



Basic mixing and purification of liquid scintillators

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Abstract

This note presents a basic introduction into the mixing of liquid scintillators and usable purification methods. We start with an introduction of some basic ingredients and their properties before we introduce possible mixing procedures. The last paragraph of this note will give an overview about possible and well proved purification methods.

1 Introduction

In order to learn more about neutrino physics we have to examine the weak interaction between normal matter and the corresponding neutrinos. Due to the very small cross section for this interaction, all experiments that try detection have to provide a sufficient high number of target protons. To monitor these events with an adequate statistics and on a reasonable time scale, recent neutrino experiments became larger in volume. The current data delivering experiment BOREXINO[2] [3] has a volume of 300t of liquid scintillator. The planned LENA experiment[4] will contain a volume of 50kt. It will be held in a cylinder which has a diameter of 30m and a height of 100m. Experiments of such dimensions will need a target, which has almost perfect optical properties in order to reach needed sensitivity. The optical properties as well as the chemical stability of these scintillator mixtures are crucial to the success of those experiments. Liquid scintillators have well proven their ability over the last years; however the future demands to the liquid scintillators are increasing. Limiting processes like scintillation light absorption or light scattering or a degradation of the scintillator itself have to be avoided or well understood at least. Since these limitations can not be avoided completely, they have to be reduced to a minimum in order to reach the demands. A proper handling of all ingredients and especially the treatment of the final scintillator is crucial to obtain a usable scintillator. All impurities still remaining in a scintillator mixture which increase light scattering or absorption have to be removed by purification.

In this note we introduce in a first step a selection of needed scintillator ingredients and their properties that are necessary for the mixing of non-doped liquid scintillators. We concentrate on possible candidates for the ANGRA experiment. We describe the scintillator mixing process along with its crucial step for different mixing procedures. Finally we provide a possibility to increase the optical properties of liquid scintillators by purification. Therefore we are introducing a tested purification system and its setup in order to enable a replication of this purification process. In addition we name two alternative purification techniques which will not be discussed in detail.

Experience shows that a proper use of all ingredients in the beginning and a careful handling of the mixture are absolutely crucial to obtain a usable Scintillator. Liquid scintillators show certain sensitivity when exposed to UV-light, Oxygen or elevated temperatures. If the mixture is subjected to one or more of such conditions over a longer time period, the scintillator base will degrade, the result is a visible yellowish color change of the scintillator.

2 Ingredients

2.1 Basic concept of scintillation in organic liquid scintillators

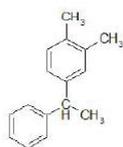
ORGANIC LIQUID SCINTILLATORS are a combination of different ingredients. Basic mixtures are a composition of a liquid solvent (scintillation base) and one wavelength shifter or fluor as it is also called. A solvent consists of molecules which include at least one benzol ring. These benzol rings and their individual bindings are the origin of the scintillation process. Each kind of molecule has its own composition and with it a special electron structure. Some structures like benzol rings for example have the ability to scintillate. This emission is a result of a degenerated electron structure which produces an energy gap between two electron band states. In case of benzol rings the structure has an electron band gap that corresponds to an energy of about 4eV. An excited electron in this ring will de-excite over this 4eV band gap and produce scintillation light in the UV-band. PXE, Phenyl-o-Xylylethane, a well known scintillator, for example, produces UV-light of about 280nm. Light of this wavelength has a high probability to get re-absorbed by other molecules of the same kind. In order to avoid re-absorption of scintillation light by the next scintillator base molecule a primary fluor is added. By adding this primary wavelength shifter the de-excitation energy is mainly transferred to the primary Fluor. This energy transfer is dominated, beneath 50 Angström, by the dexter process. This process describes a non-radiative energy transfer that is based on a dipole-dipole interaction[1]. The primary wavelength shifter, however, emits the transferred energy radioactively via photons but with a higher wavelength of about 350-400 nm. Re-absorption at this wavelength in the scintillator base is much less probable and, therefore, the transparency of the scintillator base for the shifted photons is higher. This wavelength shift can be repeated by adding a second wavelength shifter. Using a second fluor the scintillation light can be shifted into the blue part of the visible light, depending on the used fluor (i.e. up to 380-450nm for Bis/MSB). This procedure could be used to increase the detection probability for a photon because some Photomultiplier tubes (PMT) have the highest detection efficiency around 400nm.

In some applications of liquid scintillators it can be necessary to change certain properties like the light yield, number of protons, attenuation length or the density, in order to get an applicable scintillator. To change the properties of a scintillator it is possible to dilute it in a mineral oil (white oil) which has outstanding optical properties. Some unpurified mineral oils have an absorption length between 9-12m[16]. Examples are Do- or Tetradecane. Unpurified PXE as solvent, for instance, has an attenuation length between two and three meters[16]. A mixture of both liquids will produce a scintillator with an attenuation length of clearly more than 3 meters. In addition, the dilution with mineral oil will change the ratio between carbon and hydrogen molecules of the scintillator, which changes the number of possible target protons.

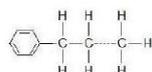
The main aspect of a scintillator is its light yield and the knowledge of its extent. Liquid scintillators and their properties are strongly dependent on the amount and fraction of their ingredients. This implies that the best possible combination of ingredients will have to be found for each application and scintillator. In order to find the perfect proportion between a maximum light output and a minimum of costs for the final scintillator, each ingredient will have to be chosen carefully as well as its proportion in the mixture. So, a higher portion of primary fluor is not always connected to a higher light output. An example shows, for the light yield curve of PXE and PPO, a saturation at 3g/L of PPO in PXE[14][1]. For this reason, a higher portion of PPO in PXE in most cases is not necessary.

2.2 Ingredients

SCINTILLATION BASES, SOLVENTS



PXE: Phenyl-o-Xylylethane[7] is, under normal conditions, a scintillating, distinct smelling solvent with a density of 0.985kg/l. It consists of carbon and hydrogen atoms. The chemical formula is $C_{16}H_{18}$. A PXE-molecule has two carbon-rings, each ring has a degenerated electron structure which produces an energy gap of about 4 eV between two electron bands. These ring molecules have three delocalized electrons which can be excited and produce the scintillation light that corresponds to an energy in the UV-band(280nm). PXE has a flash point of 160° Celsius which is, in comparison to other scintillator solvents, relatively high. This is an advantage for the use of PXE in high volume detectors. Unpurified PXE has an attenuation length of roughly 2-3m[16]. This value can be increased by purification.

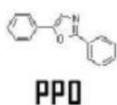


LAB

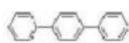
LAB: Linear alkyl benzene[8]: is under normal conditions a scintillating almost odorless solvent with a density of 0.86kg/l. The chemical formula is due to the varying number of carbon atoms not always the same. A LAB-molecule has one carbon-ring and a straight chain of $C_{10-15}H_{21-31}$ attached to it. The number of carbon atoms varies between 10 and 15 carbon atoms depending on the used product. Selling companies (example: Petresa) offer the possibility to lower this variation. Each ring has a degenerated electron structure which produces an energy band gap that corresponds to an energy in the UV-band. LAB has a flash point of 140° Celsius[8]. This, and the lower price, are an advantage for the use of LAB in high volume detectors.

Table 1 displays the most important properties of tetradecane, dodecane and PXE. The here introduced solvents, PXE and LAB, as well as the mineral oils, are not the only possible candidates for scintillator mixing. Other alternatives for scintillator bases are PC (Pseudocumene), PCH (Phenylcyclohexane) and DIN (Diisopropyl-naphthalene). An alternative to a mineral oil (or white oil) like Dodecane is the cheaper Tetradecane. It is as chemically inert and stable as Dodecane.

FLUOR, PRIMARY AND SECONDARY WAVELENGTH SHIFTERS

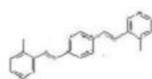


PPO



pTP

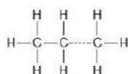
PPO, pTP: 2,5-Diphenyloxazole(PPO)[11] consists of 15 carbon- and 11 hydrogen atoms as well as a Nitrogen-oxygen group. PPO is used as a primary wavelength shifter (primary fluor) it absorbs light between 280-325nm and has its emission maximum at 370nm. The energy transfer between PXE molecules and the PPO is dominated by a non-radiating process called Dexter-process. The dexter-process is a dipole-dipole interaction that dominates the energy transfer below 50 Angström [1] (distance between PXE and PPO). PPO is, due to its structure, a polar molecule which will have to be considered when a purification of the Scintillator is planned. The reason is that some purification methods filter for polarities and would therefore lower the concentration of PPO in the scintillator. If such a reduction has to be avoided a different fluor can be used, pTP(para-Terphenyl) is such a fluor. pTP is, due to its structure, a non-polar molecule that consists of 18 carbon- and 14 hydrogen atoms. It is as well as PPO under normal condition a powder and absorbs between 250-300nm and emits between 320-370nm. A disadvantage of pTP is its lower solubility in comparison to PPO.



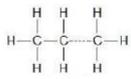
bisMSB

BIS/MSB: 1,4-bis(o-methylstyryl)-benzol[13] consists of 24 carbon and 22 hydrogen atoms. It forms a triple ring structure and is, under normal conditions, a white powder. bis/MSB is used as secondary (wavelength shifter) Fluor because it absorbs at a wavelength of 320 to 370nm (emission band of pTP and PPO) and emits between 380 and 450nm, which corresponds to the blue-band of the spectral light. The energy transfer between the primary and the secondary Fluor (from PPO to bis/MSB) is dominated by radiating processes. The energy transfer between PXE and PPO is non-radiating to avoid re-absorption in the scintillator base itself. This re-absorption is not dominant at the radiating energy transfer between PPO and bis/MSB (first and secondary Fluor) because the emission band of bis/MSB is already above the absorption band of PXE. A collection of absorption and emission bands is given in table 3.

MINERAL OILS



DODECANE: n-Dodecane[9]: is a non scintillating mineral oil that is liquid under normal conditions and not complete odor but colorless liquid. It consists of 12 carbon- and 26 hydrogen atoms, which form an inert mineral oil with outstanding optical properties, i.e., the attenuation length of some unpurified dodecane probes is in the range of 12m. The chemical structure formula of dodecane is $C_{12}H_{26}$ it has a flash point of 74°C and density of 0.749kg/l. The optical properties, the density and the inert chemical behavior of this white oil are ideal to use it as dilution i.e. lowering the density or increasing the optical properties of the final scintillator. In addition to that, dodecane offers a higher ratio of H-to-C atoms. This increases the number of Protons and with this the number of possible targets for the inverse β -decay.



TETRADECANE: Tetradecane[10] is a non scintillating mineral oil that is liquid under normal conditions an slightly smelling colorless liquid. It consist of 14 carbon-and 30 hydrogen atoms, which form an inert mineral oil with outstanding optical properties, i.e., the attenuation length of some unpurified Tetradecane probes is in the range of 10m. The chemical structure formula of Dodecane is $C_{14}H_{30}$ it has a flash point of 99°C and density of 0.762kg/l . The optical properties, the density and the inert chemical behavior of this mineral oil are ideal to use it as dilution i.e. lowering the density or increasing the optical properties of the final scintillator. In addition to that, Tetradecane offers a higher Ratio of H-to-C atoms. This increases the number of Protons and with this the number of possible targets for the inverse β -decay.

SOLVENT	PXE	LAB	DODECANE	TETRADECANE
Structure formula	$C_{16}H_{18}$	$C_{16}H_{18}$	$C_{12}H_{26}$	$C_{14}H_{30}$
Density	0.986kg/l	0.986kg/l	0.749kg/l	0.762kg/l
Molecular weight	210.2	239-243	170.34	198.4
Refractive index	1.565	1.484	1.422	1.428
flash point	160°	140°	74°	99°
CAS-Nummer	6196-95-8	68890-99-3	112-40-3	629-59-4
HMIS Rating	PXE	LAB	DODECANE	TETRADECANE
Health	1	1	1	2
Flamable	1	1	2	1
Reactivity	0	0	0	0

Table 1: Table of the most important properties of PXE, LAB, Dodecane and Tetradecane

FLUOR	PPO	PTP	BIS/MSB
Structure formula	$C_{15}H_{11}NO$	$C_{18}H_{14}$	$C_{24}H_{22}$
Molecular weight	221.26	230.31	310.44
CAS-Nummer	92-17-7	02-94-4	13280-61-0

Table 2: chemical properties of PPO, pTP and bis/MSB

The most important chemical properties of the introduced fluors are shown in tables 2 and 3. The emission spectra of PPO, bis/MSB and of its mixture are shown in Figure 1. This collection of primary and secondary fluors is just a small fraction of possible candidates. Next to the already introduced fluors are products like PBD as primary and POPOP as secondary wavelength shifter. These chemicals will always have to be chosen depending on the solvent and the planned operation

FLUOR	ABSORPTION [NM]	EMISSION [NM]
PXE	250-280	280-315
PPO	280-325	350-400
pTP	250-300	320-370
bis/MSB	320-370	380-450

Table 3: emissions- and absorptions bands of PXE and primary and secondary fluors[5]

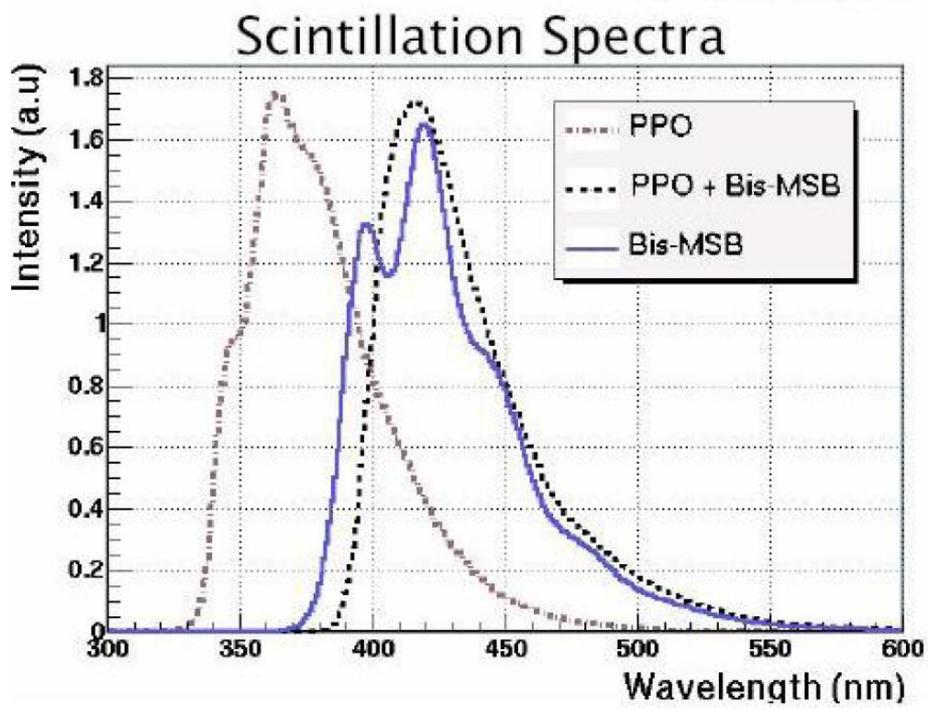


Figure 1: Emissions bands of PPO, bis/MSB and its mixture: Picture[6]

3 Mixing

3.1 Scintillator mixing

NEEDS AND PROBLEMS OF SCINTILLATOR MIXING

Liquid scintillators are mostly made of chemicals in powder form and liquids like PXE and mineral oil. Some bases are reactive to oxygen. Therefore, an inert (oxygen free) handling of these liquids and during the mixing is the preferred mode of operation. If an oxygen exposure is not avoidable during the handling and mixing process it is possible to undo the oxygen adaptation by purging the liquid with Nitrogen. After extensive exposure with oxygen the scintillator will suffer a severe degradation due to the oxidation process which will lead to a decrease of optical properties, developing a yellowish color. To guaranty the quality of all liquids it is useful to maintain a nitrogen blanket over all liquids during storage and handling.

Chemicals which come in powder form (like PPO or Bis/MSB) are known to contain suspended particles or even radioactive elements along with the particles; the degree of pollution depends on the quality of chemicals and the storage conditions. Suspended particles will reduce the optical properties of a scintillator due to Mie- and Rayleigh-scattering effects and should therefore be removed. Suspended particles produce, due to their size, mainly Mie-scattering effects which can be minimized by filtering the liquid scintillator for these particles. Radioactive pollution due to radioactive elements within the chemicals could be a result of improper storage condition. Chemicals stored in air or in a container that is not Radon tight will suffer from Radon diffusion. Radon will diffuse into the chemicals and produce daughter elements coming from the Radon decay chain. Experience coming from the BOREXINO experiment shows that this kind of radioactive pollution can be reduced by column chromatography using Silica-gel as absorbent[17][14].

The solubility of chemicals in liquids should be kept in mind if larger amounts of scintillator shall be mixed. In case of PPO the solubility in PXE is in the range of 100g/L[1][14] whereas the solubility of pTP in Pseudocumene is more difficult.

SMALL AMOUNTS OF SCINTILLATOR

The mixing of a small batch of liquid scintillator does not bring any major problems. The solubility of chemicals is not a problem because the handling of small amounts is easy, due to the small volume of the sample holder. Possible dissolving problems of the chemicals due to in-homogenous dilution can be easily solved by hard physical shaking or stirring by hand. The main challenge is the quality of the mixture and, therefore, the purity of all used ingredients as well as their handling during the mixing process. An oxygen exposure is also easy to undo by homogenous nitrogen flushing, which is not difficult with small sample holders. For very small batches it sometimes can be difficult to weigh the amount of chemicals needed. For batches with less than 500ml for instance it is very difficult to weigh the Bis/MSB (normally about 20mg per liter) exact enough to obtain the needed concentration.

BIGGER AMOUNTS OF SCINTILLATOR

The advantages of small volumes cease when large amounts of liquid have to be produced. Volumes of more than 50 Liters are not easy to handle, physical mixing is due to the high volume and mass of the liquid, very difficult and needs technical effort. Problems like stirring, homogenous dilution, liquid handling, scintillator pollution, purification as well as time and money consumption become now a problem. These problems get even worse when the amounts get bigger. For 1000L of scintillator, a stirring or a homogenous flushing process under nitrogen atmosphere is only realizable with high financial and technical effort. Large amounts of scintillator should therefore be mixed rather by combining two liquids than a powder and a liquid. The pre-mixing of a master solution is a technique which offers some advantages that can help to realize the mixing process more easily.

Mixing a master solution

This technique avoids mixing the needed chemicals with the full amount of liquid in order to lower the handled volume and avoid the physical problems of mixing, purification and handling. Therefore, the chemicals are dissolved only in a small amount of liquid. This will lead to a highly concentrated but liquid mixture called 'master solution' which offers some advantages. The volume is reduced to a minimum and the liquid handling will be significantly easier, as well as the purification of the scintillator. The purification process depends on the amount of liquid that is purified. Due to that, small volumes are purified with quicker and cheaper purification processes. In addition, a liquid master solution is easier to mix with all other liquids than a powder. A master solution offers also the possibility to produce a variety of scintillators with different properties. In order to obtain the wanted scintillator mixture (with a defined concentration of chemicals per liter or a certain density) the master solution is diluted with scintillator base or a mineral oil or both, depending on the required result.

4 Purification

4.1 Cleaning of Sample- or Probe holders

THREE-STEP-CLEANING PROCESS

Each batch mixing should start with clean sample holders. In general, everything in contact with the scintillator should be clean and made out of a material that is non-reactive to scintillator itself. If any sample holder or any other storage device was used before it could be necessary to clean it. Experience shows that 3 three step cleaning process is useful. The first step uses propanol as detergent, because alcohol dissolves oils and therefore scintillator remainders. The second step uses pure water to remove all polar and water soluble materials. The water is also a perfect indicator for oil remainders, if the previous cleaning was not successful. Oil will show clear streaks in contact with water. If no streaks are visible, the sample is clean and the holder has to be dried. This could be done by water evaporation, which takes time, or, in order to save time, it could be wiped with a non-fuzz one way paper towel. Alternatively, it is possible to dry the sample holder with the use of acetone. Acetone evaporates almost immediately in comparison to pure water. This will speed up the drying process significantly. After some minutes the acetone and the water will be visibly evaporated. In order to check whether all of the acetone is evaporated, it is helpful to detect the distinct smell of acetone. The probe holder should only be used when this smell is gone, so that a mixture of scintillator and acetone is avoided.

4.2 Purification of liquid scintillators

The optical properties a liquid scintillator are utterly important for its use in high volume detectors. Therefore, all light absorbing processes have to be either minimized or avoided. Light scattering has two main reasons: Rayleigh-scattering and Mie-scattering. The Rayleigh-process describes scattering of photons on bound electrons whereas the Mie-process describes photon scattering on particles, which are bigger than the wavelength of the scattered light. Considering the nature of both processes makes clear that Rayleigh scattering is an intrinsic property and not avoidable whereas Mie-scattering can be avoided (minimized) by reducing the number of suspended particles in the scintillator mixture. The main purification methods are shortly described in the following paragraphs. These purification procedures are:

- Column chromatography
- Vacuum distillation
- Water extraction

4.2.1 Column chromatography

The column chromatography is a standard purification procedure originated in chemistry. It uses a polar purification material through which the liquid is passed. This polar material is a very fine powder and offers the liquid a maximized surface on which the polar impurities of the liquid get attached. The now purified liquid and the purification material are physically separated by filtration. All polar impurities remain within the purification material and will be disposed with it. Liquid scintillators, however, show some sensitivity when exposed to UV-light, oxygen or elevated temperatures. If the scintillation mixture is subject to such conditions over a period of time, the scintillator base will degrade. The result of this degradation is a color change, the scintillator color turns visibly yellowish. To avoid such problems we have to take special care during purification. UV-light and temperature are easy to control. An oxygen free environment, however, implies more difficulties. Figure2 outlines an oxygen free column chromatography purification system. Two transport bottles with 3-valve-caps are connected to the purification column. The first transport bottle is pressurized with nitrogen, to push the scintillator through the system. The 3-valve cap is used to control the liquid and gas flow through the column(See detail in Figure2).

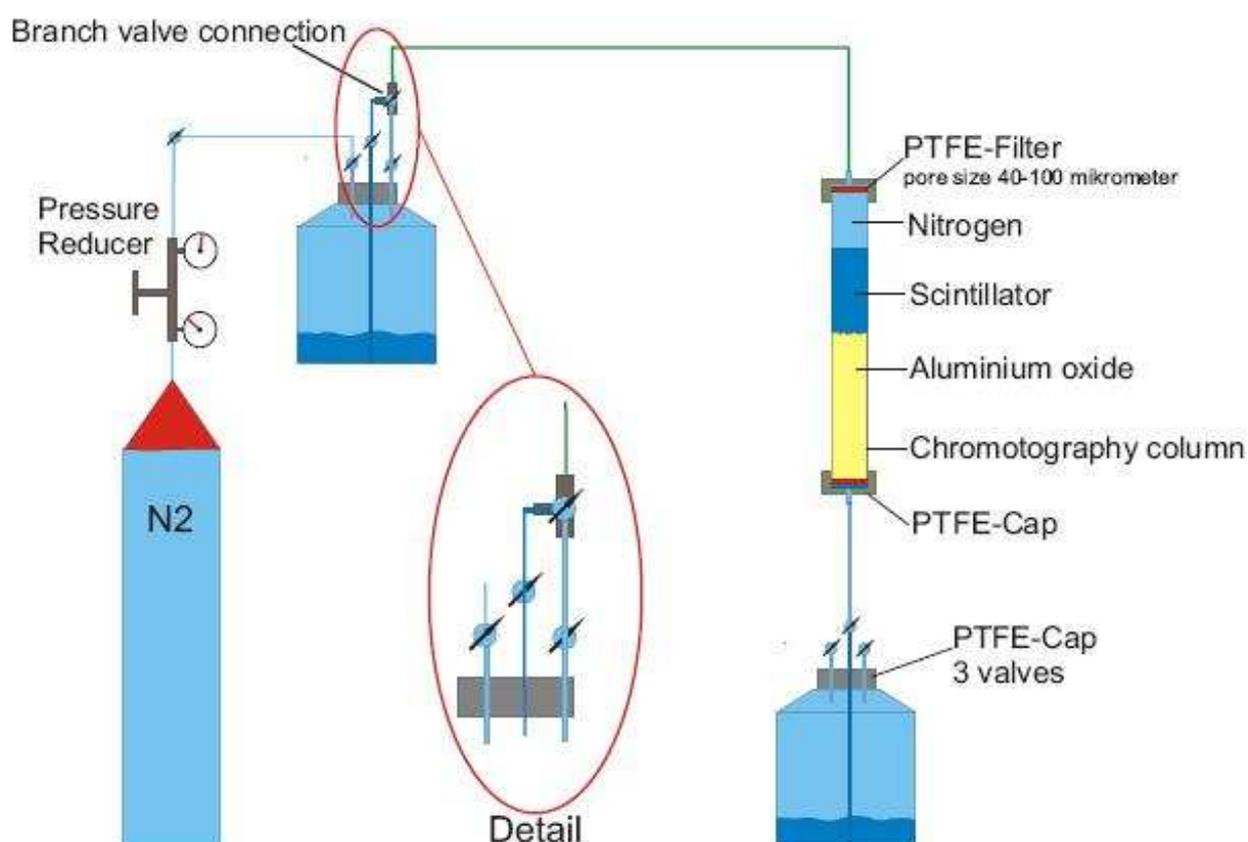


Figure 2: Oxygen free column chromatography purification setup.

The main part of this system is a pressure resistant glass column. The column is open at the top and the bottom, and can be closed air tight by two Teflon(PTFE) caps. Each cap has a removable stainless steel filter inlay and one tube connection. Each tube connection leads to a transport bottle. These 2L PE-coated glass bottles have an air tight teflon cap that offers 3 valves with tube connections (see Detail in Figure2). One of these connections has a tube running down to the bottom of the transport bottle, the other two connections end at the cap. The unpurified scintillator is stored in the upper transport bottle, under nitrogen atmosphere. By connecting a nitrogen bottle on the system and applying a slight pressure to the transport bottle, the scintillator is pushed into the upper end of the column. The liquid fills the column and is pushed through the purification material. At the bottom of the column the scintillator flows through a filter and directly into the second transport bottle. These bottles can be disconnected from the system without exposing the scintillator to oxygen. In the detail drawing of Figure2, a 3-way valve is near the 3-valve teflon caps of the transport bottle. This valve has regulation purposes: by choosing the flow path it is possible to sent either liquid in the column or nitrogen. With this layout the system has the possibility to regulate the liquid level within the column. This purification method has two working modes: a batch mode and a loop mode.

- The batch mode uses the purification material only for one batch of scintillator and deposes the material after a single purification run. This process consumes a lot purification material and handling time but shows better results in the end than the loop mode. In addition, the level of contamination will converge to a minimum and stay at this level.
- The loop mode uses the same purification material more than once. The scintillator is pumped in a circle and pushed through the same purification material again. The loop mode shows a rapid purification rate, but just until a certain level of purification is reached. If the circulation is repeated too often the material will pollute the scintillator again, and the result is a clear increase of the contamination level

PURIFICATION MATERIALS:

The standard column chromatography procedures offer standard purification materials, which are well known and reliable.

- Aluminum oxide: Al_2O_3 is one of the purification materials. It was successfully used for scintillator purification in Heidelberg(MPIK)[1] and Munich(TUM)[15]. Aluminum oxide is a white powder with a grain size between 40-90 μ m.
- Silica Gel: Si_2O is another purification material, which was successfully used in the BOREX-INO experiment[17] to purify the scintillator on pseudocumene(PC) base. Silica Gel is also a white powder with a grain size between 40-90 μ m

4.2.2 Vacuum distillation

Another possibility is vacuum distillation. The scintillator is subject to a reduced pressure environment and heated at the same time. As a result of the low pressure the boiling point of the scintillator also decreases. This is necessary because of the low flash point of the liquids. Furthermore, high temperatures are always to avoid with scintillators, since heat breaks up chemical bindings in the molecules, which leads to different scintillation properties. With this technique the pure scintillator will be distilled from all that has a higher boiling point, especially solid bodies. This is also a standard technique used in chemistry.

4.2.3 Water extraction

The water extraction purification process uses the non-solubility of oil and water as well as the polarity of water. In order to purify the scintillator it is mixed with water which has also polar properties. This mixture is then stirred, shaken or purged with nitrogen in a turbulent way to ensure a proper mixing of both liquids. This mixing ensures that the polar water molecules will get in best possible contact with the scintillator. The polar impurities will attach on the polar water molecules and remain with them. After this turbulent mixing the mixture is stored in order to allow the oil and the liquid to separate again. After this separation of oil and water, two clearly distinguishable liquid layers will have evolved. Between both liquid layers, a slight grayish layer is recognized. Oil and water layers are physically separated by using a separation funnel. The grayish layer between oil and water has to stay in the water and is disposed along with the it.

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