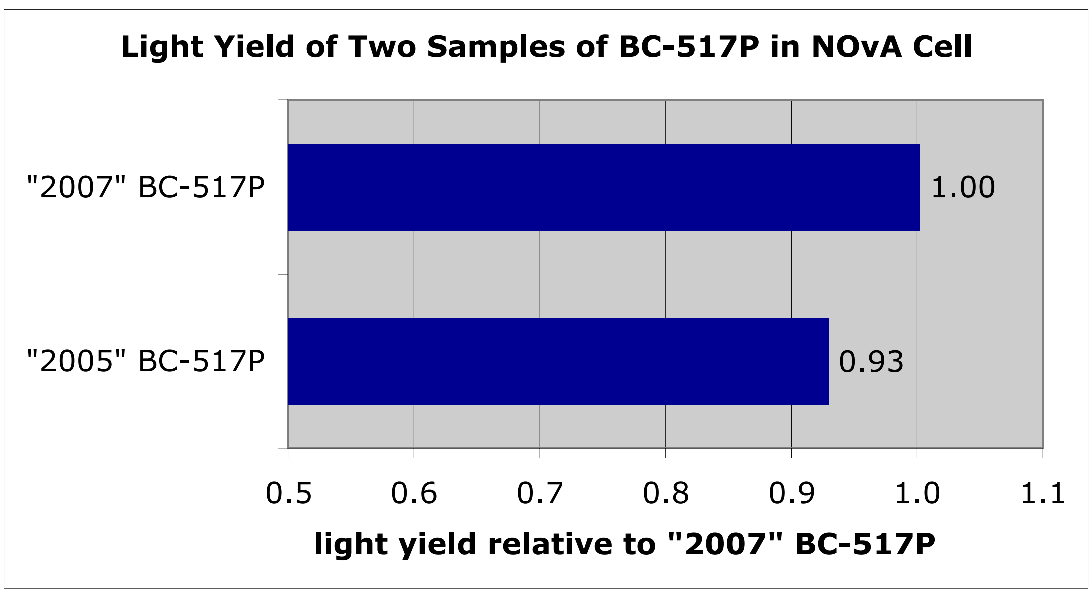

Fig. 10.19: Light yield of the two BC517P scintillators as measured in the NOνA cell.

As a second test of the α-test, we blended 4 scintillators with 100% of the fluors found in BC-517P and mineral oils with different attenuation lengths, and then compared these scintillators in the α-test. The attenuation lengths of these scintillators are shown in Figure 10.20. The differences in the attenuation length are clear.

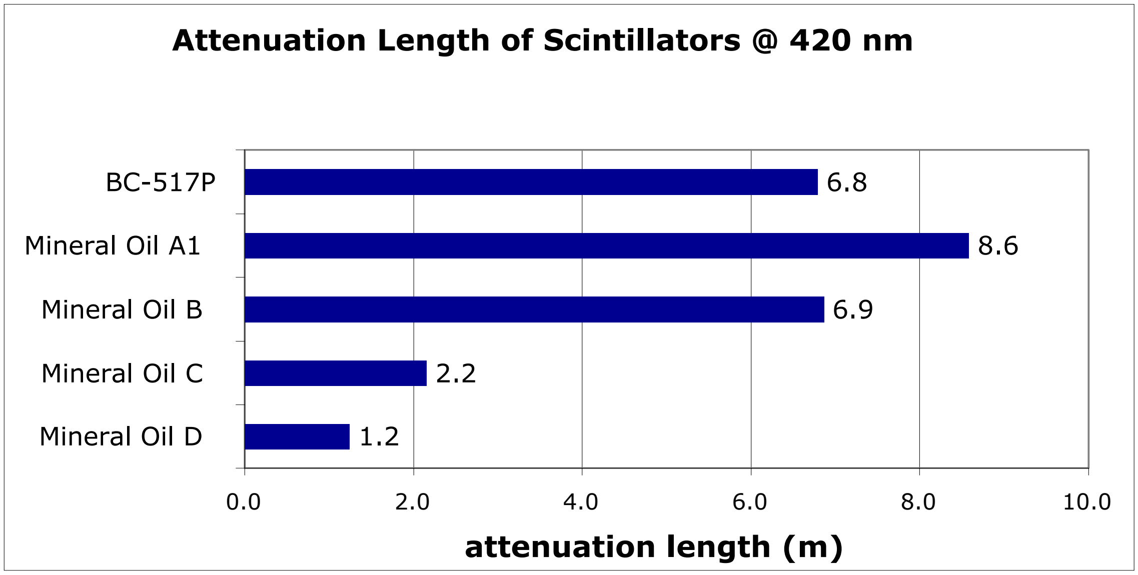

Fig. 10.20: Attenuation length of scintillators as measured in the IU Spec blended with the fluor concentration found in BC-517P and mineral oils with different attenuation lengths.

The results of the α-test for these scintillators are shown in Figure 10.21. This figure clearly shows that scintillators blended with mineral oils of very different attenuation lengths but with similar fluor concentrations give very similar results in the α-test. This test too demonstrates that the α-test is primarily a measure of composition.

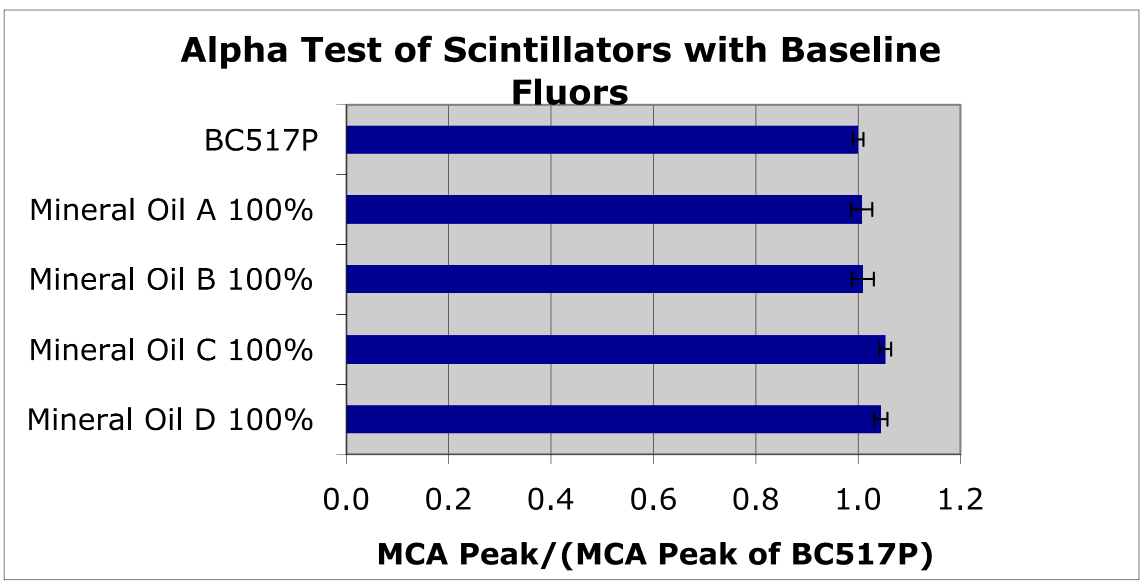

Fig. 10.21: α-test of the scintillators shown in Figure 10.19.

10.4.7.1. Precision of α-Test determination of composition

We next tested the precision to which we could measure the fluor concentration with the α-test. We blended scintillators with differing concentrations of fluors and Mineral Oil A1, and compared these scintillators with BC517P in the α-test. The results of these tests are shown in Figure 10.22. This figure shows that we can reliably detect a 3% difference in the concentration of Baseline Fluors.

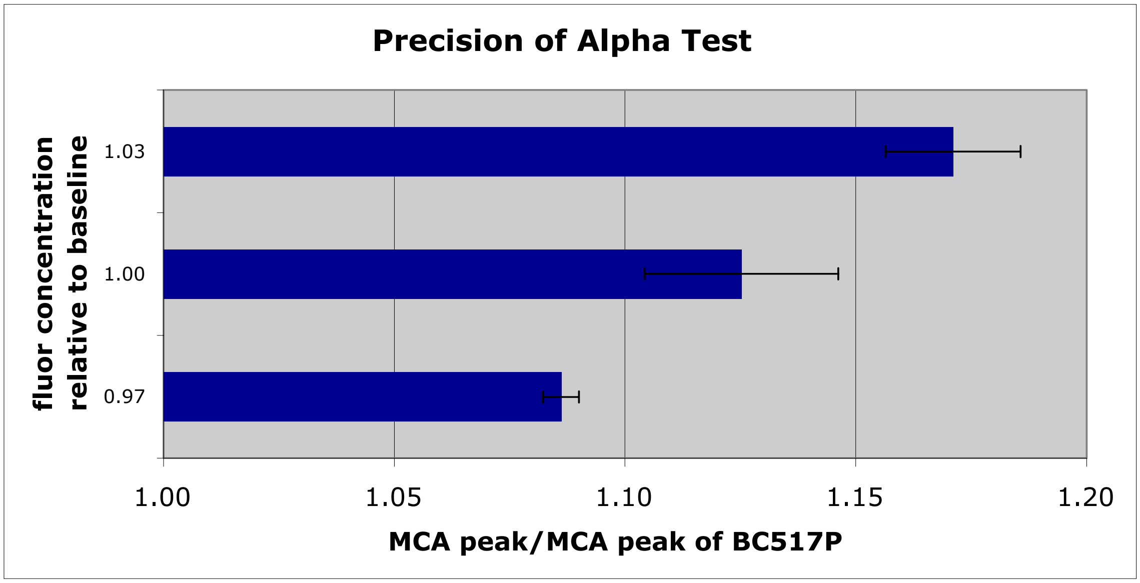

Fig. 10.22: The α-test results of the three scintillators blended with Mineral Oil A1 and differing concentrations of fluors. The error bars are statistical and reflect repeated measurements..

10.4.7.2. Radiation Safety

The α-test described above uses a 1 μCi Am-241source. These sources will be used both at the toll blender site and the Ash River site. NOνA must therefore comply with the radiation safety regulations at both locations.

Illinois is an "Agreement State", which means that Illinois has entered into an agreement with the US Nuclear Regulatory Commission, whereby the NRC has relinquished to Illinois the regulatory authority over the small quantities of nuclear materials used in the α-test. NOνA consequently is required to apply to Illinois for a license to procure, possess and use the radioactive source. After discussing this issue with officials at the Illinois Emergency Management Agency, Division of Nuclear Safety, NOνA needs only an R&D license because of the small quantity of radioactive material for the project and its research use.

The Ash River laboratory is part of the University of Minnesota and is obligated to follow the University radiation safety rules. The application and rules for "Possession and Use of Radioactive Materials" can be found at http://www.dehs.umn.edu/rpd/.

10.4.8 QC Conductivity

NOνA needs a reliable and efficient method of measuring the conductivity of blended scintillator to be confident that the scintillator conductivity is at safe levels. Currently production plans call for measurement of the conductivity before shipping at the producer. The results of these tests will be kept in the NOνA database.

Given the large volume of mineral oil required by NOνA and the necessity for a reliable and efficient method of measurement, our work has focused on the Emcee Electronics Model 1152 Digital Conductivity Meter shown in Figure 10.23. This device is a reliable and inexpensive instrument for measuring conductivity. It reads conductivity in picosiemens/meter. The meter uses a probe consisting of two concentric steel electrodes. The probe is shown in Figure 10.24. When the probe is immersed in oil, a fixed voltage is applied to the electrodes. The unit generates a current which is amplified and displayed on the meter.

The conductivity measurements shown in Figure 10.7 were made with an Emcee Conductivity Meter using the following procedure: A volumetric flask was filled with 1 liter of Mineral Oil A1 + Baseline Fluors without Stadis-425 and the contents transferred to an open mouth stainless steel container. The meter was calibrated using the standard test procedure. The conductivity meter ground lead was clipped to the edge of the stainless steel can and the meter probe inserted to the specified depth of 2.75″. Measured amounts of Stadis-425 were added and the mixture was stirred for 1 minute with a teflon paddle. The mixture was then allowed to stand for 5 minutes before the mixture was tested.

Fig. 10.23: The Emcee Electronics Model 1152 Digital Conductivity Meter

Fig. 10.24: Probe for the Emcee Electronics Model 1152 Digital Conductivity Meter

10.5 Scintillator Production

10.5.1 Production Model

The NOνA liquid scintillator will be blended at a commercial toll blending facility in the greater Chicagoland area. The production model is shown in Figure 10.25. Components are purchased by Fermilab and delivered to the blending facility by the most cost-efficient method: the mineral oil by rail or barge, the pseudocumene by rail or tanker truck, and premeasured waveshifter packages by common carrier. Blended liquid scintillator is delivered to Ash River by tanker trailers dedicated to the NOνA experiment.

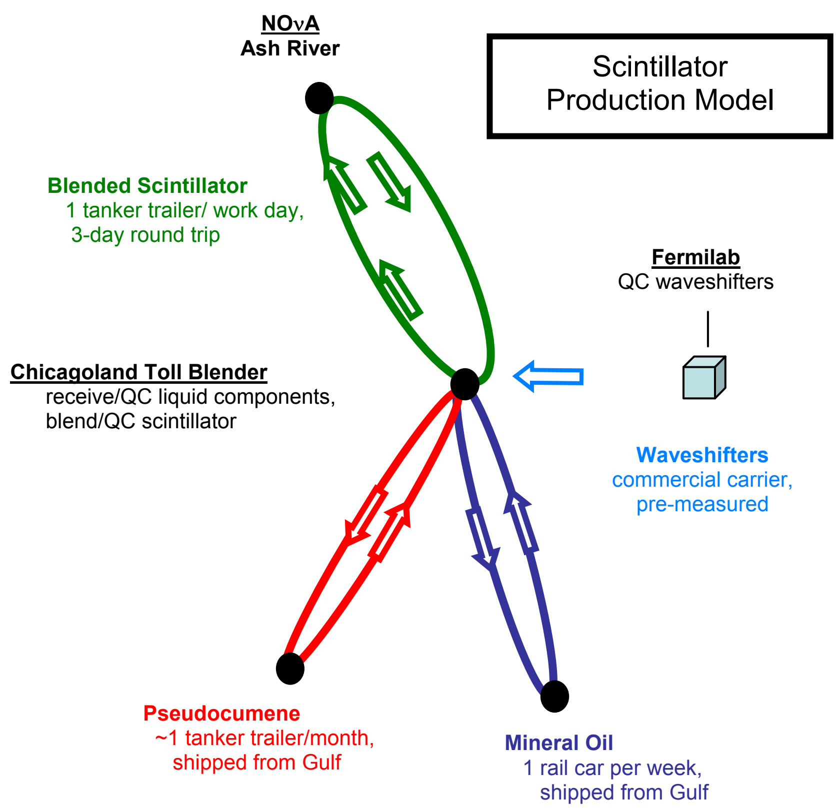

Fig. 10.25: Scintillator Production Model as described in the text.

10.5.2 Toll Blender Operations

The NOνA liquid scintillator will be blended at a commercial toll blending facility in the greater Chicago area. The blending model is shown schematically in Figure 10.26. In this model the components are purchased by Fermilab and delivered to the blending facility by the most cost-efficient method. At this facility, all the tanks used in the NOνA liquid scintillator blending will be stainless steel or epoxy-lined. It should be noted that MiniBooNE stores 250,000 gallons of very high quality mineral oil in an epoxy-lined tank "and has noticed no degradation after many years". These tanks can be heated. In addition, all transfer lines, hoses, and pumps used in the blending will be dedicated to NOνA liquid scintillator blending and locked off between deliveries for all the process steps. The lines and pumps will be insulated and fitted with filters.

The toll blender will offload the mineral oil into a storage tank with capacity in the range of 650,000 gallons. The specific gravity, kinematic viscosity, and anti-oxidant content as certified by an independent testing laboratory, with documentation supplied by the producer, will be checked against the Technical Requirements in Table 10.3 for compliance and the data added to a database. An on-site Fermilab technician will test the mineral oil for compliance with the attenuation length Technical Requirement on the NOvA Mineral Oil in Table 10.3 with the Lovibond tintometer. The attenuation length data will be added to the database.

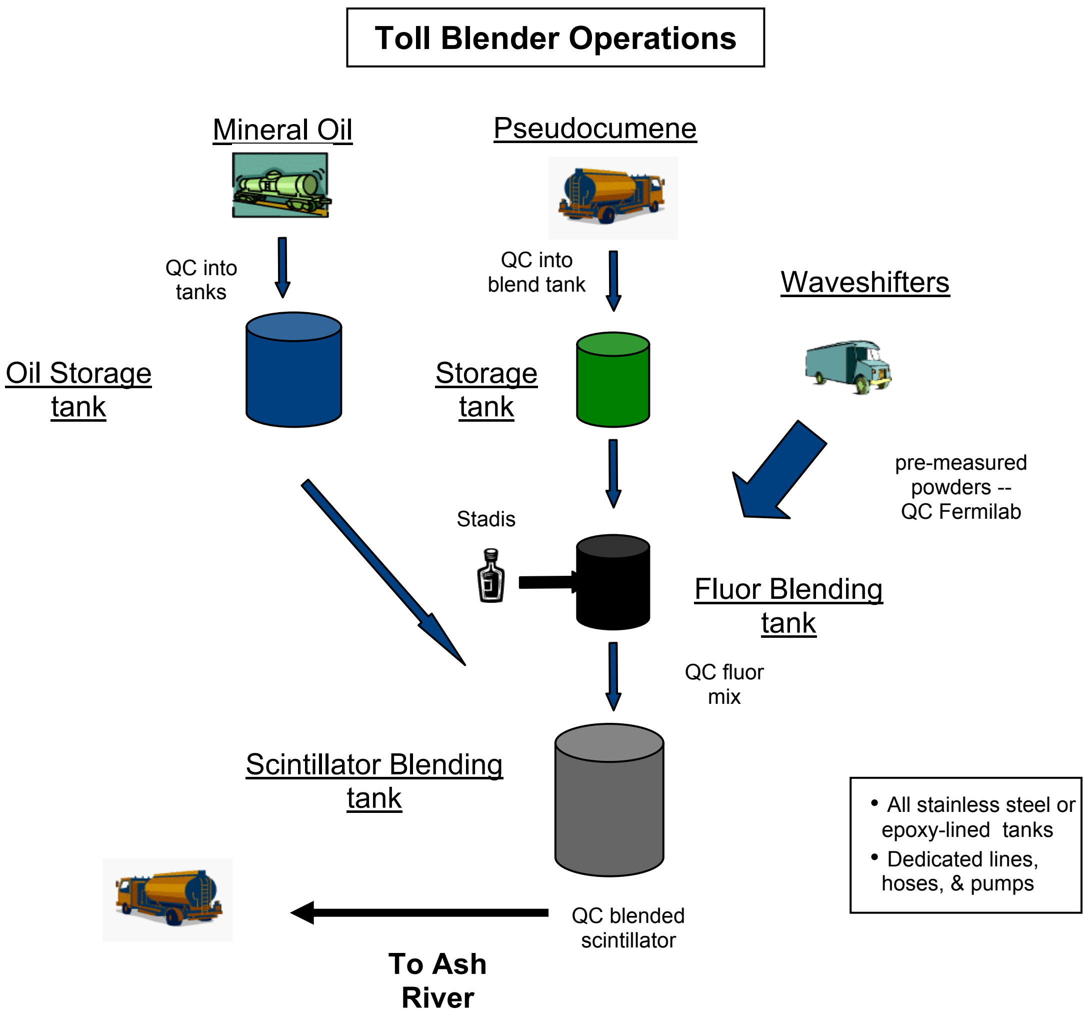

Fig. 10.26: Toll Blender Operations for the production of liquid scintillator.

Pseudocumene will be delivered to the blending facility by rail car. The kinematic viscosity as certified by an independent testing laboratory with documentation supplied by the producer will be checked against the Technical Requirement in Table 10.4 for compliance, and the data added to a database. A sample of pseudocumene will also be tested for compliance with the Technical Requirement on clarity in Table 10.4 with the Lovibond Tintometer and the data added to the database. As a final QA step before it is offloaded, a sample of the pseudocumene will be drawn and shipped to Indiana University where it will be analyzed by gas chromatography-mass spectroscopy (GCMS) to determine its composition and its compliance with the Technical Requirements on purity and sulfur content in Table

10.4. These data will also be added into the database. The pseudocumene will then be offloaded into a tank that holds of order 30,000 gallons.

Waveshifters will be delivered by commercial carrier in pre-measured packages to the Toll Blender. Before being shipped, samples of the wave shifters from each batch will be sent to Fermilab where the QA analysis will be done, as described in §10.3.1.2. Once the QA analysis establishes compliance with Technical Requirements, these packages will be transported to the blending facility where they will be combined with the pseudocumene in the Fluor Blending tank of order 30,000 gallons. The anti-static agent Stadis-425 will also be added at this step.

The blending will be done by mixing nozzles in the tank which draw from a bottom sump. After blending, a sample of the fluor mix will be drawn, mixed with mineral oil, and tested for compliance with the light yield, attenuation length, and conductivity Technical Requirements. These tests qualify the Fluor Mix. Blending in smaller batches will minimize the losses from any blending error.

The Fluor Mix is then combined with mineral oil in the Scintillator Blending tank of order 50,000 gallons. The fluids will be metered into the Scintillator Blending tank with PLCs. The blending will be done with mixing nozzles that can draw from the top or bottom of the tank. Once blended, the scintillator will be tested for compliance with the Technical on attenuation length, light yield, and conductivity. Once certified, the blended scintillator will await shipping to Ash River by tanker trrailer. The Storage tank will allow time to diagnose and correct any off-specification blends.

The baseline plan is to transport liquid scintillator by tanker trailer. The tolerances on the composition of the liquid scintillator blended from qualified components per standard 6,341 gallon tanker trailer are given in Table 10.7.

component	weight/mass per 6,341 gal	tolerance	weight/mass tolerance per 6,341 gal
mineral oil	**8,884 lbs**	**1%**	**88.8 lbs**
pseudocumene	**382 lbs**	**1%**	**3.8 lbs**
PPO	**18.5 kg**	**1%**	**185 gm**
bis-MSB	**260 gm**	**1%**	**2.6 gm**
Stadis-425	**61 gm**	**10%**	**6 gm**

Table 10.7. Blending Tolerances for 7,000 gal Tanker Trailers

10.5.3 Liquid Scintillator Delivery

The details of the NOvA liquid scintillator logistics plan are driven by the requirements on the scintillator delivery rate and the time it takes a driver to make the round trip Chicago (toll blender) – Ash River – Chicago.

The NOvA detector is made up of a large volume of liquid scintillator and this scintillator needs to be delivered at approximately the same rate as the detector is being filled in order to minimize infrastructure costs. Since this requirement closely links the delivery schedule to the detector construction schedule, the detector mass, and the funding profile, and these are not yet definite, the delivery schedule needs to remain flexible.

There are several additional constraints on the logistics of delivering scintillator. The Ash River site will only be able to take deliveries during a normal Monday through Friday work week, and there can be no assurance that the toll blender selected would not have similar restrictions. Consequently, tanker trailer deliveries and returns must be scheduled so that drivers do not make drop-offs or pick-ups on weekends (which translates into no weekend driving). In addition, to minimize driver down time, deliveries at Ash River need to be scheduled so that there is an empty tanker trailer available for the return trip immediately upon the delivery of a full tanker.

NOνA requires 3,210,584 gallons of liquid scintillator to be delivered over a period of 21 months. With the constraints described above, this schedule leads to an average delivery of 8,170 gallons per M-F work day. (At this early stage, no account need be taken yet for holidays.) Assuming standard 7,000 gallon tanker trailers, this schedule requires approximately 6 deliveries of liquid scintillator per M-F work week.

FThe distance from Fermilab to Ash River is about 600 miles. Driving at the speed limit, the driving time is about 10 hours. Since truckers must enter all driving times in their log books by law, show 1/2 hour of truck inspection per day, and take a 1/2 hr lunch break, the round trip takes 22 hours. Assuming an hour for the pick-up or drop-off time, the round trip takes 26 hours. After adding in time for traffic and weather delays, a round trip driving time of 3 days is reasonable and conservative.

Tables 10.8 and 10.9 show a logistics plan that meets the requirements for liquid scintillator delivery. There is a natural two week cadence for deliveries. Table 10.8 shows the two week cadence for a given tanker trailer.. A tanker trailer leaves the toll blender in Chicagoland in the morning and arrives at midday of the following day. The driver then picks up an empty tanker trailer in the afternoon and drives back to the toll blender, arriving at the end of the third day. This schedule enables a driver to stay clear of weekend pick-ups and deliveries.

day		**activity**	**duration**
			(days)
1	**prep/Chicago**	inspect truck, QC scintillator, load truck	1
2	**out**	travel	1
3 (AM)	**out**	travel	0.5
3 (PM),4,5,6,	**idle/Ash River**	unload truck,	5
7,8 (AM)	**idle/Ash River**	QC scintillator	
8 (PM)	**return**	travel	0.5
9	**return**	travel	1
10	**idle/Chicago**		1

Table 10.8. Two Week Tanker Truck Schedule for Delivery of NOνA Liquid Scintillator.

Table 10.9 shows the logistics plan that delivers 6 tanker trailers to Ash River/M-F work week. It operates on a two week cadence and requires 12 tanker trailers. The schedule runs from Friday-Thursday because drivers are on the road Monday-Wednesday or Wednesday-Friday, thus requiring no weekend pick ups or drop offs. Drivers end up where they began, as required.

The schedule shown in Table 10.9 is flexible and can be simply modified to adjust to needed increases or decreases in the quantities of scintillator delivered. There are two ways that we can vary the quantity of scintillator transported. First, we can vary the size of the tankers with the same schedule. The delivery schedule in Table 10.9 is based on 6,341-gallon tanker trailers. Other standard size tanker trailers have 7,000-gallon capacity and 7,500 gallon capacity. Second, the schedule in Table 10.9 can be modified to accommodate required increases or decreases in delivered scintillator. This schedule should be considered as an example. It is quite simple to alter the schedule by multiples of two tanker trailers.

	Friday	Monday	Tuesday	Wednesday	Thursday
			week 1		
prep	1,2,3		4,5,6		
out		1,2,3		4,5,6	
out			1,2,3		4,5,6
Ash River	7,8,9, 10,11,12	7,8,9, 10,11,12	1(PM),2(PM),3(PM) 7(AM),8(AM),9(AM) 10,11,12	1,2,3, 10,11,12	1,2,3, 4(PM),5(PM),6(PM) 10(AM),11(AM),12(AM)
return			7,8,9		10,11,12
return	4,5,6			7,8,9	
idle		4,5,6			7,8,9
			week 2		
prep	7,8,9		10,11,12		
out		7,8,9		10,11,12	
out			7,8,9		10,11,12
Ash River	1,2,3, 4,5,6	1,2,3, 4,5,6	1(AM),2(AM),3(AM), 4,5,6, 7(PM),8(PM),9(PM)	4,5,6, 7,8,9	4(AM),5(AM),6(AM) 7,8,9, 10(PM),11(PM),12(PM)
return			1,2,3		4,5,6
return	10,11,12			1,2,3	
idle		10,11,12			1,2,3

Table 10.9. Delivery Schedule for 12 Tanker Trailers every 2 weeks

10.6 Design Changes since the Conceptual Design Report

There have been several changes since the Conceptual Design Report.

The fluor concentration has been reduced from 100% to 75% of Bicron BC-517P. this produces a scintillator with 75% of the light output of BC-517P as required in Chapter 6.

Blending liquid scintillator is planned to take place at a commercial toll blending facility. In the Conceptual Design Report, scintillator blending was to be done at Fermilab.

The importance of making liquid scintillator semiconducting has been recognized since the Conceptual Design Report and the baseline composition of the liquid scintillator now includes the addition of Stadis-425.

We have shown that the Lovibond tintometer is a commercial device that can make rapid and consistent measurements of the transmission of mineral oil, pseudocumene, and blended liquid scintillator. The Lovibond tintometer has been adopted as the baseline measurement device for monitoring transmission of mineral oil at the producer and the toll blender, and the blended liquid scintillator at the toll blender and Ash River.

10.7 Work Remaining to Complete the Scintillator Design

There are two issues that need to be addressed with the Integration Prototype Near Detector (IPND). These issues concern production in quantity and so are not easily studied in the lab.

The first issue has to do with scintillator composition in large quantities. So far we have been blending scintillator in small batches of approximately 1-10 gallons each. For the IPND, we will be blending four batches of approximately 5,000 gallons. Two of these batches will be produced in the fall of 2007 and the second two will be produced in spring 2008. Among the issues that will be studied during these production runs will be the mixing time required to fully blend liquid scintillator in quantity and procedures for QC/QA testing during production. Since the four batches of scintillator will be blended with combinations of Mineral Oils A3 and C and Pseudocumene from producers A and B, we will be able to quantify the light yield of scintillators blended from components from multiple vendors delivered at multiple times over the next year.

The second issue concerns the blending tanks and transportation tanks. In the IPND we are using ISO tankers cleaned to chemical grade cleanliness and then cleaned once again to food grade cleanliness. The current plan calls for the tanker trailers used in transporting liquid scintillator to Ash River to be cleaned this way. The IPND will therefore provide valuable information in evaluating this cleanliness standard. Since we will be blending four batches of scintillator, we will also be able to examine the cleanliness of the blending tank after it has been used several times.

One last issue concerns the Toll Blenders in the Chicago area. We plan visits to these facilities in order to understand their capabilities. These visits will form the basis of a full RFP to select a vendor.

10.8 Chapter 10 References

[1] For example, see Birks et al., *Brit. J. Appl. Phys*, **14**, 141 (1963) for the absorption and emission spectra of PPO and POPOP. There are references in this paper to work by Kallman and others from the early 1950s. See also H. Rheinberger, “Liquid Scintillation Counters, 1950-1970”, Max Planck Institute for the History of Science, 1999.

[2] Mineral Oil Based Liquid Scintillators, www.bicron.com

[3] Mineral Oil Based Liquid Scintillators, www.eljentechnology.com/index.html

[4] NFPA 77, “Recommended Practice on Static Electricity”, 2000 ed.

[5] The MACRO Collaboration. *Nucl.Instrum.Meth*., **A486,** 665 (2002).

[6] See Kramer, Lok, and Krug, “The Evolution of Base Oil Technology”, in “Turbine Lubrication in the 21st Century”, edited by Herguth and Warne, American Society for Testing and Materials, ASTM STP#1407, 2001. See also Kramer et al., Machinery Lubrication Magazine, May 2003.

[7] J.P. Petrakis, *et al. Nucl.Instrum.Meth*., **A238,** 256 (1988).