



# Enhancing Light Collection in Scintillation Detectors

by

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## Abstract

Advances in particle-physics experiments increasingly depend on highly efficient scintillation-based detectors, as the search for rare processes and weakly interacting particles pushes detection systems to their performance limits. Improving photon collection and photon-sensing technologies is therefore essential for experiments that aim to probe hidden-sector particles, long-lived states, and other signatures of physics beyond the Standard Model.

The first part of this thesis examines a BaSO<sub>4</sub>-based diffuse reflective coating (OPRC) intended to increase photon collection in the Surrounding Background Tagger (SBT), a liquid-scintillator veto detector used in prototype configurations of the SHiP experiment. Initial laboratory tests demonstrated that OPRC achieved a diffuse reflectivity exceeding 95 % in the wavelength range  $250 \text{ nm} < \lambda < 800 \text{ nm}$ , corresponding to an increase of up to 300 % in the UV region compared to stainless steel. When implemented in a detector prototype, an approximate reflectivity of 65 % was achieved during beam operation due to the presence of rust-induced staining. Despite these conditions, the prototype reached a detection efficiency of  $\geq 99.5 \%$  at a confidence level of 68 % during a test beam campaign. The coating was evaluated through systematic reflectivity measurements, compatibility tests with detector materials, and accelerated aging studies. Although the initial results demonstrated enhanced light collection, long-term exposure to liquid scintillator revealed several degradation mechanisms, including partial dissolution of the coating, fluor deposition, and reduced reflectivity caused by swelling and increased translucency. Additional complications arose when OPRC was applied directly to corten steel, where corrosion-induced discoloration further diminished optical performance. While clearly not suitable for long-term applications as in the SHiP-SBT, OPRC lining on stainless steel could still be used over short periods of time for significantly enhancing light collection.

The second part of the thesis focuses on the development of polystyrene-based plastic scintillators for use as active light guides in the SiALG photo sensor concept. A controlled polymerization procedure was established to produce high-quality scintillators with adjustable fluor loading. Optical characterization confirmed successful incorporation of fluors, though transparency and light yield remained below commercial standards, reaching approximately 57 % of the SP32 reference performance. These results outline clear directions for improving polymer quality and fluor integration. Together, the two studies contribute to the advancement of photon-collection and photon-sensing technologies for future scintillation-based detectors.

# Table of Contents

|          |                                                                    |           |
|----------|--------------------------------------------------------------------|-----------|
| <b>1</b> | <b>Introduction</b>                                                | <b>1</b>  |
| <b>2</b> | <b>The Standard Model of Particle Physics</b>                      | <b>3</b>  |
| <b>3</b> | <b>Scintillation Detectors</b>                                     | <b>6</b>  |
| 3.1      | Organic Scintillators . . . . .                                    | 6         |
| 3.1.1    | The Scintillation Mechanism . . . . .                              | 9         |
| 3.1.2    | Wavelength-shifting with Fluors . . . . .                          | 10        |
| 3.2      | Scintillator Components . . . . .                                  | 12        |
| 3.2.1    | Linear Alkylbenzene (LAB) . . . . .                                | 12        |
| 3.2.2    | 2,5-Diphenyloxazole (PPO) . . . . .                                | 14        |
| 3.2.3    | 1,4-Bis(2-methylstyryl)benzene (bis-MSB) . . . . .                 | 15        |
| 3.3      | Photo Sensors . . . . .                                            | 17        |
| 3.3.1    | Photomultiplier Tubes . . . . .                                    | 17        |
| 3.3.2    | Silicon Photomultipliers . . . . .                                 | 18        |
| <b>4</b> | <b>Methods and Experimental Setups</b>                             | <b>20</b> |
| 4.1      | Light Absorbance Spectroscopy . . . . .                            | 20        |
| 4.2      | Fluorespectroscopy . . . . .                                       | 24        |
| 4.3      | Light Yield . . . . .                                              | 25        |
| 4.4      | Thermal Aging Tests . . . . .                                      | 27        |
| 4.5      | Reflectivity Measurements . . . . .                                | 29        |
| 4.5.1    | Fundamentals of Reflectivity . . . . .                             | 29        |
| 4.5.2    | The Reflectivity Setup . . . . .                                   | 30        |
| 4.5.3    | Measurement Principle and Analysis . . . . .                       | 33        |
| <b>5</b> | <b>The SHiP Experiment</b>                                         | <b>35</b> |
| 5.1      | Motivation . . . . .                                               | 35        |
| 5.2      | Overview of the Beam Dump Facility / SHiP Experiment at ECN3 (100) | 37        |
| 5.3      | The Surrounding Background Tagger (SBT) . . . . .                  | 39        |
| 5.3.1    | Liquid Scintillator . . . . .                                      | 40        |
| 5.3.2    | Wavelength-shifting Optical Modules (WOMs) . . . . .               | 42        |
| 5.3.3    | Reflective Coating . . . . .                                       | 44        |
| <b>6</b> | <b>Reflective Coating Studies for SHiP's SBT</b>                   | <b>47</b> |
| 6.1      | Coating Application Tests . . . . .                                | 48        |
| 6.2      | Primer Application Tests . . . . .                                 | 51        |
| 6.3      | Aging Tests . . . . .                                              | 54        |
| 6.3.1    | Aging Tests with different Primers . . . . .                       | 54        |
| 6.3.1.1  | Reflective Coating . . . . .                                       | 55        |
| 6.3.1.2  | Scintillator Quality . . . . .                                     | 59        |

|          |                                                                                   |            |
|----------|-----------------------------------------------------------------------------------|------------|
| 6.3.1.3  | Results . . . . .                                                                 | 61         |
| 6.3.2    | Aging Tests with different Scintillators . . . . .                                | 63         |
| 6.3.2.1  | Reflective Coating . . . . .                                                      | 64         |
| 6.3.2.2  | Scintillator Quality . . . . .                                                    | 67         |
| 6.3.2.3  | Results . . . . .                                                                 | 71         |
| 6.4      | Test Beam Prototypes . . . . .                                                    | 74         |
| 6.4.1    | DESY Test Beam 2022 . . . . .                                                     | 74         |
| 6.4.1.1  | Coating the 1-cell-prototype . . . . .                                            | 75         |
| 6.4.1.2  | Results from the DESY Test Beam Campaign . . . . .                                | 79         |
| 6.4.2    | CERN Test Beam 2023/24 . . . . .                                                  | 81         |
| 6.4.2.1  | Coating the 4-cell-prototype . . . . .                                            | 82         |
| 6.4.2.2  | Scintillator Study . . . . .                                                      | 87         |
| 6.4.2.3  | Results from the CERN Test Beam Campaign . . . . .                                | 89         |
| 6.5      | Summary . . . . .                                                                 | 91         |
| <b>7</b> | <b>The Silicon Photomultiplier with Active Light Guide Design (SiALG)</b>         | <b>94</b>  |
| 7.1      | Motivation and Working Principle . . . . .                                        | 95         |
| 7.2      | Styrene and Polystyrene . . . . .                                                 | 97         |
| 7.3      | Basics of Polymerization . . . . .                                                | 98         |
| <b>8</b> | <b>Plastic Scintillator Development and Characterization for the SiALG Design</b> | <b>102</b> |
| 8.1      | Plastic Scintillator Production . . . . .                                         | 102        |
| 8.1.1    | Sample Preparation . . . . .                                                      | 102        |
| 8.1.2    | Polymerization Setup and Procedure . . . . .                                      | 104        |
| 8.1.3    | Polymerization Temperature and Initiator . . . . .                                | 106        |
| 8.2      | Plastic Scintillator Characterization . . . . .                                   | 110        |
| 8.2.1    | Fluor Integration . . . . .                                                       | 110        |
| 8.2.2    | Optical Transparency . . . . .                                                    | 111        |
| 8.2.3    | Light Yield . . . . .                                                             | 114        |
| 8.3      | Summary . . . . .                                                                 | 117        |
| <b>9</b> | <b>Conclusion and Outlook</b>                                                     | <b>119</b> |
|          | <b>List of Figures</b>                                                            | <b>124</b> |
|          | <b>List of Tables</b>                                                             | <b>131</b> |
|          | <b>Use of Artificial Intelligence</b>                                             | <b>132</b> |
|          | <b>Bibliography</b>                                                               | <b>133</b> |

# 1 Introduction

*“I hate to be the one to tell you  
but I think we should pull down the sun from the sky”*

— Soulkeeper, *Smile Because It Happened*

For more than half a century, the Standard Model (SM) of particle physics has provided an extraordinarily successful framework for describing the fundamental components of matter and their interactions. Its predictive power has been demonstrated repeatedly, from the discovery of the weak neutral current to the observation of the Higgs boson. These achievements have solidified its status as one of the most rigorously tested theories in science. Yet, despite its success, the SM is widely seen as incomplete. It does not account for the observed matter–antimatter asymmetry in the Universe, provides no explanation for neutrino masses, and fails to incorporate dark matter or dark energy. Moreover, it offers no insight into the nature of gravity. These open questions strongly motivate the search for physics beyond the Standard Model (BSM), driving a broad experimental effort that extends across colliders, fixed-target experiments, neutrino facilities, and astrophysical observations.

One particularly compelling direction in the exploration of BSM physics is the search for new, very weakly interacting particles that reside in a so-called hidden sector. These particles are predicted in a variety of theoretical models addressing dark matter, the hierarchy problem, or the strong CP problem. Due to their feeble couplings to Standard Model particles, previous high-energy experiments may have been incapable of detecting the hidden sector. The Search for Hidden Particles (SHiP) experiment at CERN aims to explore this largely uncharted parameter space. Designed as a high-intensity beam-dump experiment, SHiP will probe a broad class of long-lived exotic states through their rare decays or scattering signatures. To achieve the sensitivity required to discover or constrain these particles, controlling and suppressing background events is of critical importance.

The main component of SHiP’s background mitigation strategy is the Surrounding Background Tagger (SBT). This large liquid-scintillator veto detector envelopes the experiment’s decay vessel in which hidden sector particles are expected to decay into detectable products. The SBT must identify minimum-ionizing particles (MIPs) that enter the vessel with an efficiency of at least 99 %, a requirement that imposes strict demands on scintillation light collection. Improving the probability that photons generated in the scintillator are registered in a wavelength-shifting optical module (WOM) is therefore essential. This thesis investigates an approach based on enhancing the optical reflectivity of the detector’s inner surfaces using a specialized reflective coating. Due to the coating’s efficient diffuse reflectance for visible and near-UV photons, the average photon path within the detector is extended, boosting the likelihood of detection.

The first part of this thesis in chapter 6 presents a comprehensive study of the OPRC

coating for use in the SBT. Reflectivity measurements were conducted to quantify its optical performance, and a series of chemical compatibility tests were performed to ensure long-term stability in contact with all relevant detector materials, including the liquid scintillator. In addition, accelerated aging studies were carried out to assess potential degradation over time. Building on these results, the coating was applied to two SBT prototype detectors, which were subsequently evaluated in test-beam campaigns at CERN and DESY. The performance improvements, practical considerations for large-scale application, and implications for the final SHiP detector design are discussed in detail.

The second major project, discussed in chapter 8 of this thesis, arose through a collaboration with the Eberhard Karls Universität Tübingen, and focused on the development of a novel photo sensor concept: the Silicon Photomultiplier with Active Light Guide (SiALG) design. In this approach, a polystyrene-based plastic scintillator acts as an active light guide directly coupled to a silicon photomultiplier (SiPM). By embedding the scintillating light guide into the sensor structure, the angular acceptance for incoming photons is significantly increased. Developed for the application in liquid scintillator detectors, the SiALG improves detection efficiency by guiding collected photons to the SiPM and converting them to wavelengths matched to the SiPM's peak sensitivity. To support this development, a polymerization method for producing high-quality polystyrene scintillators was established and optimized as part of this thesis. The resulting scintillator samples were characterized in terms of their optical absorbance, fluorescence properties and light yield, providing key input for the refinement of the SiALG concept.

Together, these two projects address complementary aspects of light detection in scintillation-based detectors: enhancement of photon transport via reflective surfaces and improvement of photon sensing through integrated scintillating structures. Both contribute to the broader experimental toolkit necessary for the next generation of large scale particle-physics experiments aimed at uncovering physics beyond the Standard Model.

In addition to presenting these results, this thesis is structured to provide the necessary theoretical and experimental context. Chapter 2 outlines the motivation for this work and summarizes the current status of particle physics. Chapter 3 introduces the fundamental principles of scintillation detectors, including the scintillation mechanism, relevant scintillation materials and commonly used photo sensors. Chapter 4 describes the experimental methods and setups employed throughout this thesis. Chapter 5 presents the SHiP experiment offering both a general overview of the facility and a detailed description of the Surrounding Background Tagger (SBT), which is of central importance in this work. Chapter 6 discusses the research on implementing a reflective coating in liquid-scintillator detectors in the context of the SBT, including laboratory-scale compatibility studies and the coating of two detector prototypes used in test-beam campaigns. Chapter 7 introduces the SiALG design and chapter 8 presents the development and characterization of self-manufactured plastic scintillators, including the polymerization procedure and the evaluation of their optical and scintillation properties for SiALGs.

## 2 The Standard Model of Particle Physics

*“Chaotic patterns unfold, it’s such an elegant mess”*

— Northlane, *Dream Awake*

The Standard Model of Particle Physics is a foundational concept that describes the elementary particles and the forces acting between them, shaping our understanding of the vast universe at the most basic level. Based on the work of many scientists, the Standard Model was developed over several decades during the 20th century. It stands as one of the most rigorously tested theories in physics, uniting quantum mechanics and special relativity to explain a broad variety of phenomena.

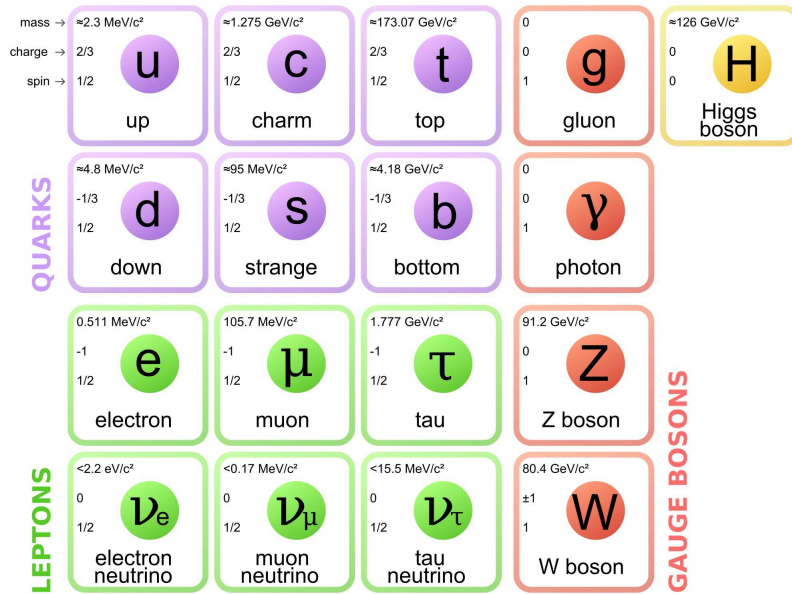


Figure 2.1: Overview of elementary particles according to the Standard Model [1].

The Standard Model identifies the elementary particles and categorizes them according to figure 2.1:

- Fermions** are particles that participate in the formation of matter. They are further divided into quarks and leptons.
 There are six types of **quarks** (Up, Down, Charm, Strange, Top, Bottom) that carry fractional charges ( $+2/3$  or  $-1/3$ ) and combine to form neutrons, protons and other hadrons.
 The category of **leptons** includes the Electron, Muon and Tau that carry a negative charge. The three corresponding neutrinos are electrically neutral and only interact through the weak nuclear force.

- **Bosons** are also known as force-carriers. This class of particles is responsible for the mediation of three of the four known forces of nature.

The fundamental forces that are included in the Standard Model are described as follows:

- The **electromagnetic force** acts on all particles that carry an electric charge and is mediated by photons. It plays a vital role in physical phenomena such as light, electricity and magnetism.
- The **weak nuclear force** affects all fermions and is responsible for processes such as the radioactive beta decay. The short-ranged nature of this force is due to the high mass of the W and Z boson that serve as mediators.
- The **strong nuclear force** provides a binding mechanism for quarks to form hadrons. While it is the strongest fundamental force, it only operates at subatomic distances. Mediation of this force occurs through the gluon.

While an explanation for **gravity** is not included in the Standard Model, it is sufficiently described by General Relativity.

The Higgs mechanism is a key component of the Standard Model, which explains how particles acquire mass through interactions with the Higgs field. The discovery of the Higgs boson in 2012 at CERN's Large Hadron Collider provided crucial evidence for this mechanism, reinforcing the validity of this fundamental theory.

Despite its widespread success, it is clear that the Standard Model is still incomplete: Phenomena such as dark matter, dark energy or the integration of gravity cannot be explained in it's current state. Furthermore, mysteries such as the non-zero mass of neutrinos and the dominance of matter over antimatter remain unresolved. Consequently, finding extensions for the Standard Model to close these gaps is of high priority in latest research in the field of particle physics. Nevertheless, the Standard Model provides a robust foundation for particle physics and continues to guide experimental and theoretical research in in this field [2][3][4].

As a part of an international collaboration that is committed to the *Search for Physics beyond the Standard Model*, the SHiP experiment (Search for Hidden Particles) aims to detect a variety of proposed particles in, so far, largely unexplored energy regions. First measurements are expected to begin in 2031 in the Experimental Cavern North 3 (ECN3) at CERN [5]. Detailed information on the motivation and setup of the experiment can be found in chapter 5 and research results related to the Surround Background Tagger (SBT) of SHiP are discussed in chapter 6.

In an effort to increase the detection efficiency of particle detectors in general, this thesis also provides research in the field of plastic scintillators. By producing conically shaped active light guides and coupling them onto Silicon Photomultipliers (SiPMs), these light sensitive components can cover a broader area of the individual detector. This approach is titled *Silicon Photomultiplier with Active Light Guide Design* (SiALG) and developed in collaboration with the *Universität Tübingen*. Chapter 7 gives an introduction to thermal polymerization, which is a key process in the production of

plastic scintillators. In chapter 8 the production of these scintillators is optimized and the results are characterized.



# 3 Scintillation Detectors

*“Alchemize, I’ll turn the dark to light”*

— Silent Planet, *Collider*

Scintillation detectors play a crucial role in particle physics, where they are widely used for detecting and analyzing the fundamental particles that make up the universe. These detectors leverage the scintillation process, where specific materials emit flashes of light when struck by high-energy particles such as electrons, protons, or other subatomic particles. This light is then captured by photodetectors, such as photomultiplier tubes (PMTs) or silicon photomultipliers (SiPMs), and converted into electrical signals that can be analyzed to determine the properties of the particles [6][7].

In particle physics experiments, scintillation detectors are often used in large-scale particle accelerators and colliders, such as the Large Hadron Collider (LHC), where they are essential for tracking the trajectories, energies, and types of particles produced in high-energy collisions [8]. Their ability to provide precise timing information and their high sensitivity make them invaluable for identifying rare particle interactions and studying phenomena such as neutrino oscillations [9], quark-gluon plasma [10], and the search for new particles beyond the Standard Model [11].

The design and material choice of scintillation detectors can be tailored to specific experimental needs, allowing researchers to optimize them for detecting particular particles or radiation types [6][12]. Their versatility and effectiveness in a variety of conditions, from deep underground laboratories to high-energy particle accelerators, make scintillation detectors a fundamental tool in the ongoing exploration of the subatomic world.

This chapter will present theoretical basics on particle detectors, providing information on organic scintillators, the scintillation process, liquid scintillator related chemicals and photosensitive equipment.

## 3.1 Organic Scintillators

Scintillators are materials that exhibit the unique property of scintillation, emitting light - designated as *luminescence* - when exposed to ionizing radiation. The term “scintillation” refers to the process where incoming radiation interacts with the scintillator material, exciting its atoms or molecules to higher energy states. As these excited states return to their lower energy states, they release photons of light. This emitted light is then detected and utilized to measure and identify the incoming radiation [6][13].

Scintillators play a pivotal role in radiation detection devices such as gamma-ray spectrometers, positron emission tomography (PET) scanners and other medical imaging equipment. Their ability to convert ionizing radiation into detectable light signals makes them relevant in fields like nuclear and particle physics and medical diagnostics.

In the context of a scintillation detector, the scintillator is also known as the active medium. The diverse range of scintillator materials available, including organic and inorganic compounds, allows for customization based on specific application requirements. Ongoing research continues to advance scintillator technology, enhancing sensitivity, efficiency, and overall performance in various radiation detection applications [14]. Organic scintillators represent a diverse class of materials with distinctive properties. Unlike their inorganic counterparts, organic scintillators are composed of carbon-based compounds, typically containing hydrogen, carbon and sometimes nitrogen. They are categorized into four types: liquids, plastics, single crystals and glass. These materials exhibit scintillation, emitting photons of light upon interaction with ionizing radiation. The proportionality between deposited energy and detected photons is utilized to identify the particle energy, while the form of the signal can give information on the particle type [7] [15].

The aromatic benzene ring, a hallmark of organic chemistry, plays a crucial role in the molecular structure of organic scintillators. The spacial orbital model of the benzene ring is depicted in figure 3.1.

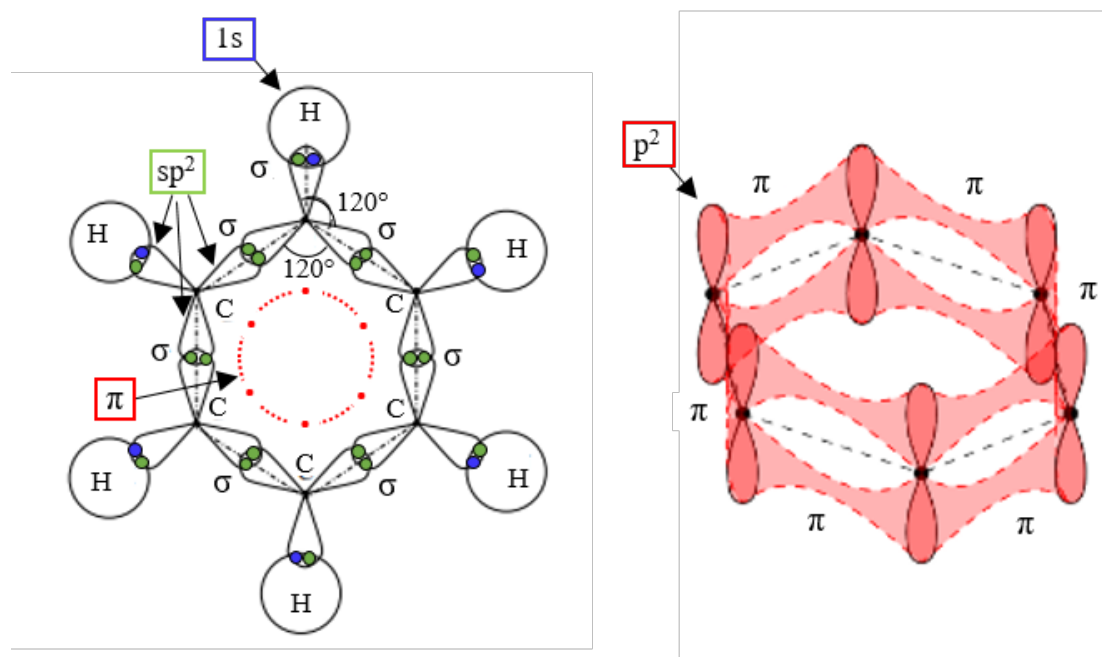


Figure 3.1: Based on [16]: **Spacial orbital model of benzene ring:** left: electron distribution within the orbital model of the benzene ring; green dots: electrons of C-atoms; blue dots: electrons of H-atoms; white orbitals: three  $sp^2$  orbitals of each C-atom forming  $\sigma$ -bonds with two adjacent carbon-atoms and one hydrogen-atom) resulting in planar structure of the benzene ring ( $120^\circ$  angle between each C-atom and each C-atom and H-atom); right: continuous cloud of  $\pi$ -electron density evenly distributed above and below the ring plane.

The following list gives an overview on how the chemical structure plays a role in

providing the molecule with scintillating properties. This part is based on sources [13], [7], [12] and [17].

- **The C-atom within the orbital model**

Carbon atoms contain six electrons in their atomic shell, two of which are bound in the inner 1s-orbital and do not contribute to binding within a molecule. The four remaining outer electrons (valence electrons) are distributed over one 2s-orbital (two electrons) and two 2p-orbitals (one electron each). Within molecules C-atoms form different electronic configurations depending on the formed bond.

- **Aromatic Stability**

The benzene ring is characterized by six carbon atoms arranged in a planar, hexagonal structure ( $120^\circ$  angle between each C-atom and each C-atom and H-atom). Within the benzene ring valence electrons of each carbon atom are configured as *sp*<sup>2</sup>-hybridorbitals containing one electron each. With these, one carbon atom forms  $\sigma$ -bonds with two adjacent carbons and one hydrogen atom. The six vertically oriented 2p-orbitals (one for each C-atom) contain one electron each and are not involved in hybridization. Within the benzene ring they form a conjugated system of six  $\pi$ -electrons above and below the plane. This results theoretically in three single and two three bonds between C-atoms evenly distributed within the benzene ring. The resonance hybrid of the various possible resonance structures imparts stability to the benzene ring, a phenomenon known as aromatic stability.

- **Delocalization of  $\pi$ -Electrons**

The key feature of the benzene ring is the delocalization of its  $\pi$ -electrons throughout the entire ring. This delocalization occurs due to the resonance of electrons between adjacent carbon atoms, resulting in a continuous cloud of  $\pi$ -electron density above and below the ring plane. This delocalized system of electrons enhances the stability of the benzene ring, making it resistant to reactions that typically break  $\pi$ -bond conjugation.

- **Contribution to Scintillation**

The delocalized  $\pi$ -electron system in the benzene ring allows for efficient absorption of energy from ionizing radiation. When a high-energy particle interacts with the scintillator material, it can excite these  $\pi$ -electrons to higher energy states, initiating the scintillation process.

- **Tailoring Scintillation Properties**

The ability to modify the molecular structure of organic scintillators, including the benzene ring, allows for tailoring of scintillation properties. By introducing specific functional groups or altering the conjugated system, the response of the scintillator to different types of ionizing radiation can be defined. Therefore, scintillators can be chosen for a relevant application, in regard to characteristics such as light yield, decay time, energy resolution and emitted wavelength.

In this thesis, the focus lies on liquid scintillators in the context of the SHiP Surround Background Tagger in chapter 6. Plastic scintillators also play a vital role in this thesis, as they are produced via thermal polymerization for the application as an active light guide in the SiALG design. Chapters 7 and 8 provide insights on plastic scintillator production and results concerning transparency, life time and light yield. The following chapter details the scintillation process in the context of organic scintillators.

### 3.1.1 The Scintillation Mechanism

When a charged, ionizing particle enters an organic scintillator, it interacts with  $\pi$ -electrons by exciting them. Via different processes such as scattering and ionization, the particle deposits its kinetic energy into the active medium, while it travels through the detector. The energy deposition of a particle over a distance  $-dE/dx$  in a specific material is best described with the Bethe-Bloch formula:

$$-\frac{dE}{dx} = \frac{4\pi n z^2}{m_e c^2 \beta^2} \left( \frac{e^2}{4\pi\epsilon_0} \right)^2 \left[ \ln \left( \frac{2m_e c^2 \beta^2}{I(1-\beta^2)} \right) - \beta^2 \right] \quad (3.1)$$

This formula contains  $\beta = v/c$ ,  $v$  as the current velocity of the particle,  $c$  as the speed of light,  $E$  as the energy of the particle,  $x$  as the distance traveled in the medium,  $z$  as the charge number of the particle,  $\epsilon_0$  as the vacuum permittivity,  $e$  as the elementary charge,  $n$  as the electron density of the material,  $m_e$  as the mass of the electron, and  $I$  as the mean excitation potential [12].

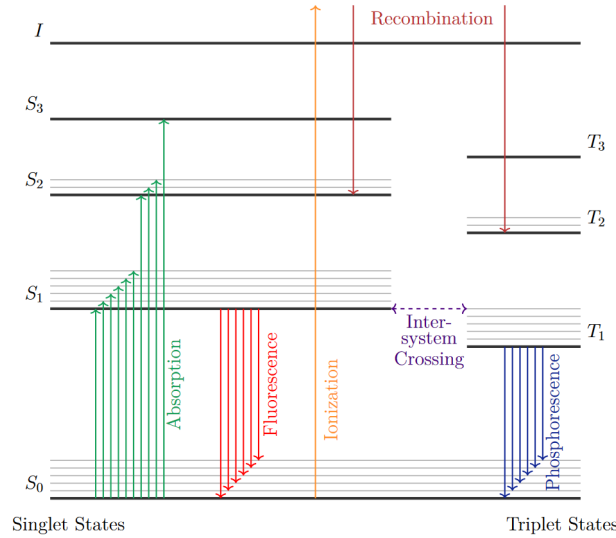


Figure 3.2: Jabłonski diagram: Schematic overview of molecular energy levels. The thick black lines represent spin states and the ionization energy while the grey lines refer to the vibrational sub-states. Possible transitions between energy levels are indicated by colored arrows [18].

Figure 3.2 shows a Jabloski diagram, a typical energy diagram for  $\pi$ -molecular orbitals

in an organic scintillator molecule. The  $\pi$ -electron within an aromatic compound exists in a ground state  $S_0$  and can be excited to higher energy states by interacting with ionizing particles. While  $S_i$  ( $i \geq 1$ ) are the excited singlet spin states,  $T_i$  ( $i \geq 1$ ) are the excited triplet spin states. Grey lines represent vibrational sub-levels.

Singlet spin-states can be directly populated by energy absorption, as the spin orientation is equal to that in the ground state  $S_0$ . The molecule rapidly dissipates from its excited state  $S_i$  to the  $S_1$  state by collision with neighboring molecules, also called internal degradation. The electron then de-excites to  $S_0$  by emitting a characteristic photon known as fluorescence. The life time of this excited state is typically a few nanoseconds, as there is ideally no selection rule that prohibits this transition.

Triplet states have an anti-parallel spin compared to the singlet states. Therefore, they cannot be excited directly from ground state, due to the prohibition of electromagnetic transitions with spin change. Inter-system crossing allows for the formation of an excited triplet state from a previously excited singlet state through a non-radiative transition. As the decay from an excited triplet state to ground state is highly forbidden, life times for this de-excitation are in the order of microseconds. Photons that are released in this decay is called phosphorescence. Alternatively, two excited triplet states  $T_1$  can interact and form an  $S_1$  and  $S_0$  state. This leads to the release of delayed fluorescence, next to the prompt fluorescence, adding a delayed component to the scintillator signal [13][2].

The energy of photons that are emitted from the scintillator molecules is lower than the excitation energy due to non-radiative transitions from the vibrational sub-levels of the  $\pi$ -electrons. This leads to longer wavelengths, to which the material offers a higher transparency.

Generally the scintillator emits photons in a distribution that corresponds to the individual emission spectrum of the material. This spectrum results from various de-excitation paths which a  $\pi$ -electron can undergo, while giving off different amounts of energy non-radiatively. While a part of these emitted photons no longer provides enough energy to lead to further excitation of molecules, photons with higher energies may be subject to self-absorption in the scintillator, effectively reducing the signal. In figure 3.5 this effect is shown in the overlap of absorption and emission spectrum of linear alkylbenzene.

To counteract this effect, it is common practice to add so called wavelength-shifters or fluors to the scintillator. These additives provide a shift to the scintillation light, further reducing the energy of the emitted light and lowering the probability for self-absorption [12][13][2]. The process of wavelength-shifting is detailed in the following chapter.

### 3.1.2 Wavelength-shifting with Fluors

In an effort to reduce self-absorption, wavelength shifters or fluors are added to the base scintillator. Wavelength-shifting describes the mechanism, where a fluor molecule absorbs a UV-photon (provided by the scintillator base) and re-emits the photons with less energy and therefore a longer wavelength. This results in photons in the visible range, to which the scintillator is more transparent. The process can be described

via the Franck-Condon principle, which is illustrated in figure 3.3. The graph shows the ground spin state  $S_0$  and the first excited spin state  $S_1$  as Morse potentials of a  $\pi$ -electron. The corresponding wave function of vibrational sub-levels  $v_i$  in both spin states are included.

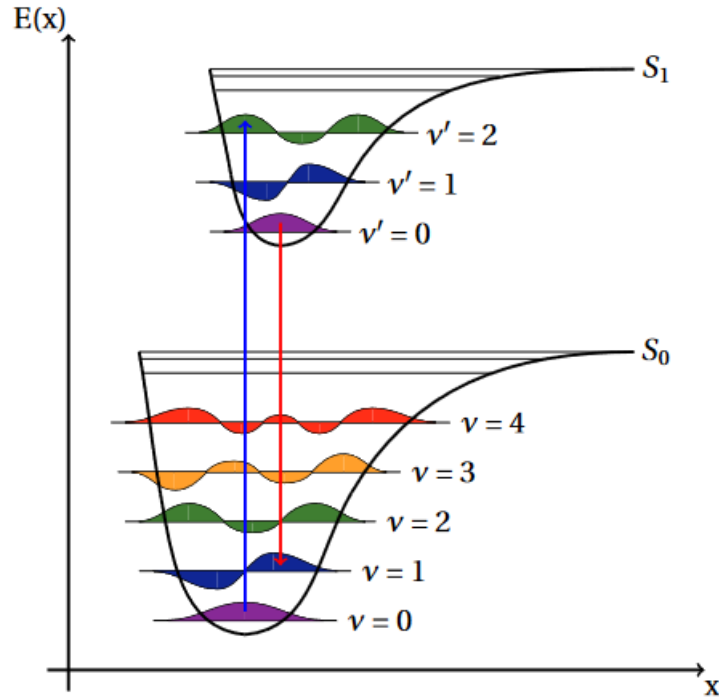


Figure 3.3: Illustration of the Franck-Condon principle [16][13]

When a  $\pi$ -electron is excited from ground state, it can only occupy a higher energy state, when the wave functions of both states overlap in space. This excitation is illustrated by the blue arrow. Due to a change in wave function, when this higher state is occupied, the electron can no longer de-excite via the same path as it was excited. Through non-radiative transitions, the electron begins to de-excite to the lowest vibrational level in the excited spin state. When an overlap in wave functions allows for transition to the ground state, the electron emits a photon.

Due to vibrational transitions, the energy of the emitted photon is lower than the absorbed photon which is also called a Stokes shift. By adding two or more fluors to the scintillator, the wavelength-shift can occur multiple times, as long as the emission of the primary fluor overlaps with the absorption spectrum of the secondary fluor. With this practice, the emission wavelength of the scintillator can be tuned to the individual application. This is especially helpful to match the emission wavelength to the sensitivity of the photosensitive equipment. This chain of wavelength-shifting is illustrated in figure 3.5 with a base scintillator of Linear Alkylbenzene and the added fluors 2,5-Diphenyloxazole and 1,4-Bis(2-methylstyryl)benzene. The following chapter will introduce these scintillating chemicals [12][13][2].

## 3.2 Scintillator Components

Liquid scintillators are typically a combination of a solvent and one or more fluors. While there is a wide variety of options that can benefit the individual experiment, this part of the thesis lays focus on the scintillating chemicals that are used in the SHiP SBT. They are presented in the following chapters.

### 3.2.1 Linear Alkylbenzene (LAB)

Linear Alkylbenzenes (LAB) is the name of a family of organic compounds that has gained popularity in the recent past as a liquid scintillator solvent for large scale particle detectors. Especially neutrino experiments, such as JUNO, SNO+ or the Daya Bay Reactor Neutrino Experiments, have made use of its beneficial properties [16][19][20].

Because of its high transparency, high light yield and typically low radioactive impurities it shows optimal properties for the use in scintillator physics. LAB is also known for its excellent solvent properties, chemical stability and compatibility with other organic compounds which are essential properties for the utilization in multi-component scintillators [21][22].

Additionally, it has a high flash point and there are no concerns for human health or the environment, in contrast to previously used compounds (such as Pseudocumene [23][24]). This allows for safe handling of the substance and less environmental impact through potential leakage of the detector [25][21]. LAB is typically produced as a cost-effective intermediate for detergents and surfactants which makes LAB widely available [26].

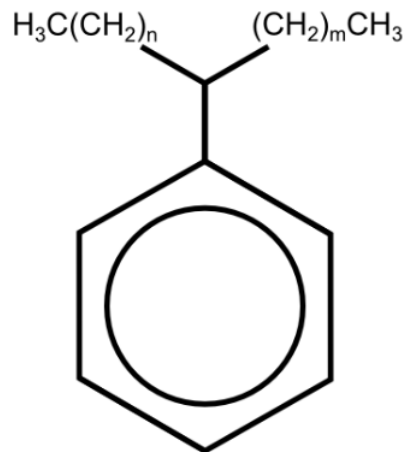


Figure 3.4: Chemical structure of Linear Alkylbenzene [16]

As shown in figure 3.4, the molecular structure of LAB consists of a benzene ring, which is necessary for fluorescence, and two hydrocarbon chains. The lengths of these two chains vary and are described by  $n$  and  $m$  respectively. In this thesis, a particular

LAB with a cut of  $n + m = 7 - 10$  (SASOL Hyblene 113) was used which leads to a linear alkyl chain length (all combined hydrocarbon chains) of  $C_{10} - C_{13}$  [27][28]. Important chemical and physical properties of LAB can be found in table 3.1.

|                    |                                                     |
|--------------------|-----------------------------------------------------|
| Name               | Linear Alkylbenzene (LAB) ( $C_{10} - C_{13}$ )     |
| Chemical Formula   | $C_9H_{12}(CH_2)_{m+n}$ with $7 \leq n + m \leq 10$ |
| Density            | $0.857 \text{ kg/l}$                                |
| Absorption Maximum | $260 \text{ nm}$                                    |
| Emission Maximum   | $283 \text{ nm}$                                    |
| Hazards            | flammable                                           |
| Flash Point        | $140^\circ \text{C}$                                |

Table 3.1: Chemical and physical properties of LAB [28][17]

Figure 3.5 shows absorption and emission spectra of the most important liquid scintillator components that were used during this thesis. LAB absorbs and emits light in the UV-region with a large spectral overlap which leads to a high probability of self-absorption which in turn reduces the transparency of the scintillator drastically.

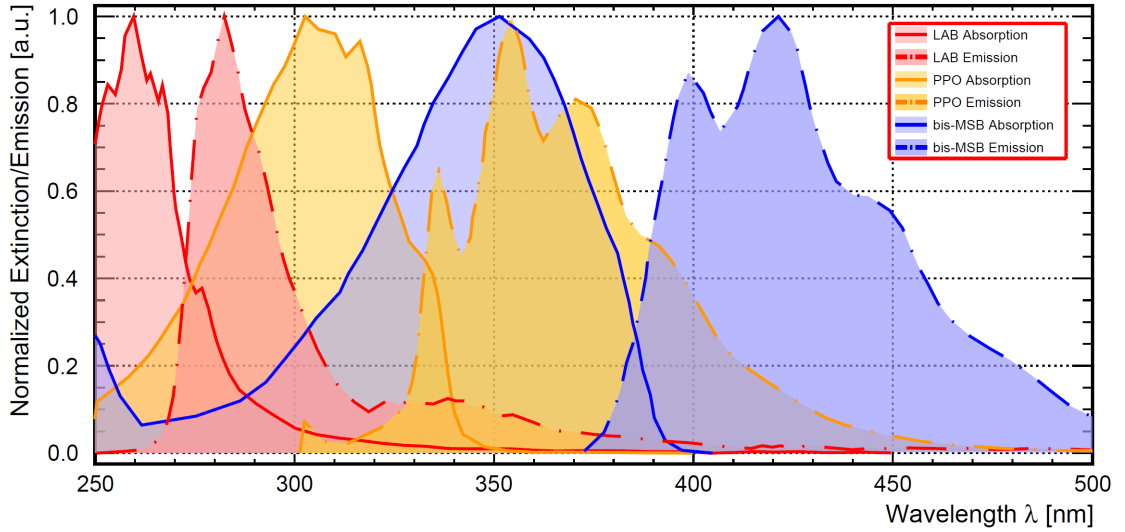


Figure 3.5: Absorption- and emission spectra of LAB (red), PPO (yellow) and bis-MSB (blue) [16]

As discussed in chapter 3.1.2, it is common practice to add fluors to scintillators to shift emitted photons to a higher wavelength, to prevent the loss of fluorescence and increase the transparency of the scintillator for the scintillation light. This effect of increased transmission at longer wavelengths is clearly shown in figure 3.6.

The following sections will go into detail on fluors that are used during this thesis.



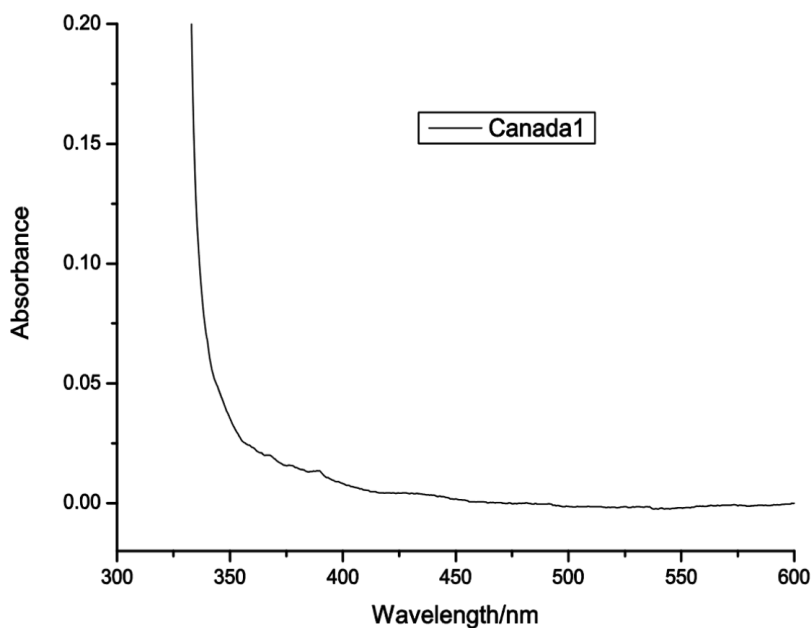


Figure 3.6: Absorption spectrum of a purified LAB sample [29]

### 3.2.2 2,5-Diphenyloxazole (PPO)

PPO is frequently used as a primary fluor in liquid- and plastic scintillators. Figure 3.7 shows the chemical structure of PPO which contains an oxazole ring and two benzene rings. These provide a large number of  $\pi$ -electrons that can be excited for fluorescence.

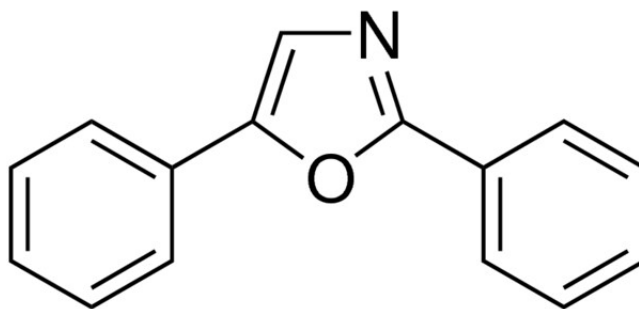


Figure 3.7: Chemical structure of PPO [30]

Figure 3.5 shows the absorption spectrum of PPO covering the complete LAB emission spectrum. The respective peaks are located only 20 nm apart, ensuring efficient energy transfer from the solvent to the fluor [31]. Table 3.2 shows that the absorption and emission peaks of PPO are located 52 nm from each other. Not only does this lead to minimal spectral overlap and therefore a low probability of self-absorption, but it also introduces a Stokes shift to wavelengths at the lower end of the visible electromagnetic

spectrum. Light in this spectral region is typically suitable for the detection by photomultipliers or other photosensitive equipment [32][33].

|                    |                           |
|--------------------|---------------------------|
| Name               | 2,5-Diphenyloxazole (PPO) |
| Chemical Formula   | $C_{15}H_{11}NO$          |
| Absorption Maximum | 303 nm                    |
| Emission Maximum   | 355 nm                    |

Table 3.2: Chemical and physical properties of PPO [34]

PPO is known for a relatively high light yield which is crucial for scintillation detectors to provide high sensitivity and efficiency. A low fluorescent life time of 1.4 ns makes it suitable for the application in high-energy physics, where rapid signal detection is a necessity. PPO-based scintillators also achieve good energy resolution, allowing for distinction between energy levels of incident radiation [35][36].

The solubility of PPO in various organic solvents makes it suitable for incorporation into liquid and plastic scintillators and its compatibility with other materials and ease of processing contribute to its versatility in scintillator design [36].

PPO is generally stable under typical operating conditions, but the long-term stability can be influenced by factors such as temperature, exposure to light or the presence of solved oxygen. As these factors typically present a danger to all kinds of organic scintillators, additional protective measures are often employed including proper encapsulation or the addition of antioxidants [37][38].

By adding another shift to the fluorescence spectrum of PPO the attenuation length of the photons within the scintillator is increased even further. The following section will look into 1,4-Bis(2-methylstyryl)benzene which is often used in scintillators also containing PPO [38][39][40][37].

### 3.2.3 1,4-Bis(2-methylstyryl)benzene (bis-MSB)

Bis-MSB is a typical addition to scintillators that also contain PPO as the absorption spectrum of the secondary fluor overlaps with most of the PPO emission spectrum, as seen in figure 3.5. The chemical structure of Bis-MSB is shown in figure 3.8.

Further information on the compound is listed in Tab 3.3.

While bis-MSB is often included in liquid scintillators with LAB and PPO, in this thesis, it is used in polystyrene-based plastic scintillators (see chapter 7) and in wavelength-shifting optical modules (see chapter 5.3.2).

|                    |                                         |
|--------------------|-----------------------------------------|
| Name               | 1,4-Bis(2-methylstyryl)benzol (bis-MSB) |
| Chemical Formula   | $C_{24}H_{22}$                          |
| Absorption Maximum | 347 nm                                  |
| Emission Maximum   | 420 nm                                  |

Table 3.3: Chemical and physical properties of bis-MSB [42]

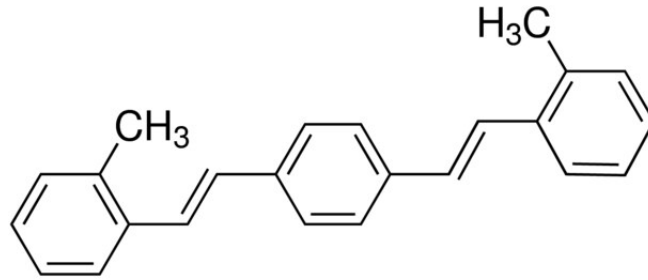


Figure 3.8: Chemical structure of Bis-MSB [41]

Bis-MSB shares many advantages with PPO such as high light output, good energy resolution and relatively low cost of production [43]. Under the correct conditions (oxygen-free environment, room temperature) it is also chemically stable for the use in detectors involved in long term measurements [42].

Especially in combination with silicon photomultipliers, the shift to 420 nm is useful as it is close to the maximum photo detection efficiency of the instrument. This is discussed in more detail in chapter 5.3.2 and, more specifically, in figure 5.7.

### 3.3 Photo Sensors

Photo sensors are components that detect and measure light intensity by converting photons into an electrical signal. They are integral devices to a variety of applications, such as medical imaging, environmental monitoring or industrial automation. This thesis focuses specifically on the application of these photo sensors in the context of scintillation detectors.

As discussed in chapter 3, particle detectors consist of an active medium, the scintillator, which emits a characteristic radiation, when energy is deposited by a traversing particle. The form and intensity of the light signal determines the particle type, the deposited energy and the particle interaction that occurred in the active medium. Photo sensors are responsible for the conversion of incoming light into an amplified electric signal (gain). The sensitivity of a photo sensor describes the ability to detect low-intensity light.

Two commonly used photo sensor technologies are Photomultiplier Tubes (PMTs) and Silicon Photomultipliers (SiPMs) which are often employed in highly sensitive light detection scenarios. The following sections will explain the basic working principles of the two components and compare them for the application in liquid scintillator-based detectors.

#### 3.3.1 Photomultiplier Tubes

Photomultiplier tubes (PMTs) operate based on the photoelectric effect and secondary electron emission and are the current standard in light detection. They are designed to detect and amplify very low light levels, often down to single photons, with high sensitivity and efficiency. The working principle of a PMT is explained according to figure 3.9.

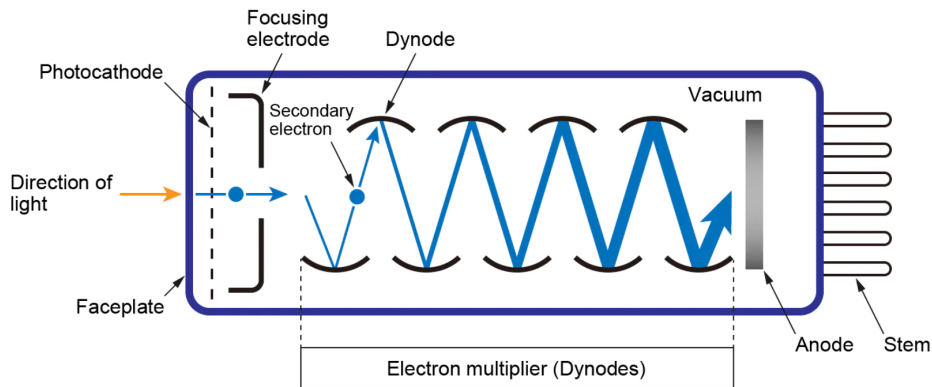


Figure 3.9: Schematic depiction of the working principle of a PMT [44]

When light enters through a glass window at the front end of the PMT, it hits a photocathode that emits electrons when struck by photons. This process is based on the photoelectric effect, where the energy of the photon causes the ejection of

electrons from the photocathode. These incident electrons are then accelerated and focused onto a series of dynodes through an electrical field. Maintained at progressively higher voltages, these dynodes release multiple secondary electrons when struck by a photoelectron. This causes a cascading amplification as the secondary electrons are accelerated to the next dynode where they produce further electrons. After passing all cathodes, the multiplied electrons are collected at the anode which converts them into an electrical signal that is proportional to the incident light intensity.

PMTs are comparatively large in size and fragile due to a faceplate consisting of UV-transparent glass. The electron acceleration requires a voltage in the order of  $10^3\text{V}$  to achieve a gain in the order of  $10^8$ . The operation under a vacuum ensures low noise which is of vital importance to high sensitivity measurements. Due to the influence of external magnetic fields on the trajectory of the photo electrons and secondary electrons, the performance of a PMT can change if not properly shielded [45] [46].

### 3.3.2 Silicon Photomultipliers

Silicon photomultipliers (SiPMs) are highly sensitive solid-state photo detectors. They consist of an array of avalanche photodiodes (APDs), also named microcells, working in Geiger mode. The absorption of a photon leads to an amplified electrical signal through an avalanche multiplication process. Figure 3.10 shows a schematic to clarify the working principle of a SiPM.

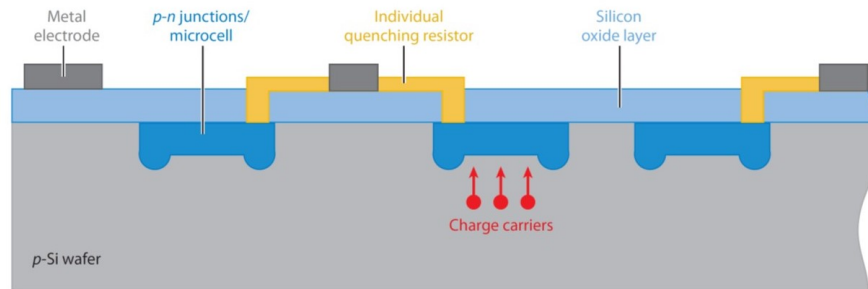


Figure 3.10: The working principle of a SiPM [47]

Silicon belongs to the class of semi-conductors, which are materials that present electrical conductivity between insulators and conductors. This is due to a band gap separating valence and conduction bands. The band gap describes the energy difference between the two bands. Only electrons in the conduction band are able to travel freely in the material, meaning the band gap also describes the amount of energy an electron needs to absorb to contribute to the electrical conductivity of the material. When this promotion of an electron occurs, a positively charged "hole" is created simultaneously due to charge conservation [48].

SiPMs are fabricated by doping silicon wafers to form pn-junction diodes, both electrically and optically isolated and oriented in an array. Each of these diodes are reversed biased above the breakdown voltage of the diode to create a strong electrical

field within each microcell. When a photon is absorbed in the SiPM, it creates an electron-hole pair that is accelerated in the electrical field. The collision of these charge carriers with silicon atoms produces further electron-hole pairs through impact ionization. This is known as the avalanche multiplication effect, which enables single photons to trigger measurable electrical pulses. To stop the indefinite continuation of this avalanche, each microcell contains a quenching resistor to temporarily reduce the voltage below breakdown voltage. This leads to a binary output pulse from each individual microcell. The final output of the SiPM is the sum of all microcell pulses which leads to a signal proportional to the number of incident photons [49] [50] [51]. In comparison to the fragility of PMTs, SiPMs have the advantage of a compact and robust build. The operating voltage in the order of  $10 - 100\text{ V}$  is lower than the voltage that is necessary for PMTs while the gain of the electric signal can reach the order of  $10^6$  (lower end of PMT gain). A disadvantage of SiPMs is the thermal noise which can be minimized by cooling the component.

The spectral range of the two photodetector types are compared in 5.3.2 for the application in wavelength shifting optical Modules for the SHiP Surrounding Background Tagger.

This concludes the overview on scintillation detectors and the individual components. The following chapter will present Methods and experimental setups that are applied throughout this thesis.

## 4 Methods and Experimental Setups

*“Feel like I wanna tear this body apart and make it whole again”*

— Greyhaven, *Night In October*

The characterization of scintillators, in the context of particle detectors, mostly involves measuring the optical properties. Absorption measurements convey how far light can travel within the scintillator. Fluorescent measurements record the emissive behavior of the scintillator including the range of emitted wavelengths and timing of the emission. These measurement methods are applied throughout this thesis with liquid and plastic scintillators.

In order to investigate the impact of long term aging on the scintillator quality, thermal aging tests are conducted. By incubating scintillator samples, chemical reactions are accelerated. This allows for testing of the compatibility of different materials in contact with the scintillator. During these aging tests, the optical properties of the scintillator are measured to identify potentially harmful combinations of materials. This method is applied in chapter 6.3, in the context of the SHiP Surround Background Tagger.

To measure the diffuse and specular reflectivity of different materials, a self constructed reflectivity setup is improved during this thesis. This method is used in chapter 6 to evaluate the reflectivity of different combinations of reflective coating on steel, with the goal to improve the reflectivity of the inner walls of the Surround Background Tagger. By combining this approach with thermal aging tests, the coating quality can be assessed after long term contact with liquid scintillator.

This chapter will present different experimental setups and explain in detail the individual methods for sample preparation, measuring and data analysis.

### 4.1 Light Absorbance Spectroscopy

Wavelength dependent light absorption is a measure to quantify the change in light intensity when traveling through a medium. It is an important property of any given scintillator to assess light propagation within any detector setup. To achieve a signal of high intensity, it is vital that the scintillator is highly transparent to the emitted scintillation light, minimizing self-absorption. This method also gives insight into the scintillator quality as impurities and aging processes typically lead to a change in the absorption spectrum. Therefore, this method is not only employed in basic scintillator research but also in studies aimed at scintillator purification or in aging tests that investigate chemical compatibility of different components of a setup.

The light absorption of a sample in general is measured by irradiating it with a light source with the intensity  $I_0$  and measuring the intensity  $I_1$  after the light passes through the sample. The spectrometer *Lambda 850* of the company *Perkin Elmer* is used for all absorption measurements in this thesis. To provide a sufficiently intense spectrum throughout the wavelength range from 175 nm - 900 nm, a combination of a

Tungsten-halogen and Deuterium lamp is used in this setup. In figure 4.1 a screenshot of the software *UV Winlab* is shown that details the different optical components of the spectrometer. A gridless PMT is responsible for the light detection [52].

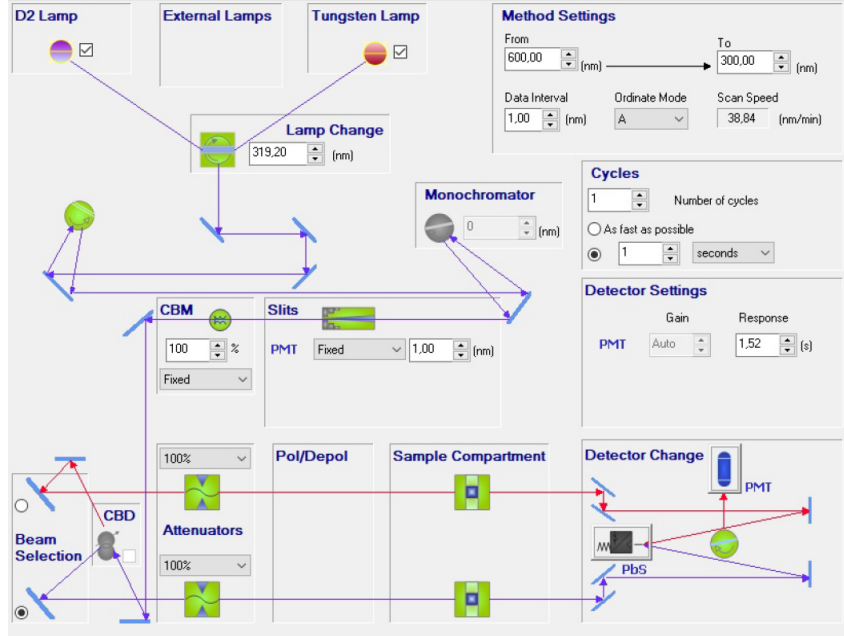


Figure 4.1: Screenshot of the *UV Winlab* software detailing the optical components and different settings in the spectrometer

In this thesis, the spectrometer is employed to measure both liquid and plastic scintillators. To achieve precise absorption measurements, sample preparation is of great importance.

### Sample Preparation

Liquid scintillators are prepared by degassing them with an ultrasonic cleaner. This removes solved gases from the sample, including oxygen, which can induce quenching effects and therefore falsify the measurement. Liquid scintillator samples are transferred into a 10 cm cuvette by *Hellma* made of High Performance Quartz Glass (QS) that features high and constant transmission throughout the entire lamp spectrum [53]. After cleaning the interfaces of the cuvette, it is fixed in a cuvette holder in the spectrometer.

Plastic scintillators which are polymerized in a cylindrical shape, are sanded and polished to guarantee parallel and transparent interfaces. The finalized plastic scintillator is then placed onto a dedicated solid sample holder which is fixed in the sample compartment of the spectrometer.



### Spectrometer Measurements

The spectrometer begins the measurement by recording the initial light intensity  $I_0$  without a sample. This is followed by a measurement of  $I_1(x)$ , the light intensity after transitioning through the sample. The spectrometer provides a value for the absorbance  $A$  for each wavelength.  $A(x)$  is defined in formula 4.1.

$$A(x) = \log_{10} \left( \frac{I_0}{I_1(x)} \right) == \log_{10} \left( \frac{1}{T(x)} \right) = \epsilon c x \quad (4.1)$$

with  $T$  as the transmission and  $\epsilon$  as the molar absorption coefficient. According to the *Lambert-Beer Law*, the absorbance is proportional to the molar concentration  $c$  of a colored solution. This is helpful to identify unknown concentrations of fluors or contaminants in scintillators by comparing them to other known samples [2]. Figure 4.2 shows an absorbance spectrum of a representative liquid scintillator that was measured during this thesis. The plot also gives a comparison between the raw data from the spectrometer and the corrected results which are detailed in the following paragraph.

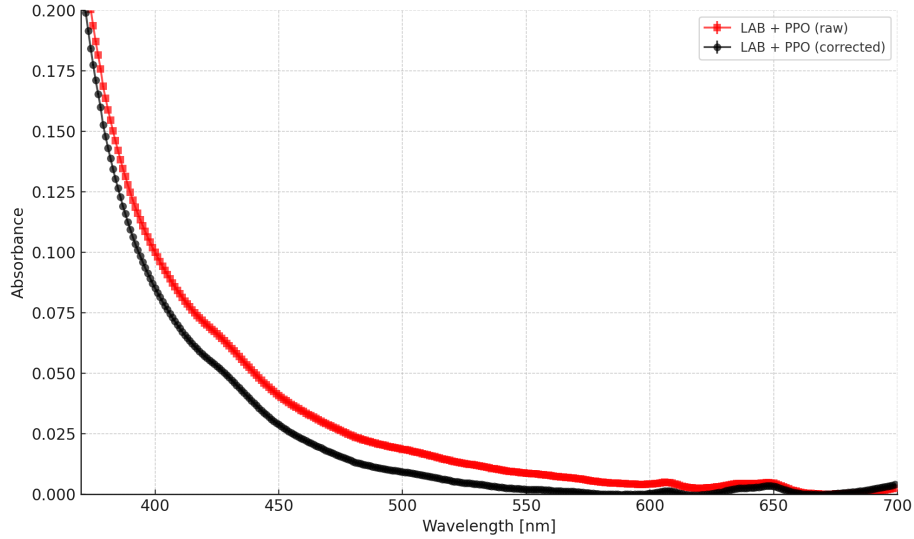


Figure 4.2: Absorbance spectrum of a liquid scintillator sample in a 10 cm cuvette containing LAB and 2 g/l of PPO. The red curve shows the raw data as given by the spectrometer, while the black curve represents the corrected data.

### Measurement Correction and Uncertainty Assessment

When the light transitions from one medium to another, a proportion of light is reflected, while the rest transmissions through the interface. In a cuvette filled with liquid scintillator, light undergoes four transitions; two from air to glass and two from glass to liquid. This leads to undetected light at the end of the light path. To compensate for the undetected light, an absorption spectrum of an empty cuvette

is recorded. This corrective measurement includes four transitions from air to glass. The transmission  $T$  and reflection proportion  $R$  can be computed with the refractive indices  $n_i$

$$\begin{aligned} n_{air} &\approx 1 \\ n_{LAB} &\approx 1.5 \text{ [54]} \\ n_{cuvette} &\approx 1.5 \text{ [53]} \end{aligned}$$

and the following equation, derived from the Fresnel Equations under the condition, that the incident angle is  $0^\circ$ .

$$T = 1 - R = 1 - \frac{(n_1 - n_2)^2}{(n_1 + n_2)^2}. \quad (4.2)$$

By inserting the corresponding values, following results for the two different transitions are computed:

$$\begin{aligned} T_{Air,qs} &\approx 0.96 \\ T_{air,liquid} &\approx 1 \end{aligned}$$

These result in the full-cuvette-transmissions:

$$\begin{aligned} T_{empty} &\approx 0.96^4 \\ T_{filled} &\approx 0.96^2 \end{aligned}$$

Due to the logarithmic absorbance shown in formula 4.1, the following rule applies:

$$T_{filled} = \sqrt{T_{empty}} \iff A_{filled} = \frac{1}{2} A_{empty} \quad (4.3)$$

The correction for the absorbance measurements is computed with the following formula

$$A_{corr} = A_{measured} - \frac{1}{2} A_{empty} \quad (4.4)$$

Finally, after the corrections for the light transitions are applied, the results are compensated for streak formation in the liquid scintillator. These result from small temperature fluctuations within the liquid scintillator or residual alcohol from cleaning the cuvette. The streaks lead to increased light refraction within the liquid, affecting short wavelengths more significantly than long wavelengths. This can lead to a tilt in the measurements. Experimental observations in this thesis have shown that LAB's spectrum reaches close to zero absorbance at 585 nm and 665 nm. The final spectrum is calculated by adjusting the measurement through rotation to fulfill this requirement at both specified wavelengths [55].

Measurements with the UV-Vis spectrometer present several sources of systematic errors. Small inaccuracies in sample preparation can unintentionally change the absorbance of the liquid and imprecise cleaning of the cuvette can affect the measurement accuracy. Due to a small tolerance in cuvette positioning within the sample holder,

the incident angle can deviate from orthogonality, leading to higher light refraction at the optical interfaces than estimated in the corrections. These errors are quantified by applying the standard deviation of a minimum of three measurements of the same sample. The cuvette is removed from the spectrometer, cleaned with isopropanol on the exterior and repositioned in the sample holder between these repeated absorbance scans. Given that the statistical photon counting error is negligible compared to the dominant systematic errors, it is not taken into account in this analysis.

## 4.2 Fluorospectroscopy

Fluorospectroscopy is an analytical technique used to study the fluorescence properties of a substance. When a scintillating material is irradiated by light (usually ultraviolet or visible light), it leads to the excitation of molecules. When these molecules return to ground state, they emit characteristic photons that correspond to the fluorescence spectrum of the scintillator.

In the context of a particle detector, an overlap between the emission spectrum of the scintillator and the acceptance of the photo detector is necessary. Therefore, this method is used in the characterization of new scintillator mixtures and the optimization of the fluor concentration.

The emission properties of all scintillators in this thesis are measured with the *FS5 Spectrofluorometer* from *Edinburgh Instruments*. It employs a xenon arc lamp to irradiate the sample and excite the scintillating molecules. Three different detectors are operated in this setup, including a reference detector that corrects for light fluctuations, a transmission detector and a single-photon counting detector (PMT) for the fluorescence measurements. A detailed overview of the FS5 setup can be seen in figure 4.3. The software *Fluoracle* is used to read out and visualize data from the spectrometer. Further analysis is conducted with a python script.

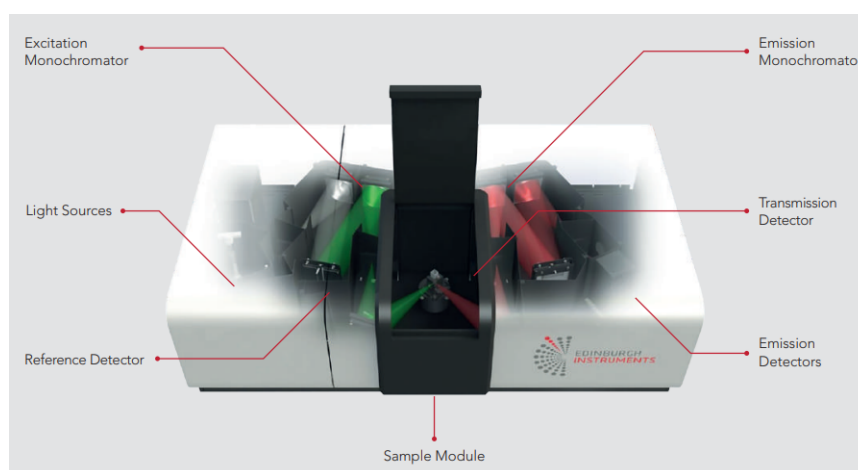


Figure 4.3: Visualization of the FS5 spectrofluorometer including all components and the light path [56]

The sample is irradiated by the lamp and the emitted light is measured at a  $90^\circ$  angle to exclude unabsorbed light from the light source. The uncertainty of these measurements is computed by applying the standard counting error shown in formula 4.5 to the number of detected photons  $N(\lambda)$ .

$$\Delta N(\lambda) = \sqrt{N(\lambda)} \quad (4.5)$$

An exemplary fluorescent spectrum of a liquid scintillator is shown in figure 4.4.

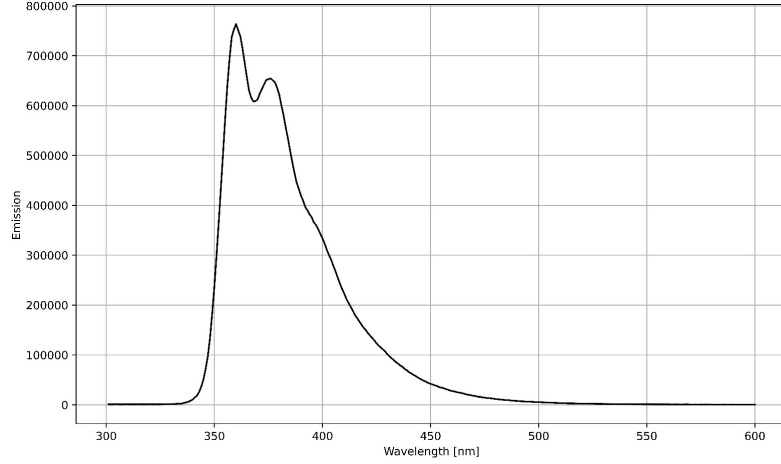


Figure 4.4: Fluorescent spectrum of a liquid scintillator sample consisting of LAB and 2 g/l of PPO

### 4.3 Light Yield

When ionizing radiation deposits energy in a scintillating material, only a fraction of that energy is converted into photons. The light yield is a measure that quantifies this conversion efficiency and is typically given as the number of scintillation photons produced per unit of deposited energy (photons/MeV). This fundamental parameter of scintillator performance directly determines the achievable energy resolution, signal-to-noise ratio, and overall detector sensitivity.

The light yield depends on the material, including the scintillation mechanism, molecular structure, quenching processes, and the efficiency of energy transfer between solvent and wavelength shifters. It is also influenced by external conditions such as temperature, oxygen content, and optical purity. In the context of detector systems, the measured light yield is further influenced by photon-collection efficiency and the spectral response of the photo detector. Accurate measurement of the light yield is therefore essential for comparing different scintillator materials and optimizing the detector design.

The measurements are conducted using a dedicated experimental setup designed to register emitted light under controlled irradiation. The system combines PMTs, a well-defined scintillation cell, a gamma source and an external gamma-ray reference

detector to record coincident events and identify Compton-scattered energy depositions in the scintillator sample. A schematic overview of the key components is shown in figure 4.5.

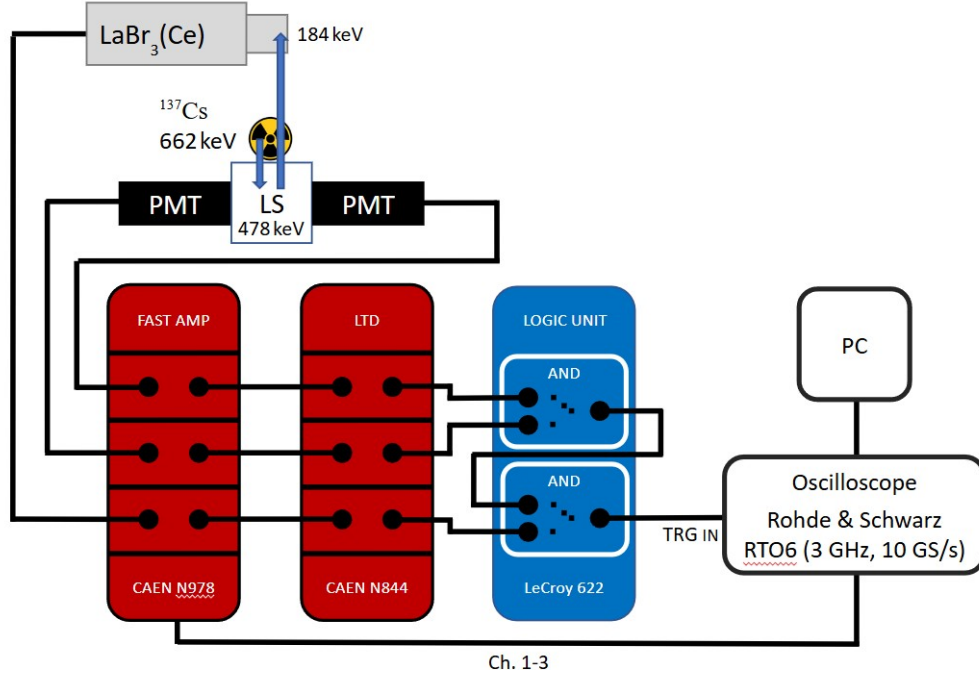


Figure 4.5: Schematic overview of the light yield setup [57]

The scintillator sample is contained in a 1-inch cylindrical PTFE cell, chosen for its high reflectivity and chemical compatibility with organic scintillators. By diffusely reflecting photons towards the PMTs, PTFE enhances light-collection efficiency in this setup. Two ETEL high-quantum-efficiency PMTs are mounted on opposite sides of the cell to measure the intensity of scintillation light emitted from the sample. The scintillator is irradiated via a  $^{137}\text{Cs}$  radioactive source that provides 662 keV gamma-rays. Since the deposited energy in the scintillator is determined by Compton scattering, an additional  $\text{LaBr}_3(\text{Ce})$  detector is employed to tag backscattered gamma rays and thereby fix the deposited energy. Coincidence between the PMTs and the  $\text{LaBr}_3(\text{Ce})$  detector selects events that correspond to an energy deposition of 478 keV in the scintillator sample. This method significantly suppresses background and ensures that all samples are compared under identical energy deposition. The charge of each PMT pulse is obtained by numerical integration over a 250 ns window. The resulting events are plotted in a histogram which gives the gamma-spectrum recorded through the tested scintillator. This spectrum includes the Compton spectrum and the backscatter peak. The Compton spectrum originates from false coincidence events and is therefore regarded as background. An exemplary gamma spectrum is shown in figure 4.6. The light yield of the scintillator sample is determined by fitting a Gaussian to the backscatter peak and extracting the mean value. Although the coincidence condition

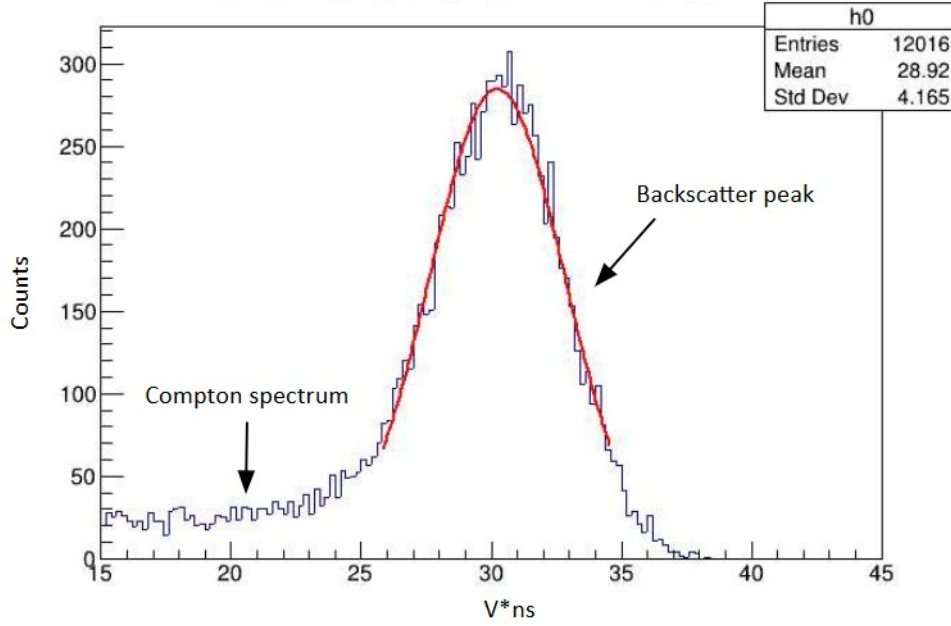


Figure 4.6: Example of a gamma spectrum recorded with the light yield setup with a Gaussian distribution fitted to the backscatter peak

selects events with a nominal deposited energy of 478 keV, statistical fluctuations in scintillation photon production, light collection, and detector response broaden this monoenergetic line into a Gaussian-shaped peak. In addition, the  $\text{LaBr}_3(\text{Ce})$  detector has finite energy resolution and the coincidence geometry accepts a range of scattering angles around  $180^\circ$ , so the deposited energy is not perfectly fixed but distributed over a narrow interval.

The Compton background is not explicitly fitted or subtracted. Although it contributes an underlying continuum that slightly elevates the baseline under the backscatter peak, its shape is determined by the source and coincidence geometry and is therefore essentially identical for all samples. As a result, the background does influence the absolute light yield, however, it does not bias the relative comparison of light yields which is of main interest in this thesis.

#### 4.4 Thermal Aging Tests

Liquid scintillators in general are relatively sensitive when it comes to long term stability if adequate measures for scintillator conservation are not met. Exposure to oxygen, for example, causes oxidation of fluor molecules. This not only alters the absorption and emission spectra of the scintillator due to a lower concentration of fluor but also leaves behind remnants of oxidized molecules which lead to additional light absorption [37].

During preparation, storage and in the final detector, the scintillator is in contact with

different materials for shorter or longer periods of time. To ensure chemical stability of the scintillator, these materials need to be tested before usage. A thermal aging test is a simple method to check for chemical compatibility between different materials, which are in contact with each other. By storing samples of the material inside of vials filled with scintillator, potential interactions between the materials are accelerated in a controlled environment and then identified via optical measurements.

These vials are stored in an incubator of the type IN30 by the manufacturer Memmert. This incubator can store samples at a constant temperature over long periods of time with maximum temperature fluctuations of 0.1 °C. By increasing the temperature, chemical processes are accelerated which allows for studies of long-term effects on a manageable time scale.

In this thesis, aging tests are applied in the context of the Surround Background Tagger for the SHiP experiment. To increase the reflectivity of the inner detector walls, they are spray-coated with a reflective coating. With aging tests, it is possible to quantify aging processes that can lower the scintillator quality or damage the reflective coating over long periods of time. As the reflective coating is designed to withstand temperatures up to 60 °C, this is the chosen incubation temperature.

The acceleration of chemical processes can be described by the *Van't-Hoff Law* which states that an increase in temperature by 10 K leads to an increased reaction rate by a factor of 2-4. This is also described by formula 4.6 [2].

$$Q_{10} = \frac{k_2}{k_1} e^{\frac{10 K}{T_2 - T_1}} \quad (4.6)$$

$Q_{10}$  is the factor by which the reaction rate is increased when the temperature is increased by 10 K. The reaction rate constants  $k_1$  and  $k_2$  correspond to the temperatures  $T_1$  and  $T_2$ .

The *Van't-Hoff Law* is an approximation and becomes less accurate for higher temperature differences. For this reason this thesis will always refer to the lower limit of this law to provide a minimum time of aging which equates to the worst case. For example, by storing these samples for 100 days at 60 °C, the rate of chemical processes is increased by 16-fold which induces aging of more than four years.

Before the incubation begins, scintillator samples are degassed in an ultra sonic cleaner, aiming to purge the sample of oxygen. If solved oxygen remains in the scintillator, energy deposition (i.e heat) can produce highly reactive singlet oxygen which in turn leads to the oxidation of fluors. The oxidized fluors form radicals which in turn oxidize other fluor molecules. This effect has a high impact on optical properties of the scintillator as the products of oxidation lead to an increase in absorption. Oxidized fluors also no longer participate in wavelength-shifting, additionally lowering the efficiency of the scintillator [37]. Sample preparation can also involve intentionally contaminating the sample with oxygen or the addition of an antioxidant.

After the desired time has passed, absorption measurements (see chapter 4.1) are conducted on the scintillators and compared to a reference. If there is a significant deviation in the results, the material in question can be identified as harmful towards the scintillator and should be substituted for an alternative. In the case of extreme

oxidation, the color of the scintillator typically turns yellow to deep orange (see figure 4.7).

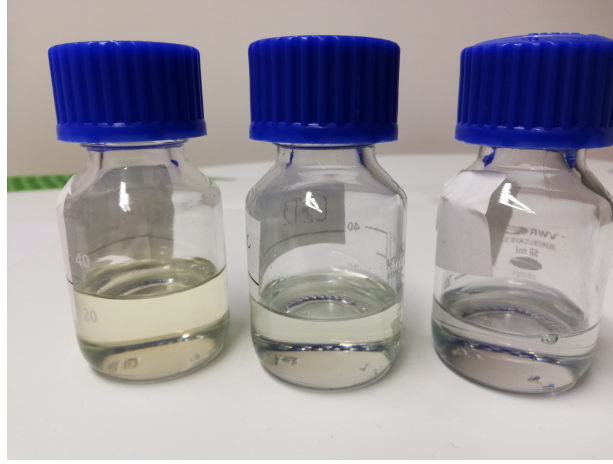


Figure 4.7: Liquid scintillators showing different degrees of oxidation [58]

## 4.5 Reflectivity Measurements

To increase the light yield of liquid scintillator detectors, one approach involves researching different diffuse reflective coatings that are applied to the inner walls of the detector. By maximizing reflectivity in the wavelength region of the scintillator's fluorescence, photons can traverse the detector multiple times, increasing the probability for detection by a photo sensor.

To measure and compare the reflectivity of different surfaces, a self-constructed experimental setup was implemented that measures the reflected light intensity with individually adjustable incident and reflecting angle. This allows for the separation of diffuse and specular reflectivity.

This chapter details the theoretical background of light reflection, the experimental setup, the measurement principle and analysis of the data.

### 4.5.1 Fundamentals of Reflectivity

The *Huygens-Fresnel principle* states that every point on a wave front is itself a source of wavelets. These wavelets retain the same speed as the original wave and spread out in a forward direction. The new wave front then forms on a tangent to all wavelets [59]. An illustration of this principle is shown in figure 4.8.

The reflectivity of a surface is a unit that gives the proportion of reflected light intensity  $\Phi_R$  compared to the incident light intensity that is radiated onto the sample  $\Phi_I$ . It is defined as:



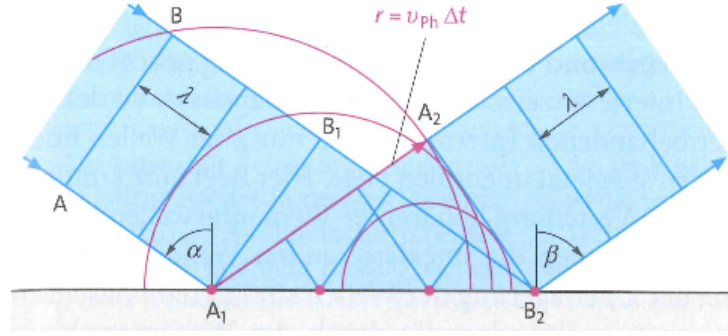


Figure 4.8: Illustration of the Huygens-Fresnel principle [59]

$$R = \frac{\Phi_R}{\Phi_I} \quad (4.7)$$

There are two general different types of reflectivity, which depend on the surface texture of the reflecting body:

**Specular reflectivity** can be observed when the reflecting surface is smooth and polished. This includes mirrors or polished stainless steel. Reflectivity at all angles is nearly zero, except for one appropriate angle: the reflected light leaves the specular reflector at the same angle to the surface normal as the incident light on the opposite side of the surface normal.

**Diffuse reflectivity** originates from matte and rough surfaces, such as white paint. Ideally, light is reflected uniformly at all angles, which is also known as "Lambertian reflectance".

In reality, most reflective surfaces do not fully correspond to one or the other category due to imperfections. A mirror-like surface will usually not reflect light in a single specular peak but rather in a spread out *specular lobe* that follows a Gaussian distribution. The diameter of this lobe becomes larger and the proportion of diffuse reflection increases with the roughness of the surface. Subsequently, all practical diffuse reflectors will not reflect completely uniformly but will also feature a specular proportion. All of these cases are shown in figure 4.9 [60].

With this knowledge, an experimental setup is designed to measure the relative reflectivity of different surfaces, such as stainless steel, corten steel and different combinations of reflective coatings. The measurement principle that is applied in this context is detailed in the following section.

#### 4.5.2 The Reflectivity Setup

The requirements for a reflectivity setup in the context of the SBT are to measure the wavelength dependent reflectivity of different samples with variable incident and

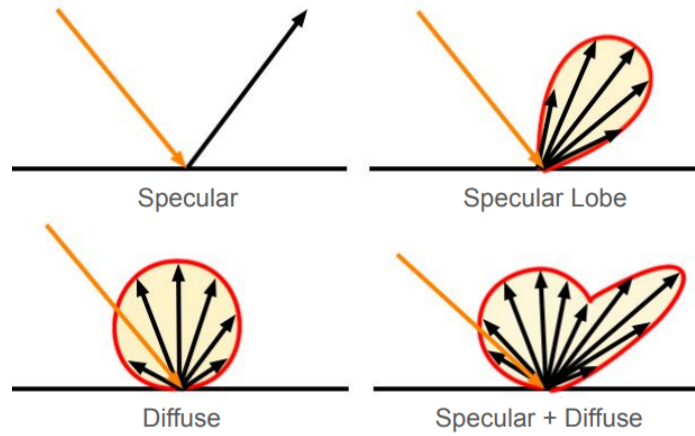


Figure 4.9: Illustration of different types of reflectivity [61]

reflecting angle. This allows for the separation of the diffuse and specular proportions of the reflected light.

In figures 4.10 and 4.11 models of the experimental setup are presented from a side view and from a top view. The individual components are marked and labeled. Both figures are designed with the software *Solidworks* with the support of Rainer Othegraphen.

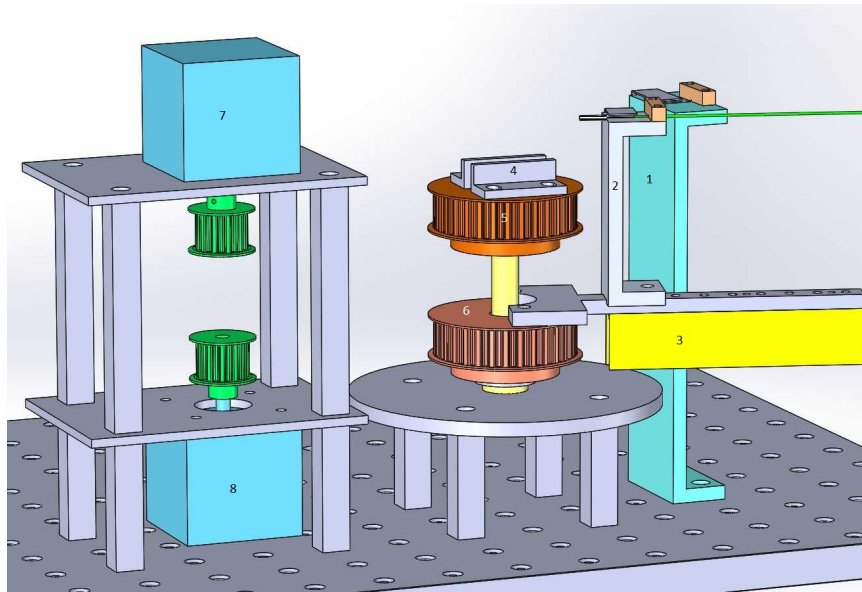


Figure 4.10: SolidWorks model of the reflectivity setup (side view). 1: fiber holder (light source); 2: fiber holder (spectrometer); 3: turning table extension (spectrometer); 4: sample holder; 5: turning table (sample); 6: turning table (spectrometer); 7/8: stepper motors [58]

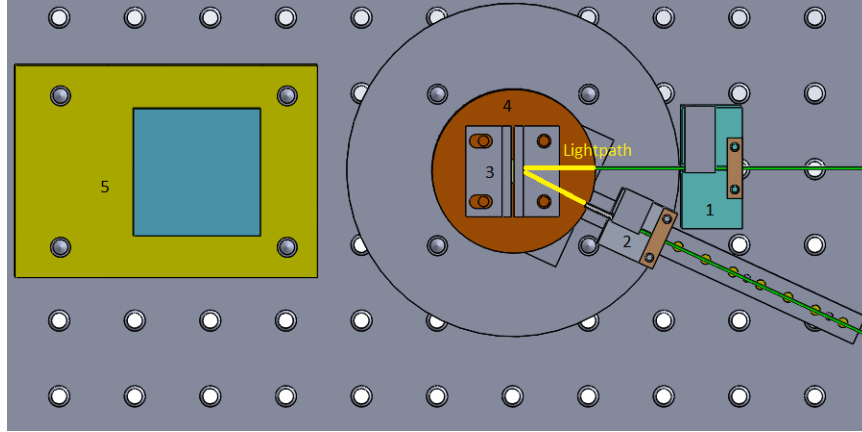


Figure 4.11: SolidWorks model of the reflectivity setup (top view). 1: fiber holder (light source); 2: fiber holder (spectrometer); 3: sample holder; 4: turning table (sample); 5: stepper motor holder [58]

A deuterium light source of the type 'SL3' is coupled into the setup via optical fibers and directed towards the reflective surface. The lamp spectrum is shown in figure 4.12. The reflected light is collected by a second optical fiber which guides the signal to a 'Black-Comet' UV-Vis Concave Grating Spectrometer. The manufacturer of both of these optical instruments is *StellarNet Inc.* The spectrometer measures the light intensity for the selected wavelength range and sends the results to a computer via USB. The software *SpectraWiz* is used to visualize the results and save the data for further analysis.

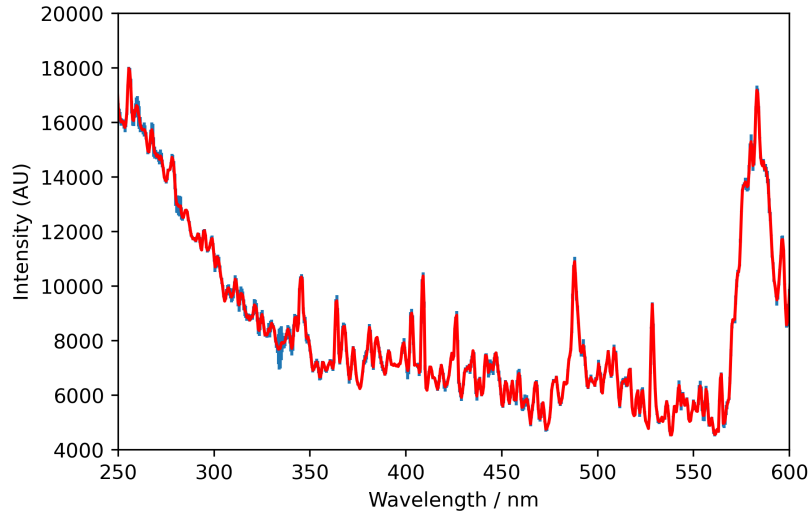


Figure 4.12: Spectrum of the deuterium light source SL3 used in the reflectivity setup

The main mechanical components of this reflectivity setup are two turning tables

positioned above each other sharing the same center axis. Both of these tables are turned individually using stepper motors that are connected with toothed belts. The top table uses a holder to fix the sample, which is typically a 15 mm  $\times$  30 mm steel sample with a reflective coating. By rotating the sample holder, the incident angle of the reflection is set.

The optical fibers leading from the light source and from the spectrometer to the setup are fixed in position by screwing the ends into designated fiber holders. The holder for the spectrometer cable is connected to the lower turn table which allows for adjusting the reflecting angle, while the irradiating fiber is in a fixed position.

The stepper motors (Nema 17 by *TopDirect*) are controlled through a 'Physical-Computing-Plattform' of the type *Arduino UNO R3* and a CNC-Shield V3 with A4988 stepper motor drivers. A script for the stepper motors is programmed on the Arduino IDE Software to turn the motors by the intended number of steps. The step size is adjusted through the CNC-Shield. The entire setup is mounted onto an optical breadboard from *Thorlabs* that is shielded against stray light by an optical enclosure. The measurement principle and analysis method concerning the reflectivity setup is presented in chapter 4.5.3.

### 4.5.3 Measurement Principle and Analysis

The previously introduced reflectivity setup is used throughout this thesis to assess mainly diffuse reflectivity of the Optopolymer Bariumsulfate (BaSO<sub>4</sub>) Coating OPRC by the manufacturer *Berghof Fluoroplastics*. Comparisons are made between applications on different steel surfaces and in combination with different primers. The impact of the long term exposure of the coating to the liquid scintillator on the surface reflectivity is also observed with this setup.

This setup is not capable of making direct absolute reflectivity measurements as this requires the collection of all reflected light. By using a highly efficient reflective standard with a well-known reflectivity spectrum and comparing the measurement with that of the sample, a quantitative approximation can be made while still being able to measure specular and diffuse components separately.

This reference sample is chosen to fit the individual measurement: a well-documented PTFE sample is used as a reference, so the measurement is independent of the lamp spectrum. The reflectivity spectrum of the PTFE standard is shown in figure 4.13.

After OPRC is identified as a promising candidate for the application in the SBT and the reflectivity is not distinguishable from PTFE in this setup, the focus shifts to finding changes in reflectivity due to aging or the addition of a primer. For these measurements an unaged OPRC sample is used as a standard.

Before every measurement the setup is put into a marked default setting, where the incident and reflecting angle are known. Both stepper motors are then operated for the required number of steps to reach the desired angles for the sample and the spectrometer fiber. A low resolution of the angles is sufficient, as the light beam is not focused onto the sample and features a significant expansion, when the spectrometer fiber is reached. This leads to a large area of the specular lobe with almost constant

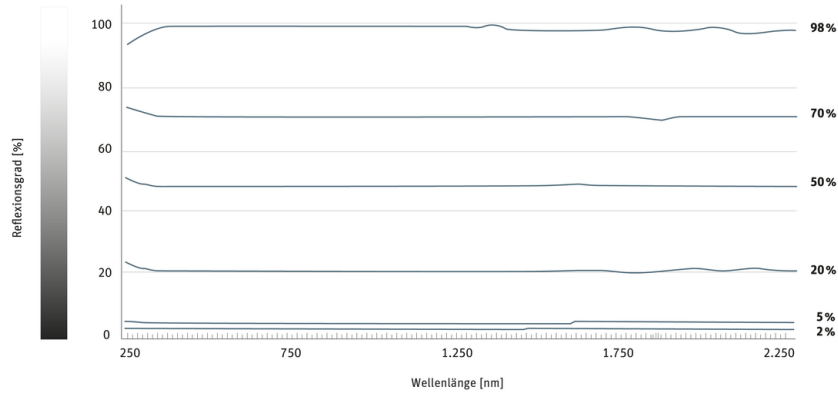


Figure 4.13: Reflectivity spectrum of the PTFE standard: The graph shows different grayscale of the diffuse reflector. The sample with the highest reflectivity is chosen as a reference for the reflectivity setup [62].

light intensity. As long as the measurement is within this region, specular reflectivity is measured. For the diffuse part of the reflection, the spectrometer fiber is positioned well outside of the specular lobe where the light intensity has dropped significantly, typically at an angular configuration of  $0^\circ - 45^\circ$  (incident-reflecting angle).

The integration time of the spectrometer for the entire measurement series is determined by placing the reference in the sample holder and identifying an integration time where the signal is just below the saturation threshold of the spectrometer. Exceeding this threshold would prevent additional counts in the individual channels and distort the measurement. As the reference typically features the highest reflectivity of all samples, this ensures that the spectrometer does not reach saturation during the entire measurement series.

The uncertainty is estimated by repeating every measurement at least twice. In between these reiterations the sample is removed from and reinserted into the setup to account for inconsistent alignment. The stepper motors are also reset to their default positions and then brought back to the measurement positions, which accounts for inaccurate angle settings. With these multiple measurements, the standard deviation is computed and the reflectivity error is determined via Gaussian error propagation.

The results are exported from the SpectraWiz software and are then analyzed independently and plotted with a python script.

## 5 The SHiP Experiment

*“It’s darker now, darker than I realized  
As I crossover behind the bend,  
I step past the horizon of who I’ve been,  
but not all is unfamiliar”*

— Boundaries, *From the Departed, Dear or Otherwise*

The *Search for Hidden Particles* (SHiP) experiment is an effort to explore, so far, hidden sectors of particle physics. Discoveries in recent decades that cannot be explained with the Standard Model have given hints for physics beyond the Standard Model. By colliding a high-intensity proton beam from the CERN Super Proton Synchrotron (SPS) with a fixed target optimized for the creation of heavy D- and B-mesons in a dedicated beam dump facility (BDF), a variety of secondary particles are created in the impact. A low background decay volume is provided, where hidden sector particles can decay. The resulting decay products and their scattering signatures can be identified in a spectrometer to gain information about the incident particle.

SHiP will be sensitive to feebly interacting particles with energies in the GeV-scale, allowing for potential verification of models with heavy neutral leptons (HNL), light dark matter, dark photons, dark scalars and axion-like particles. Additionally, SHiP intends to contribute to neutrino physics by performing measurements of tau neutrinos and neutrino-induced charm production with an independent detector [63] [64] [65].

As of March 2024 the SHiP experiment is pre-approved by the CERN Research Board and construction is expected to begin in 2027 in the Experimental Cavern North 3 (ECN3). First measurements are planned to begin in 2031 [5].

### 5.1 Motivation

Even though the Standard Model of particle physics has been tested to great precision and delivers an explanation for almost all observed particles and interactions, there are still open questions that cannot be answered with the fundamental theory as we know it.

Current approximations divide all energy in the universe into baryonic matter (5%), dark matter (27%) and dark energy (68%). The latter two are not included in the Standard Model. While dark energy was introduced to explain the observed accelerating expansion of the universe, the physical nature is still not agreed upon in the scientific community. While verification has not been achieved so far, observations in the universe such as the dynamics of galaxy clusters and the Gravitational Lense Effect cannot be explained solely by the visible matter and suggest the existence of an invisible type of matter. Dark matter could consist of undiscovered particles that are expected to be electrically neutral, stable and only able to interact with baryonic matter via gravitational force. This makes the detection of dark matter especially

challenging. Several extensions to the Standard Model that can be verified with SHiP conveniently provide candidates for dark matter [66] [67] [68].

The *Baryon Asymmetry of the Universe* is another problem that challenges the Standard Model. When the universe formed during the Big Bang, energy was abundant, which led to a high rate of particle-antiparticle pair creation. Following the Standard Model, one would expect an equal number of particles and antiparticles to annihilate, leaving behind a universe exclusively filled with radiation and absent of matter. As this is not the case and there is an absence of antimatter in the observable universe, the reason for this unexplained asymmetry in matter and antimatter needs explanation [69] [70]. The SHiP collaboration intends to construct an experiment to explore physics beyond the Standard Model, operating at the *intensity-frontier*. SHiP is a proposed general-purpose experiment to be implemented at the CERN SPS-Beam Dump Facilities and is set to search for weakly interacting long-lived particles and different types of light Dark Matter (LDM). The high intensity of the SPS and the 400 GeV proton beam allows a search in a broad energy range (MeV-GeV) covering unprecedentedly low coupling constants. The large region of parameter space, that SHiP will be sensitive to, cannot be reached by any other experiment up to this point. Measurements conducted with the SHiP experiment will be complementary to collider measurements at the energy-frontier and therefore support the international effort for the search for physics beyond the Standard Model [63]. Examples of proposed extensions to the Standard Model that can potentially be verified by the SHiP Experiment are listed below:

**The Neutrino Minimal Standard Model** is an extension to the Standard Model by the Seesaw mechanism, which postulates the existence of three additional sterile neutrinos. As these are right-handed particles, sterile neutrinos are only affected by the gravitational force and potentially undiscovered forces. Also known as Heavy Neutral Leptons (HNL), their associated masses are lower than the electroweak scale. The lightest of the three HNL is considered a Dark Matter candidate with a life time longer than that of the universe and can therefore not be studied by SHiP. The two heavier HNL fall into the sensitivity range of SHiP and could explain the Baryon asymmetry of the universe through a process that is called Leptogenesis [71] [72].

At SHiP, the most likely production mechanisms for HNLs are weak decays of flavored mesons produced in the target. HNLs are then expected to decay into Standard Model particles, primarily through the two-body decay channel  $\text{HNL} \rightarrow \mu\pi$ , within the SHiP decay vessel.

**Axion-like particles (ALPs)** are pseudoscalar particles that appear in many proposed extensions of the Standard Model. They are generally weakly-interacting and couple to the electromagnetic force. Low-mass ALPs are considered a candidate for dark matter [73] [74].

**Dark Photons** are hypothetical particles that are equivalent to photons as force exchange particles in electromagnetism but interacting with Dark Matter. Through Kinetic Mixing with ordinary photons, Dark Photons could weakly couple to

| Physics model                     | Final states                                                            |
|-----------------------------------|-------------------------------------------------------------------------|
| HNL                               | $\ell^\pm \ell^\mp \nu, \pi \ell, \rho \ell, \pi^0 \nu, q \bar{q} \ell$ |
| ALP (fermion coupling)            | $\ell^+ \ell^-, \pi \pi, q \bar{q}$                                     |
| ALP (gluon coupling)              | $\pi \pi \pi, 3\pi, \eta \pi \pi, \gamma \gamma$                        |
| ALP (photon coupling)             | $\gamma \gamma$                                                         |
| Dark photons                      | $\ell^+ \ell^-, 2\pi, 3\pi, 4\pi, K K, q \bar{q}, D \bar{D}$            |
| Neutrino-induced charm production | $D_s^\pm, D^\pm, D^0, \bar{D}^0, \Lambda_c^+, \bar{\Lambda}_c^-$        |

Table 5.1: Physics models and their associated final states that SHiP is sensitive to [78]. In this context  $\ell = e, \mu, \tau$ .

charged particles which allows for their detection [75] [76].

In addition to exploring the Hidden Sector, SHiP will also feature a rich neutrino program with a dedicated Scattering and Neutrino detector (SND). In this context the tau neutrino will be distinguishable from the anti-tau-neutrino by performing electric charge measurements of the  $\tau$  decay products, thus providing the first observation of the anti-tau-neutrino [77]. Through high statistics, the detector is expected to measure the tau neutrino cross section. The experiment is also suitable for measurements of neutrino-induced charm production [63].

Table 5.1 gives examples of final states for the corresponding physics models that SHiP is sensitive to.

The following chapters will give an overview of SHiP and describe the essential components that the experiment will be composed of. The "Surrounding Background Tagger", a dedicated liquid scintillator veto detector, is of main interest for this thesis and will be presented in detail.

## 5.2 Overview of the Beam Dump Facility / SHiP Experiment at ECN3 (100)

SHiP is a general-purpose, intensity-frontier experiment, which is in preparation to be operated in beam-dump mode at the CERN SPS accelerator. By taking full advantage of  $4 \times 10^{19}$  mostly unused protons per year with an energy of 400 GeV, SHiP will search for "hidden" feebly interacting particles (FIPs) at a GeV-scale while also conducting measurements on neutrinos.

The entire experiment can be divided into two parts: the beam dump facility (BDF) and the SHiP detector. An overview of the most relevant components of the experiment is shown implemented into the ECN3 area in figure 5.1.

In the BDF, the proton beam impinges upon a high-density target optimized for the production of heavy mesons. This is followed by two measures to reduce background from the proton beam in the SHiP detector: A magnetic hadron absorber, made of iron and 5 m in length, absorbs hadrons and electromagnetic radiation; and an active muon shield that deflects muons from the detector fiducial volume, which were produced in the beam-target interactions.



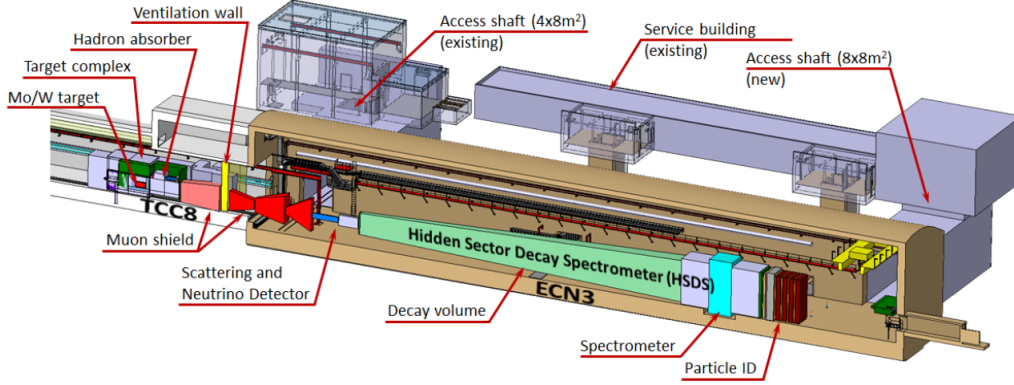


Figure 5.1: Overview of the SHiP Experiment with the associated BDF implemented into the SPS ECN3 area [79].

Located downstream of the BDF is the SHiP detector, which in turn, can be divided into the Scattering and Neutrino detector (SND) and the Hidden Sector Decay Spectrometer (HSDS). The SND will search for LDM scattering and perform measurements on the  $\nu_\tau$  and the anti- $\nu_\tau$ .

The HSDS will measure the final states of fully reconstructible FIP decays and partially reconstructible decays with neutrinos behind the 50 m-long decay vessel. The decay volume has a rectangular cross section of increasing area to accommodate the growing beam diameter. To eliminate background from neutrinos interacting with the decay volume, a vacuum of 1 mbar is sustained within. Alternatively, a newer, mechanically more practical approach aims to fill the decay volume with helium at atmospheric pressure.

SHiP is intended as a zero background experiment which necessitates the suppression of and discrimination against all background events. The Surrounding Background Tagger (SBT) is a system of liquid scintillator-based background taggers that envelops the decay volume to provide a veto against background from muon and neutrinos interacting in the vessel walls or surrounding structures. It also identifies particles entering the vessel from the outside, such as cosmic muons. As one of the main focuses of this thesis is detector optimization in the context of the SBT, it is presented in more detail in chapter 5.3.

A spectrometer is located behind the decay volume. It is combined with a straw tracker to determine decay vertex, mass and impact parameter of the reconstructed FIP trajectory at the proton target. To discriminate between a wide range of models that include FIPs, particle identification is necessary and is provided by an electromagnetic calorimeter with integrated particle identification and a muon detector [65] [63] [80].

### 5.3 The Surrounding Background Tagger (SBT)

In the SHiP experiment, it is essential to control, suppress and tag all different sources of background. While the 50 m long decay vessel is evacuated (or filled with Helium, following a novel approach) to eliminate background from neutrinos interacting within the decay volume, particles that enter the volume from outside of the experiment, such as cosmics cannot be shielded by such measures. Additionally, muons from the proton target, that are back scattered in the walls surrounding the experiment, can enter the decay vessel. The vessel walls are also susceptible for muon or neutrino interactions. To prevent these sources of background from interfering with measurements in the SHiP experiment, the outer walls of the decay vessel need to be instrumented with a system of highly efficient background tagging detector cells.

To fulfill the requirements of the SHiP experiment, a veto efficiency of more than 99% for minimum ionizing particles (MIPs) is required. A time resolution in the nanosecond range and good spatial resolution need to be provided to locate the source of background reactions in time and space. The veto detector needs to offer full coverage around the entire decay vessel. This is achieved by surrounding the vessel with liquid scintillator, thus providing the name Surround Background Tagger (SBT).

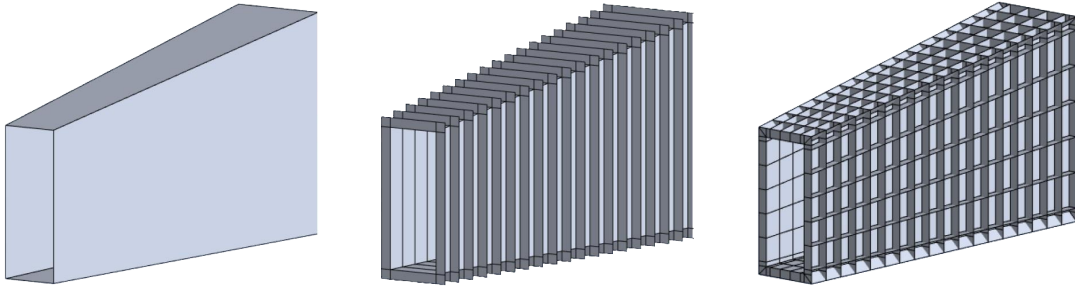


Figure 5.2: Illustration of the support structure of the SHiP decay vessel, providing a division into cells that is used for the SBT.

In figure 5.2 the SHiP decay vessel is shown schematically. Due to a corten steel support structure, using horizontal and vertical stiffening members, the walls of the decay vessel are naturally divided into a cell-like structure. These cells are welded shut with a sheet of corten steel and filled with liquid scintillator as an active detector medium. Liquid scintillator was chosen specifically for this purpose as it can perfectly fill out all gaps of the support structure, providing optimal coverage of the decay vessel. Each cell is equipped with two *Wavelength-shifting Optical Modules* (WOMs) (see chapter 5.3.2), that are inserted from the outside into the cell, separated from the liquid scintillator with a UV-transparent PMMA vessel. The WOMs are inserted at equal distance from the two vertical stiffening members of the cell which is detailed in figure 5.3. The cylindrical geometry of the WOMs ensures efficient collection of scintillation light, originating from any point in the cell. This light collection is enhanced further by increasing the reflectivity of the inner walls of the detector cells with a  $\text{BaSO}_4$  coating. The optimization of this reflective coating is of main focus in this thesis.

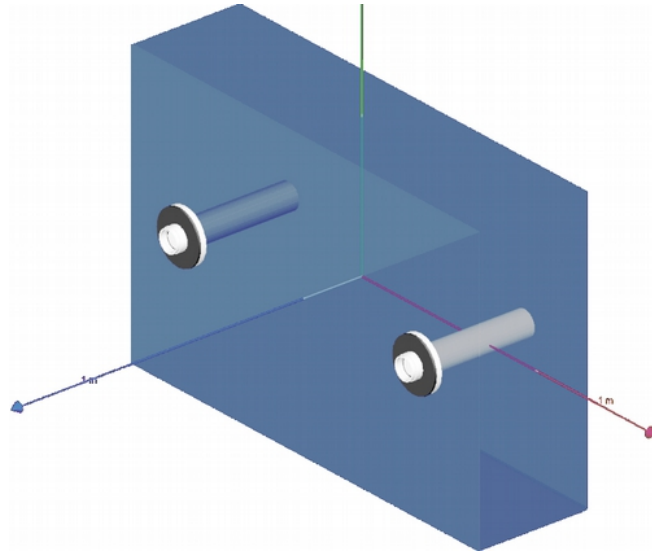


Figure 5.3: Illustration of a single detector cell of the SBT showing the position of the two WOMs.

While the surface area of each cell increases due to the frustum shape of the decay vessel, the thickness of all cells is 20 cm. This amounts to an overall number of around  $\sim 850$  cells in the SBT containing a total of  $150 \text{ m}^3$  of LS [81].

To reach the proposed detection efficiency of 99 % for MIPs, all components of the SBT are optimized to provide the highest light output possible while also ensuring chemical compatibility. For this reason, two large-scale detector prototypes were constructed and employed in test beam measurements during this thesis to characterize and understand the performance. These tests aided in the study of detector performance and also in improving equipment such as a large scale liquid scintillator handling system. Following extensive laboratory testing, the application of a diffuse reflective coating (see chapter 5.3.3) to the inner detector walls was conducted for both test beam measurements. This allowed for optimization of the application method, performance of the coating and investigating long-term stability. A 1-cell-prototype was employed in October 2022 at the DESY II test beam area 24. In 2023/2024, a 4-cell-prototype was tested at the CERN test Beam area east, T9. The different components of the SBT are detailed in the following sections.

### 5.3.1 Liquid Scintillator

The liquid scintillator (LS) will be ionized by the initial particle energy and will produce a fluorescent signal with an intensity proportional to the amount of deposited energy. In recent decades, LS has been used in large scale detectors searching for neutrinos, which has led to a broad availability of information through research in these projects. Especially the combination of the solvent linear alkylbenzene (LAB) with the fluor 2,5-diphenyloxazole (PPO) is a popular choice due to well known benefits such as high transparency, fast fluorescence time, low price and low toxicity which are detailed in

chapter 3.2.

With a concentration of 2 g/l of PPO, a primary light yield of  $\sim 10^4$  photons per MeV can be achieved with an emission maximum at  $\sim 380 - 390$  nm. In this range, LAB typically has a light attenuation length of several meters, which can be further increased through purification in aluminum oxide columns. In this process, organic impurities are removed that would otherwise absorb light in the near-UV-region. A picture of the laboratory scale setup that was used in the purification of 300 l of LAB for the 2022 test beam prototype is displayed in figure 5.4. Absorbance spectra demonstrate the optical performance when comparing raw LAB to purified LAB.

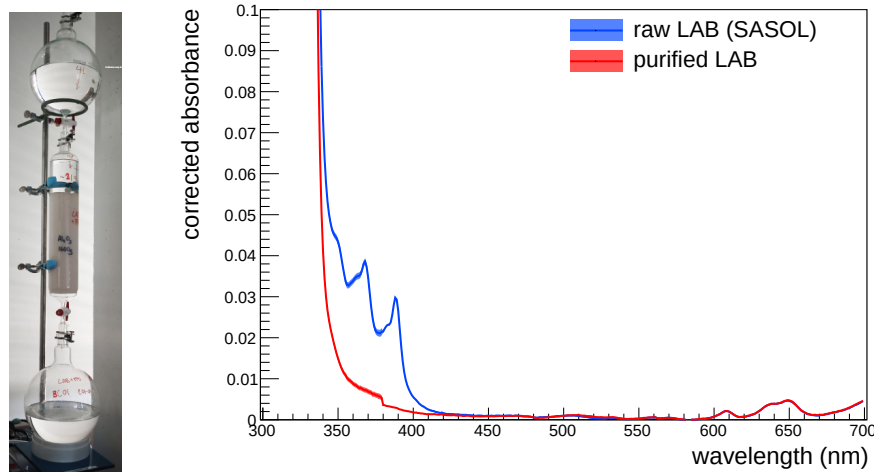


Figure 5.4: **Left:** Laboratory setup for the purification of LAB. The column is filled with 1.6 kg of Aluminum oxide to purify 25 l of LAB, which was manually divided into batches and refilled by hand. A small vacuum pump is employed to increase the purification speed. **Right:** UV-vis absorbance spectra of raw and purified LAB measured in a 100 mm Suprasil glass cuvette. An improved absorbance is achieved, especially, in the region of scintillation light (360 nm-400 nm) by removing organic impurities.

In 2023, purification of 1000 l of LAB was conducted in a large-scale setup at the *Max Planck Institut für Kernphysik* in Heidelberg for the 4-cell-prototype test beam. Due to direct transfer from one IBC to another and an increase in column size, this method is more practical compared to the small laboratory setup while achieving comparable or better results. A picture of the large scale purification setup is shown in figure 5.5. Apart from LAB and PPO, Tocopherol is also added to the scintillator for its antioxidant properties. As aging processes ultimately lead to the oxidation of fluor molecules, which reduces the light yield of the scintillator, Tocopherol can delay the loss in scintillator quality. The antioxidant is added at a concentration of 0.001 wt.%. This value has shown minimal immediate impact on the transparency of the scintillator while significantly reducing oxidation over time [58]. Due to SHiP's minimum operation time of 5 years, it is necessary to ensure the long-term stability of the scintillator. In preparation for both test beams, the additives were mixed with purified LAB in a high concentration master solution. A *liquid handling system*, consisting of a pump and



Figure 5.5: LAB purification setup at the *Max Planck Institut für Kernphysik* in Heidelberg for the purification of 1000l of LAB. Raw LAB is pumped from one IBC, through aluminum oxide columns into another IBC, where the purified LAB is collected. Two columns with 10 kg of Aluminum oxide each are sufficient to purify up to 350l of scintillator.

a circuit of valves and pipes, was employed at the test beam site to ensure safe transfer of the liquid scintillator from the transportation container to the detector prototypes. Additionally, the system allowed for circulation of the scintillator, facilitating the mixing of the master solution into the bulk scintillator. Nitrogen connections to the detector and to the storage containers maintained a nitrogen atmosphere within the entire liquid handling system, to prevent early oxidation and quenching from introduced oxygen. A sketch with all components of the liquid handling system is shown in figure 5.6.

In the SBT, a portion of the scintillation light is absorbed by a WOM which is also responsible for guiding the photons to a SiPM-array. The following section details the working principle of these devices.

### 5.3.2 Wavelength-shifting Optical Modules (WOMs)

Wavelength-shifting optical modules (WOMs) are transparent tubes that are dip-coated with a wavelength-shifting (WLS) paint. Originally designed for the upgrade of the IceCube detector [82], for the SBT specifically, the tubes are made of PMMA and the paint, that contains bis-MSB as a WLS, is applied on both the inside and the outside of the tube. This results in a large active surface with a maximum absorption probability of the scintillation light. In figure 5.7 a schematic overview of a WOM is shown to clarify the working principle. The emission spectra of the scintillator and the bis-MSB are also compared.

When a UV-photon, emitted by the liquid scintillator, hits the layer of WLS paint on

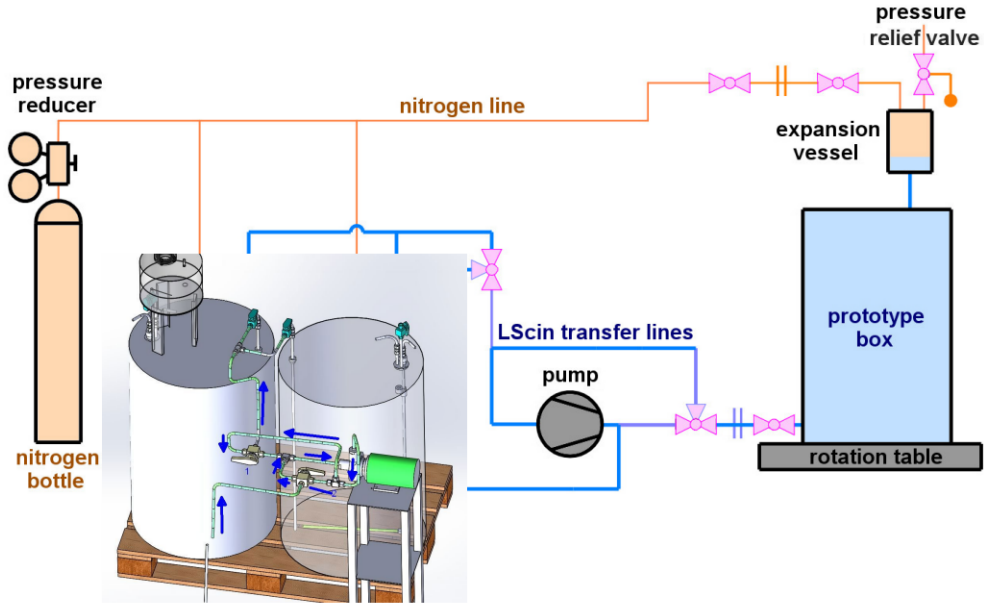


Figure 5.6: Sketch of the liquid handling system used for the SHiP test beam in 2022. A system of valves and pipes allows for filling and emptying of the prototype and circulation of the scintillator under a nitrogen atmosphere.

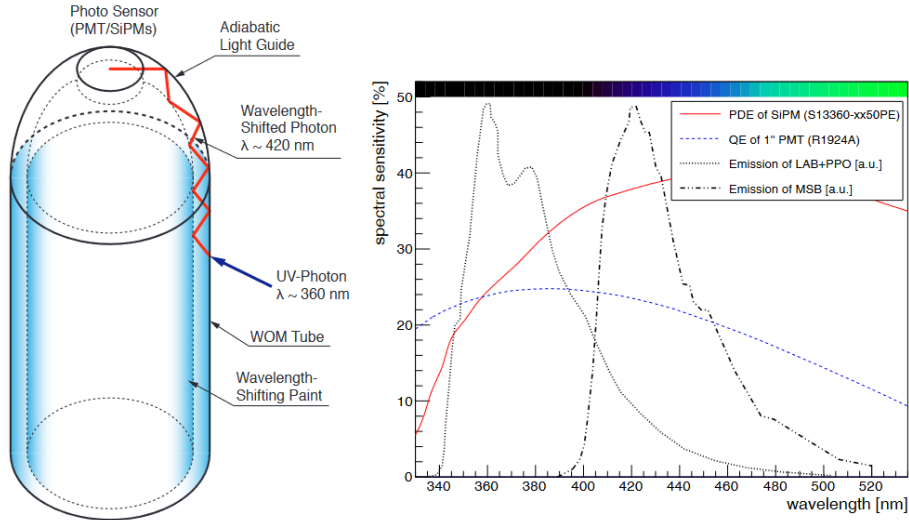


Figure 5.7: **Left:** The working principle of a WOM. **Right:** Emission spectra of the liquid scintillator and the wavelength-shifting paint used on the WOMs containing bis-MSB. Also shown is the quantum efficiency of a photo multiplier and the photo detection efficiency of the SiPM. The overlap of the bis-MSB emission peak with a high photo detection efficiency of the SiPMs indicates good compatibility of the components [83].

the WOM, it is absorbed by a bis-MSB molecule. A secondary photon is re-emitted isotropically with a longer wavelength  $\sim 420$  nm. As the WOM is separated from the LAB by a UV-transparent PMMA-vessel, it is surrounded by air, which has a lower refractive index than the PMMA of the WOM. This results in the total reflection of a large fraction of secondary photons within the wall of the WOM. The tube acts as an active light guide, leading up to 74.6 % of secondary photons (neglecting absorption and scattering) to an optically coupled photo sensor.

The SBT will employ two WOMs per detector cell with a tube length of 200 mm, an outer diameter of 60 mm and a wall thickness of 3 mm. A ring-shaped array of 40 3 mm x 3 mm-SiPMs is connected directly to one end of every WOM. Bis-MSB is used in the WLS paint, as it acts as an efficient absorber in the emission range of the liquid scintillator. The emission range of bis-MSB also coincides with a maximum photo detection efficiency of the SiPMs resulting in a overall optimized detection probability.

### 5.3.3 Reflective Coating

An additional measure to maximize light collection in the SBT is to enhance the reflectivity of the inner detector walls, allowing scintillation photons to traverse the active detector medium multiple times before reaching a WOM. By applying a diffusely reflective coating on the inner cell walls of the SBT, the probability for the absorption of scintillation light in the WOMs and the consequent detection becomes substantially higher.

The cell walls of the SBT will be comprised of corten steel, which, in an unoxidized state, has a comparable surface to unpolished stainless steel. This gives the reflectivity of the inner detector walls a high specular proportion with a low reflectivity for wavelengths below 400 nm. Corten steel surfaces typically oxidize at a high rate when in contact with the environment, which reduces the reflectivity significantly. Additionally, the rust does not develop evenly, which leads to unpredictable surface conditions and location-dependent reflectivity. By applying a highly diffuse UV-reflective layer to the inner walls of the detector the overall reflectivity can be uniformly increased.

Three main factors are considered for the application of a reflective layer:

**Application:** As the final SBT will contain a number of individual detector cells in the order of 850, a practical, reproducible and efficient application of the reflective layer is significant to minimize work load. The application of the reflective layer before the assembly of the cells will almost certainly lead to damages due to the welding of the steel. As this would also introduce particles from the reflective layer into the scintillator, this option was dismissed. The only remaining method of application, although less practical, is to use the two insertion holes for the WOMs to access the inner walls of the cells after the assembly and welding.

Without creating further or larger openings, this rules out any type of reflective foil. Even though diffusely reflective foils are commercially available and based on PTFE, which is known to be chemically compatible with the scintillator, no practical method of application within the welded cell could be identified.

A reflective coating that could be applied with a spray gun using a special



nozzle that allows for the distribution of coating at an angle of  $90^\circ$  fulfills these requirements. By varying the insertion angle of the spray gun, all areas on the inside of the closed detector cells are accessible for coating. This process can be verified by using an endoscopic camera to check for coverage.

**UV-Vis Reflectivity:** Photons that are reflected on the inner detector walls will typically have undergone a Stokes-shift through absorption and emission by PPO. The wavelength of the emitted light is therefore generally in the range of 360-400 nm with an emission peak at 380 nm. Therefore, the reflective coating must show high reflectivity for wavelengths below 400 nm.

**Compatibility:** The reflective coating needs to be chemically compatible with the scintillator and corten steel, as they will be in direct contact for the duration of the experiment, i.e a baseline of 15 years. This includes that there is no significant loss in scintillator quality through chemical reactions with the coating. The impact of the scintillator on the reflective coating also needs to be taken into account. Possible difficulties in this context include a reduction of reflectivity over time or reduced physical stability which could lead to crumbling or dissolving of the reflective coating into the scintillator.

The compatibility of the scintillator with different materials with and without anti-oxidants is tested in laboratory aging tests. By storing these materials in scintillator in a heated environment, i.e an incubator, the rate of chemical reactions can be increased, allowing the observation of long term aging within a few weeks. By monitoring the reflectivity of the reflective samples and the absorbance of the scintillator, negative impacts can be identified. These aging tests are analyzed and discussed in chapter 6.3.

In previous research, reflective paints containing Titanium-oxide ( $\text{TiO}_2$ ) and Barium-sulfate ( $\text{BaSO}_4$ ) pigments were investigated. While the diffuse reflectivity of widely-used  $\text{TiO}_2$ -paint was just below 100 % in the visible spectrum, for wavelengths below 400 nm, the reflectivity was measured at around 10 %, which is far from sufficient for the application in the SBT [58].

The  $\text{BaSO}_4$ -based paint *Optopolymer Reflective Coating (OPRC)* by Berghof Fluoroplastics is documented to reach a diffuse reflectivity of 95 – 98 % at 300 – 1300 nm. The reflectivity spectrum, provided by the manufacturer, is shown in figure 5.8.

Due to the high reflectivity in the emission region of the scintillator, OPRC is of main interest for research in this thesis. The coating can resist temperatures up to  $60^\circ\text{C}$  and is non-toxic. OPRC can be applied to many different surfaces and complex geometries using any compatible spray gun system. Berghof Fluoroplastics recommends the use of a SATAjet 1000 B RP spray gun with a 1.6 mm nozzle. By utilizing an extended  $360^\circ$ -radial-nozzle and a  $90^\circ$ -angled-nozzle, the application of the coating in the welded detector cells is possible.

When compared to stainless steel (shown in figure 5.9), the reflectivity of the inner detector walls is increased significantly (factor  $\sim 3$ ) by coating them with OPRC.

The chemical resistance and mechanical stability of OPRC within liquid scintillator



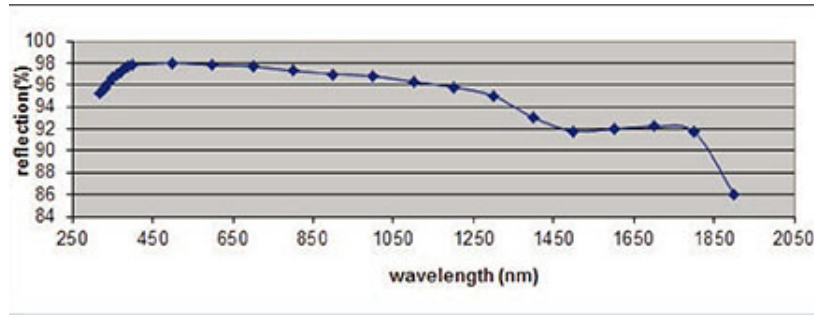


Figure 5.8: Reflectivity spectrum of the reflective  $\text{BaSO}_4$  coating OPRC provided by the manufacturer [84]

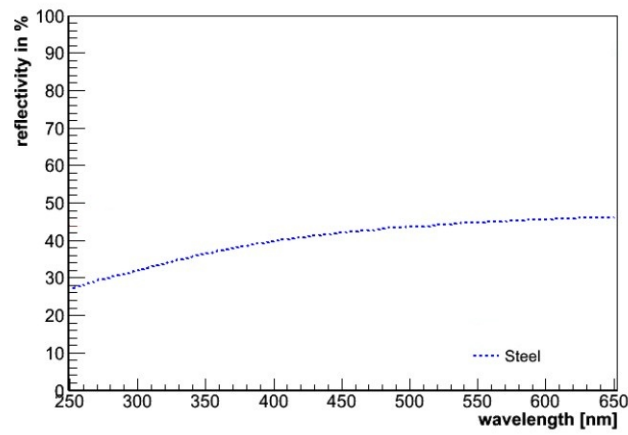


Figure 5.9: Diffuse reflectivity spectrum of stainless steel [85]

detectors is investigated in dedicated laboratory aging tests and in full-size prototypes documented in chapter 6.

## 6 Reflective Coating Studies for SHiP's SBT

*“I spend most of my time arguing with my own reflection.  
For no apparent reason.”*

— Counterparts, *Reflection*

The SHiP experiment aims to explore so far unknown particle physics by colliding a 400 GeV proton beam, provided by the CERN SPS accelerator, with a fixed target. This interaction provides the energy for the creation of a variety of proposed, hidden sector particles, that are expected to decay into known and detectable products in a vacuum decay vessel. Reconstruction is achieved by measuring the final states of these decays with a spectrometer which in turn provides information on the incident particle.

While the proton-target interactions are expected to provide a high intensity of hidden sector particles, the same reactions will lead to a high flux of minimal ionizing particles (MIPs), with a large proportion of muons. The direct impact of this background source is prevented by magnetically deflecting the charged particles from the beam path with an active muon shield. Most of these muons will leave the vicinity of the experiment, while others are back-scattered towards the decay vessel through interactions in the cavern walls of ECN3. This is the main source of background in the SHiP experiment. The SBT is a liquid scintillator detector, designed to envelope the decay vessel of the SHiP experiment. By identifying and tagging background events induced by MIPs in the vicinity of the decay vessel, they are isolated from the desired signals of hidden particle decays. To reach the proposed physics goals, that are detailed in chapter 5.1, a detection efficiency of 99% for MIPs needs to be achieved by the SBT. This necessitates the employment of a highly optimized detector system for the SBT.

Approaches to increase the light yield, and therefore the detection efficiency of the SBT, include the enhancement of the liquid scintillator (see chapter 5.3.1) and WOMs (see chapter 5.3.2). Another method focuses on the inner walls of the detector (see chapter 5.3.3): The SBT will be constructed of corten steel which shows poor reflectivity in the UV-region. By increasing the reflectivity of the surfaces within the SBT, scintillation photons can traverse the detector several times before reaching a WOM, increasing the number of registered photons per event.

Early research has shown that the BaSO<sub>4</sub> Optopolymer reflective coating (OPRC) from Berghof Fluoroplastics can fulfill the requirements of the SBT concerning the reflective capabilities (see figure 5.8). To verify the use in detector prototypes, the compatibility of the reflective coating with the corten steel and the liquid scintillator need to be fully investigated over a period of time in the order of several years. This chapter documents all results concerning the application of OPRC in the context of a liquid scintillator detector.

Chapter 6.1 studies the application of OPRC to corten steel. Due to oxidation on the steel surface during the drying of the coating, rust particles are solved, which leads to yellow stains that lower the reflectivity.

The addition of different primers is investigated in chapter 6.2 to reduce the staining of the coating.

The long term effects on the reflectivity and the physical and chemical stability of the coating, when in constant contact with liquid scintillator, is investigated in chapter 6.3 with laboratory-scale aging tests. By storing coated steel samples in liquid scintillator at increased temperatures, aging effects of many months are simulated within a few weeks. By measuring the absorbance of the scintillator and the reflectivity of the coating, the quality of either is monitored to identify harmful influences.

Chapter 6.4 documents the application of OPRC to a 1-cell and 4-cell detector prototype for test beam measurements at DESY and CERN. The condition of the coating before and after these measurements is compared to observe short term aging under conditions that apply to the use in the SBT. Scintillator quality is also monitored in the case of the 4-cell prototype.

Chapter 6.5 gives a summary of all results and discusses advantages and disadvantages of the use of OPRC in the SBT.

## 6.1 Coating Application Tests

In preparation for the application of the *Optopolymer Reflective Coating* (OPRC) to the inner walls of liquid scintillator detector prototypes, preliminary tests were conducted in a laboratory setting. This allows for the optimization of spray gun parameters, such as pressure and nozzle settings to achieve a uniformly covered surface. Additionally, the adherence of the coating to the steel is examined and the reflectivity is verified and compared to unpolished stainless steel.

Initial application tests were conducted on stainless steel with the SATAjet 1000 B RP spray gun, the standard 1.6 mm nozzle and a pressure of 1.5 bar. The steel was coated with two layers of the reflective paint, allowing the paint to dry for two hours after each layer. Figure 6.1 shows three samples that are examined in this chapter.

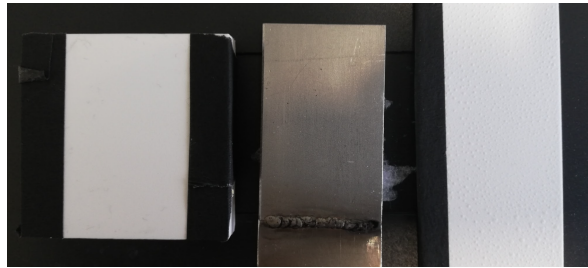


Figure 6.1: Investigated samples in this chapter. From left to right: PTFE Standard, stainless steel, stainless steel coated with OPRC.

In order to compare the reflectivity of OPRC and pure stainless steel, the samples are

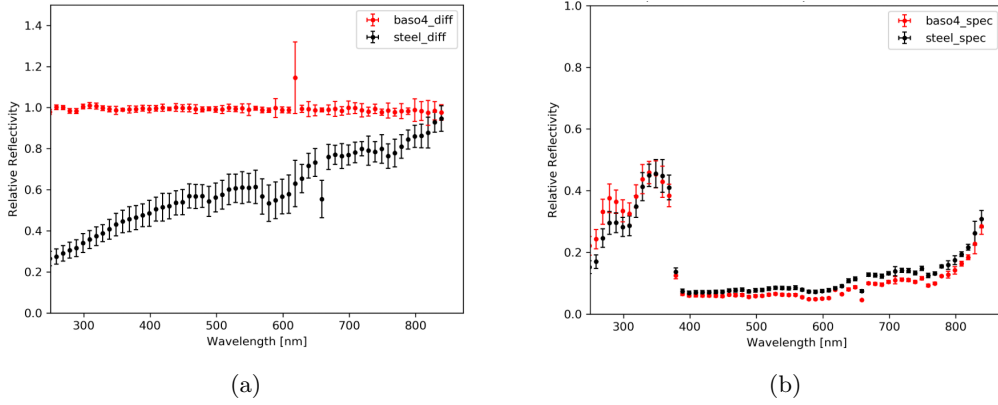


Figure 6.2: (a) Diffuse Reflectivity (diff) and (b) Specular reflectivity (spec) of unpolished stainless steel and the BaSO<sub>4</sub>-based reflective coating. The measurements are normalized to a PTFE sample.

measured in the reflectivity setup, which is detailed in chapter 4.5.2. Figure 6.2 shows the diffuse and specular reflectivity spectra of both samples. The diffuse reflectivity is plotted relative to the PTFE standard ( $R > 98\%$ ) while the specular reflectivity is compared to a DF2000MA-foil standard.

In this measurement, the OPRC sample is not distinguishable from the PTFE standard proving that the reflective coating acts as a nearly perfect diffuse reflector and verifying the reflectivity spectrum provided by the manufacturer in figure 5.8. A comparison of the diffuse reflectivity shows that applying OPRC to stainless steel results in an increase of 100 – 300 % for  $\lambda < 450$  nm, with the most pronounced improvement observed in the UV region. The measured diffuse reflectivity of stainless steel, especially for higher wavelengths, is increased compared to figure 5.9. Due to variations in surface texture of different steel samples, these deviations are expected. The measurements for specular reflectivity do not indicate a significant difference between the coating and stainless steel. This shows that the addition of the reflective coating on the inner walls of a detector can increase the light collection significantly.

Since corten steel was the dedicated building material for the detector prototypes and the SBT, the tests are repeated on the metal alloy to check for compatibility with the coating. Corten steel (type S355JO(J2/K2)W), also known as weathering steel, is typically used for outdoor structures as it presents increased resistance to atmospheric corrosion. In contrast to the unreactive stainless steel, the surface of corten steel forms rust at a high rate. Underneath this layer of rust, a barrier of adherent sulfates and phosphates is formed, that impedes further oxidation. This mechanism prevents the rust from penetrating the steel which would lead to a loss in structural integrity. While the oxidation of the complete surface can take several months in a dry environment, the process is accelerated to a few hours in the presence of moisture. A photo of a corten steel sample with a partially oxidized surface is shown in figure 6.3.

Within ten minutes of applying the coating to the corten steel, signs of yellowing

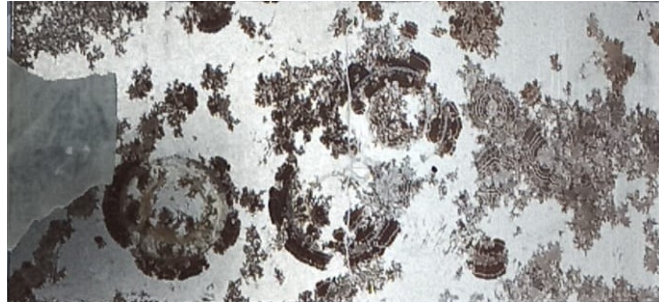


Figure 6.3: Sample of S355JO(J2/K2)W corten steel showing typical signs of surface oxidation

developed that turned darker during the remaining drying time. These stains remained fully visible in the final coating result which is shown in figure 6.4.



Figure 6.4: Dried result of first OPRC application on corten steel: The orange rust stains originate from oxidation of the corten steel surface during the drying of the reflective coating.

Due to the water content in OPRC, the wet paint on the corten steel surface provides an environment that encourages oxidation. These oxidized molecules are solved into the still drying coating which leads to a discoloration of the surface. Typically, a discoloration of any surface changes the reflective properties. A negative effect of the rust stains on the reflectivity, especially at low wavelengths, is shown in reflectivity measurements in figure 6.6.

To prevent rust from staining the coating, a protective layer between the corten steel and the reflective coating was investigated. As the use of a primer is already encouraged to increase the adhesion of the coating to the steel, a promising approach is the application of a primer that prevents oxidation.

In first primer application and compatibility tests, two different acrylic- and anti-rust primers are applied to corten steel in combination with OPRC. The effects of these primers on the resulting reflectivity of the samples are investigated, as well as long

| Sample Name | Primer Name                    | Manufacturer | Color      |
|-------------|--------------------------------|--------------|------------|
| A1          | "Acryl Multigrund"             | Hornbach     | white [86] |
| A2          | "Premium acrylic spray primer" | Maston       | gray [87]  |
| R1          | "Rostschutz Grundierung"       | Hornbach     | white [88] |
| R2          | "Anti Rust Primer"             | Maston       | gray [89]  |

Table 6.1: Information on different primers used in laboratory tests

term aging effects, while in contact with liquid scintillator. The results are presented in chapters 6.2 and 6.3.

## 6.2 Primer Application Tests

In preparation for the application of the reflective coating OPRC on the inner walls of detector prototypes, laboratory tests were carried out. Corten steel was cut into 15 mm x 30 mm samples which were coated with two different anti-rust primers (R1, R2) and two acrylic primers (A1, A2). Details on these primers are shown in table 6.1. The primers A1 and R1 were applied with the spray gun and the standard nozzle with 1.5 bar. A2 and R2 were applied using the spray cans they are packaged in. The primed samples were dried in the deduction for an hour.

Out of four samples for every primer, two were additionally coated with OPRC. The successfully primed and coated corten steel samples are displayed in figure 6.5. There are no signs of discoloration through oxidation on the surfaces of all samples.

Slight staining of the coating only took place around the edges of the sample, where the primer did not fully cover the steel. This allowed for direct contact between the steel and the coating allowing oxidation to take place.

To assess the effects of rust stains covering the entire reflective coating, an additional corten steel sample was coated with OPRC, without the use of a primer to intentionally induce oxidation. A photo of the sample is shown in figure 6.8.

Diffuse reflectivity measurements were conducted at an angular configuration of ( $0^\circ - 45^\circ$ ) on the samples to investigate whether the underlying primer has an effect on the coating reflectivity. A stainless steel sample was measured to represent the reflectivity of unoxidized corten steel. An improved reflectivity setup detailed in chapter 4.5.2 with an added collimator to the illuminating fiber was used for these measurements. The reflectivity relative to an OPRC standard is shown as a function of the wavelength in figure 6.6.

The measurements are compared at  $\lambda = 380\text{ nm}$  which is in the area of expected maximum emission of the scintillator. The results are displayed in table 6.2.



Figure 6.5: Primer and coating application tests on corten steel: Each row uses a different primer, indicated by R1, R2 (anti-rust primer) and A1, A2 (acrylic primer). In each row, the two left samples are only primed, while the two right samples are primed and coated with OPRC.

| Sample           | $R(380\text{ nm})$ |
|------------------|--------------------|
| A1 + OPRC        | $1.005 \pm 0.012$  |
| A2 + OPRC        | $0.990 \pm 0.018$  |
| R1 + OPRC        | $1.027 \pm 0.012$  |
| R2 + OPRC        | $1.033 \pm 0.026$  |
| No Primer + OPRC | $0.714 \pm 0.011$  |
| Stainless steel  | $0.271 \pm 0.011$  |

Table 6.2: Comparison of relative diffuse reflectivity measurements for primed and coated corten steel samples

The stainless steel sample achieved the lowest reflectivity of 0.271 in this test, once again highlighting the need for improvement.

The reflectivity of all combinations of primers with OPRC performed as well as the standard and were indistinguishable with the reflectivity setup. Therefore, the results indicate no significant advantages between the individual primers. The average reflectivity of all primed and coated samples results to 1.013.



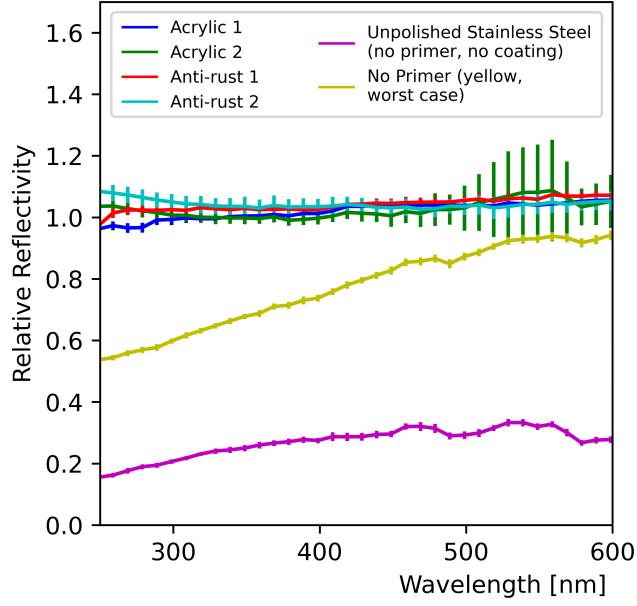


Figure 6.6: Relative diffuse reflectivity spectrum of four differently primed (acrylic/anti-rust) and coated corten steel samples, uncoated stainless steel and an unprimed coated corten steel with signs of oxidation. The wavelength-dependent reflectivity is measured relative to a  $\text{BaSO}_4$ -standard with an angular configuration of  $(0^\circ - 45^\circ)$ .

The coated sample with oxidation shows a reflectivity of 0.714 at 380 nm. The corresponding reduction in coating reflectivity by 29 % would negatively impact the detection efficiency when employed in the context of a scintillator detector. This sample represents the worst case scenario that can occur when applying the coating, while in practice, the reflectivity will likely lie between this lower approximation and that of the standard.

This comparison shows that the worst outcome of the coating still increases the reflectivity by a factor of almost 3. The best possible result could increase the reflectivity by a factor 4. To minimize the risk of oxidation, it is necessary to achieve full coverage with the primer before applying the reflective coating as any unprimed area can introduce rust stains.

These results reflect only the initial performance of the coating. In practical detector applications, however, the coating is continuously exposed to liquid scintillator over extended periods. To evaluate long-term degradation under such conditions, accelerated aging tests were performed in the laboratory that are documented in the following chapter.



## 6.3 Aging Tests

As the SHiP experiment is planned to be operational for at least 15 years, it is necessary to check for long-term stability of all detector components when in direct contact with each other. As the compatibility of the liquid scintillator with different primers, the reflective coating OPRC or corten steel is largely unknown, laboratory thermal aging tests are vital to this project. If signs of instability are found in the coating or scintillator quality, they need to be quantified to assess the impact this could have on the detection efficiency of the SBT.

Thermal aging tests are performed at a laboratory scale by storing individual glass vessels containing coated steel samples in liquid scintillator. The scintillator samples are kept in an incubator at 60°C, as this is the maximum temperature the coating can withstand according to the manufacturer.

At this temperature, the *Van't-Hoff Law* states that the reaction rate in these samples is increased by a factor of 16-256 compared to room temperature. Since this is a broad range, the decision is made to focus on the minimum equivalent time ( $t_{min}$ ) of this estimation to be conservative. A detailed description of the Van't-Hoff Law, thermal aging test preparation and analysis can be found in chapter 4.4.

All aging tests have been conducted between 2022 and 2024, initially evaluating various primer-coating combinations, followed by testing the best-performing candidate on different liquid scintillator mixtures. Alongside these laboratory tests, the coating is also applied to two detector prototypes used in test beam measurements, in order to study its performance under realistic conditions and its impact on detection efficiency (see chapter 6.4).

This chapter will discuss reflectivity measurements of coated corten steel samples aged in liquid scintillator. By comparing the results with the initial reflectivity, changes within the given time span can be quantified. Additionally, absorption measurements are conducted on the liquid scintillator samples, to investigate changes in scintillator quality.

### 6.3.1 Aging Tests with different Primers

First application tests of OPRC to corten steel in chapter 6.1 have shown that the water content of the coating induces rapid oxidation of the steel surface. These oxidized molecules are solved into the coating during the drying process, leading to a yellow discoloration and lower reflectivity. Several primers are identified in chapter 6.2 that mitigate this problem while showing no influence on the reflectivity of the coating.

Before the reflective coating can be applied to the inner walls of a liquid scintillator-based detector prototype, the long term compatibility of the coating, primers and scintillator are investigated in aging tests.

The liquid scintillator, in which the reflective samples are submerged, consists of unpurified LAB and 2 g/l of PPO. The same scintillator is also employed in the detector prototypes. To mitigate oxidation of the scintillator, the liquid is purged of oxygen through ultra-sonic degassing. The empty space in the individual containers

| Sample Name | Description                                                 |
|-------------|-------------------------------------------------------------|
| A1          | A1 primer on corten steel                                   |
| A2          | A2 primer on corten steel                                   |
| R1          | R1 primer on corten steel                                   |
| R2          | R2 primer on corten steel                                   |
| A1F         | A1 primer on corten steel + OPRC                            |
| A2F         | A2 primer on corten steel + OPRC                            |
| R1F         | R1 primer on corten steel + OPRC                            |
| R2F         | R2 primer on corten steel + OPRC                            |
| CT          | Blank, unoxidized, corten steel                             |
| F           | OPRC on corten steel, no primer, completely stained by rust |
| SSF         | OPRC on stainless steel                                     |
| Ref         | Scintillator without reflective sample, reference           |
| start       | Scintillator before aging                                   |

Table 6.3: Details on all samples compared in the 2022 aging tests

is filled with nitrogen to prevent oxygen from entering the sample. An extensive description of thermal aging tests is given in chapter 4.4.

All reflective samples included in this aging test series are listed and detailed in table 6.3. The samples are stored in an incubator at 60 °C for 120 days which results in a minimum equivalent aging time of  $\sim 5$  years. After incubation, the absorbance of the scintillator samples and the reflectivity of the coatings are measured and compared to initial values. The results of these measurements are discussed in the following chapters.

### 6.3.1.1 Reflective Coating

After a minimum equivalent aging time of  $\sim 5$  years, the scintillator samples are removed from the incubator and the included reflective samples are extracted. In this chapter, the state of the reflective coating is assessed through visual comparison and reflectivity measurements. By comparing the aged samples to an unaged reference, the changes during incubation are quantified. Figures 6.7 and 6.8 show photos of all primed and coated steel samples before and after aging.

Scratches and missing coating can be seen on several samples after aging. This is mainly due to handling and extraction of the reflective samples from the scintillator. Since there is no physical handling of the coated surface in the context of a detector prototype, this issue is mostly irrelevant for the intended employment of the coating. Additionally, extended exposure to the liquid scintillator leads to softening of the coating as the scintillator is absorbed into its porous surface. This explains why samples coated with OPRC over a grey primer (A2F, R2F) or directly on stainless steel (SSF) exhibit signs of the coating losing its opacity and becoming translucent, exposing the darker underlying surface. Wrapping these samples in a paper towel and

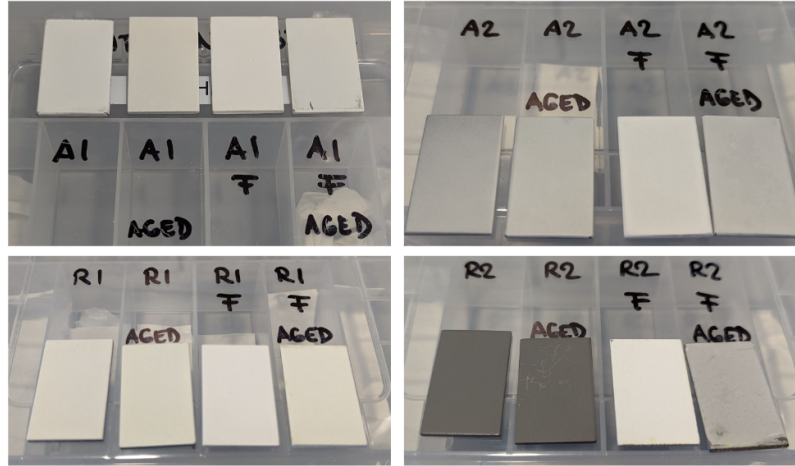


Figure 6.7: Photos of primed and coated corten steel samples before and after aging

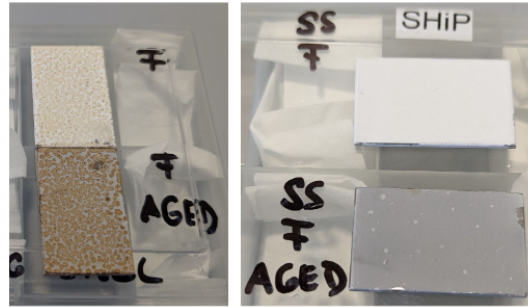


Figure 6.8: Fotos of samples F and SSF before and after aging

allowing them to dry for a few days enables the scintillator to be reabsorbed, partially restoring the coating's opacity. This effect is illustrated in figure 6.9.

All white primers (A1, R1) and coatings applied over white primers (A1F, R1F) exhibit varying degrees of yellowing after aging. This is likely due to surface oxidation of the corten steel. Since the samples showed no signs of yellowing prior to aging, it is concluded that the softening of the coating and primer allows oxidized molecules to migrate into the coating, resulting in yellow discoloration. This is supported by sample F, which demonstrates that preexisting rust stains darken over time when exposed to the scintillator. The absence of yellowing in sample SSF (coated stainless steel) clearly indicates that the reactive surface of corten steel is the primary cause of the yellowing effect. Samples coated with the grey primer (A2, R2, A2F, R2F) likely also underwent yellowing during aging. However, this discoloration is not visible due to the grey background.

While visual inspection can reveal certain issues with the coating, reflectivity measurements are necessary to quantitatively assess the coating's condition. The experimental

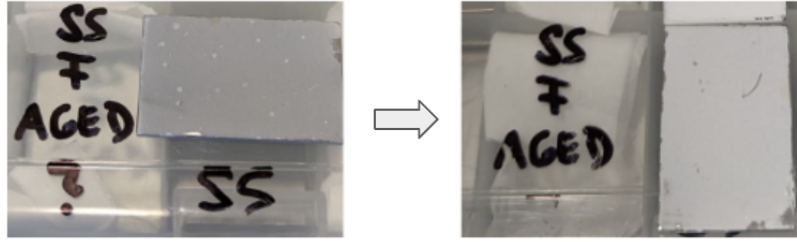


Figure 6.9: After wrapping the samples in a paper towel to dry, the opaqueness of the coating is partly restored. Here shown is sample SSF.

setup and analysis are described in chapter 4.5.

Figure 6.10 shows a representative reflectivity measurement of sample R1F after incubation and drying. The spectrum distinctly exhibits the characteristic emission peak of PPO, indicating that the fluor is incorporated into the coating. This fluorescence leads to a distortion of the reflectivity spectrum as light from lower wavelengths is shifted to the typical double peak structure of PPO, increasing the intensity of light in this region.

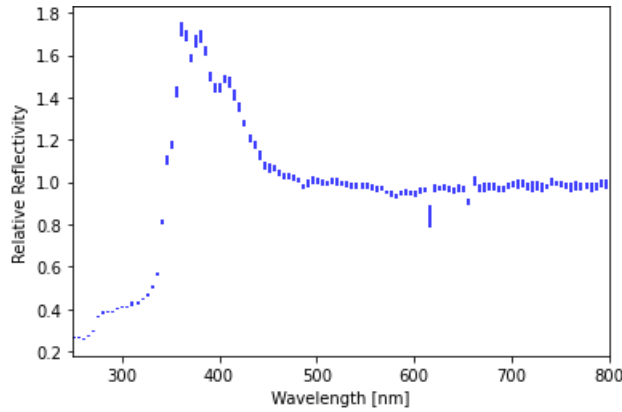


Figure 6.10: The reflectivity spectrum of aged coating shows the typical double peak structure of PPO fluorescence, indicating that the fluor has been deposited in the coating.

To avoid excitation of the fluor in the coating, a polished PMMA piece is positioned between the light source and the sample. The PMMA effectively absorbs all light below  $\lambda = 390$  nm, thereby blocking wavelengths within the PPO excitation range while allowing efficient transmission of longer wavelengths. This setup prevents reflectivity measurements below 400 nm, despite the scintillator's expected peak emission occurring at  $\lambda = 380$  nm. Given that the majority of light reflected from the detector's inner surfaces is spectrally shifted into the PPO emission range, this analysis is not suitable for evaluating the reflectivity of a large fraction of the reflected light. While most of PPO emission lies below 400 nm, a small portion of the full intensity can reach wavelengths of up to 460 nm (see figure 3.5). Therefore, the investigation of reflectivity for wavelengths above 400 nm, is not ideal, but still relevant for the application in a liquid scintillator

| Sample | $R_{unaged}(425 \text{ nm})$ | $R_{aged}(425 \text{ nm})$ |
|--------|------------------------------|----------------------------|
| A1F    | 1                            | $0.88 \pm 0.08$            |
| A2F    | 1                            | $0.86 \pm 0.07$            |
| R1F    | 1                            | $0.88 \pm 0.07$            |
| R2F    | 1                            | $0.81 \pm 0.07$            |
| SSF    | 1                            | $0.82 \pm 0.08$            |
| F      | $0.8 \pm 0.01$               | $0.6 \pm 0.05$             |

Table 6.4: Relative diffuse reflectivity of primed and coated corten steel samples before and after aging for  $\lambda = 425 \text{ nm}$ . The wavelength-dependent reflectivity is measured relative to a  $\text{BaSO}_4$ -standard with an angular configuration of  $(0^\circ - 45^\circ)$ .

detector. Due to measurement instabilities in the range  $400 \text{ nm} < \lambda < 425 \text{ nm}$ —likely caused by residual absorbance of the PMMA—the comparison is conducted at  $\lambda = 425 \text{ nm}$ . This method is applied to all samples measured in this chapter. The diffuse reflectivity spectra of all aged reflective samples are displayed in figure 6.11. They are compared to a  $\text{BaSO}_4$  standard that is measured under identical conditions.

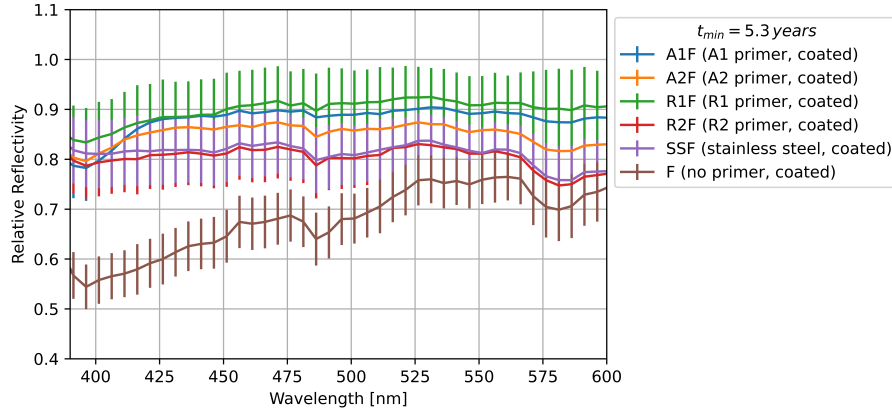


Figure 6.11: Relative diffuse reflectivity spectra of all relevant aged samples with PMMA to block  $\lambda < 390 \text{ nm}$ . The wavelength-dependent reflectivity is measured relative to a  $\text{BaSO}_4$ -standard with an angular configuration of  $(0^\circ - 45^\circ)$ .

The reflectivity before aging of the samples is shown in figure 6.6. The change in reflectivity is evaluated in table 6.4.

Overall, the uncertainties of the measurements are too large to draw definitive conclusions about the performance differences among all primer/coating combinations. However, all primed samples, except R2F, exhibited higher remaining reflectivity than the coated sample without primer (SSF), suggesting that applying a primer can enhance the coating's stability. The best results were observed in samples A1F and R1F, both retaining 88 % of their initial reflectivity. This is likely due to the primers A1 and R1 containing  $\text{TiO}_2$  which can compensate for lost reflectivity in the visible region due to translucence of the coating.

Sample F experienced a 25 % reduction in reflectivity, the greatest loss among all aged samples. This indicates that coatings showing rust stains prior to aging are more likely to suffer greater reflectivity degradation, confirming the visual comparison.

These results strongly support the use of a primer in combination with the OPRC coating, with R1F and A1F demonstrating the best reflectivity retention after aging. The following chapter will investigate the scintillator absorbance of every individual sample to identify potentially harmful processes during incubation.

### 6.3.1.2 Scintillator Quality

During aging tests, coated and primed corten steel samples are stored in individual containers filled with liquid scintillator. By measuring the absorbance of the scintillator samples and comparing them to an identical scintillator sample before aging, changes in the spectrum can be identified. An increased absorbance within the range of fluor emission correlates with reduced light collection. Therefore, efforts are focused on minimizing these adverse effects.

The reference scintillator sample (AgedRef) is stored in the incubator at the same conditions as all other samples, excluding a reflective sample, to isolate effects of aging that are independent of added materials. The start sample is measured before aging to provide a reference for the full absorbance change that occurs during incubation. A detailed description on the execution and analysis of absorbance measurements is given in chapter 4.1.

Generally speaking, an increase of absorbance through aging is related to two processes: Impurities, such as solved coating, can be introduced into the scintillator or chemical reactions, such as oxidation, can change the molecules of the scintillator over time, which changes the absorption and emission properties. The absorption spectra of all aged samples of this test series are displayed in figure 6.12.

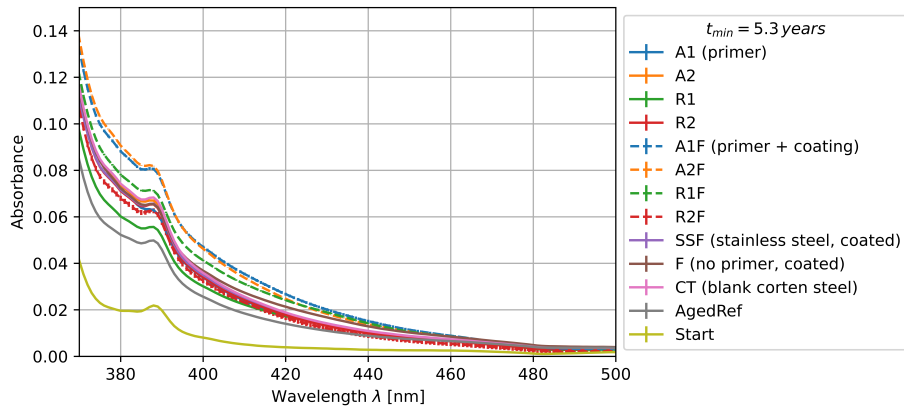


Figure 6.12: Absolute absorbance measurements of all aged scintillator samples from this aging test

As expected, the start scintillator sample shows the least absorbance, followed by Aged-Ref, with the lowest absorbance of all incubated samples. This validates the method

of thermal induced aging. The plot demonstrates that the presence of any foreign material in the scintillator accelerates aging, with the effect being more pronounced at shorter wavelengths.

Figure 6.13 shows the absorbance change of all aged scintillator samples containing corten steel with different primers and primer/coating-combinations. These samples are of main interest as they are primary candidates for the implementation in detector prototypes.

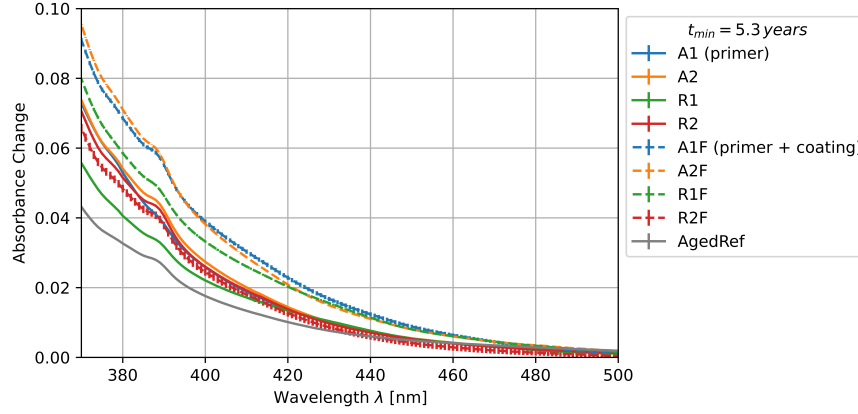


Figure 6.13: Absorbance change of aged scintillators compared to the "start" sample. This plot displays all aging samples with different primers and primer/coating-combinations. AgedRef is displayed to quantify aging that takes place without a reflective sample.

When comparing the samples, excluding sample R2F, all scintillators containing primed and coated corten steel exhibit higher absorbance than those containing only primed steel. This observation could suggest that the reflective coating contributes to increased aging in these samples. However, samples SSF and F, which are coated without primer (as shown in figure 6.14), display absorbance levels comparable to those of primed but uncoated samples and blank corten steel (CT). This finding supports the conclusion that the reflective coating alone is not responsible for the increased degradation of the scintillator.

The comparable increase in absorbance seen in samples SSF, F, and CT relative to the reference suggests that the incorporation of stainless steel or corten steel leads to elevated absorbance levels, independent of OPRC application. Additionally, this confirms that unoxidized corten steel does not cause greater aging than stainless steel. In general, the acrylic primers (A1, A2) performed worse than the rust protection primers (R1, R2), in regards to preserving scintillator quality. This implies that the combinations R1F and R2F are the most promising candidates for the implementation in a liquid scintillator detector.

The following section summarizes the key findings from this aging test. By integrating the results from the reflectivity measurements of the coated steel samples with the absorbance measurements of the corresponding scintillators, a recommendation is provided for their implementation in a detector prototype.

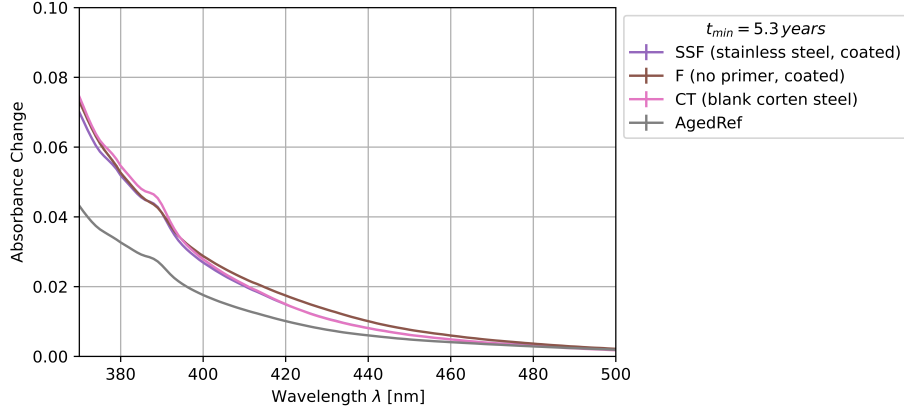


Figure 6.14: Absorbance change of aged scintillators compared to the "start" sample. This plot displays all unprimed, coated aging samples and a sample containing blank corten steel. AgedRef is displayed to quantify aging that takes place without a reflective sample.

### 6.3.1.3 Results

In this chapter, the compatibility of all components was evaluated through thermal aging tests, in which individually contained steel samples with different primers and coatings were immersed in liquid scintillator and stored in an incubator at 60 °C. The results from this series of aging tests form the research basis for the application of the reflective coating in a 1-cell prototype for test beam measurements at DESY in 2022. A detailed description of this project is given in chapter 6.4.1.

Overall, this research has shown that both the reflective coating and the scintillator quality are affected over time when combined. This will also happen in the context of a detector.

Visual comparison showed slight yellowing in some of the reflective samples that developed during aging. The coating is softened due to scintillator absorption, allowing the migration of oxidized molecules from the corten steel surface into the coating leading to a yellow discoloration. This also causes fluor deposition and partial translucency of the coating which is reversed by drying the samples. If the coating presents preexisting rust stains, they are darkened during aging leading to increased reflectivity reduction. After a minimum equivalent aging time of  $t_{min} = 5.3$  years, samples primed with the acrylic primer A1 and the anti-rust primer R1 with the OPRC coating (A1F, R1F) showed the highest remaining reflectivity for  $\lambda = 425$  nm of 88 %.

Absorbance measurements on the scintillator samples are able to verify the increased aging of scintillators containing a steel sample compared to a reference sample. The aging rate shows an upward trend when comparing simply primed samples to primed and coated samples. This leads to the conclusion that the scintillator ages faster, the more materials are added. Whether the coating itself is responsible for increased absorbance of the scintillator remains inconclusive as blank and coated corten steel produce similar results. A comparison of coated stainless steel and coated corten steel shows similar results, indicating that both metals are similarly compatible. Overall,



the analysis of the scintillator quality concludes that anti-rust primers have a lower impact on the absorbance than acrylic primers.

In the context of detector prototypes, these results lead to the recommendation for the application of the R1 primer onto the inner detector walls before adding the reflective coating. This configuration showed the highest reflectivity stability while having a low impact on the scintillator. It is of high priority to prevent oxidation of the corten steel surface during the coating process as this can lead to rust stains that darken over time. This is achieved by ensuring full coverage when priming the corten steel as any direct contact with the coating induces oxidation.

Chapter 6.3.2 describes further aging tests conducted on the anti-rust primer R1 and OPRC coating in preparation for a 4-cell prototype detector for test beam measurements at CERN in 2023. These tests focus on the utilization of different scintillator treatments to reduce degradation during aging.

### 6.3.2 Aging Tests with different Scintillators

To evaluate the utilization in large scale detector prototypes, aging tests were conducted in chapter 6.3 to investigate long term stability of the reflective coating OPRC when applied to corten steel and submerged in liquid scintillator. Due to oxidation on the corten steel surface, that is promoted by the water content of the coating, different primers were tested to enhance the stability of the coating and form a barrier between the steel and coating. This approach prevents the premature yellowing of the coating that is caused by oxidized molecules from the corten steel. In this chapter, further research is conducted on the anti-rust primer R1, which performed best in the previous tests.

During the preparation for the coating of a 4-cell prototype in 2023, which is used in test beam measurements at CERN and detailed in chapter 6.4.2, two series of aging tests are conducted. These tests aim to quantify the aging effects on identically primed (R1) and coated (OPRC) corten steel samples immersed in differently treated scintillators. While all scintillators included in this research consist of LAB and 2 g/l of PPO, as proposed for SHiP's SBT, they differ in treatment and antioxidant addition. The standard in scintillator preparation is to ensure that the scintillator does not contain solved oxygen. This is due to the fact that oxidation of the fluor is the main driver of scintillator degradation during aging. Through this process, the absorbance of the scintillator increases in the range of 350–450 nm, as the products of oxidation absorb light in this region. Advanced oxidation typically leads to a visible yellowing of the scintillator which could transfer to the coating. Due to the direct impact on the scintillator's light yield, the aging tests in this chapter are focused on quantifying the effects of oxidation by inducing and suppressing oxidation through different methods.

| Sample Name | Description                                                       |
|-------------|-------------------------------------------------------------------|
| DG-P        | Degassed, purified LAB                                            |
| DG-PT       | Degassed, purified LAB, 0.001% Tocopherol                         |
| DG-UP       | Degassed, unpurified LAB                                          |
| O2-P        | Solved oxygen, purified LAB                                       |
| O2-PT       | Solved oxygen, purified LAB, 0.001% Tocopherol                    |
| DG-P-rust   | Degassed, purified LAB, oxidized corten steel (no primer/coating) |
| XX-aged-Ref | Aged references without corten steel samples                      |
| XX-start    | Before aging                                                      |

Table 6.5: Details on all scintillator samples for aging tests

Degassing (DG) of scintillators, for the removal of oxygen, is conducted with an ultrasonic cleaner. The addition of the antioxidant, in this case  $\alpha$ -Tocopherol (T), offers further protection against this invasive process. By oxygenating (O2) the scintillator, oxidation is encouraged. This is achieved by shaking the sample for several minutes. While most scintillators in these aging tests use purified LAB (P), one sample is left unpurified (UP) to investigate whether this leads to additional aging.

All scintillators, except those mentioned specifically, contain a sample of corten steel

that is primed with R1 and coated with OPRC. The coated surface area per volume of scintillator is equivalent to the proportions in the 4-cell prototype. An overview of samples from aging tests in this chapter is given in table 6.5.

The samples are aged in an incubator at 60 °C. The first aging series (S1) is stored for 56 days which is equivalent to 896 - 4096 days at room temperature. This results in a minimum aging of around  $t_{min} = 2.5$  years. The second aging series (S2) is measured twice, after 11 days and after 43 days, which is equivalent to a minimum of 176 days (0.5 years) and 688 days (1.9 years) at room temperature.

The results of these aging tests are separated into reflectivity measurements of the coated steel samples and scintillator absorption measurements and are discussed in the following chapters.

### 6.3.2.1 Reflective Coating

Corten steel samples that are primed with R1 and coated with OPRC are aged in individually treated liquid scintillators for different time spans. To compare the reflectivity after aging, visual comparison of the samples and diffuse reflectivity measurements are carried out. This section presents the impact of aging on the reflective coating taken from two separately conducted aging test series which are detailed in chapter 6.3.2.

Just as in chapter 6.3.1.1 the coated samples are extracted from the individual liquid scintillators after storage in an incubator. The samples are dried in a paper towel for several days to remove most of the absorbed scintillator from the coating, before the reflectivity is measured.

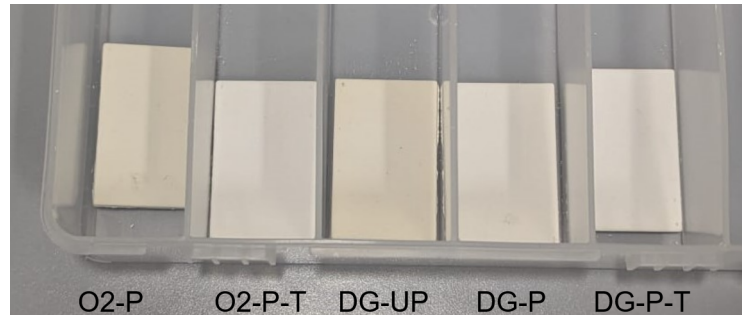


Figure 6.15: Visual comparison of primed and coated corten steel samples, aged in liquid scintillator for a minimum equivalent time of 2.5 years

Figure 6.15 shows a visual comparison of all aged reflective samples after a minimum equivalent aging time of 2.5 years from test series S1. Samples O2-P and DG-UP show signs of yellowing while all other samples remain white after aging. The discoloration is likely caused by oxidized compounds from the corten steel surface. While, in theory, a highly oxidized scintillator could contribute to yellowing of the reflective coating, the observed results suggest otherwise. In the oxygenated sample (O2-P), oxidation of the scintillator floor is actively promoted, whereas in the degassed sample (DG-UP), this reaction is suppressed. However, both samples exhibit yellowing on their respective

reflective surfaces, indicating that the discoloration is more likely attributable to preexisting surface oxidation on the corten steel segments. This is supported by the fact that the scintillators show no apparent signs of yellowing. The softening of the coating during incubation allows for the migration of the oxidized particles from the steel to the coating, turning it yellow in the process.

Tocopherol is an antioxidant used in the samples O2-PT and DG-PT to prevent the oxidation of the scintillator. While this helps to protect the coating from yellowing caused by scintillator oxidation, it does not prevent discoloration resulting from steel oxidation, as the latter occurs before and during the coating process.

The visual comparison of reflective samples from test series S2 reveal no noticeable changes in coating appearance, supporting the conclusion that the yellowing observed in samples O2-P and DG-UP from S1 are outliers.

All coated steel samples from both test series are measured in the reflectivity setup that is detailed in chapter 4.5. The addition of a block of polished PMMA prevents light below 390 nm from illuminating the sample and exciting the deposited fluor in the coating. This approach is detailed in chapter 6.3.1.1.

The reflectivity spectra of all samples from test series S1 after a minimum equivalent aging of 2.5 years are given in figure 6.16. The reflectivity spectra for the samples from aging test series S2 are shown in figures 6.17 (0.5 years) and 6.18 (1.9 years).

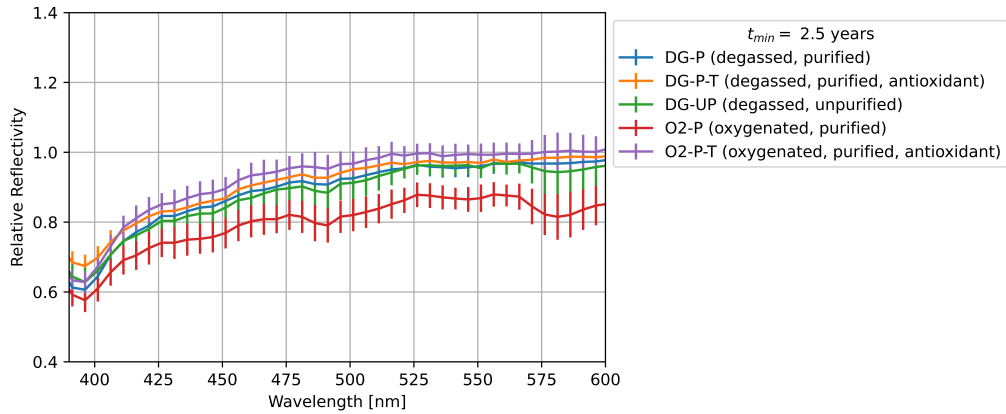


Figure 6.16: Relative diffuse reflectivity spectra of all aged samples of aging test series S1. The minimum equivalent aging is 2.5 years. The wavelength-dependent reflectivity is measured relative to a  $\text{BaSO}_4$ -standard with an angular configuration of  $(0^\circ - 45^\circ)$ .

The measurements are compared at  $\lambda = 425 \text{ nm}$ , as this is still within the emission spectrum of the scintillator but far from the cut-off of the shielding PMMA. The results are shown in table 6.6.

| Sample | $R_{2.5}$ (S1)  | $R_{0.5}$ (S2)  | $R_{1.9}$ (S2)  |
|--------|-----------------|-----------------|-----------------|
| DG-P   | $0.82 \pm 0.03$ | $0.81 \pm 0.03$ | $0.76 \pm 0.02$ |
| DG-PT  | $0.83 \pm 0.03$ | $0.79 \pm 0.03$ | $0.73 \pm 0.02$ |
| DG-UP  | $0.80 \pm 0.04$ | $0.81 \pm 0.04$ | $0.76 \pm 0.02$ |
| O2-P   | $0.74 \pm 0.04$ | $0.80 \pm 0.04$ | $0.77 \pm 0.01$ |
| O2-PT  | $0.85 \pm 0.03$ | $0.77 \pm 0.03$ | $0.73 \pm 0.01$ |

Table 6.6: Relative diffuse reflectivity of primed and coated corten steel samples after aging for  $\lambda = 425$  nm. The wavelength-dependent reflectivity is measured relative to a  $\text{BaSO}_4$ -standard with an angular configuration of  $(0^\circ - 45^\circ)$ . S1 and S2 distinguish the two individual test series.

Overall, the three individual measurements show no significant deviations among themselves, apart from sample O2-P in S1, proving again, that this sample is an outlier. A correlation between the preservation of reflectivity and the scintillator treatment could therefore not be established.

S2 exhibits a significantly higher aging rate compared to S1. Such deviations can occur when the initial conditions differ between test series—for example, variations in scintillator batches or additive content, insufficient degassing, and similar factors.

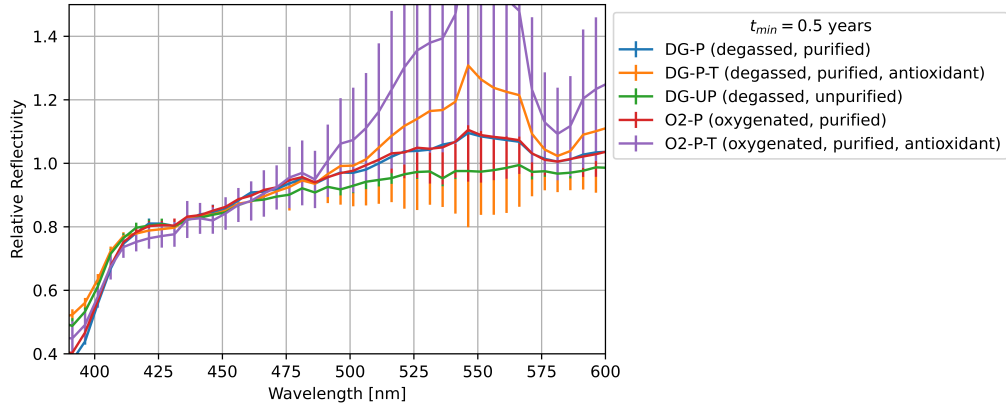


Figure 6.17: Relative diffuse reflectivity spectra of all aged samples of aging test series S2. The minimum equivalent aging is 0.5 years. The wavelength-dependent reflectivity is measured relative to a  $\text{BaSO}_4$ -standard with an angular configuration of  $(0^\circ - 45^\circ)$ .

As this second series of aging tests is measured twice over the course of incubation, an exponential decay of reflectivity over time can be identified. Even though the second incubation period is significantly longer than the first, the reflectivity loss is minimal in comparison. This leads to the conclusion that the main reduction of coating reflectivity occurs within the first 0.5 years or possibly sooner.

As seen in previous aging tests in chapter 6.3.1.1, the reduction in reflectivity for all aged samples is higher for  $\lambda < 500$  nm than for longer wavelengths. This effect is caused by the degradation of the coating, resulting in reduced opacity and potential exposure and light reflection on the underlying titanium dioxide-based primer. Contact

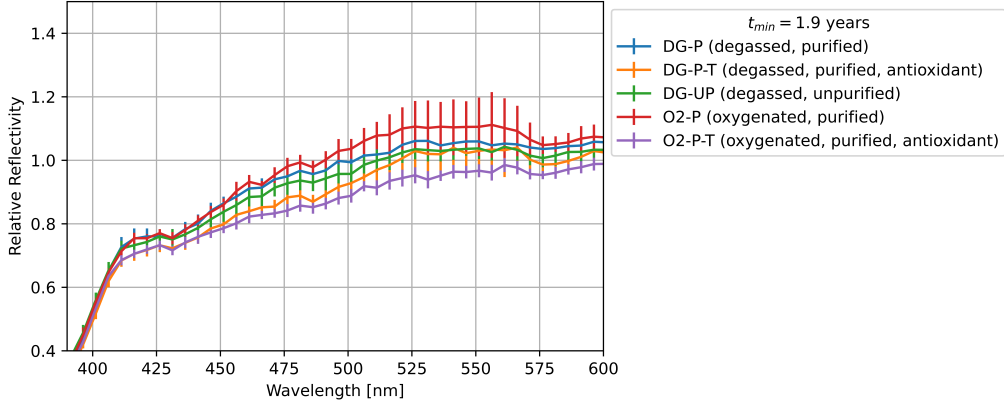


Figure 6.18: Relative diffuse reflectivity spectra of all aged samples of aging test series S2. The minimum equivalent aging is 1.9 years. The wavelength-dependent reflectivity is measured relative to a  $\text{BaSO}_4$ -standard with an angular configuration of  $(0^\circ - 45^\circ)$ .

with LAB ( $n \approx 1.5$ ) penetrates the coating and swells the binder holding the  $\text{BaSO}_4$  ( $n \approx 1.65$ ) particles. This reduces the refractive index difference between the  $\text{BaSO}_4$  and the binder resulting in a more translucent coating, allowing more light to reach the underlying  $\text{TiO}_2$  primer. The primer remains largely unaffected due to the significantly higher refractive index of  $\text{TiO}_2$  ( $n \approx 2.61$ ).  $\text{TiO}_2$  typically reaches a reflectivity of  $\sim 80\%$  compared to  $\text{BaSO}_4$  for  $\lambda = 425\text{ nm}$ . Since the reflectivity measurements indicate a stagnation of reflectivity at  $73\% - 85\%$  relative to the  $\text{BaSO}_4$ -standard, it is likely that, upon advanced aging, some of the light is reflected from the primer rather than the coating. Given that both  $\text{BaSO}_4$  and  $\text{TiO}_2$  are equally efficient reflectors for  $\lambda > 500\text{ nm}$ , the reflectivity in this region remains largely unchanged. This is also supported by the drop in reflectivity for  $\lambda < 410\text{ nm}$ , as  $\text{TiO}_2$  is known to have lower reflectance in the UV region compared to  $\text{BaSO}_4$ .

These results are compared to the reflectivity of unpolished stainless steel shown in figure 6.6 which gives a diffuse reflectivity of  $26\%$  for  $\lambda = 425\text{ nm}$ . In this context, the primer-coating combination maintains a reflectivity that is  $3.17 - 3.27$  times greater, even after extensive aging. Since the surface condition of corten steel can vary significantly—ranging from a texture similar to stainless steel to a fully oxidized state—its reflectivity is, on average, lower than that of stainless steel. This suggests that the actual increase in reflectivity is likely higher than estimated.

### 6.3.2.2 Scintillator Quality

The aging tests summarized in chapter 6.3.2 are specifically designed to induce scintillator oxidation in some samples while inhibiting it in others. Oxidation is generally the primary mechanism of scintillator degradation, emphasizing the importance of studying this process. The presence of oxygen or the introduction of oxidized compounds promotes oxidation of the scintillator fluor, whereas degassing the scintillator or adding an antioxidant can effectively suppress this reaction. This chapter presents

absorbance measurements of scintillator samples of these two aging test series. To quantify the degradation of the scintillator, the measurements of the aged samples are compared to measurements conducted before aging ("start"). Additionally, two scintillator reference samples ("Ref") are incubated without reflective samples to distinguish aging caused by the reflective samples from natural aging. A detailed description of the measurement principle and the analysis related to absorption measurements can be found in chapter 4.1.

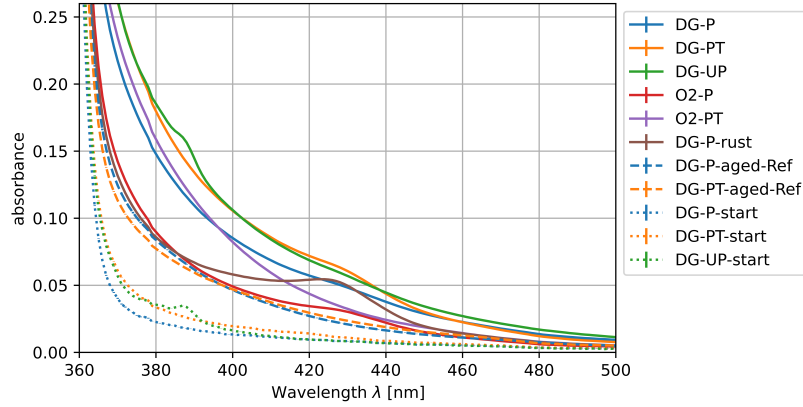


Figure 6.19: Absolute absorbance spectra of all aged scintillator samples from test series S1 after a minimum equivalent aging time of 2.5 years and the corresponding "start" measurements.

Figures 6.19 and 6.20 show the absolute and relative absorbance spectra of the aged scintillators from aging series S1 after a minimum equivalent aging time of 2.5 years, along with the corresponding initial measurements taken before incubation.

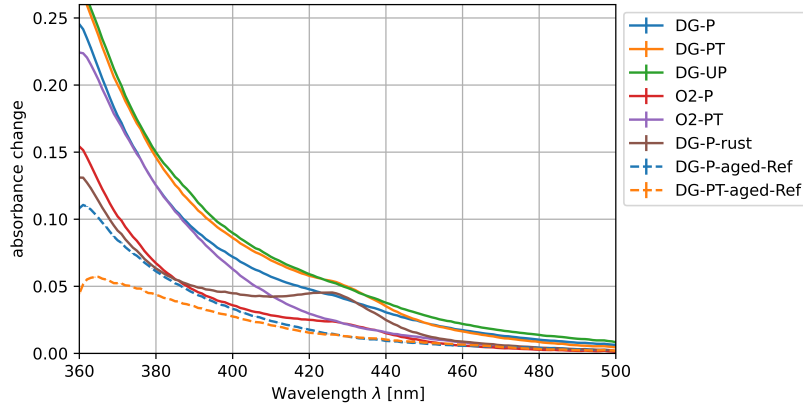


Figure 6.20: Absorbance change of all aged scintillator samples from test series S1 after a minimum equivalent aging time of 2.5 years. The measurements are compared to the individual start sample.

A comparison of the "start" measurements reveals the impact of scintillator purification. The unpurified sample (DG-UP-start) shows a higher absorbance for 370 nm

$< \lambda < 400$  nm than the purified sample (DG-P-start) with a small absorbance peak at  $\lambda \approx 385$  nm. This peak is also visible in the aged sample (DG-UP). Sample DG-PT-start also shows increased absorbance compared to DG-P-start due to the addition of the antioxidant Tocopherol. As anticipated, all aged samples exhibit increased absorbance compared to their corresponding 'start' samples, indicating degradation through effects of aging.

The lowest absorbance of all aged scintillators is found in the reference sample containing Tocopherol (DG-PT-aged-Ref), closely followed by the reference without the antioxidant (DG-P-aged-Ref). As both samples are degassed before incubation, oxidation is mitigated in these samples. The similarity between the spectra indicates that antioxidant effects are negligible in the absence of oxidation-promoting factors. As there is no additional material that can present compatibility issues in these samples, this is the minimum degradation that the individual scintillators undergo during incubation. When focusing on the aged samples for  $\lambda < 390$  nm, there is no correlation between protective scintillator treatment (degassing/antioxidants) and lower degradation during incubation. The degassed samples DG-PT, DG-UP and DG-P show the highest absorbance of the test series, despite the preventative measures applied against scintillator oxidation. Overall, the oxygenated samples show lower absorbance compared to the degassed counterparts. This leads to the conclusion that this wavelength region is unaffected by oxidation.

The results suggest that a process occurs during aging which causes the absorbance spectra of the scintillators to shift upward, regardless of how the samples were prepared. This shift affects shorter wavelengths more than longer ones. The effect is probably caused by impurities that enter the scintillator during sample preparation or through the degradation of the reflective coating.

Since the two reference samples show a similar effect, though less pronounced, it is likely that the impurities are not only introduced from the coating, primer, or corten steel. Contamination from cleaning residues in storage containers or absorption cuvettes may also play a role. However, because the shift is larger in scintillators with reflective samples, it is likely that these materials introduce more impurities. In this case, the effects on the scintillator depend on subtle differences in the quality of the reflective samples, as all reflective samples were produced using the same method.

Signs of oxidation become evident when comparing samples O2-P and DG-P-Rust to the other aged samples in the region of  $390 \text{ nm} < \lambda < 450 \text{ nm}$ , where the two feature a distinctive peak. While DG-P-Rust contains a degassed scintillator, which should inhibit oxidation, it also includes a sample of corten steel with a fully rusted surface. When comparing the height of the peaks, the oxidation of the scintillator is further advanced in DG-P-Rust than in O2-P. This is due to the fact that dissolved oxygen alone is relatively unreactive under normal conditions. While it can contribute to slow oxidation, especially under light or radiation exposure, it does not readily generate highly reactive species on its own. In contrast, the presence of oxidized metal introduces catalytic surfaces and iron ions, which can accelerate oxidative degradation through redox reactions. These reactions produce reactive oxygen species, which rapidly break down scintillator components, leading to faster discoloration, reduced light yield, and



chemical instability.

The absorbance peaks of the two samples lie well within the emission spectrum of the scintillator (see figure 4.4), making it a critical factor to consider as it can lead to an overall lower light yield and detection efficiency. Since none of the other samples show signs of oxidation, the implemented preventative measures can be considered effective. To gather more data, the aging test is repeated using newly prepared samples made in the same manner as in test series S1. The absorbance of the samples is measured after a minimum equivalent aging time of 0.5 years and 1.9 years to distinguish between short-term and long-term scintillator degradation. The absorbance change during the 0–0.5 year period is shown in figure 6.21, while figure 6.22 illustrates the change over the 0.5–1.9 year period.

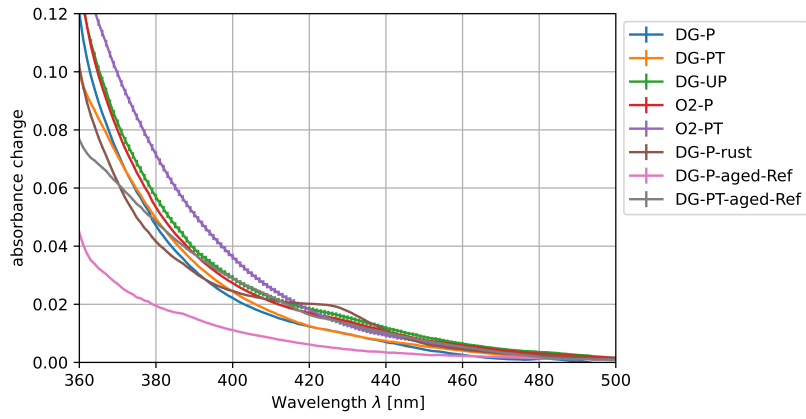


Figure 6.21: Absorbance change of all aged scintillator samples from test series S2 after a minimum equivalent aging time of 0.5 years. The measurements are compared to the individual start sample.

As these measurements exhibit the same absorbance shift caused by contamination, as discussed in the context of S1, this analysis focuses specifically on the absorbance peak associated with scintillator oxidation.

Oxidation during the short incubation period is only identified in sample DG-P-rust, confirming the direct negative impact of previously oxidized molecules on the scintillator. In contrast, the early measurement of sample O2-P shows no signs of oxidation proving the delayed onset of oxidation in oxygenated scintillators.

Figure 6.22 displays the absorbance change of the second incubation period of test series S2. Varying degrees of oxidation are observed in samples DG-P-rust, O2-P, DG-P, and DG-UP. The high oxidation levels in DG-P-rust and, to a lesser extent, O2-P are consistent with previous observations. While the second incubation period is approximately three times longer than the first, the absorbance at  $\lambda = 425$  nm increases by a factor of 8.5 for sample DG-P-rust, confirming that oxidation accelerates over time. This occurs because oxidation products can initiate subsequent oxidation reactions.

The comparatively higher oxidation shown in the degassed samples DG-P and DG-UP

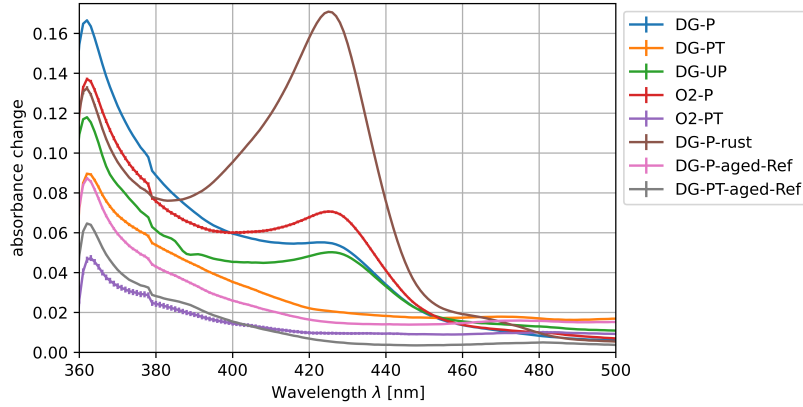


Figure 6.22: Absorbance change of all aged scintillator samples from test series S2 after a minimum equivalent aging time of 1.9 years. The measurements are compared to the samples with a minimum equivalent aging time of 0.5 years to separate short-term and long-term scintillator degradation.

is unexpected, as the corresponding samples in test series S1 did not exhibit this behavior. Since the aged reference sample DG-P-aged-ref shows no signs of oxidation, residual dissolved oxygen or air ingress into the sample containers can be excluded as the primary cause. Instead, this suggests that the introduction of the reflective samples consistently promotes scintillator oxidation. The most probable cause in these cases is the presence of undetected surface oxidation on the corten steel samples used in S2.

The addition of Tocopherol successfully suppressed oxidation in all scintillator samples. This provides direct evidence of the antioxidant effects of tocopherol, even under strongly oxidative conditions as seen in O2-PT.

The following section provides a comprehensive overview of all results and conclusions from the aging tests in this chapter.

### 6.3.2.3 Results

This chapter investigates the long-term compatibility of differently treated liquid scintillators (LAB + PPO) with corten steel, primed with an anti-rust primer (R1) and coated with the reflective coating OPRC from Berghof Fluoroplastics. The reflective samples are placed into separate containers, submerged in scintillator and incubated at 60 °C. The scintillator samples differ in the preventative measures applied to inhibit oxidation.

It is known that liquid scintillators are highly susceptible to oxidation which is promoted by oxygenating the scintillator or introducing oxidized materials to the scintillator. Oxidation is decreased by degassing the scintillators or adding antioxidants. Scintillators that are subjected to oxidation typically feature increased absorption in the range of  $350 \text{ nm} < \lambda < 450 \text{ nm}$  (see figure 6.22). In the absorption spectrum, this feature appears as a growing "bump" with a peak around  $\sim 425 \text{ nm}$ . As the degree of

oxidation increases, the bump becomes more pronounced and broadens accordingly. This behavior is assumed to be caused by fluor oxidation products, which increase light absorption in this range. These effects result in a lower scintillator quality and reduced detection efficiency.

The absorbance of the different scintillators and the reflectivity of the coated corten steel are measured after different aging times to assess the degree of degradation of the materials. The key findings are summarized in this section.

Examinations revealed that the liquid scintillator is absorbed into the reflective coating during incubation, softening it in the process. This softening led to visible yellowing of the coating in two cases, likely due to the migration of oxidized compounds from the steel surface into the coating.

A consistent shift in the absorbance spectra of all scintillators—affecting shorter wavelengths more strongly—indicates varying degrees of contamination during incubation. As this effect is more pronounced in samples containing a reflective sample, the primary source of contamination is identified as soluble components from the coating or corten steel.

Additionally, the  $\text{BaSO}_4$  coating becomes translucent when exposed to the scintillator. When in contact with LAB ( $n \approx 1.5$  [90]), the coating is penetrated leading to swelling of the binder holding the  $\text{BaSO}_4$  ( $n \approx 1.65$  [91]) particles. This swelling reduces the difference in refractive index between the  $\text{BaSO}_4$  particles and the binder, which lowers the amount of light scattered by the coating. As a result, the coating loses some of its opacity, allowing more light to pass through and reach the underlying  $\text{TiO}_2$ -based primer. While  $\text{TiO}_2$  ( $n \approx 2.61$  [92]) has a significantly higher refractive index and therefore provides strong reflectivity, it is less efficient at reflecting UV-light, which is the main emission range of the scintillator. This causes a decrease in the overall reflectivity and efficiency of the coating system in the UV region.

These effects result in an exponential reduction in reflectivity of 15–27% at  $\lambda = 425$  nm within the first two years of aging, with the most significant decline occurring within the first 0.5 years. Even greater losses are estimated at shorter wavelengths. However, no clear correlation was found between the extent of scintillator oxidation and the degree of reflectivity loss, suggesting that the long-term reflective performance is primarily determined by the initial quality of the reflective coating.

The introduction of corten steel with an oxidized surface leads to the highest rate of scintillator oxidation, resulting in degradation that is more than 2-3 times greater than that observed in all other oxidized scintillator samples. The oxidized steel exposes the scintillator to a catalytic surface and highly reactive species, that directly induce oxidation of the scintillator. As corten steel, in general, is highly susceptible to surface oxidation, the construction of a liquid scintillator detector of the material would have strong implications on the long term stability of the scintillator.

Although the oxygenated samples generally exhibited greater degradation, some degassed scintillators also experienced oxidation during incubation. This is attributed to undetected surface oxidation on the corten steel samples, as none of the comparable reference samples exhibited similar behavior. This demonstrates that scintillator degassing is only effective in suppressing oxidation when no pre-oxidized compounds are

present.

The addition of tocopherol successfully suppressed oxidation in all scintillator samples, even in sample O2-PT in which oxidation is encouraged through solved oxygen. This supports the general recommendation to include antioxidants in liquid scintillator formulations to enhance long-term stability.

Figure 6.19 shows that the reduction in transparency achieved through purification of the scintillator is negligible in comparison to the increase in absorbance due to aging. Since there are no indications of increased oxidation caused by impurities in the scintillator, the necessity of extensive scintillator purification is questionable in the light of the results.

This concludes the analysis of laboratory aging tests. The following chapter presents the two prototype detectors, that were both coated with OPRC for test beam measurements conducted at DESY and CERN. In this context, the setup, spray coating process and the results of the test beams are discussed.

## 6.4 Test Beam Prototypes

To enhance the overall light yield of detector systems, one effective strategy is to increase the reflectivity of the inner detector walls. This can lead to a significant increase in detection efficiency, especially when the building material (corten steel) features low reflectivity in the UV-region.

In the context of the SHiP SBT, the reflective coating OPRC was studied through a combination of small-scale laboratory tests and large-scale prototype evaluations conducted in parallel. The laboratory investigations focused on diffuse reflectivity, chemical compatibility, aging effects, the role of primers, and various application techniques, as discussed in chapters 6.1 and 6.3. Simultaneously, large-scale tests on two detector prototypes were performed to apply and assess the coating under realistic conditions within a closed volume. These tests provided valuable insight into both the practical aspects of spray application and the impact of improved wall reflectivity on detection efficiency.

Chapter 6.4.1 presents the results on the application of the reflective coating in a 1-cell-prototype. This detector is employed in test beam measurements at DESY at the end of 2022. Even though oxidation led to the development of rust stains in the coating, a reflectivity of 65 % is achieved. The results concerning the 1-cell-prototype have been published in [81].

Chapter 6.4.2 shows the results on the application of OPRC in a 4-cell-prototype. By increasing air flow in the detector and using an anti-rust primer, rust stains are reduced to a minimum in the first state after application. This larger detector was used in test beam measurements at CERN over the course of 2023 and 2024.

The overall state of the coating in these detectors was inspected with an endoscopic camera to assess changes in appearance over time. This revealed that rust stains appear darker when in contact with liquid scintillator. Additionally, a study on the scintillator quality was conducted on the 4-cell prototype over a period of two months proving that a portion of the coating is solved into the scintillator over time.

### 6.4.1 DESY Test Beam 2022

A full-scale SBT cell prototype (1-cell-prototype) has been constructed in 2022 for characterization in a test beam in October 2022 at the DESY II area 24 [93]. The detector is tested with positrons of varying energies between 1.4 GeV and 5.4 GeV. While the positron beam does not trigger the same detector response as MIPs, it is still well suited for initial detector characterization. In this context the first large scale application of the reflective coating OPRC is conducted on the inner detector walls. A CAD drawing of the 1-cell-prototype is shown in 6.23. It is constructed of corten steel (S355 J2W 1.8965) sheets of 10 mm thickness with all possible welding seams inside the cell. The dimensions are representative for a vertical side segment of the SBT. From one side to the other, the height of the prototype increases from 1224 mm to 1246 mm, which corresponds to the widened shape of the decay vessel. The depth of the cell, and therefore the thickness of LS is 250 mm. Two circular openings were

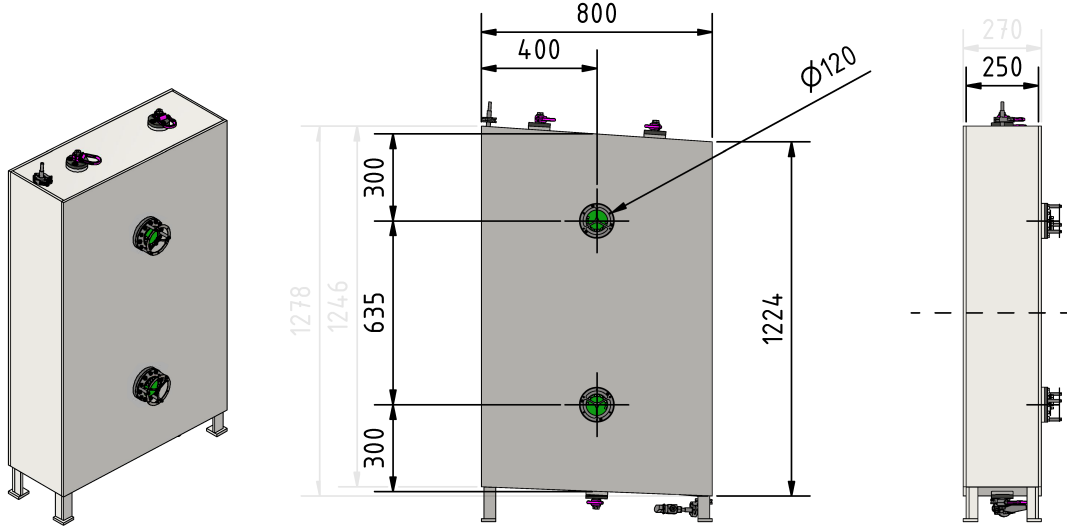


Figure 6.23: CAD drawing of the full-size 1-cell-detector prototype for the Test Beam 2022 at DESY [81]

cut out from the front of the detector to equip the cell with corten steel flanges that hold the double-walled PMMA vessels, which in turn house the WOMs.

The detector features four stands on the bottom to support the prototypes upright position despite its irregular shape. Two crane hooks on the top and one at the bottom allow for transportation. A connection at the lowest point of the box is used to fill the cell with LS. Another connection at the highest point allows for the attachment of an expansion vessel. When filling the cell with LS and leaving the expansion vessel half empty, it is ensured that the detector is filled to the top even if the volume changes due to small temperature variations. In the final setup, a gas line is connected to the expansion vessel to upkeep a nitrogen atmosphere above the scintillator. A pressure relief valve ensures that excess nitrogen can escape the detector. During the test beam, the vertical position of the beam is varied by lifting and lowering the prototype with a DESY-table. A turn table was used to change the incident angle.

Chapter 6.4.1.1 documents the process of coating the inner detector walls with the reflective coating and provides insight on the state of the reflective coating after the test beam. The results are summarized in 6.4.1.2.

#### 6.4.1.1 Coating the 1-cell-prototype

The 1-cell-prototype made of corten steel is coated in September 2022 over a two week period in an empty laboratory space at the JGU in Mainz. In figure 6.24, the detector is shown in preparation before coating.

The inner detector walls are roughly cleaned by spraying isopropyl alcohol onto the surface and then wiping them with paper towels. Before the coating process, the inside of the detector is documented with an endoscopic camera. As shown in figure 6.25,



Figure 6.24: The 1-cell-prototype in preparation for coating

the detector presents small areas of oxidation on mostly unoxidized corten steel. The application of the reflective coating follows research results from chapter 6.3.1.



Figure 6.25: Endoscopic photos showing selected areas of the inner detector walls with and without oxidation.

To prevent oxidation on the corten steel surface, when in contact with the  $\text{BaSO}_4$  coating, previous laboratory tests have shown that a primer can act as a protective barrier between the OPRC and the steel, mitigating oxidation. The anti-rust primer

R1 shows the best performance in aging tests when comparing remaining reflectivity and scintillator quality. Due to the fact that the anti-rust primer leads to blockage of the spray gun nozzle during several attempts which could only be solved through complete disassembly of the spray gun, the A1 primer is used as the next best option. The inner walls of the cell are coated by using a radial nozzle inserted through the WOM holes. While this does not allow for visual inspection of the surfaces during coating, by varying the insertion angle of the spray gun, all surfaces within the detector are accessible. Due to the importance of full coverage of the corten steel surface with primer and coating, both were applied in two layers. As the airflow within the detector is restricted and the blind application leads to higher amounts of primer and coating applied, the drying time is increased. While previous open-faced coating tests dried within 1-2 hours, the individual layers in the detector were given 48 h each to fully dry. The inside of the detector is inspected with an endoscopic camera after each layer. This is especially helpful to achieve complete coverage with the primer, due to the visual difference between the steel and the white primer. However, the dried OPRC coating and the primer are both white in appearance and have similar reflectivity in the visible spectrum. Therefore they are difficult to distinguish with the endoscopic camera. To identify uncoated areas on a primed background, the surfaces were inspected shortly after coating to find dry spots, where the coating was not applied. These areas are then targeted specifically with the reflective coating.

With this method of application, the detector is successfully primed and coated. Due to the increased drying time, the wet (water-based) acrylic primer is in contact with the corten steel for a longer period of time compared to open-faced tests. This leads to oxidation of the steel surface and dark orange rust stains in the first layer of primer. These stains appear in areas, independently of previous oxidation on the corten steel. As a removal of the primer was not an option, the decision was made to continue with the coating process in the hopes of covering the rust stains with the following layers of primer and OPRC. While the second layer of primer did have this effect, the first layer of reflective coating caused a drastic increase in affected areas. With the final layer of OPRC the rust stains changed color to a lighter orange. Endoscopic photos of the inside of the prototype after every layer of primer and coating are shown in figure 6.26. The top half of the detector (figure 6.27) shows larger areas of rust stains than the bottom half (figure 6.26). The reason for this is unknown, as one would expect, the wet primer and coating to run down to the lower half of the detector and cause more oxidation at the bottom.

As shown in laboratory aging tests in chapter 6.3, the quality of the reflective coating changes over time when in direct contact with liquid scintillator leading to reduced reflectivity. Therefore, inspection of the coating after the test beam is of great interest. As the SHiP experiment will be taking data for at least 15 years, it is crucial that the performance of the SBT does not drop below a level, where the set goal of  $> 99\%$  detection efficiency can no longer be guaranteed.

To conduct actual reflectivity measurements, the prototype would have to be destroyed in order to cut samples out of the detector walls. This is not an option as it will be employed at the Albert-Ludwigs-Universität Freiburg for measurements of cosmic rays.



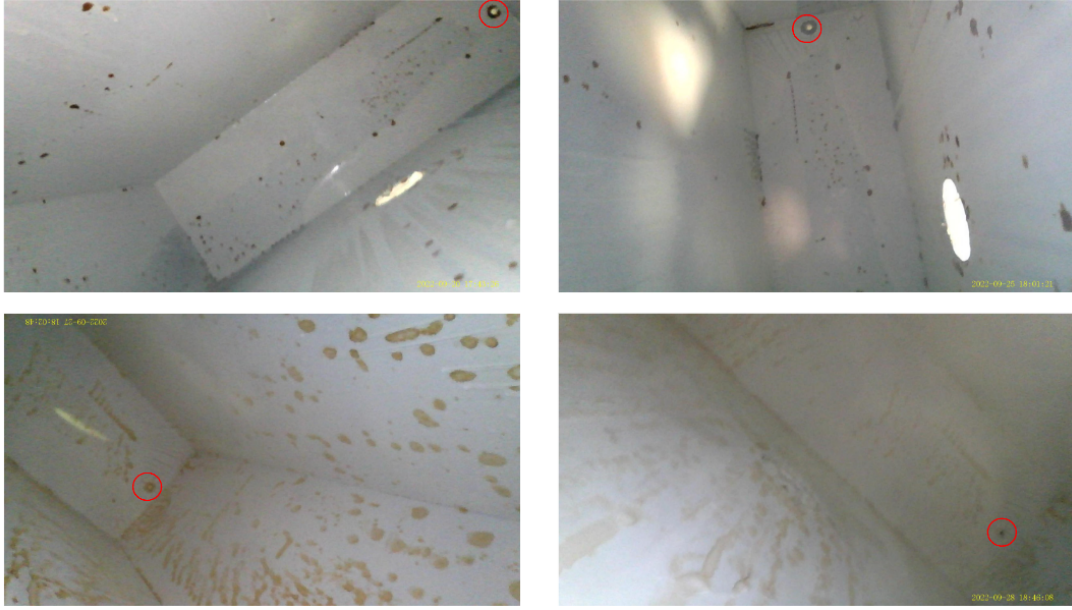


Figure 6.26: Endoscopic photos of the bottom half of the 1-cell-prototype after every layer of primer and coating. For orientation, the connection that is used for filling is marked in every photo. **Top left:** first layer of primer **Top right:** second layer of primer **Bottom left:** first layer of reflective coating **Bottom right:** second layer of reflective coating



Figure 6.27: Final coating result of the top half of the 1-cell-prototype

Another test beam measurement with this prototype is not intended, so a repeat of the method to determine the reflectivity during test beam is not possible.

The only option to estimate, whether the coating has changed compared to the previous result, is to empty the detector and to record new pictures with the endoscopic camera. These are compared to recordings made shortly after the coating process was complete. Aging tests have shown that rust stains become darker over time and even white areas will develop a yellow tint.

In figure 6.28, selected areas within the detector are compared in their appearance shortly after coating and two months later, after the cell was filled with scintillator during the test beam.

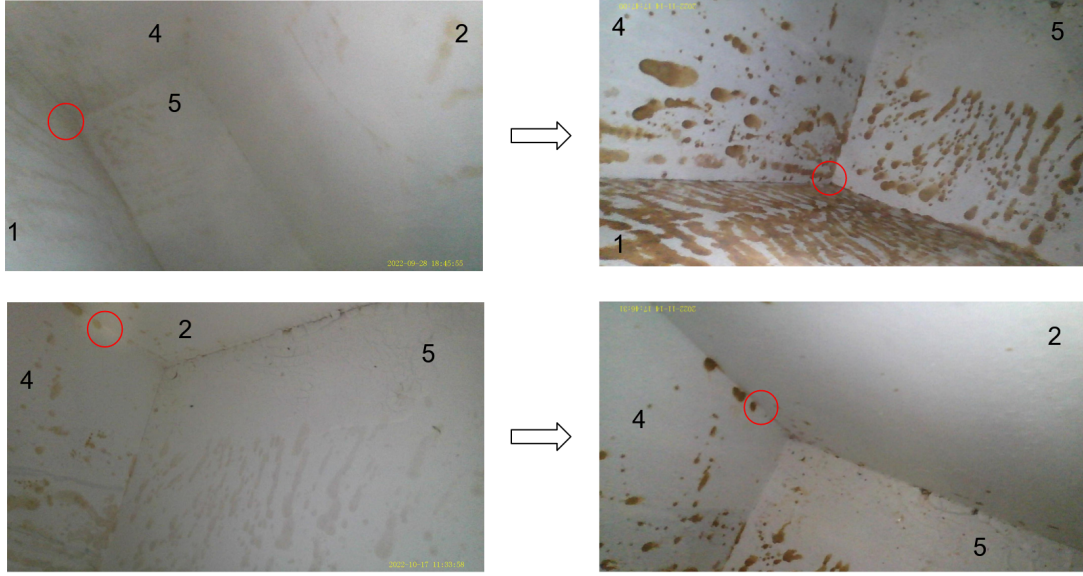


Figure 6.28: Comparison of endoscopic photos to assess the condition of the reflective coating. **Left:** After completion of spray coating. **Right:** After test beam. A number is assigned to the individual walls and a red circle marks a characteristic area in both photos to provide orientation.

As expected, the rust stains are significantly darker after being in contact with liquid scintillator during the test beam. Therefore a further loss in reflectivity can be assumed. Chapter 6.4.1.2 will present results for the achieved reflectivity and detection efficiency in the 1-cell-prototype.

#### 6.4.1.2 Results from the DESY Test Beam Campaign

In preparation for the October 2022 test beam at DESY, the inner surfaces of a one-cell prototype detector were coated with the reflective paint OPRC to enhance reflectivity. An acrylic primer was applied directly to the corten steel to improve coating adhesion and reduce rust staining. However, rust stains appeared during the drying of the first primer layer and persisted through subsequent primer and paint layers. The water content of the acrylic primer accelerated oxidation of the steel surface, while limited air ventilation inside the detector prolonged drying times. As a result, oxidized particles were released into the primer, producing orange discoloration.

The reflectivity of the inner walls of the detector prototype is estimated by comparing the detector signals from the test beam with signals from a Geant4 Monte-Carlo simulation using varying wall reflectivity. The closest agreement is reached when setting the simulated reflectivity to 65 % of the full potential of the coating [94]. These results are displayed in figures 6.29 and 6.30. This is not only a rough approximation but also gives a mean value for the overall reflectivity of the prototype. In reality, depending on the exact location in the detector, the reflectivity will vary strongly. This result seems compatible with those of reflectivity measurements conducted in

the lab in Mainz. These have shown that stained areas feature a lower reflectivity of  $R(400\text{ nm}) = 0.74 \pm 0.01$  compared to white areas of the coating, which is detailed in chapter 6.3.1.1. The reflectivity of the coating, in all cases, is reduced further, when in contact with the liquid scintillator.

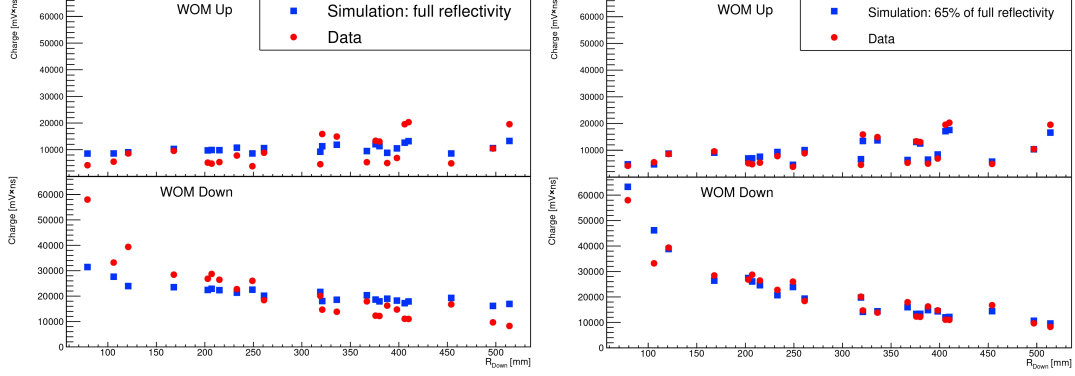


Figure 6.29: Comparison of Monte-Carlo simulation with test beam data: the plots show the signal charge depending on the beam distance to the upper and lower WOM. **Left:** simulation data with full reflectivity **Right:** simulation data with 65 % reflectivity [94]

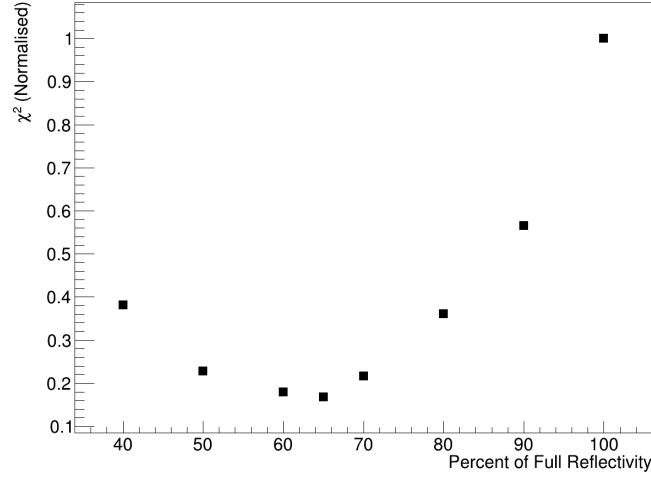


Figure 6.30: Normalized  $\chi^2$  plotted against the reflectivity of the inner detector walls. The minimum at 65 % indicates the best fit between simulation and data [94].

During test beam measurements, different positions of the detector are targeted with positrons of varying energies between 1.4 GeV and 5.4 GeV. The detection efficiency over the entire detector is found to be  $\geq 99.3\%$  at a confidence level of 68 % when using only one WOM. With the requirement for at least one of the two WOMs to register a signal above dark-count level the detection efficiency reached  $\geq 99.5\%$  at a confidence level of 68 %. Overall, these results are in agreement with the requirements for the SBT and have been published in [81].

### 6.4.2 CERN Test Beam 2023/24

In 2023, a (2x2) 4-cell-prototype is constructed for test beam measurements at CERN. In this beam time, the detector response to electrons and muons is characterized. By employing four cells, instead of a single cell, more precise information on the particle trajectory, such as the crossing point or the incident angle, can be gathered. The main purpose of this test beam campaign was to study edge effects that occur when a particle crosses two cells close to the border. The irregular size of all four cells is representative for a side section of the SBT. A detailed CAD drawing of the prototype is shown in figure 6.31.

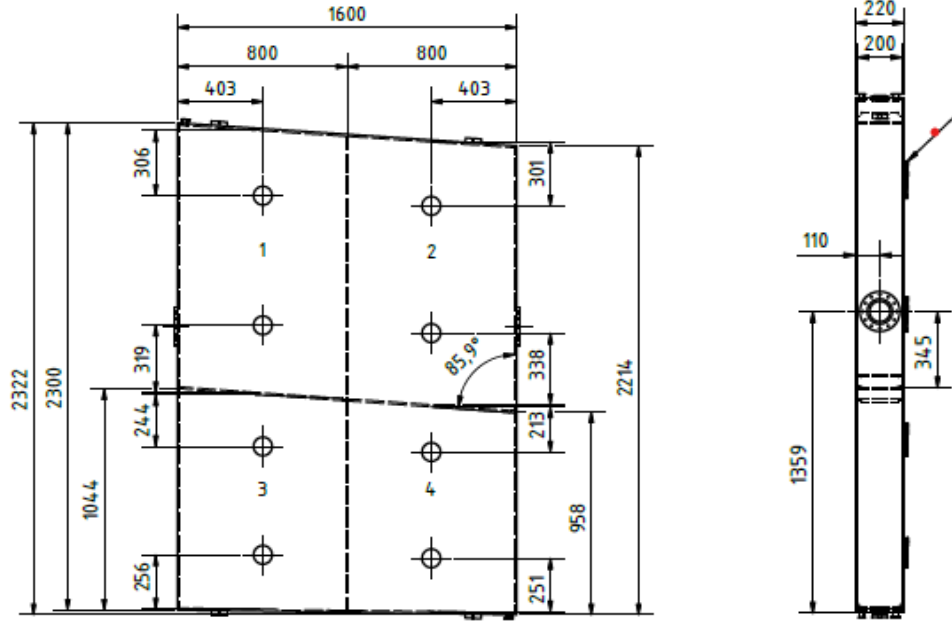


Figure 6.31: CAD drawing of the 4-cell-prototype for the test beam 2023/24 at CERN. The inner and outer walls are made of 10 mm thick corten steel. The four cells of irregular size represent a side section of the SBT, with the height of the detector cells following the widened shape of the decay vessel. All lengths are given in mm.

The prototype measures 2322 mm in height and 1600 mm in width. The cell depth was reduced to 200 mm, as previous beam-time results demonstrated high detection efficiency, suggesting that a thinner layer of liquid scintillator would suffice. All of the corten steel walls have a thickness of 10 mm. The entire prototype holds a total of 700 l of liquid scintillator. This allows for testing of scintillator purification, handling and filling on a much larger scale.

The walls that separate the cells within the detector feature small holes in the corners so the detector can be filled through a single connection at the bottom. A small expansion vessel with a nitrogen connection ensures that the detector is completely filled and under nitrogen atmosphere at all times. A photo of the 4-cell-prototype,

fully equipped and positioned in the test beam area, is shown in figure 6.32.



Figure 6.32: The 4-cell-prototype fully equipped with WOMs, SiPMs and readout electronics at the CERN test beam area

The inner walls are fully coated with an anti-rust primer and the  $\text{BaSO}_4$ -based reflective coating OPRC. The coating process, as well as any changes in coating quality observed over the following months, are described in chapter 6.4.2.1. Chapter 6.4.2.2 presents changes in scintillator quality over the course of the test beam measurements. Chapter 6.4.2.3 summarizes the findings.

#### 6.4.2.1 Coating the 4-cell-prototype

A 4-cell-detector prototype, constructed of corten steel was spray coated with an anti-rust primer (R1) and the reflective coating OPRC in preparation for test beam measurements at CERN in 2023/24. The coating process was carried out in a designated spray booth at the Albert-Ludwigs-Universität Freiburg.



In contrast to the application in the 1-cell-prototype, several improvements were made to reduce the risk of coating yellowing. To shorten drying time, pressurized air was connected to the top of the prototype. By closing the WOM holes and opening a valve at the bottom of the detector, the air was forced to flow through all 4 cells of the prototype. This was intended to minimize the drying time, during which corten steel remains in contact with wet primer or coating, in order to reduce oxidation and the dissolution of oxidized compounds from the steel surface into the coating.

Even though the inner walls of the prototype were covered in rust and writing from the construction (see figure 6.33), cleaning of the inner surfaces was not conducted with this prototype. This was mainly due to safety concerns when dispersing volatile substances such as isopropyl alcohol in a closed volume as this can create an explosive atmosphere. Non-volatile cleaning agents would either accelerate the oxidation of the steel surface or lead to potential scintillator contamination.



Figure 6.33: This figure shows a selection of endoscopic photos taken from the inside of the 4-cell-prototype before the coating process. The inner walls are covered in rust and writing from the construction of the prototype.

As the anti-rust primer was not applicable with the radial nozzle in the 1-cell prototype due to the high viscosity, the nozzle was replaced with a 90° angled nozzle featuring a larger diameter. This change required adjustments to the application technique, as the nozzle's orientation had to be altered multiple times at each insertion point to ensure complete coverage of the inner walls. The primer not only improves the physical stability of the coating but also acts as a protective barrier, preventing contact between the corten steel and the wet reflective coating. Due to the water content of the reflective coating, which can rapidly oxidize the steel surface and lead to coating discoloration, complete coverage with primer is critical. The primer and coating were

applied in two layers with at least 24 h of drying time in between.

During the drying of the first primer layer, a malfunction in the moisture extractor of the pressurized air system went unnoticed. As a result, small amounts of water entered the detector through the pressurized air valve, causing a large yellow stain in the primer around the valve (see figure 6.34).



Figure 6.34: **Left:** A large yellow stain developed in the first layer of primer due to water in the pressurized air. **Right:** The following layers of primer and coating were able to fully cover this area.

After a temporary substitution for the pressurized air system was found, the application of the following layers could occur without complications. As shown in 6.34 the subsequent layers of primer and coating fully covered the yellow stain around the air valve.

The final coating results show no obvious rust stains from oxidation on the surface areas of the steel. An assortment of endoscopic photos that are taken after the final layer of coating has dried is presented in figure 6.35.

Upon closer inspection, slight yellowing could be identified around some of the weld seams and connecting holes between the individual cells. Compared to the detector's rough surface structure, the weld seams are significantly smoother. This reduced surface texture, combined with potential oil or gas residues from the welding process, can cause the anti-rust primer to bead up and partially repel from these seams during application. As a result, small gaps in primer coverage may occur, allowing the reflective coating to come into direct contact with the underlying corten steel. This direct contact promotes oxidation, which manifests as yellowing of the coating. The coverage of the primer inside the connecting holes is insufficient due to the limitation of the spray nozzle. While these stains are light in color and difficult to distinguish with the endoscopic camera, examples of affected areas are marked in figure 6.36.

As these stains are known to become darker or spread out over time, when in contact with liquid scintillator, the state of the reflective coating after the first test beam measurements is investigated. As the scintillator is absorbed into the coating, it softens the material and increases its translucency. This, in turn, allows more reflections from the underlying  $\text{TiO}_2$ -based primer, which is less efficient at reflecting UV light. Over time, this process can also lead to the formation of new rust stains and the darkening of stains introduced during coating application. As all of these effects lead



Figure 6.35: A selection of endoscopic photos taken of the inside of all cells of the 4-cell-prototype after completion of the coating process



Figure 6.36: Some areas around weld seams and connecting holes between the cells show slight yellow discoloration in the final coating result. This is due to the primer showing low adherence to the seams and not being able to fully cover the connecting holes with primer.

to a reduction in reflectivity which negatively effects the detector's efficiency, the state



of the reflective coating in the 4-cell prototype is examined in March 2024, after the detector has been filled with liquid scintillator for a time span of 4 months.



Figure 6.37: This figure presents endoscopic photographs of the inner walls of the 4-cell prototype, taken in March 2024. By this time, the reflective coating had been in contact with the liquid scintillator for four months. Each photograph corresponds to one of the individual cells, showing the condition of the reflective coating in that cell.

The photographs shown in figure 6.37 give a broad overview of the individual cells of the detector. While the coating on the large surface areas remains mostly white, the regions presenting yellow discoloration are highlighted in figure 6.38.

As discussed, the limitations of applying the coating with the 90° nozzle become particularly evident when attempting to cover the interior of the connecting holes between the individual detector cells. In addition, the welding seams caused the primer to bead up and fail to adhere properly. In both cases, this led to direct contact between the water-based reflective coating and the corten steel, which in turn induced oxidation on the steel surface. Figure 6.38 clearly shows dark rust stains concentrated mainly in these areas.

In comparison to the coating application described in chapter 6.4.1.1 in the 1-cell prototype, the new method using an anti-rust primer in combination with pressurized air to reduce drying time has produced significantly better results.

Apart from the reflective coating, the liquid scintillator is another component of the detector that undergoes aging and may suffer performance losses over time. This aspect is investigated in chapter 6.4.2.2.



Figure 6.38: This figure presents additional endoscopic photographs of the 4-cell prototype, taken in March 2024. The focus here is on highlighting areas of the reflective coating that exhibit pronounced yellowing.

#### 6.4.2.2 Scintillator Study

In addition to planning, constructing, and preparing the detector prototype, test beam measurements also provide an opportunity to evaluate the liquid scintillator. This includes preparing the scintillator through purification, mixing in additives on sight and testing of a large-scale liquid handling system responsible for filling the detector. While these procedures are discussed in chapter 5.3.1, this chapter will focus on how the scintillator quality changes over the time span of two months during the test beam at CERN.

1000l of LAB are transported and stored at the test beam site in an Intermediate Bulk Container (IBC), in which the additives (PPO and Tocopherol) are mixed in with a magnetic stirrer. A gas line that is connected to the IBC and submerged in the liquid allows for nitrogen flushing to remove solved oxygen from the scintillator. Additionally, a nitrogen atmosphere is retained in the IBC, the liquid handling system

and the detector prototype to prevent oxygen from entering the scintillator. During the test beam, six scintillator samples were extracted from the IBC and the detector in order to carry out absorption measurements with the UV-Vis spectrometer which is detailed in chapter 4.1. The results are presented in figure 6.39.

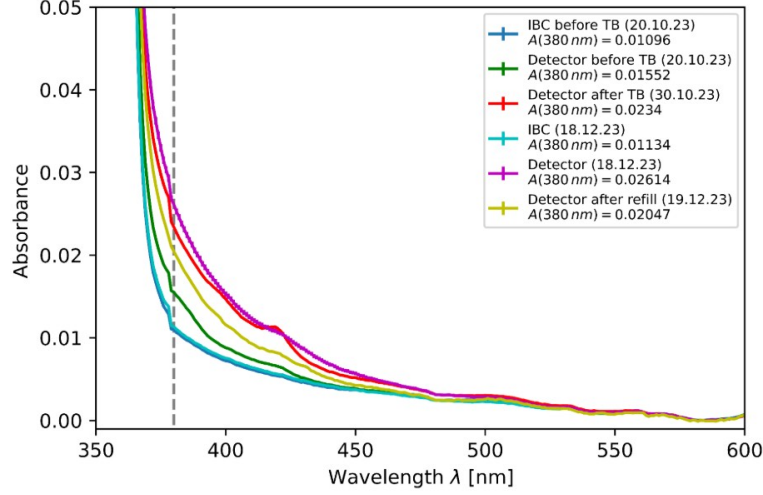


Figure 6.39: This plot shows absorption measurements taken from different scintillator samples that were collected during the test beam campaign in 2023 at CERN.

The first sample was taken from the IBC, after the additives were successfully mixed into the scintillator. This measurement is seen as a reference to all following samples to identify changes in scintillator quality.

The second sample is extracted from the detector, shortly after it was filled into the detector. Given that the detector holds a volume of  $\sim 700\text{l}$ ,  $300\text{l}$  of liquid scintillator remain in the IBC after this procedure. The plot shows a clear increase in absorbance by 50 % for  $\lambda = 380\text{ nm}$  compared to the reference. Since the change in absorbance is wavelength-dependent and comparatively small at higher wavelengths, this suggests that remnants from the welding of the detector and parts of the coating were released into the scintillator. This is reinforced by the fact that the inner detector surfaces were not cleaned before or after the coating process. Flushing the detector with scintillator not intended for operation would prevent the initial introduction of contaminants into the active scintillator.

After the first phase of test beam measurements concluded, another sample was collected from the detector, to assess changes that occurred during the ten-day-period. This absorption measurement shows a increase in absorbance by  $\sim 130\%$  for  $\lambda = 380\text{ nm}$  compared to the reference sample indicating that more coating is solved into the scintillator over time. A small oxidation peak (first described in chapter 6.3.2) around  $\lambda = 420\text{ nm}$  is also visible in this measurement. Since scintillator oxidation in this short time span of aging is improbable and the following sample taken from the detector does not present this feature, the oxidation is attributed to inadequate storage of the sample between extraction and measurement.

Around two months later, two additional scintillator samples are extracted from both the IBC and the detector. The absorption measurement of the scintillator that was stored in the IBC is almost identical to the initial IBC sample. Therefore, the IBC is considered a suitable option for long-term scintillator storage.

Compared to the absorbance increase observed within the first 10 days, the scintillator sampled from the detector after two months showed only minor changes. This supports the hypothesis that the primary source of contamination is particles released from the coating. The increase in absorbance over the course of two months amounts to 139 % or  $\Delta A = 0,01518$  for  $\lambda = 380 \text{ nm}$  which is in the order of absorbance reduction achieved through scintillator purification  $\Delta A_{\text{purification}} \approx -0,015$  (see figure 5.4). Consequently, for future application in SHiP, additional precautions would have to be taken, such as pre-cleaning or rinsing of the cell walls with LAB, or circulation of liquid scintillator while filtering.

After the detector's content is pumped out into the IBC and refilled into the detector, another sample is collected. This final measurement represents a mixture of  $\sim 30 \%$  IBC scintillator and  $\sim 70 \%$  detector scintillator. Since the measured result of  $A(380 \text{ nm}) = 0.02047$  is close to the estimation of  $(0.3 * A_{\text{detector}}) + (0.7 * A_{\text{IBC}}) = 0.0217$ , this confirms the viability of the method applied in this chapter.

The following chapter gives a concise overview of the results from the CERN test beam campaign.

#### 6.4.2.3 Results from the CERN Test Beam Campaign

In 2023, a 4-cell detector prototype made of corten steel was constructed for a test beam campaign at CERN. To increase the efficiency of the detector, the reflectivity of the inner walls is improved by coating them with the  $BaSO_4$ -based paint OPRC by *Berghof Fluoroplastics*. Since the corten steel surface begins to oxidize rapidly when in contact with water which is included in the coating, a protective and stabilizing primer is applied between the two. This measure aims to prevent yellowing of the coating which is introduced through oxidized particles that are transferred from the corten steel to the coating.

The coating process (detailed in chapter 6.4.2.1) was carried out with a spray gun using the standard nozzle and a  $90^\circ$ -nozzle to access all inner detector walls through the WOM holes. In contrast to the coating procedure of the 1-cell prototype (see chapter 6.4.1) two measures were implemented to reduce oxidation in the 4-cell prototype. Instead of applying an acrylic primer, an anti-rust primer is used in this detector. To increase airflow and to reduce drying time of the primer and coating layers, pressurized air is forced through all four cells of the prototype. A comparison of the final coating results from both detectors indicates that these precautions effectively inhibited oxidation of the corten steel and the consequent discoloration of the coating. While the coating of the 1-cell prototype was covered in rust stains, the only visible yellowing of the coating in the 4-cell prototype was localized around some of the welding seams and connecting holes between the individual cells. These issues resulted from inadequate primer adhesion at the welding seams and the limited angular reach of the spray gun,

which prevented primer application within the connecting holes. As previously seen in the 1-cell prototype, these rust stains appear darker and more pronounced when exposed to liquid scintillator.

Throughout the first two months of the test beam campaign, scintillator samples were extracted from the detector to monitor changes in absorbance (see chapter 6.4.2.2). This study concluded that the absorbance increase for  $\lambda = 380$  nm during this time period ( $\Delta A = 0.01518$  or 239 % of the initial value) was almost equal to the absorbance decrease achieved through scintillator purification. The primary source of scintillator contamination originates from dissolved particles of the coating. This is indicated by the fact that shorter wavelengths are affected more than longer wavelengths. Additionally, the contamination rate slows down with time, with a significant increase in absorbance immediately following detector filling. The impact of this contamination source could be reduced significantly by flushing the detector with liquid scintillator prior to filling the active medium.

A discussion of all results concerning the reflective coating OPRC in the context of a liquid scintillator detector is presented in chapter 6.5.

## 6.5 Summary

This chapter evaluates the use of the BaSO<sub>4</sub>-based reflective coating OPRC by Berghof Fluoroplastics in the context of liquid scintillator detectors with a focus on the Surrounding Background Tagger of the SHiP experiment. The proposed physics goals of the SHiP experiment require the SBT to achieve a detection efficiency of 99 % for minimum ionizing particles.

Corten steel, which was intended as building material for the detector, presents low reflectivity in the UV-region which is the main emission region of commonly used liquid scintillators, such as LAB + PPO. As BaSO<sub>4</sub> acts as a nearly perfect diffuse reflector ( $95\% \leq R$ ) in the relevant wavelength region, the application on the inner detector walls can lead to a significant increase in detection efficiency. This chapter presents an overview of results gathered from a series of laboratory tests and large scale coating application in two detector prototypes examining the coating process and compatibility of all involved materials.

**Rust stains and yellowing** of the coating was observed when OPRC was directly applied to corten steel. This occurrence is attributed to the surface of the metal alloy which promotes the formation of a protective rust layer that impedes oxidation on the inside of the steel. This process is accelerated due to the water content of the coating. The resulting oxidized particles are solved into the coating causing yellow discoloration. A sample that featured pronounced yellowing exhibited 80 % of the standard OPRC reflectivity for  $\lambda = 425$  nm. These stains appear darker when the coating is in long term contact with liquid scintillator reducing the reflectivity even further to 60 %. The application of a **rust-protection primer** between the corten steel and OPRC prevented the staining of the coating.

Laboratory aging tests showed, that the **coating reflectivity** is reduced by 15-27 % ( $\lambda = 425$  nm) within an equivalent aging time of 2 years while submerged in liquid scintillator. Two processes were identified that lead to this degradation in reflectivity. The scintillator is absorbed into the coating causing a deposition of the fluor and a swelling of the coating. This turns the coating translucent as the swelling reduces the difference in refractive index between the BaSO<sub>4</sub> particles and the binder which lowers the proportion of light scattering on the coating. This is only possible due to the similarity of refractive indices of  $n_{LAB} \approx 1.5$  and  $n_{BaSO_4} \approx 1.65$ . The underlying TiO<sub>2</sub>-based primer is not affected since  $n_{TiO_2} \approx 2.61 \gg n_{LAB}$ . This leads to less reflections on the coating and more on the TiO<sub>2</sub> primer which is an inefficient reflector in the UV-region compared to BaSO<sub>4</sub>.

The liquid scintillator also dissolves parts of the coating. This is proven by the fact that the absorbance of the scintillator increases, affecting shorter wavelengths more than longer wavelengths. While the main absorbance increase and decrease in coating reflectivity is observed during short aging times, these values stagnate after the initial changes, further reinforcing the claim. The increase in scintillator absorbance measured in a two month time frame during the test beam of the 4-cell-prototype was almost equal to the initial absorbance reduction through purification ( $\Delta A = 0.01518$ ).

Apart from dissolved particles from the coating, another source for **scintillator degra-**

**ation** is the oxidation of the fluor. This causes a complete loss of the typical absorption and emission properties of the molecule and reduces the light emission and efficiency of the scintillator. Laboratory tests showed that an increase in fluor oxidation is indicated by a growing absorption "bump" in the range of  $350\text{ nm} < \lambda < 450\text{ nm}$ . Since this reaction relies on the presence of oxygen, degassing the scintillator and storing it under a nitrogen atmosphere is an effective preventative measure. The addition of the antioxidant Tocopherol at a concentration of 0.001% proved to be the most reliable method for inhibiting oxidation. The incorporation of previously oxidized molecules in the form of rusted corten steel into the scintillator led to significantly accelerated scintillator oxidation.

The **coating application** was conducted with a SATAjet 1000 B RP spray gun and a nozzle with a diameter of 1.6 mm. As it was necessary to coat the inside of the detectors after they were welded shut through the open WOM holes, a nozzle was employed that allowed for application in a  $90^\circ$ -angle. This method achieved full coverage in both detector prototypes.

The first application of the coating in the 1-cell-prototype led to an approximate reflectivity of 65 % during the test beam measurements. This was due to the use of an acrylic primer which presented promising results in laboratory tests but caused rust stains due to the prolonged drying time in the closed detector. Despite the suboptimal outcome, the detector achieved an efficiency of  $\geq 99.5\%$  at a confidence level of 68 % for positrons with energies between 1.4 GeV and 5.4 GeV with the requirement for at least one of the two WOMs to register a signal. These results comply with the SBT specifications and have been published in [81].

The coating process for the 4-cell-prototype implemented pressurized air to ventilate the inside of the detector and reduce the drying time. The acrylic primer was exchanged for an anti-rust primer. These additional measures achieved a nearly stainless coating. Due to insufficient adhesion of the primer to the welding seams and incomplete coverage of the connecting holes between the cells, light yellowing of the coating was caused in these areas. This emphasizes the importance of complete primer coverage before the application of the coating. As of the submission of this thesis, the final results from the 4-cell prototype test beam have not yet been published.

Even though the coating of the 4-cell-prototype was a success, the reflective coating OPRC is no longer considered for the implementation in SHiP's SBT. The reduced reflectivity of the coating and the contamination of the scintillator through dissolved coating can lead to lowered detection efficiency over time. This is not acceptable, especially in an experiment which is intended to record data for at least 15 years. The practicability of coating all  $\sim 850$  cells of the SBT is questionable as this would require extensive working hours to complete. Also, the underlying corten steel will cause local staining of the coating if the task is not carried out with diligence.

The results in this chapter prove that corten steel, in general, is an unsuitable building material for liquid scintillator detectors. Especially, if the detector is left uncoated, oxidation on the corten steel surface leads to increased oxidation of the scintillator which reduces the emissive properties of the active medium. In any case, the addition of an antioxidant to the scintillator is recommended as it counteracts this process while

having a negligible effect on the scintillator absorbance.

During the research conducted in this thesis, the intended design for the SHiP experiment planned for an evacuated Decay Vessel to achieve maximum background suppression. Following feasibility and detector optimization studies, the Decay Vessel was redesigned to contain Helium at atmospheric pressure to minimize neutrino interactions within the volume. Consequently, the building material was reevaluated since structural stability for the containment of a vacuum was no longer necessary.

Since then, the results of a study [95] were published comparing the performance of three different detector prototypes. The first detector consisted of stainless steel with the inner walls covered in sheets of PTFE. The other two detectors were built of aluminum (AlMg4.5) with one featuring polished inner walls while the inside of the other prototype remained unpolished. The required light yield and time resolution for the SBT was achieved with both the PTFE-covered detector and the polished aluminum prototype. The best performance concerning the uniformity of the detector response across the entire detector, which is also crucial for SHiP, was accomplished with the polished aluminum prototype. These results will affect the ongoing research concerning the final design of SHiP's Surrounding Background Tagger.



## 7 The Silicon Photomultiplier with Active Light Guide Design (SiALG)

*"My eyes open to a crepuscular vision,  
fluorescent lights invade my unsuspecting eyes"*

— 156/Silence, *Entropy*

In modern detector design, Photomultiplier Tubes (PMTs) are the conventional standard for low-light detection. While PMTs are capable of detecting single photon events, they also present several disadvantages. Their operation relies on a fragile vacuum tube, they are expensive and prone to defects due to overexposure. Additionally, their large physical size and low angular acceptance lead to a restriction in efficiency, especially, in application that require wide-field detection. Consequently, large-scale detectors that are designed to measure rare events, such as neutrino interactions, require a large number of PMTs to provide full coverage, significantly increasing the cost and complexity.

The "Silicon Photomultiplier with Active Light Guide Design (SiALG)" presents a compact and cost-effective alternative to PMTs. Developed in cooperation with the Eberhard Karls Universität Tübingen, this design uses Silicon Photomultipliers (SiPMs), which consist of Single Photon Avalanche Diodes that are mounted in an array for an increased photosensitive area. To enhance the angular acceptance of incident photons, the SiPM array is coupled to dedicated light guides. Simulations indicate that conically shaped light guides maximize photon collection efficiency [96] [97]. By taking advantage of total reflection on the light guide's interfaces, the incoming light is focused onto the SiPM array.

Active light guides are essentially plastic scintillators that are shaped according to the application. They combine a polymer matrix with scintillating molecules, that absorb incoming photons and re-emit them within a characteristic spectrum. This allows for the tailoring of the light guide to the spectral detection efficiency of the SiPMs by selecting appropriate fluors.

While the electronics of the SiALGs and the post-processing of the light guides are developed by the Eberhard Karls Universität Tübingen, the production of light guides is conducted in Mainz. In this context, polystyrene-based plastic scintillators are fabricated via thermal polymerization of styrene, enabling a cost-efficient method for the production of highly transparent scintillating light guides.

This chapter discusses the motivation and operating principles of the SiALG Design and provides the theoretical background on polymerization.

## 7.1 Motivation and Working Principle

In order to detect particles that have a low cross section when interacting with matter, it is typical to provide a large volume of active medium to increase interaction probability within the detector. Especially in neutrino physics, experiments such as ANNIE [98], JUNO [99], Borexino [9] and SNO [100] are required to monitor signals produced by scintillator quantities of up to 20.000 t. In the IceCube experiment, a volume of more than one cubic kilometer of ice in the south pole region is equipped with photosensitive equipment for the detection of neutrino interactions [101].

PMTs are the current standard in light detection of very weak signals. Main disadvantages of these photo sensors include mechanical fragility, sensitivity towards magnetic fields, large physical size and high price. SiPMs consist of Single Photon Avalanche Diodes and are currently emerging as an alternative to PMTs. They are small and rugged by design, the production cost is comparatively low and they are insensitive to magnetic fields. A large photosensitive area is efficiently equipped by aligning many SiPMs in an array. To increase the angular acceptance, an active light guide is optically coupled to the array. This describes the working principle of the SiALG [102]. For more detailed information on both types of photo sensors see chapter 3.3. The quantum efficiency of a typical PMT and the photon detection efficiency of SiPMs are displayed in figure 5.7. While PMTs achieve their highest efficiency near 380 nm and become less sensitive at longer wavelengths, SiPMs reach peak efficiency around 500 nm, making fluors that shift emission toward longer wavelengths significantly more effective.

In collaboration with the Eberhard Karls Universität Tübingen, research and development is conducted for the SiALG design. The goal of this project is the production of compact photo sensors, that allow for the separation of Cherenkov and scintillation radiation. Cherenkov photons are emitted when charged particles traverse a dielectric medium at a greater speed than the phase velocity of light in that medium [103]. Scintillation photons emanate from the excitation and deexcitation of molecules with scintillating abilities (see chapter 3.1.1). As Cherenkov radiation typically precedes scintillation photons by 1-10 ns, a high timing resolution of the SiALG is of vital importance [104]. Polystyrene-based scintillators, which usually reach timing resolutions below 500 ps, are therefore the main focus of this study [105]. This polymer is also known to be cost-effective and highly transparent in the region of maximum detection efficiency of SiPMs, making it an excellent candidate for use as light guides for this project.

The contribution of this thesis to the SiALG design, which is illustrated in figure 7.1, lies in the optimization and production of these active light guides.

An inactive light guide consists of a transparent medium with a higher optical density than the surrounding medium. When photons enter the light guide, they are contained within due to inward reflections on the lateral interfaces and are focused onto the SiPM. An active light guide incorporates scintillating molecules (fluor), that shift the light to higher wavelengths within the material. An illustration of possible light paths within the (in)active light guide is shown in figure ???. As the transparency

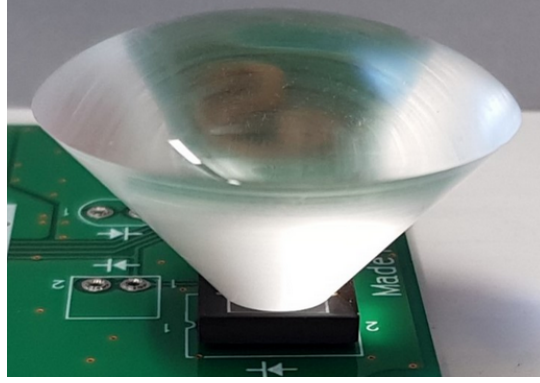


Figure 7.1: Illustration of the SiALG Design consisting of a light guide coupled to a SiPM [106]

of polystyrene increases with higher wavelengths, the addition of fluors increases the number of photons that reach the SiPM. By selecting fluors with emission spectra matching the SiPM's sensitivity range, the light guide can be optimized. In principle, active light guides are plastic scintillators that are shaped accordingly.

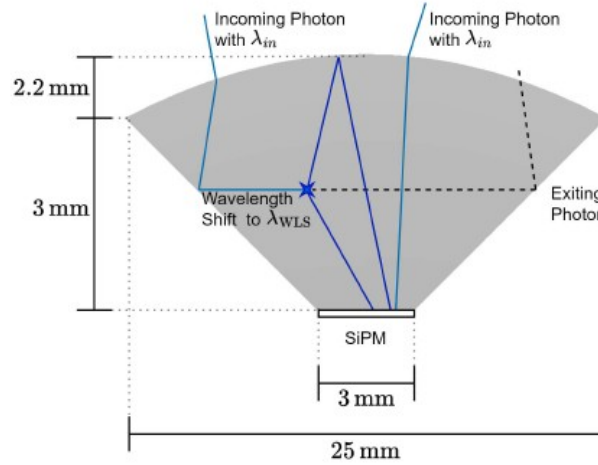


Figure 7.2: Schematic illustration of a SiALG module with corresponding light paths within the (in)active light guide: Light-blue paths indicate photons that either reach the SiPM directly or are initially absorbed by the wavelength shifter. After absorption, photons are re-emitted at longer wavelengths with an isotropic angular distribution. Detection-relevant trajectories of re-emitted photons are shown in dark blue and include both direct transport to the SiPM and redirection toward the photo sensor via reflection from the parabolic upper surface of the light collector. An additional trajectory, depicted by a dashed black line, represents photons emitted at an angle  $\theta_{in}$  smaller than the critical angle  $\theta_{crit}$ . In this case, total internal reflection does not occur, and the photon escapes the light-guide structure rather than being guided toward the SiPM [107].

The motivation for this project is not limited to the SiALG design, as the ability to independently produce high quality plastic scintillators is beneficial for numerous

applications. Commercially produced plastic scintillators are expensive and the ability to tune the scintillator to the individual need, shows great potential.

While the method for producing plastic scintillators through extruded polystyrene is efficient and simple in application, the thermal polymerization of styrene to polystyrene produces a polymer that is superior in transparency. Light Yield measurements have shown that 3D-printed scintillators provide a signal of less than 40 % when compared to an identical sample that is polymerized in one piece [108]. Plastic scintillators are known for their high light yield and signal decay times in the nanosecond range, which are both crucial for the application in the SiALG design.

Due to the overlap of high detection efficiency of the SiPMs with the emission spectrum of bis-MSB, it is chosen as the secondary fluor. This is discussed in more detail in chapter 5.3.2. Due to the low energy transmission between polystyrene and bis-MSB, PPO is added as a primary fluor. The scintillation process is described in chapter 3.1.1. While both fluors are introduced in chapter 3.2, an overview of chemical and physical properties of styrene and polystyrene is given in the following chapter.

## 7.2 Styrene and Polystyrene

Polystyrene plays an essential role in the development of plastic scintillators due to its favorable properties in detecting ionizing radiation. It is a synthetic aromatic polymer made from the monomer styrene which exhibits scintillating properties [109]. The structure of both molecules is displayed in figure 7.3. Important physical and chemical properties are listed in tables 7.1 and 7.2.

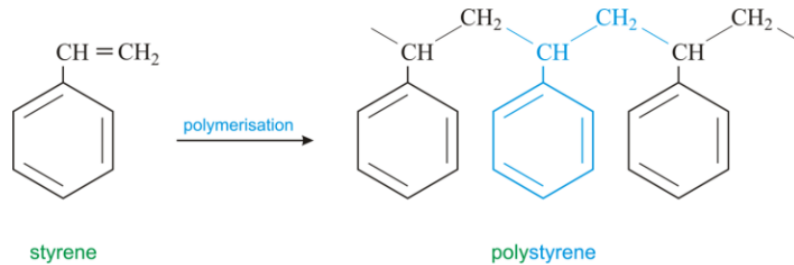


Figure 7.3: Chemical structure of Styrene and Polystyrene [110]

Polystyrene is a versatile thermoplastic that can be easily molded into different shapes, making it suitable for various applications such as light guides. Due to their high light yield (up to 10.000 photons per MeV) and fast signal decay time in the order of nanoseconds, polystyrene-based scintillators are capable of detecting low levels of radiation and present a time resolution that is sufficient for the separation of scintillation- and Cherenkov radiation in the SiALG design. While the polymer features high light transmission in the visible range, this is not the case for the characteristic emission range of polystyrene, which calls for the addition of fluors to prevent self-absorption [111].

|                       |                                         |
|-----------------------|-----------------------------------------|
| Name                  | Styrene                                 |
| Chemical Formula      | $C_8H_8$                                |
| Flash Point           | 34 °C                                   |
| Boiling Point         | 145 °C                                  |
| Lower Explosive Limit | 0.9 %                                   |
| Hazards               | flammable, toxic, probably carcinogenic |

Table 7.1: Chemical and physical properties of Styrene [112]

|                    |              |
|--------------------|--------------|
| Name               | Polystyrene  |
| Chemical Formula   | $(C_8H_8)_n$ |
| Melting Point      | 240 °C       |
| Absorption Maximum | 261 nm       |
| Emission Maximum   | 328 nm       |

Table 7.2: Chemical and physical properties of Polystyrene [113][114]

Styrene is liquid at room temperature which allows for the dissolution of fluors and other additives. The mixture is polymerized to solid polystyrene, which provides a matrix for the suspension of the fluor molecules. When ionizing radiation passes through the polystyrene, it leads to the excitation of the polymer molecules. The energy is then transferred to the dopants non-radiatively, which re-emit this energy as visible light [109].

Thermal polymerization of the styrene–fluor mixture is applied to produce the scintillator. However, due to the high volatility of styrene at room temperature and the toxicity of the vapor, strict safety precautions are necessary during preparation. The low flash point and explosive limit further complicate the polymerization process, as discussed in the following chapters.

### 7.3 Basics of Polymerization

Polymerization is a chemical process in which small molecules called monomers join together to form larger, more complex structures known as polymers. This transformation is fundamental to the creation of a vast range of materials that are essential to modern life, from everyday plastics and synthetic fibers to high-performance materials used in engineering and medicine. Polymers are characterized by their repeating structural units, which give them unique properties such as strength, flexibility, and durability [115].

Plastic scintillators, in general, are a scintillating material in which the fluor is suspended in a base that consists of a solid polymer matrix. To achieve the integration of the fluor, it is typically dissolved in the liquid base before polymerization. Aromatic plastics such as polyvinyltoluene or polystyrene are commonly used in plastic scintillators as they exhibit scintillating properties in the presence of ionizing radiation.

Due to low transparency to the own emission, the use of fluors is necessary to achieve reasonable light yield in combination with these plastics [116]. As the quality of the polymer matrix is of vital importance to the emissive properties of the plastic scintillator, the polymerization process is explained in this chapter.

Polymerization mechanisms are divided into two main classes:

- **Step-growth polymerization** refers to a mechanism where monomers form short chains (i.e dimers, trimers) individually before combining to longer chains later in the reaction.
- **Chain-growth polymerization** describes a mechanism, where monomers add to an active site of an existing chain one at a time. The limitation of active sites throughout the process gives this polymerization technique its characteristic. These active sites can be of different nature (e.g radicals, ions) depending on the monomer [117][115].

As the main focus in this thesis lies on the production of plastic scintillators with a polystyrene base, this chapter will detail the radical chain-growth polymerization in the context of styrene polymerization. The mechanism is separated into three stages:

- The **initiation reaction** presents the beginning of the formation of a polymer chain with the creation of an active site. Monomers are neutrally charged so they do not attract other monomers or present a point for other monomers to connect. By introducing energy into the system, the dissolution of bonds within the monomers is induced, which leads to the creation of highly reactive radicals. The reactivity stems from an unpaired electron in the remaining monomer that can bond to other neutral monomers [115]. In the case of styrene polymerization in this thesis, the initiation energy is introduced thermally, while other methods include energy deposition through radiation or chemical reactions. To lower initiation energy and therefore gain more radicals at a lower temperature, an initiator can be added. The initiator decomposes to two radicals with a lower energy threshold than the monomers. The provided radicals can then react with the monomers, creating active sites in the process. Benzoyl peroxide (BPO) is an initiator that is typically used in styrene polymerization [118].
- The **propagation reaction** describes the reaction of an active site with a monomer, leading to the addition of the molecule to the polymer chain. As the new polymer molecule features an unbound electron, it serves as an active site for the next monomer [117]. This process is repeated until a termination reaction takes place.

Chain transfer is a polymerization reaction that can take place during propagation. By shifting the unpaired electron from the previous active site of the chain to another molecule, chain branching can occur to form more complex structures [115].

- The **termination reaction** occurs when the active site of a growing polymer chain is neutralized and propagation is therefore terminated. This can occur through the combination of two active chain ends, the combination of an active chain end with a radical or the interaction with impurities such as oxygen [117].

All reactions that are involved in chain-growth polymerization are illustrated in figure 7.4.

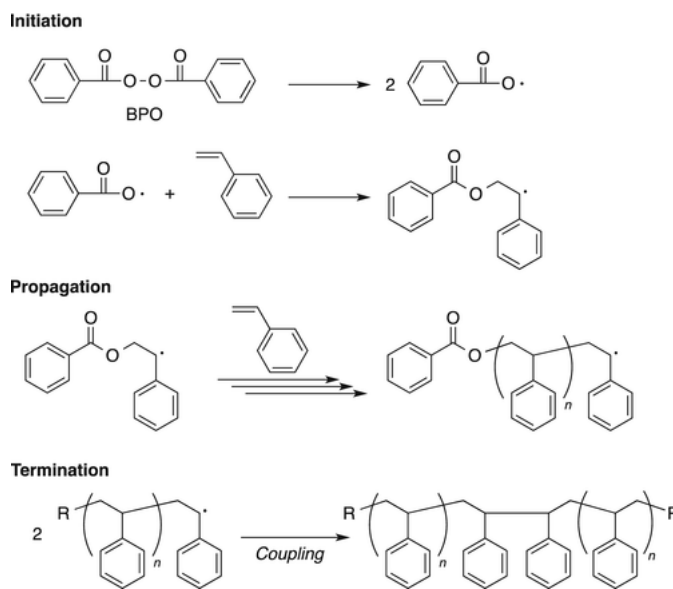


Figure 7.4: Illustration of the three reactions involved with the radical chain-growth polymerization of styrene. The initiator Benzoyl peroxide is added to increase the production of radicals at lower temperatures [118].

Polymerization is a chaotic chain reaction that can be influenced by changing the conditions in which the process takes place. By increasing the polymerization temperature or increasing initiator concentration, the reaction occurs at a faster rate while the average length of resulting polymer chains is reduced. This is connected to the availability of radicals during polymerization as they are decisive for the initiation and termination processes during the formation of a polymer chain. In an environment with a high radical concentration, the probability of termination reactions increases, resulting in shorter polymer chains. In contrast, a lower radical concentration favors propagation reactions, leading to the formation of longer chains. Short chain lengths lead to a softer or brittle polymer with lower molecular weight while longer chains tend to form a harder and more stable product [115].

Initiator use can introduce additional disadvantages such as yellowing of the polymer and bubble formation due to byproducts that originate from decomposition [119]. In the specific case of BPO, the decomposition produces  $\text{CO}_2$  which may become trapped within the polystyrene matrix [120].

It is also important to note, that polymerization is a highly exothermic reaction.

Styrene generates  $71 \frac{\text{kJ}}{\text{mol}}$  during the polymerization process [121]. If the heat dissipation from the sample is not adequate, temperature buildups can occur that impact the polymerization reaction.

This concludes the theoretical basics about polymerization. The following chapter discusses the method of thermal polymerization of styrene in a laboratory setup and presents the results of the scintillator characterization.



## 8 Plastic Scintillator Development and Characterization for the SiALG Design

*"Star baby, did you deep-fry a piece of the Great Barrier Reef?"*

— The Callous Daoboys, *Star Baby*

The SiALG design introduces a novel type of photo sensor that combines an active plastic light guide with a SiPM. Due to its conical geometry, the light guide provides a substantially enhanced angular acceptance compared to conventional PMTs and SiPMs. In this context, a method for the small-scale fabrication of plastic scintillators intended for use as active light guides was developed.

Chapter 8.1 describes the laboratory polymerization setup and outlines the individual steps required to produce plastic scintillators. The influence of different temperatures and initiator concentrations on the resulting polymer is also examined.

Chapter 8.2 presents the characterization of the fabricated scintillators, including measurements of fluorescence, optical absorption, and light yield. The results are compared to the commercial scintillator SP32.

Finally, chapter 8.3 summarizes the findings related to the plastic scintillators and highlights potential directions for future research in this field.

### 8.1 Plastic Scintillator Production

In order to produce active light guides for the SiALG design, a method for the fabrication of polystyrene-based plastic scintillators was established in this thesis. The polymerization of the styrene-fluor mixture is achieved thermally in a vacuum oven. Sample preparation is detailed in chapter 8.1.1. This includes steps such as purification and degassing of styrene to produce a highly transparent polymer. The addition of fluors and polymerization initiators is also discussed in this chapter.

Chapter 8.1.2 details the laboratory setup for thermal polymerization including safety precautions that are applied due to the low explosive limit of styrene.

Chapter 8.1.3 focuses on the effects of different polymerization temperatures and initiator concentrations on the resulting polymer. For the production of high-performance plastic scintillators, the composition of the polymer matrix is vital. By changing the two parameters, the average chain length and therefore the stability, hardness and transparency of the scintillator are influenced. The optimal combination of temperature and initiator content is determined, while maintaining a practical polymerization time.

#### 8.1.1 Sample Preparation

As the optimal performance of any scintillator depends on the use of a solvent with high transmission for the scintillation light, the styrene undergoes several steps of

preparation before polymerization. The styrene used in this thesis is supplied by *Sigma Aldrich*. It is stabilized with 4-tert-butylcatechol to prevent premature polymerization during storage.

The first preparation step involves the removal of the stabilizer and potential impurities from the styrene through aluminum oxide chromatography. Due to polar interactions and the stationary phase, separation of the medium occurs. While the non-polar styrene passes rapidly through the column, polar impurities and the stabilizer are retained in the upper layers. This procedure employs a 20 mm x 400 mm column filled with active, basic aluminum oxide (mean pore size 9 nm, *Millipore*). The styrene (~200 ml per column) is introduced from a reservoir flask from which it continuously flows through the column. The purified styrene is then filtered to remove residual aluminum oxide and collected in a flask. A photograph of the laboratory setup is shown in figure 8.1.

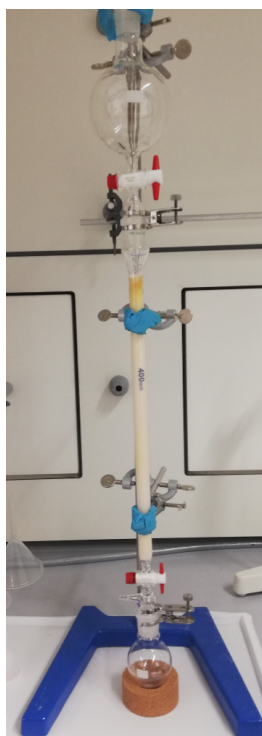


Figure 8.1: Laboratory setup for the purification of styrene

The next step involves the removal of dissolved oxygen from the styrene. This is essential for the preservation of the fluors added in subsequent stages, as oxidation of fluors in oxygen-rich environments leads to degradation of their phenyl rings, resulting in reduced scintillator performance. Additionally, dissolved gases can escape during polymerization, causing bubble formation in the solidified scintillator.

Degassing can be accomplished using two different techniques: nitrogen flushing and ultra sonic degassing. Nitrogen flushing displaces dissolved oxygen by creating buoyancy.

|                  |                                                           |
|------------------|-----------------------------------------------------------|
| Name             | Benzoyl Peroxide (BPO)                                    |
| Chemical Formula | $C_{14}H_{10}O_4$                                         |
| Density          | $1.33 \text{ g/cm}^3$                                     |
| Molar Mass       | $242.23 \text{ g/mol}$                                    |
| Melting Point    | $70^\circ\text{C}$                                        |
| Hazards          | Explosive, flammable, health hazard, environmental hazard |

Table 8.1: Chemical and physical properties of BPO [122]

The disadvantage of this method is an increased contamination risk, since it requires direct insertion of a needle and gas stream into the sample.

In contrast, ultra sonic degassing avoids direct contact with the styrene. The beaker containing the styrene is partially immersed in an ultrasonic bath. The sonication promotes the formation and release of gas which was previously dissolved in the liquid. Due to the cleanliness and efficiency of this method, it is applied to all styrene samples in this thesis.

During the final stage of ultra sonic degassing, additives are solved into the styrene. This includes different fluors (see chapter 3.2) or the polymerization initiator benzoyl peroxide (BPO, see table 8.1). Sonication leads to the disturbance of the mixture, which encourages the mixing and dissolution of these additives.

Following these preparation steps, polymerization is initiated by heating the styrene in a vacuum oven. The polymerization setup and procedure are described in detail in the following chapter.

### 8.1.2 Polymerization Setup and Procedure

The thermal polymerization of styrene requires strict safety precautions due to the volatility, flammability and toxicity of the substance. Accumulation of styrene vapor in closed volumes, especially in the presence of oxygen, must be minimized and monitored to prevent the formation of explosive mixtures. Also, isolation of sensitive equipment from the styrene vapors is essential to prevent potential damage.

These safety requirements are fulfilled by polymerizing the styrene in a vacuum oven manufactured by *Bender*. The internal pressure of the oven is regulated using a type PC 3000 vacuum pump by *vacuubrand* and monitored via an integrated pressure gauge. This configuration enables complete and controlled gas exchange from the oven chamber, facilitating the removal of oxygen prior to polymerization and the safe extraction of styrene vapors following the procedure.

A cold trap is installed between the oven and the vacuum pump, where the harmful styrene vapors condense in order to avoid damaging the vacuum pump. The oven chamber can also be filled with nitrogen through a dedicated gas inlet. The entire setup for plastic scintillator polymerization is displayed in figures 8.2 and 8.3. Temperature profiles for the entire polymerization process are programmed directly via the oven's control interface.

Following sample preparation, the styrene solution is transferred into a mold. During



Figure 8.2: Vacuum oven used for the polymerization of plastic scintillators. The upper compartment houses the oven chamber, while the lower compartment contains the cold trap and the vacuum pump.



Figure 8.3: Cold trap (left) and vacuum pump (right) located in the lower, ventilated compartment of the polymerization setup.

this thesis, polymerization is carried out in laboratory glass beakers. The filled molds are placed inside the vacuum oven and covered with watch glasses to minimize evaporation.

The pressure within the oven chamber is reduced to approximately 40 mbar, minimizing the oxygen content and removing residual dissolved gases from the styrene. After 10 min,

the oven is filled with nitrogen to prevent the formation of an explosive atmosphere during polymerization. Once normal pressure is restored, the polymerization reaction is initiated by heating the oven.

Once polymerization is complete, the oven chamber is evacuated and the styrene vapors are collected in the cold trap. The resulting polymerized scintillator is extracted from the glass mold by immersing it in water at room temperature while it is still warm. The thermal expansion of the polymer causes the glass to detach from the scintillator. Examples of polystyrene-based scintillators that were produced using this setup, each containing different fluors, are shown in 8.4.

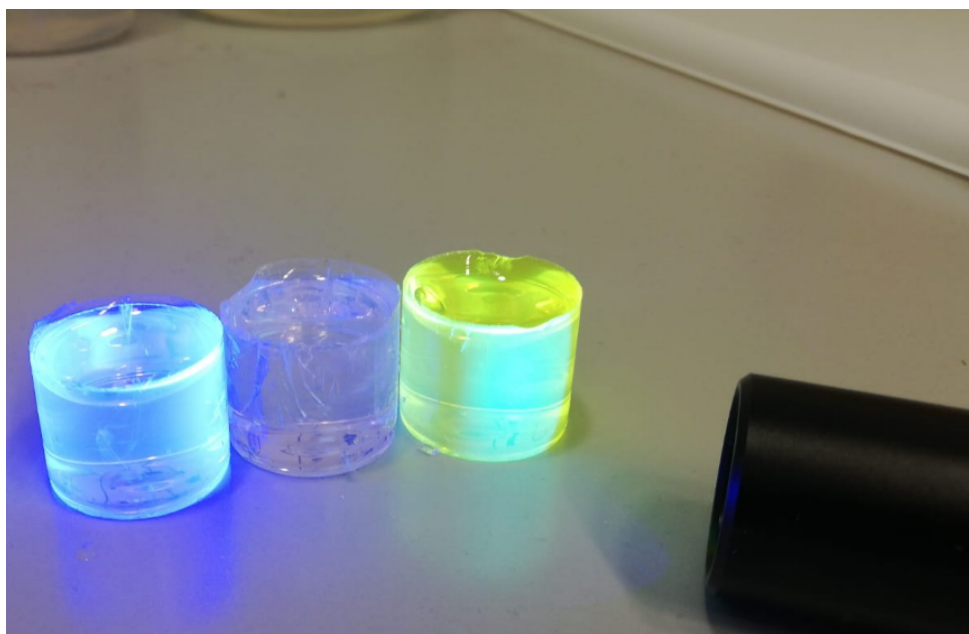


Figure 8.4: Polystyrene-based plastic scintillators with different fluors (L-R: Bis-MSB, PPO, BPEA) are excited by a UV-lamp causing the emission of their characteristic fluorescent light.

This concludes the description of the thermal polymerization of plastic scintillators. Post-processing steps, including sanding to achieve parallel surfaces and polishing to enhance light transmission through the interfaces, are performed separately. A discussion of the ideal polymerization parameters is given in the following chapter.

### 8.1.3 Polymerization Temperature and Initiator

When determining optimal polymerization parameters, temperature plays a decisive role in the duration of the process and the quality of the resulting polymer. In general, higher temperatures lead to a higher polymerization rate, which is necessary to produce plastic scintillators in a reasonable time. However, high temperatures can also lead to favorable conditions for bubble formation. Polymerization is an exothermic reaction which leads to an increase in temperature in the sample. While small-volume samples can polymerize evenly without forming bubbles, larger samples tend to show lower heat

dissipation which can lead to local heat accumulation where styrene is vaporized at a high rate. If the polymerization process is already advanced in the vicinity, increasing the viscosity, this vapor can encapsulate as a bubble in the final product. As these bubbles lead to a disturbance of light transmission within the polymer, they need to be avoided. If small bubbles form close to the surface of the scintillator, they can be sanded off, while bubbles in the center of the polymer can render it useless. Figure 8.5 shows three polystyrene samples of different sizes polymerized under the same conditions, demonstrating that the number of bubbles increases with sample size. For the application as a light guide in the SiALG design, 50 ml beakers are utilized as a mold to produce cylindrical scintillators with a diameter of 32-34 mm and a height of 30 mm.



Figure 8.5: Three identically produced polystyrene samples show the dependency of bubble formation on the volume of the individual samples

Excessive heat can also cause a lower mean polymer chain length as the termination process is favored due to a higher number of radicals available. This results in a more fragile scintillator. To achieve a balance in short polymerization time, low bubble formation and high stability, several parameters were varied in initial tests.

Full polymerization was achieved within 3 days at a temperature of 100 °C. An increase to 130 °C in the final hour of the process ensured full polymerization without introducing aging effects such as yellowing of the scintillator. Samples prepared under these conditions, however, often exhibited bubbles distributed in the central and lower regions.

Several attempts were made to consistently produce bubble-free polystyrene samples:

- Polymerization temperature variations in the range of 90 – 130 °C appeared to have no impact on the probability of bubble formation. Temperatures below 90 °C could not achieve full polymerization within 5 days.
- Increasing the temperature over the course of 24 h from room temperature to the

intended polymerization temperature could not consistently provide bubble-free samples.

- Reducing the pressure in the oven chamber to 500 mbar during polymerization had no apparent influence on bubble formation.

The addition of the polymerization initiator benzoyl peroxide (BPO) allowed for a temperature reduction to 75 °C while achieving full polymerization within 48 h. This is possible due to the increased availability of radicals that originate from the decomposition of BPO. Figure 8.6 shows the results of styrene polymerization with varying weight fractions of BPO at otherwise identical conditions.



Figure 8.6: Plastic scintillator samples with different weight fractions of BPO added as an polymerization initiator (L-R: 0.5 wt.%, 1 wt.%, 0.1 wt.%).

The sample with 0.1 wt.% of BPO shows small bubbles and did not fully polymerize within 48 h. The sample containing 1 wt.% of BPO presents cracks from the extraction of the polymer from the mold. This is a clear indication that the amount of BPO is too high. Due to an over-abundance of radicals during the polymerization process the mean polymer chain length is reduced, affecting the mechanical stability. The sample with 0.5 wt.% shows the best polymerization result as there are no visible cracks, discolorations or bubbles in the sample. Therefore, all plastic scintillators that were produced following these results use 0.5 wt.% of BPO. Although bubble encapsulation occasionally occurred at a BPO concentration of 0.5 wt.% near the bottom of the polymer, these imperfections were easily removed by sanding, resulting in only a few millimeters of material loss.

To assess the impact of BPO on the absorbance of the resulting polymer, comparative measurements were conducted with the UV-Vis spectrometer (detailed in chapter 4.1) on a sample that was polymerized with 0.5 wt.% of BPO at 75 °C and another sample, that was polymerized without BPO at 100 °C. Both scintillators were cylindrical, 20 mm in height, with sanded and polished surfaces. Figure 8.7 shows the raw absorption spectra of both polystyrene samples.



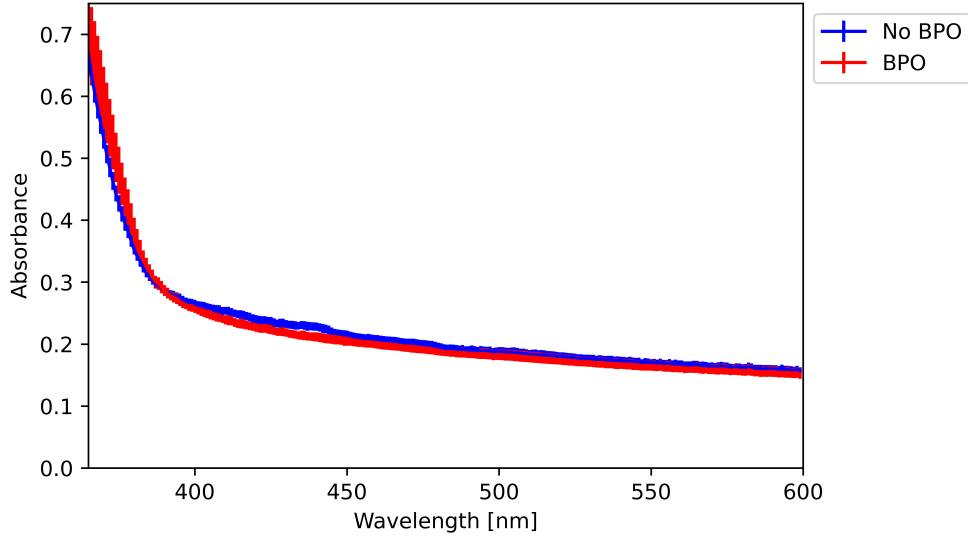


Figure 8.7: Absorbance spectra of polystyrene with and without the the addition of BPO as a polymerization initiator

In the 400–450 nm region, the sample without BPO exhibits higher absorbance than the one polymerized with the initiator. Since this wavelength range corresponds to the maximum emission of bis-MSB, which is intended as an additive in SiALG light guides, high transmission in this region is crucial for the scintillator’s performance. The measurements indicate a small improvement in scintillator transparency for  $\lambda > 400$  nm when BPO is used as a polymerization initiator. This is likely due to a narrow chain length distribution caused by a more controlled polymerization reaction due to the addition of BPO. Polymerization without an initiator tends to yield a broader chain length distribution as the availability of radicals for the termination process can vary locally. This can cause inhomogeneities in the refractive index of the polymer which leads to increased light scattering.

Following the identification of a stable polymerization method, the next chapter presents the characterization of these custom-made scintillators.



## 8.2 Plastic Scintillator Characterization

Plastic scintillators are a vital part of the SiALG design, and their performance is determined by a combination of optical, chemical, and scintillation properties. A thorough characterization is therefore essential to assess whether the lab-manufactured materials meet the requirements for efficient light collection and wavelength shifting in large-scale detector environments. This chapter presents a systematic study of the key properties of scintillator performance: fluor integration, optical transparency, and scintillation light yield.

The results provide a comprehensive understanding of how the material composition affects the optical behaviour of the scintillators and how the in-house-produced samples compare to a commercial reference. Together, these measurements form the basis for evaluating the suitability of the developed plastic scintillators for integration into the SiALG detector design.

### 8.2.1 Fluor Integration

The designated area of application of SiALGs is in liquid scintillator detectors that use PPO or a compound with similar emission characteristics as a primary fluor. The active light guides include bis-MSB as it efficiently absorbs the light emitted from the liquid scintillator and shifts it to higher wavelengths with a maximum at  $\lambda = 420$  nm. Polystyrene exhibits greater transparency at longer wavelengths, and the SiPM shows its highest sensitivity within the bis-MSB emission range. Consequently, bis-MSB is well suited as the wavelength-shifting fluor for this application. This mechanism is analogue to the working principle of WOMs which is detailed in figure 5.7.

Since the fluor is dissolved into the styrene and is therefore exposed to the high temperatures that are necessary for the polymerization process the fluorescent properties of the resulting plastic scintillator needs to be studied. In this chapter, two polystyrene-based scintillators with different bis-MSB concentrations (0.0001 wt% and 0.005 wt%) are measured with the fluorospectrometer (detailed in chapter 4.2). Figures 8.8 and 8.9 show the resulting emission–excitation maps for both samples.

The emission–excitation maps for the two polystyrene-based scintillators show a clear dependency of the optical response on the concentration of the fluor. Increasing the bis-MSB concentration results in a substantial enhancement of the overall emission intensity. The maximum signal more than doubles when the concentration is raised from 0.0001 wt% to 0.005 wt%. The higher concentrated sample exhibits a broader and more pronounced emission region, indicating a more complete population of bis-MSB molecules within the scintillator. Despite the changes in magnitude and spatial distribution, the position of the emission maximum remains centered around 420–440 nm for both samples. This proves that the fluorescence spectrum of bis-MSB itself is not significantly altered within this range by higher concentrations or increased temperatures during polymerization. The excitation profiles intensify with increasing bis-MSB content, demonstrating more efficient energy transfer from PPO to the secondary fluor. Overall, these results confirm that higher bis-MSB concentrations improve the scin-

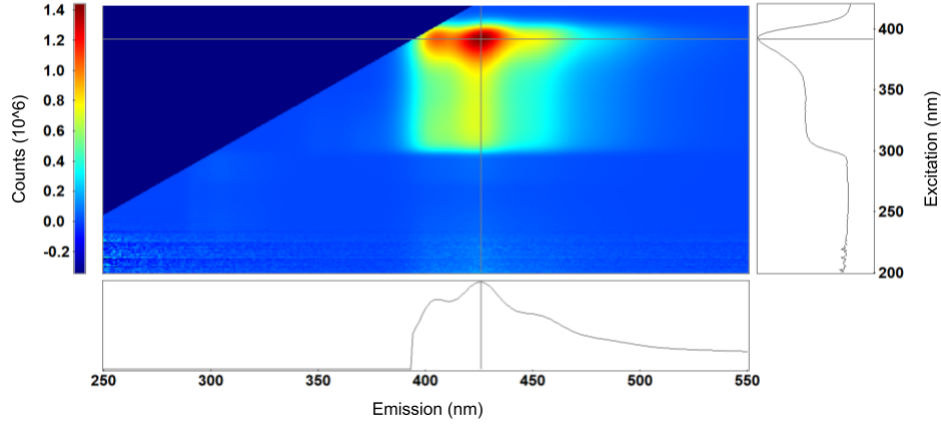


Figure 8.8: Emission–excitation map of a polystyrene-based scintillator containing 0.0001 wt% of bis-MSB

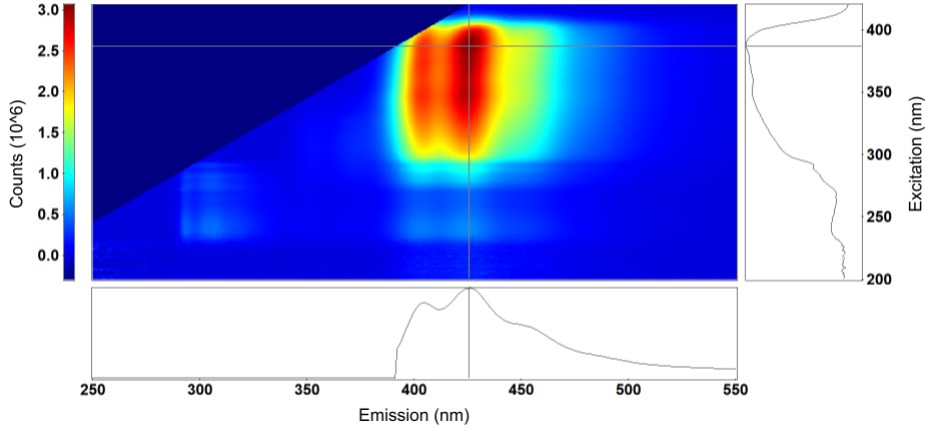


Figure 8.9: Emission–excitation map of a polystyrene-based scintillator containing 0.005 wt% of bis-MSB

tillation light yield and increase the energy-transfer efficiency, while maintaining the emission wavelength characteristic of bis-MSB.

### 8.2.2 Optical Transparency

This chapter presents the optical characterization of the lab-manufactured plastic scintillators using UV–Vis absorption spectroscopy, as described in detail in chapter 4.1. Since the optical transparency of a scintillator directly affects its light-transport efficiency and therefore its overall light yield, UV–Vis measurements provide essential insight into the material’s quality and its suitability for application in the SiALG detector design. In particular, the absorption spectrum is highly sensitive to residual monomers, chemical impurities, or scattering centers introduced during synthesis,

making it a powerful tool for evaluating the success of the fabrication process.

To establish a reference for the lab-manufactured samples, the commercially available plastic scintillator SP32 from *Nuviotech Instruments* was measured under identical experimental conditions. Additionally, an independent absorption measurement of SP32 from literature [123] was included to validate the performance of the UV–Vis spectrometer used in this work.

The SP32 scintillator consists of a polystyrene matrix doped with p-terphenyl and POPOP as the primary and secondary fluors [108]. Although these fluors differ from the PPO and bis-MSB used in the lab-manufactured scintillators, the underlying polystyrene base is identical and therefore directly comparable. Since all fluor-related absorption occurs below approximately 425 nm, the transparency of the polymer matrix itself can be compared reliably at wavelengths above this value. It is assumed that any remaining absorption above 425 nm originates from surface reflections, micro-irregularities, or intrinsic polymer coloration rather than from the fluors.

All scintillators measured in this chapter (lab-manufactured samples and the SP32 reference) were prepared with a thickness of 1 inch (25.4 mm). In contrast, the literature measurement of SP32 was performed on a significantly thinner 6 mm sample. To minimize surface-induced scattering, all lab-manufactured scintillator samples were sanded and polished prior to measurement. The surface of the SP32 sample was processed by the manufacturer. The resulting transmission spectra are shown in figure 8.10.

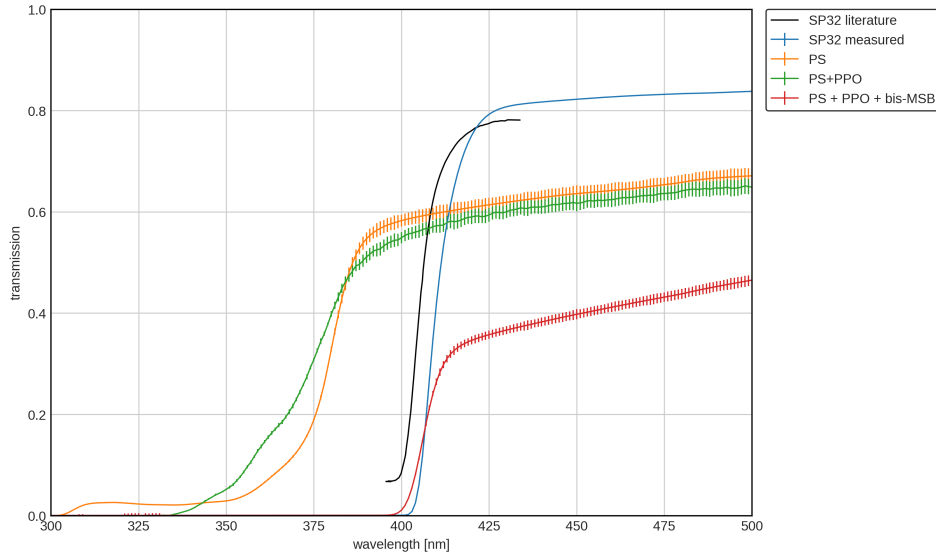


Figure 8.10: Transmission spectra of lab-manufactured plastic scintillators PS, PS+PPO and PS+PPO+bis-MSB and the commercial plastic scintillator SP32

Overall, the transmission measurements clearly reflect the fluor composition of each scintillator. The fluor-free PS sample represents the behaviour of pure polystyrene, which absorbs strongly below 300 nm and exhibits a steep rise in transmission beginning

| Scintillator Sample      | T(425 nm) | T/T(SP32) |
|--------------------------|-----------|-----------|
| SP32 literature (6 mm)   | 0.776     |           |
| SP32 measured (25.4 mm)  | 0.793     | 1         |
| PS (25.4 mm)             | 0.614     | 0.774     |
| PS+PPO (25.4 mm)         | 0.594     | 0.750     |
| PS+PPO+bis-MSB (25.4 mm) | 0.357     | 0.450     |

Table 8.2: Transmission comparison of different plastic scintillators for  $\lambda=425$  nm

at 350 nm, reaching a plateau at approximately 390 nm. The PS+PPO sample contains the primary fluor PPO, whose characteristic absorption band extends up to about 335 nm. Above this wavelength, the transmission increases until it levels off near 390 nm, similar to pure PS.

In the PS+PPO+bis-MSB and SP32 samples, the presence of secondary fluors broadens the absorption region further. Complete absorption extends up to roughly 400 nm, followed by a sharp rise in transmission. The plateau is reached at around 415 nm for PS+PPO+bis-MSB and between 420–425 nm for SP32, which is consistent with the known absorption characteristics of their respective fluor systems.

Since the plateau of these measurements is of main interest to assess the quality of the polystyrene, the transmission is compared at  $\lambda=425$  nm in table 8.2.

Despite the substantial difference in thickness between the two SP32 samples, the transmission for  $\lambda=425$  nm differs by only 1.7 %. Interestingly, the thicker SP32 sample measured in this work exhibits slightly higher transmission than the measurement taken of the thinner sample from literature. Since polystyrene is highly transparent in the visible wavelength range, the influence of sample thickness on the transmission above 425 nm is expected to be minimal. In contrast, differences in surface preparation, that are not detailed for both SP32 samples, are a more likely explanation for the observed deviation. Unlike the SP32 measurement presented here, the literature data show incomplete absorption below 400 nm. Although this discrepancy may be attributed to the thinner sample, the corresponding spectral feature is not discussed in the original publication. Overall, the close agreement between the two measurements indicates that the polystyrene base of SP32 possesses high optical quality and further validates the reliability of the UV–Vis spectrometer used in this study.

In general, the lab-manufactured plastic scintillators exhibit lower transmission than the SP32 standard, indicating that the optical quality of the underlying polymer matrix does not yet match that of the commercial material. At  $\lambda = 425$  nm, the PS and PS+PPO samples reach 77.4 % and 75.0 % of the transmission of SP32, respectively, while the fully doped PS+PPO+bis-MSB sample achieves only 45.0 % of the SP32 transmission. These results suggest that the polymerization process used for the lab-manufactured scintillators introduces additional absorption or scattering losses.

The reduced transparency may originate from several factors, including incomplete polymerization, residual monomers, microscopic inhomogeneities, or surface imperfections that were not fully removed during polishing. Since all samples share the

same fluor-independent absorption region above 425 nm, the differences observed at this wavelength primarily reflect the intrinsic optical quality of the polystyrene matrix rather than the influence of the dopants.

The particularly poor performance of the PS+PPO+bis-MSB sample, despite being polymerized under the same conditions as the other lab-manufactured scintillators, indicates that a higher fluor loading could affect the polymerization dynamics. The increased concentration can interfere with chain growth or reduce polymerization efficiency leading to increased scattering. Additionally, the radical polymerization of styrene is inherently stochastic, and factors such as local temperature gradients and the random formation of micro-inhomogeneities can introduce noticeable sample-to-sample variations, even when all external production conditions are kept identical. While this aspect can be further investigated by producing more scintillator samples with identical content, due to the constraints of this thesis, further research was not conducted.

A comparison of measurement uncertainties reveals that the lab-manufactured scintillators exhibit higher uncertainty than the SP32 scintillator. The uncertainty was calculated by applying the standard deviation to three repeated measurements of the same sample, with the sample being removed and reinserted into the sample holder between each measurement. This suggests that the polymer structure of the SP32 scintillator is more homogeneous than that of the lab-manufactured samples. Additionally, surface treatment of the samples may introduce variability, contributing to deviations in repeated measurements of the same sample.

In summary, while the spectral shapes of the lab-manufactured scintillators confirm the correct incorporation of the intended fluors, the lower transmission relative to SP32 indicates that further optimization of the polymer synthesis is required to achieve a polymer quality comparable to commercial standards.

### 8.2.3 Light Yield

This chapter presents the light yield analysis of a polystyrene-based scintillator that was manufactured with the polymerization setup presented in chapter 8.1. Using the experimental setup and procedures described in chapter 4.3, the goal is to quantify how efficiently the material converts deposited radiation energy into optical photons. The plastic scintillator under study contains 0.005 wt% bis-MSB and 0.22 wt% PPO. Since the SiALG light guides are intended to operate with PPO-based liquid-scintillator systems, it is not necessary for the light guides themselves to contain PPO; the excitation energy is transferred directly from the PPO in the liquid scintillator to the bis-MSB in the light guides. However, during light yield measurements, polystyrene molecules are primarily excited. As the energy transfer from polystyrene to bis-MSB is inefficient, PPO is incorporated into the measured light guide to provide an effective intermediate step in the energy-transfer chain, ensuring that excitation energy is efficiently passed on to the bis-MSB.

The performance of the tested scintillator is compared to the commercially available polystyrene-based scintillator SP32 by *Nuviatech Instruments* with a documented light yield of  $8750 \frac{\text{photons}}{\text{MeV}}$  [124] [123].

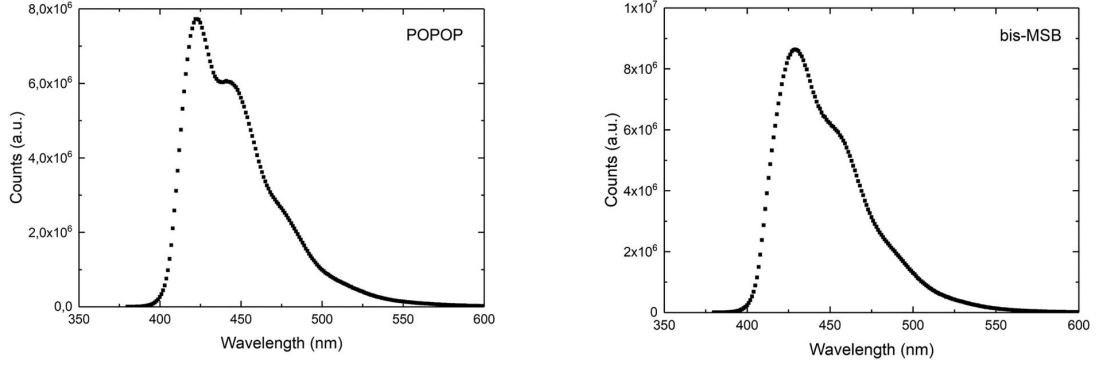


Figure 8.11: Fluorescent spectra of polystyrene-based plastic scintillators containing POPOP (left) and bis-MSB (right) [111].

Unlike the scintillator under study, this reference scintillator uses p-terphenyl as the primary fluor and POPOP as the secondary fluor [108]. However, as the emission spectrum of POPOP is sufficiently similar to that of bis-MSB (see 8.11), a comparable detection efficiency of the PMTs is assumed.

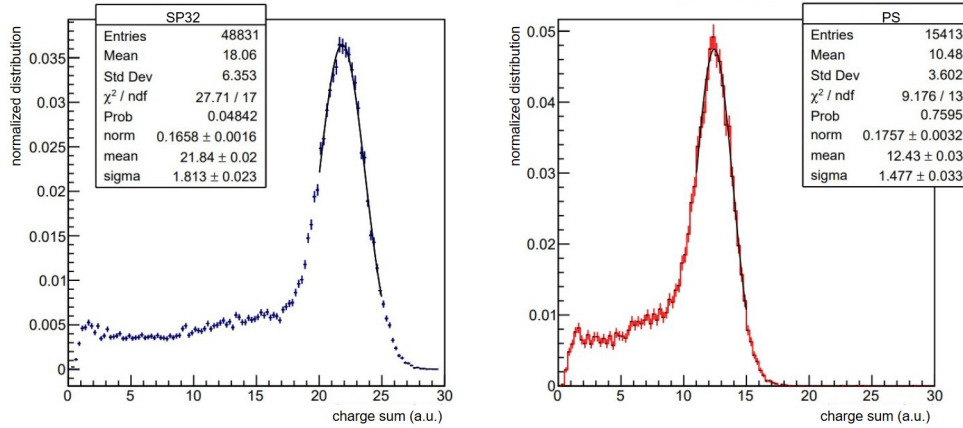


Figure 8.12: Gamma spectra of the SP32 scintillator and a lab-manufactured scintillator with corresponding Gaussian fits

The resulting gamma spectra recorded with both plastic scintillators are displayed in figure 8.12. The backscatter peaks that represent the fixed energy events are fitted with a Gaussian. While the Compton background from accidental coincidences is included in these fits resulting in a slightly increased mean charge sum for both scintillators, a subtraction of the background would shift both peaks downward by the same fraction, leaving the relative light yield unaffected.

These measurements lead to the conclusion that the plastic scintillator that was manufactured in the polymerization setup achieves a light yield of  $\sim 57\%$  relative to the SP32 scintillator resulting in  $4980 \frac{\text{photons}}{\text{MeV}}$ .

Although the exact fluor concentration of the SP32 scintillator is not disclosed, com-

mercially available plastic scintillators generally contain fluor loadings that are higher by a factor of 5–10 compared to the lab-manufactured scintillator, which accounts for its reduced light yield [125] [126] [127]. Additionally, the UV–Vis measurements presented in chapter 8.2.2 demonstrate that the polymer used in SP32 exhibits higher light transmission than the in-house-produced plastic scintillators. Since optical transparency directly influences the achievable light yield, this difference must be considered in the overall performance assessment.

### 8.3 Summary

This chapter presented the complete development and characterization of polystyrene-based plastic scintillators produced for use as active light guides in the SiALG design. Beginning with the establishment of a reliable small-scale polymerization method, this work demonstrated that high-quality scintillators can be fabricated in a laboratory environment when appropriate purification, degassing, and controlled thermal curing procedures are applied. The optimization of polymerization temperature and initiator concentration, particularly the identification of 0.5 wt% BPO as an effective initiator, proved essential for achieving mechanically stable, bubble-free, and optically transparent polymers. In practice, a polymerization temperature of 75 °C was selected enabling full polymerization within 48 h.

The subsequent characterization of the scintillators provided a detailed assessment of their optical and scintillation properties. Fluorescence measurements confirmed the successful integration of bis-MSB and demonstrated that its emission characteristics remain stable after polymerization. Additionally, these measurements also showed that higher bis-MSB concentrations lead to more intense fluorescence and provide more efficient energy-transfer.

UV–Vis absorption spectroscopy revealed that, while the spectral shapes of the lab-manufactured scintillators match expectations based on their fluor composition, their overall transparency is lower than that of the commercial SP32 reference. While the fluor-free polystyrene sample reached 77.4 % of the reference transmission and the sample containing PPO achieved 75.0 %, the scintillator doped with both PPO and bis-MSB attained only 45.0 %. These results indicate that further refinement of the polymerization process is required in order to achieve transparencies comparable to commercial scintillators. Differences in surface treatment may also contribute to the reduced transparency, since the reference sample was finished by the manufacturer, whereas the lab-produced scintillators were sanded and polished manually. It remains unclear whether the particularly low transmission of the fully doped scintillator is caused by the higher fluor concentration or whether it represents an outlier arising from the stochastic nature of radical polymerization. Resolving this question will require additional systematic testing beyond the scope of this work.

Finally, the light yield measurements demonstrated that the lab-manufactured scintillator achieves approximately 57 % of the light output of SP32. This result is compatible with the lower fluor loading and reduced optical transparency observed in the UV–Vis measurements. While the achieved light yield is sufficient for proof-of-concept studies, it underlines the importance of improving both polymer quality and fluor incorporation to reach commercial-grade performance.

Overall, the results of this chapter show that the laboratory-produced scintillators successfully capture the essential behaviour of commercial materials but still fall short in optical quality and light yield. The findings provide a clear roadmap for future improvements, including optimization of polymerization conditions and the production of scintillators with a higher fluor loading. These developments are crucial for realizing high-performance SiALG light guides suitable for large-scale detector applications.



The complete SiALG concept was evaluated by [REDACTED] at the Eberhard Karls Universität Tübingen, and the corresponding results were published in his dissertation [107]. For his work, several inactive light guides made of pure polystyrene and active light guides doped with bis-MSB were supplied for further investigation. The workshop in Tübingen machined the cylindrical light guides into the required conical geometry for the SiALG. The tested SiPMs achieved time resolutions between 0.1 and 0.6 ns, a performance level that is essential for distinguishing scintillation light from Cherenkov light. While having only a minimal impact on the SiPM timing response, incorporating both active (ALC) and inactive light guides (PLC) increased the number of detected photons as presented in figure 8.13. The figure also indicates that SiALGs equipped with active light guides produce higher signal levels than those with inactive guides. This behavior aligns with the additional wavelength shifting provided by bis-MSB, which moves the emitted light into a spectral region where polystyrene is more transparent.

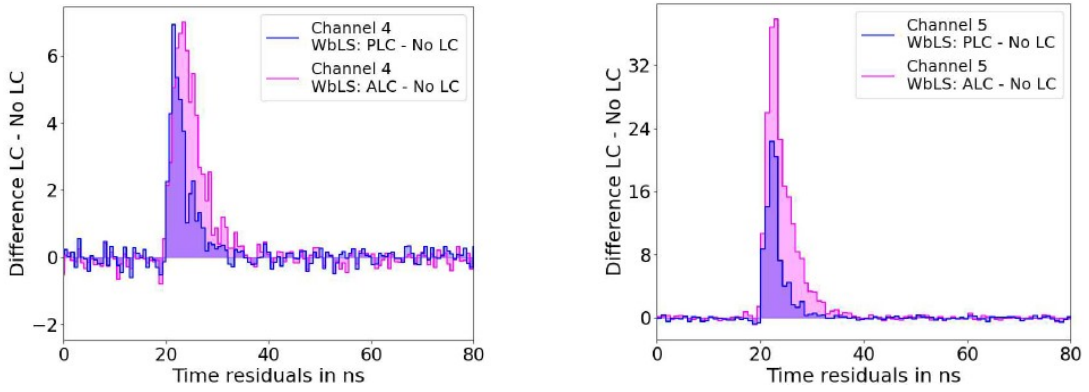


Figure 8.13: Time-dependent differences in signal intensity between SiALGs with active light guide containing bis-MSB (ALC) and inactive light guide (PLC) compared to a SiPM without light guide (No LC). These plots were calculated from time residual distributions normalized to their background level. The different versions of the photo sensor were integrated in a water-based liquid scintillator detector with the fluor PPO [107].

The enhanced signal obtained with the SiALG leads to an improved effective signal-to-noise ratio through increased photon collection efficiency. As the signal amplitude increases while the dark count rate remains unchanged, the relative impact of dark noise is reduced, allowing more reliable temporal discrimination between genuine events and uncorrelated noise. This improvement is particularly beneficial for low-rate experiments searching for neutrinos or dark matter interactions.

Altogether, this study successfully demonstrates the feasibility of the SiALG concept.

## 9 Conclusion and Outlook

*"I could cry because it's over  
Think i'll smile because it happened"*

— Soulkeeper, *Smile because it happened*

The work presented in this thesis was motivated by the demanding performance requirements of next-generation experiments searching for physics beyond the Standard Model. Rare-event searches and high-performance background rejection require detector systems that combine high efficiency, long-term stability, and precise optical performance over extended operational lifetimes. In scintillation-based detectors, light collection performance directly determines the detection efficiency. Enhancing photon collection is therefore not just a matter of optimization, but a central design challenge. This thesis addressed this challenge through two complementary approaches. The first investigated the enhancement of photon transport in the context of SHiP's Surrounding Background Tagger (SBT) by increasing the reflectivity of the inner surfaces. The second focused on optimization of photon collection at the sensor level through the development of polystyrene-based active light guides for the Silicon Photomultiplier with Active Light Guide (SiALG) concept. Together, these studies contribute to a deeper understanding of the interplay between optical properties, material stability, and detector geometry in scintillation systems.

### Reflective Coating Studies for the SHiP Surrounding Background Tagger

The SBT of the SHiP experiment is designed to achieve a detection efficiency of at least 99 % for minimum ionizing particles. Meeting this requirement places rigorous demands on scintillation light collection, particularly given the large detector volume and the need for uniform response. In this context, the BaSO<sub>4</sub>-based reflective coating OPRC, manufactured by Berghof Fluoroplastics, was evaluated as a potential solution to increase diffuse reflectivity of the inner detector walls.

BaSO<sub>4</sub> is known to act as an almost ideal diffuse reflector in the relevant wavelength range, with reflectivities of  $R \gtrsim 95\%$  around  $\lambda = 425\text{ nm}$  under laboratory conditions. Initial measurements confirmed the high diffuse reflectance of the coating in the visible and near-UV region, where the liquid scintillator consisting of LAB + PPO emits the majority of light. The application of the coating to detector prototypes was therefore expected to significantly increase the average photon path length and, consequently, the probability of photon detection by the wavelength-shifting optical modules (WOMs). However, systematic aging studies revealed that the reflectivity of the coating degrades when submerged in liquid scintillator. Laboratory measurements showed a reduction in reflectivity of 15–27 % at  $\lambda = 425\text{ nm}$  within an equivalent aging time of approximately

two years. This degradation was traced to two principal mechanisms: (1) absorption of scintillator into the coating, leading to swelling and reduced opacity, and (2) partial dissolution of coating constituents into the scintillator.

In addition, pronounced yellowing of the coating was observed when OPRC was applied directly to the corten steel from which the SBT vessel is constructed. Samples exhibiting discoloration showed a reflectivity decrease to approximately 80 % of the standard value at  $\lambda = 425$  nm, and prolonged contact with liquid scintillator reduced this value further to approximately 60 %. The discoloration was attributed to corrosion processes at the steel surface, induced by the water content of the coating, which led to the formation of rust layers and oxidized particles that were subsequently incorporated into the coating matrix. The use of an anti-rust primer successfully mitigated staining; however, the chemical interaction between coating and scintillator remained problematic.

The influence of coating degradation on scintillator quality was also quantified. Over a two-month test beam campaign with the four-cell prototype, an increase in scintillator absorbance of  $\Delta A = 0.01518$  was measured, nearly matching the absorbance reduction achieved through initial scintillator purification. This was attributed to coating dissolution. Furthermore, in laboratory tests, oxidation of the fluors was identified as an additional degradation channel, which was significantly accelerated upon contact with rusted corten steel. These tests also showed that the addition of 0.001 % Tocopherol effectively suppressed fluor oxidation, while degassing and nitrogen storage proved essential preventive measures.

Two large-scale detector prototypes were constructed and tested in beam campaigns at CERN and DESY. In the 1-cell prototype, a reflectivity of approximately 65 % was achieved during beam operation, which was lower than the values measured under laboratory conditions. This was attributed to prolonged drying times of the acrylic primer during detector application, which promoted oxidation of the corten steel and resulted in staining of the coating. Despite these limitations, the detector achieved an efficiency of  $\geq 99.5$  % at a confidence level of 68 % for positrons with energies between 1.4 GeV and 5.4 GeV, provided that at least one of the two WOMs registered a signal. The initial coating discoloration was prevented in the 4-cell prototype by increasing ventilation and applying an anti-rust primer. However, prolonged contact between the coating and the scintillator during the test beam campaign led to the appearance of rust stains in the coating.

While these results demonstrate that high detection efficiencies are achievable, the long-term degradation of reflectivity and the practical challenges of large-scale coating application led to the conclusion that OPRC is not a viable long-term solution for SHiP's SBT. The experiment is designed for data-taking periods of at least 15 years. The reflectivity loss over extended time scales would inevitably reduce photon statistics and compromise detection efficiency. Moreover, the coating process itself is labor-intensive, requiring extended drying times, careful ventilation, and meticulous surface preparation.

Following the completion of this thesis, an alternative design concept employing aluminum as the structural material for the SBT was identified. Detector configurations using polished AlMg4.5 demonstrated enhanced reflectivity and improved response

uniformity in a subsequent test beam campaign. Although these investigations were conducted beyond the scope of the present work, the results obtained in this thesis provided essential input to the design development of the SBT and supported the transition toward mechanically robust and chemically stable solutions.

## **Development of Polystyrene-Based Scintillators for the SiALG Concept**

The second major research focus of this thesis addressed photon detection efficiency at the sensor level. Within a collaboration with the University of Tübingen, the SiALG concept was developed. The SiALG integrates a polystyrene-based plastic scintillator directly coupled to a SiPM, thereby combining photon collection, wavelength shifting, and detection within a compact architecture. By increasing the angular acceptance for incident photons, the active light guide enhances the overall detection probability in liquid scintillator detectors, thereby reducing the number of required photo sensors. Furthermore, the enhanced photon collection efficiency improves the signal-to-noise ratio, which is particularly advantageous for low-rate detectors employed in neutrino and dark matter searches.

A laboratory-scale polymerization method for polystyrene-based scintillators was established and optimized. Through the addition of benzoyl peroxide and a polymerization temperature of 75°C, suitable conditions were identified for achieving mechanically stable, bubble-free, and optically transparent samples. Fluorescence measurements confirmed successful incorporation of bis-MSB and demonstrated enhanced fluorescence intensity for increased bis-MSB concentrations.

UV-Vis absorption spectroscopy revealed that the spectral shapes of lab-produced samples were consistent with expectations based on fluor composition. However, the measured transmission values remained below those of a commercial scintillator, reaching only 45.0 %-77.4 % of its value. The reduced transparency and inconsistent results between the lab-produced scintillators were attributed to surface finishing differences and potential residual polymerization imperfections, indicating the need for further refinement of the polymerization process.

Light yield measurements showed that the lab-produced scintillators achieved approximately 57 % of the light output of commercial SP32 scintillator. Although sufficient for proof-of-concept validation, this result highlights the importance of improved polymer purity, optimized fluor concentrations, and enhanced optical finishing to reach commercial-grade performance.

Integration tests of the lab-manufactured active- and inactive light guides confirmed the central advantage of the SiALG concept. SiPMs achieved time resolutions between 0.1 ns and 0.6 ns, enabling discrimination between scintillation and Cherenkov light. Active light guides doped with bis-MSB increased the number of detected photons compared to inactive guides. Quantification of this improvement will require further research. This validates the principle of embedding fluors directly into the light guide to improve photon capture and transport efficiency.

Overall, the SiALG research demonstrates the technical feasibility of integrating active light guides with SiPMs. While further optimization is required to improve transparency and light yield of the light guides, the achieved performance establishes a clear path toward scalable, high-efficiency photon detection modules suitable for large liquid scintillator detectors.

## Final Remarks

The research presented in this thesis demonstrates that enhancing light collection in scintillation detectors requires a comprehensive consideration of optical performance, material compatibility, and long-term stability. Improvements in reflectivity or photon yield alone are insufficient if accompanied by chemical degradation, mechanical instability, or operational impracticalities. Instead, detector optimization must balance photon transport efficiency with durability under realistic experimental conditions.

The reflective coating studies provide a detailed analysis of the interplay between diffuse reflectivity, scintillator chemistry, and substrate interactions. While high initial reflectance values and successful proof-of-principle beam tests demonstrated the potential of BaSO<sub>4</sub>-based coatings, the observed long-term degradation mechanisms ultimately limited their suitability for sustained operation in SHiP's SBT. These results underscore the importance of extended aging studies and material compatibility tests prior to large-scale detector implementation.

In contrast, the SiALG investigations highlight the promise of material-level innovation and sensor-integrated light collection. The successful development of laboratory-scale polystyrene scintillators, combined with measurable performance gains from active light guides, demonstrates that targeted modifications at the photo sensor level can significantly enhance signal intensity. Although further improvements in optical transparency and light yield are required to match commercial standards, the results establish a solid foundation for continued development of integrated scintillation-SiPM systems.

Future detector concepts for rare-event searches will increasingly rely on precise control of photon propagation, efficient wavelength shifting, and robust long-term material stability. The findings of this thesis contribute to this objective by clarifying the limitations of certain approaches while identifying viable directions for further optimization. In doing so, this work supports the ongoing effort to refine scintillation detector technology for experiments operating at the frontiers of particle physics.



# List of Figures

|      |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |    |
|------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----|
| 2.1  | Overview of elementary particles according to the Standard Model [1].                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | 3  |
| 3.1  | Based on [16]: <b>Spatial orbital model of benzene ring</b> : left: electron distribution within the orbital model of the benzene ring; green dots: electrons of C-atoms; blue dots: electrons of H-atoms; <b>white orbitals: three sp<sup>2</sup> orbitals of each C-atom forming <math>\sigma</math>-bonds with two adjacent carbon-atoms and one hydrogen-atom</b> resulting in <b>planar structure of the benzene ring</b> (120° angle between each C-atom and each C-atom and H-atom); right: <b>continuous cloud of <math>\pi</math>-electron density evenly distributed above and below the ring plane</b> . . . . . | 7  |
| 3.2  | Jabłoński diagram: Schematic overview of molecular energy levels. The thick black lines represent spin states and the ionization energy while the grey lines refer to the vibrational sub-states. Possible transitions between energy levels are indicated by colored arrows [18]. . . . .                                                                                                                                                                                                                                                                                                                                  | 9  |
| 3.3  | Illustration of the Franck-Condon principle [16][13] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | 11 |
| 3.4  | Chemical structure of Linear Alkylbenzene [16] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | 12 |
| 3.5  | Absorption- and emission spectra of LAB (red), PPO (yellow) and bis-MSB (blue) [16] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               | 13 |
| 3.6  | Absorption spectrum of a purified LAB sample [29] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | 14 |
| 3.7  | Chemical structure of PPO [30] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | 14 |
| 3.8  | Chemical structure of Bis-MSB [41] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | 16 |
| 3.9  | Schematic depiction of the working principle of a PMT [44] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | 17 |
| 3.10 | The working principle of a SiPM [47] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | 18 |
| 4.1  | Screenshot of the <i>UV Winlab</i> software detailing the optical components and different settings in the spectrometer . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | 21 |
| 4.2  | Absorbance spectrum of a liquid scintillator sample in a 10 cm cuvette containing LAB and 2 g/l of PPO. The red curve shows the raw data as given by the spectrometer, while the black curve represents the corrected data. . . . .                                                                                                                                                                                                                                                                                                                                                                                         | 22 |
| 4.3  | Visualization of the FS5 spectrofluorometer including all components and the light path [56] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | 24 |
| 4.4  | Fluorescent spectrum of a liquid scintillator sample consisting of LAB and 2 g/l of PPO . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | 25 |
| 4.5  | Schematic overview of the light yield setup [57] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | 26 |
| 4.6  | Example of a gamma spectrum recorded with the light yield setup with a Gaussian distribution fitted to the backscatter peak . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | 27 |
| 4.7  | Liquid scintillators showing different degrees of oxidation [58] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | 29 |
| 4.8  | Illustration of the Huygens-Fresnel principle [59] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | 30 |
| 4.9  | Illustration of different types of reflectivity [61] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | 31 |

|      |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |    |
|------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----|
| 4.10 | SolidWorks model of the reflectivity setup (side view). 1: fiber holder (light source); 2: fiber holder (spectrometer); 3: turning table extension (spectrometer); 4: sample holder; 5: turning table (sample); 6: turning table (spectrometer); 7/8: stepper motors [58] . . . . .                                                                                                                                                                                                                                              | 31 |
| 4.11 | SolidWorks model of the reflectivity setup (top view). 1: fiber holder (light source); 2: fiber holder (spectrometer); 3: sample holder; 4: turning table (sample); 5: stepper motor holder [58] . . . . .                                                                                                                                                                                                                                                                                                                       | 32 |
| 4.12 | Spectrum of the deuterium light source SL3 used in the reflectivity setup                                                                                                                                                                                                                                                                                                                                                                                                                                                        | 32 |
| 4.13 | Reflectivity spectrum of the PTFE standard: The graph shows different grayscale of the diffuse reflector. The sample with the highest reflectivity is chosen as a reference for the reflectivity setup [62]. . . .                                                                                                                                                                                                                                                                                                               | 34 |
| 5.1  | Overview of the SHiP Experiment with the associated BDF implemented into the SPS ECN3 area [79]. . . . .                                                                                                                                                                                                                                                                                                                                                                                                                         | 38 |
| 5.2  | Illustration of the support structure of the SHiP decay vessel, providing a division into cells that is used for the SBT. . . . .                                                                                                                                                                                                                                                                                                                                                                                                | 39 |
| 5.3  | Illustration of a single detector cell of the SBT showing the position of the two WOMs. . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                  | 40 |
| 5.4  | <b>Left:</b> Laboratory setup for the purification of LAB. The column is filled with 1.6 kg of Aluminum oxide to purify 25 l of LAB, which was manually divided into batches and refilled by hand. A small vacuum pump is employed to increase the purification speed. <b>Right:</b> UV-vis absorbance spectra of raw and purified LAB measured in a 100 mm Suprasil glass cuvette. An improved absorbance is achieved, especially, in the region of scintillation light (360 nm-400 nm) by removing organic impurities. . . . . | 41 |
| 5.5  | LAB purification setup at the <i>Max Planck Institut für Kernphysik</i> in Heidelberg for the purification of 1000 l of LAB. Raw LAB is pumped from one IBC, through aluminum oxide columns into another IBC, where the purified LAB is collected. Two columns with 10 kg of Aluminum oxide each are sufficient to purify up to 350 l of scintillator. . . . .                                                                                                                                                                   | 42 |
| 5.6  | Sketch of the liquid handling system used for the SHiP test beam in 2022. A system of valves and pipes allows for filling and emptying of the prototype and circulation of the scintillator under a nitrogen atmosphere.                                                                                                                                                                                                                                                                                                         | 43 |
| 5.7  | <b>Left:</b> The working principle of a WOM. <b>Right:</b> Emission spectra of the liquid scintillator and the wavelength-shifting paint used on the WOMs containing bis-MSB. Also shown is the quantum efficiency of a photo multiplier and the photo detection efficiency of the SiPM. The overlap of the bis-MSB emission peak with a high photo detection efficiency of the SiPMs indicates good compatibility of the components [83]. . . . .                                                                               | 43 |
| 5.8  | Reflectivity spectrum of the reflective BaSO <sub>4</sub> coating OPRC provided by the manufacturer [84] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                               | 46 |
| 5.9  | Diffuse reflectivity spectrum of stainless steel [85] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | 46 |



|      |                                                                                                                                                                                                                                                                                                                                                               |    |
|------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----|
| 6.1  | Investigated samples in this chapter. From left to right: PTFE Standard, stainless steel, stainless steel coated with OPRC. . . . .                                                                                                                                                                                                                           | 48 |
| 6.2  | (a) Diffuse Reflectivity (diff) and (b) Specular reflectivity (spec) of unpolished stainless steel and the BaSO <sub>4</sub> -based reflective coating. The measurements are normalized to a PTFE sample. . . . .                                                                                                                                             | 49 |
| 6.3  | Sample of S355JO(J2/K2)W corten steel showing typical signs of surface oxidation . . . . .                                                                                                                                                                                                                                                                    | 50 |
| 6.4  | Dried result of first OPRC application on corten steel: The orange rust stains originate from oxidation of the corten steel surface during the drying of the reflective coating. . . . .                                                                                                                                                                      | 50 |
| 6.5  | Primer and coating application tests on corten steel: Each row uses a different primer, indicated by R1, R2 (anti-rust primer) and A1, A2 (acrylic primer). In each row, the two left samples are only primed, while the two right samples are primed and coated with OPRC. . . . .                                                                           | 52 |
| 6.6  | Relative diffuse reflectivity spectrum of four differently primed (acrylic/anti-rust) and coated corten steel samples, uncoated stainless steel and an unprimed coated corten steel with signs of oxidation. The wavelength-dependent reflectivity is measured relative to a BaSO <sub>4</sub> -standard with an angular configuration of (0° – 45°). . . . . | 53 |
| 6.7  | Photos of primed and coated corten steel samples before and after aging                                                                                                                                                                                                                                                                                       | 56 |
| 6.8  | Fotos of samples F and SSF before and after aging . . . . .                                                                                                                                                                                                                                                                                                   | 56 |
| 6.9  | After wrapping the samples in a paper towel to dry, the opaqueness of the coating is partly restored. Here shown is sample SSF. . . . .                                                                                                                                                                                                                       | 57 |
| 6.10 | The reflectivity spectrum of aged coating shows the typical double peak structure of PPO fluorescence, indicating that the fluor has been deposited in the coating. . . . .                                                                                                                                                                                   | 57 |
| 6.11 | Relative diffuse reflectivity spectra of all relevant aged samples with PMMA to block $\lambda < 390$ nm. The wavelength-dependent reflectivity is measured relative to a BaSO <sub>4</sub> -standard with an angular configuration of (0° – 45°). . . . .                                                                                                    | 58 |
| 6.12 | Absolute absorbance measurements of all aged scintillator samples from this aging test . . . . .                                                                                                                                                                                                                                                              | 59 |
| 6.13 | Absorbance change of aged scintillators compared to the "start" sample. This plot displays all aging samples with different primers and primer/coating-combinations. AgedRef is displayed to quantify aging that takes place without a reflective sample. . . . .                                                                                             | 60 |
| 6.14 | Absorbance change of aged scintillators compared to the "start" sample. This plot displays all unprimed, coated aging samples and a sample containing blank corten steel. AgedRef is displayed to quantify aging that takes place without a reflective sample. . . . .                                                                                        | 61 |
| 6.15 | Visual comparison of primed and coated corten steel samples, aged in liquid scintillator for a minimum equivalent time of 2.5 years . . . . .                                                                                                                                                                                                                 | 64 |

|      |                                                                                                                                                                                                                                                                                                                                                                                                  |    |
|------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----|
| 6.16 | Relative diffuse reflectivity spectra of all aged samples of aging test series S1. The minimum equivalent aging is 2.5 years. The wavelength-dependent reflectivity is measured relative to a BaSO <sub>4</sub> -standard with an angular configuration of (0° – 45°). . . . .                                                                                                                   | 65 |
| 6.17 | Relative diffuse reflectivity spectra of all aged samples of aging test series S2. The minimum equivalent aging is 0.5 years. The wavelength-dependent reflectivity is measured relative to a BaSO <sub>4</sub> -standard with an angular configuration of (0° – 45°). . . . .                                                                                                                   | 66 |
| 6.18 | Relative diffuse reflectivity spectra of all aged samples of aging test series S2. The minimum equivalent aging is 1.9 years. The wavelength-dependent reflectivity is measured relative to a BaSO <sub>4</sub> -standard with an angular configuration of (0° – 45°). . . . .                                                                                                                   | 67 |
| 6.19 | Absolute absorbance spectra of all aged scintillator samples from test series S1 after a minimum equivalent aging time of 2.5 years and the corresponding "start" measurements. . . . .                                                                                                                                                                                                          | 68 |
| 6.20 | Absorbance change of all aged scintillator samples from test series S1 after a minimum equivalent aging time of 2.5 years. The measurements are compared to the individual start sample. . . . .                                                                                                                                                                                                 | 68 |
| 6.21 | Absorbance change of all aged scintillator samples from test series S2 after a minimum equivalent aging time of 0.5 years. The measurements are compared to the individual start sample. . . . .                                                                                                                                                                                                 | 70 |
| 6.22 | Absorbance change of all aged scintillator samples from test series S2 after a minimum equivalent aging time of 1.9 years. The measurements are compared to the samples with a minimum equivalent aging time of 0.5 years to separate short-term and long-term scintillator degradation. . . . .                                                                                                 | 71 |
| 6.23 | CAD drawing of the full-size 1-cell-detector prototype for the Test Beam 2022 at DESY [81] . . . . .                                                                                                                                                                                                                                                                                             | 75 |
| 6.24 | The 1-cell-prototype in preparation for coating . . . . .                                                                                                                                                                                                                                                                                                                                        | 76 |
| 6.25 | Endoscopic photos showing selected areas of the inner detector walls with and without oxidation. . . . .                                                                                                                                                                                                                                                                                         | 76 |
| 6.26 | Endoscopic photos of the bottom half of the 1-cell-prototype after every layer of primer and coating. For orientation, the connection that is used for filling is marked in every photo. <b>Top left:</b> first layer of primer <b>Top right:</b> second layer of primer <b>Bottom left:</b> first layer of reflective coating <b>Bottom right:</b> second layer of reflective coating . . . . . | 78 |
| 6.27 | Final coating result of the top half of the 1-cell-prototype . . . . .                                                                                                                                                                                                                                                                                                                           | 78 |
| 6.28 | Comparison of endoscopic photos to assess the condition of the reflective coating. <b>Left:</b> After completion of spray coating. <b>Right:</b> After test beam. A number is assigned to the individual walls and a red circle marks a characteristic area in both photos to provide orientation. . . . .                                                                                       | 79 |
| 6.29 | Comparison of Monte-Carlo simulation with test beam data: the plots show the signal charge depending on the beam distance to the upper and lower WOM. <b>Left:</b> simulation data with full reflectivity <b>Right:</b> simulation data with 65 % reflectivity [94] . . . . .                                                                                                                    | 80 |

|      |                                                                                                                                                                                                                                                                                                                                                       |    |
|------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----|
| 6.30 | Normalized $\chi^2$ plotted against the reflectivity of the inner detector walls. The minimum at 65 % indicates the best fit between simulation and data [94]. . . . .                                                                                                                                                                                | 80 |
| 6.31 | CAD drawing of the 4-cell-prototype for the test beam 2023/24 at CERN. The inner and outer walls are made of 10 mm thick corten steel. The four cells of irregular size represent a side section of the SBT, with the height of the detector cells following the widened shape of the decay vessel. All lengths are given in mm. . . . .              | 81 |
| 6.32 | The 4-cell-prototype fully equipped with WOMs, SiPMs and readout electronics at the CERN test beam area . . . . .                                                                                                                                                                                                                                     | 82 |
| 6.33 | This figure shows a selection of endoscopic photos taken from the inside of the 4-cell-prototype before the coating process. The inner walls are covered in rust and writing from the construction of the prototype. . .                                                                                                                              | 83 |
| 6.34 | <b>Left:</b> A large yellow stain developed in the first layer of primer due to water in the pressurized air. <b>Right:</b> The following layers of primer and coating were able to fully cover this area. . . . .                                                                                                                                    | 84 |
| 6.35 | A selection of endoscopic photos taken of the inside of all cells of the 4-cell-prototype after completion of the coating process . . . . .                                                                                                                                                                                                           | 85 |
| 6.36 | Some areas around weld seams and connecting holes between the cells show slight yellow discoloration in the final coating result. This is due to the primer showing low adherence to the seams and not being able to fully cover the connecting holes with primer. . . . .                                                                            | 85 |
| 6.37 | This figure presents endoscopic photographs of the inner walls of the 4-cell prototype, taken in March 2024. By this time, the reflective coating had been in contact with the liquid scintillator for four months. Each photograph corresponds to one of the individual cells, showing the condition of the reflective coating in that cell. . . . . | 86 |
| 6.38 | This figure presents additional endoscopic photographs of the 4-cell prototype, taken in March 2024. The focus here is on highlighting areas of the reflective coating that exhibit pronounced yellowing. . . . .                                                                                                                                     | 87 |
| 6.39 | This plot shows absorption measurements taken from different scintillator samples that were collected during the test beam campaign in 2023 at CERN. . . . .                                                                                                                                                                                          | 88 |
| 7.1  | Illustration of the SiALG Design consisting of a light guide coupled to a SiPM [106] . . . . .                                                                                                                                                                                                                                                        | 96 |

|      |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |     |
|------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| 7.2  | Schematic illustration of a SiALG module with corresponding light paths within the (in)active light guide: Light-blue paths indicate photons that either reach the SiPM directly or are initially absorbed by the wavelength shifter. After absorption, photons are re-emitted at longer wavelengths with an isotropic angular distribution. Detection-relevant trajectories of re-emitted photons are shown in dark blue and include both direct transport to the SiPM and redirection toward the photo sensor via reflection from the parabolic upper surface of the light collector. An additional trajectory, depicted by a dashed black line, represents photons emitted at an angle $\theta_{\text{in}}$ smaller than the critical angle $\theta_{\text{crit}}$ . In this case, total internal reflection does not occur, and the photon escapes the light-guide structure rather than being guided toward the SiPM [107]. | 96  |
| 7.3  | Chemical structure of Styrene and Polystyrene [110] . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    | 97  |
| 7.4  | Illustration of the three reactions involved with the radical chain-growth polymerization of styrene. The initiator Benzoyl peroxide is added to increase the production of radicals at lower temperatures [118]. . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | 100 |
| 8.1  | Laboratory setup for the purification of styrene . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | 103 |
| 8.2  | Vacuum oven used for the polymerization of plastic scintillators. The upper compartment houses the oven chamber, while the lower compartment contains the cold trap and the vacuum pump. . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | 105 |
| 8.3  | Cold trap (left) and vacuum pump (right) located in the lower, ventilated compartment of the polymerization setup. . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | 105 |
| 8.4  | Polystyrene-based plastic scintillators with different fluors (L-R: Bis-MSB, PPO, BPEA) are excited by a UV-lamp causing the emission of their characteristic fluorescent light. . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | 106 |
| 8.5  | Three identically produced polystyrene samples show the dependency of bubble formation on the volume of the individual samples . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | 107 |
| 8.6  | Plastic scintillator samples with different weight fractions of BPO added as an polymerization initiator (L-R: 0.5 wt.%, 1 wt.%, 0.1 wt.%). . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | 108 |
| 8.7  | Absorbance spectra of polystyrene with and without the the addition of BPO as a polymerization initiator . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               | 109 |
| 8.8  | Emission–excitation map of a polystyrene-based scintillator containing 0.0001 wt% of bis-MSB . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | 111 |
| 8.9  | Emission–excitation map of a polystyrene-based scintillator containing 0.005 wt% of bis-MSB . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            | 111 |
| 8.10 | Transmission spectra of lab-manufactured plastic scintillators PS, PS+PPO and PS+PPO+bis-MSB and the commercial plastic scintillator SP32 .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      | 112 |
| 8.11 | Fluorescent spectra of polystyrene-based plastic scintillators containing POPOP (left) and bis-MSB (right) [111]. . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | 115 |
| 8.12 | Gamma spectra of the SP32 scintillator and a lab-manufactured scintillator with corresponding Gaussian fits . . . . .                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            | 115 |

|                                                                                                                                                                                                                                                                                                                                                                                                                                                  |     |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| 8.13 Time-dependent differences in signal intensity between SiALGs with active light guide containing bis-MSB (ALC) and inactive light guide (PLC) compared to a SiPM without light guide (No LC). These plots were calculated from time residual distributions normalized to their background level. The different versions of the photo sensor were integrated in a water-based liquid scintillator detector with the fluor PPO [107]. . . . . | 118 |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|

# List of Tables

|     |                                                                                                                                                                                                                                                                                                                           |     |
|-----|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----|
| 3.1 | Chemical and physical properties of LAB [28][17] . . . . .                                                                                                                                                                                                                                                                | 13  |
| 3.2 | Chemical and physical properties of PPO [34] . . . . .                                                                                                                                                                                                                                                                    | 15  |
| 3.3 | Chemical and physical properties of bis-MSB [42] . . . . .                                                                                                                                                                                                                                                                | 15  |
| 5.1 | Physics models and their associated final states that SHiP is sensitive to [78]. In this context $\ell = e, \mu, \tau$ . . . . .                                                                                                                                                                                          | 37  |
| 6.1 | Information on different primers used in laboratory tests . . . . .                                                                                                                                                                                                                                                       | 51  |
| 6.2 | Comparison of relative diffuse reflectivity measurements for primed and coated corten steel samples . . . . .                                                                                                                                                                                                             | 52  |
| 6.3 | Details on all samples compared in the 2022 aging tests . . . . .                                                                                                                                                                                                                                                         | 55  |
| 6.4 | Relative diffuse reflectivity of primed and coated corten steel samples before and after aging for $\lambda = 425$ nm. The wavelength-dependent reflectivity is measured relative to a $\text{BaSO}_4$ -standard with an angular configuration of $(0^\circ - 45^\circ)$ . . . . .                                        | 58  |
| 6.5 | Details on all scintillator samples for aging tests . . . . .                                                                                                                                                                                                                                                             | 63  |
| 6.6 | Relative diffuse reflectivity of primed and coated corten steel samples after aging for $\lambda = 425$ nm. The wavelength-dependent reflectivity is measured relative to a $\text{BaSO}_4$ -standard with an angular configuration of $(0^\circ - 45^\circ)$ . S1 and S2 distinguish the two individual test series. . . | 66  |
| 7.1 | Chemical and physical properties of Styrene [112] . . . . .                                                                                                                                                                                                                                                               | 98  |
| 7.2 | Chemical and physical properties of Polystyrene [113][114] . . . . .                                                                                                                                                                                                                                                      | 98  |
| 8.1 | Chemical and physical properties of BPO [122] . . . . .                                                                                                                                                                                                                                                                   | 104 |
| 8.2 | Transmission comparison of different plastic scintillators for $\lambda=425$ nm                                                                                                                                                                                                                                           | 113 |

## Use of Artificial Intelligence

| AI-Tool    | Use                             | Reason                      | Where         |
|------------|---------------------------------|-----------------------------|---------------|
| Chat GPT   | Text revision and rephrasing    | Improved readability        | Entire thesis |
| Chat GPT   | Explanation of different topics | Clarification of questions  | Chapter 3     |
| MS Copilot | Aid in Python programming       | Improved data visualization | All plots     |

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