# Set Up of a Purification and Measurement System for Organometallic Liquids for a Future PET Detector and Test Measurements with Tetramethylsilane

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# 1 Introduction

Positron Emission Tomography (**PET**) is a nuclear medicine imaging technique widely used in medical research and diagnostics. PET utilizes unique properties of the  $\beta^+$ decay in order to reconstruct a 3D image based on an artificially introduced radioactive tracer within an examined volume. One application is to examine metabolic processes within a living organism. Here a positron ( $\beta^+$ ) emitter is used to label a biochemically active molecules which are then injected into the bloodstream of the subject. After an initial accumulation time the subject is placed in the PET scanner where a series of opposing detectors scan for coincident photons produced in the annihilation of positrons, emitted from the labeled molecules. The acquired data then allows to reconstruct a 3D image based on the intersection lines of the individual events, which correspond to the distribution of the biochemically active molecule.

Due to its ability to quantify biochemical activity, PET finds many applications in medicine, mainly the detection of tumors, diagnostics of degenerative brain diseases and the development of new medical compounds. Improving the image quality of a PET scanner requires very efficient detectors with good spatial, energy and time resolution. A new type of  $\gamma$ -ray detector proposed in [YRT<sup>+</sup>14] shows great potential for PET applications. Using a heavy organometallic liquid, TriMethylBismuth (TMBi), as the detector medium, the CaLIDSO detector efficiently converts 511 keV photons into photo

detector medium, the CaLIPSO detector, efficiently converts 511 keV photons into photoelectrons and achieves fast timing by detecting Cherenkov photons created by these. Additionally secondary electrons that are created by inelastic scattering of the photoelectrons in the medium are extracted and detected on a segmented anode, providing position and energy information. Drifting charges to the anode requires liquids with a high level of purity, since electronegative impurities catch electrons and prevent them from contributing to the signal.

This master thesis introduces a purification and measurement system for organometallic liquids that was developed in close cooperation with the European Institute for Molecular Imaging in Muenster, and the CEA in Paris. The setup is tested with TetraMethylSilane (TMS), a liquid that is already well researched and hence allows to benchmark the detector system and compare obtained values to the literature.

# 2 Positron Emission Tomography

PET is a very versatile imaging technique, its applicability far exceeds the scope of this thesis. Therefore just a short introduction into one of its main applications, cancer diagnostics, is given to explain the general operating principle of PET.

### 2.1 Function Principle

In cancer diagnostics the most commonly used tracer for PET is Fludeoxyglucose (FDG), a glucose analog. In FDG the 2-hydroxyl group (hydroxyl group at the C-2 Position, see figure 2.1b) is substituted with <sup>18</sup>F, a  $\beta^+$  emitter with a half life of 110 min (figure 2.1b). Behaving almost exactly like glucose FDG participates in the metabolism of the body. Thus the FDG concentration is an excellent indicator of the glucose uptake within the body.

The  $\beta^+$  emitted during the decay of the <sup>18</sup>F nucleus constantly loses energy due to



Figure 2.1 – differnce between glucose (a) and FDG (b). The hydroxyl group at the C-2 Position is replaced with  $^{18}$ F.



**Figure 2.2** – The positron emitted from the nucleus during  $\beta^+$ -decay forms a hydrogen like state with an electron, called positronium. Electron and positron annihilate approximately at rest, resulting in the emission of two photons with energies corresponding to the electrons rest mass. In the rest frame of the onium and the case of two photons the emission is back to back (collinear).



**Figure 2.3** – In the laboratory frame the momentum of the onium is not 0, resulting in a non-collinearity of both photons. This and the finite positron range create a physical limit on the position resolution achievable in PET. Choosing a  $\beta^+$  emitter with a low endpoint energy reduces the positron range, improving the achievable resolution.

multiple scattering with electrons of the surrounding atoms. Once most of its energy is lost the positron forms an hydrogen like state with one of the surrounding electrons, called **positronium**, a metastable onium<sup>1</sup> with a mass of  $1.022 \,\mathrm{MeV}$ . Depending on the spin orientation of electron and positron this state can be differentiated into two configurations, **para-** and **ortho-**positronium. While in para-positronium the spins are antiparallel resulting in a total spin of  $S_p = 0$ , the parallel spins in ortho-positronium add up to a spin of  $S_o = 1$ . Positronium has a finite lifetime, since electron and positron annihilate. In the rest frame of the positronium the net momentum of the onium is 0 so it can only annihilate into two or more photons in order to conserve the momentum. Due to spin conservation parapositronium can only decay into even numbers and orthopositronium only into odd numbers of photons. Higher order annihilation into more than two photons only occurs with a probability of 0.003% [CDEP06]. In the case of two photons, due to momentum conservation, both are emitted back to back and have the same energy of 511 keV. Detecting the photons with a detector ring as seen in figure 2.2 allows to determine a line of response (LOR) connecting the two detectors marked in green. Since both photons were emitted back to back the LOR contains the point of annihilation. Given enough statistics a 2D or 3D image of the activity within the body can be reconstructed from the detected LORs. Depending on the energy of the  $\beta^+$  decay the positron first travels a distance through the tissue before it annihilates, resulting in an uncertainty in the measurement of the point of decay. Isotopes with a low endpoint of the beta spectrum have a lower range, making them more interesting for PET applications. Another uncertainty factor is due to the fact that the onium in the laboratory frame still has a finite momentum, resulting in a non-collinearity of the photons emitted (figure 2.3). Though non collinearity, and positron range provide a physical boundary of what resolution is achievable, most modern PET scanners are limited due to noise in the data, generated by false coincidences (figure 2.4). A way to quantify the influence of these effects is to calculate the Noise Equivalent Count Rate (NECR):

$$NECR = \frac{T^2}{T + S + kR}.$$
(2.1.1)

T, S and R are the rates of true, scatter and random events respectively and k is a

<sup>&</sup>lt;sup>1</sup>bound state of particle and anti particle

parameter that depends on the random correction method [MAMNK<sup>+</sup>11]. A higher NECR value corresponds to a better image contrast.



**Figure 2.4** – Types of coincidences in PET. (a) True coincidence, here LOR and point of annihilation coincide. (b): Scattered coincidence, one or both photons undergo Compton scattering before detection, resulting in a wrongly reconstructed LOR. Detectors with good energy resolution can be used to discriminate between scattered and unscattered photons. (c): Random coincidence, photons from more than one event are detected within the coincidence time window. Good timing resolution allows to make a smaller coincidence window and timing resolutions in the sub nanosecond regime can be used for TOF measurements, drastically improving the image quality (see figure 2.6). (d): Multiple coincidences, three or more detectors give a signal within the coincidence time frame, a good timing resolution also improves the distinction capability of the PET scanner.

### 2.2 Detector Setup

A key component in PET is the detector system. Depending on the type of interaction highly energetic photons create electrons with different energy distributions within the detector medium (figure 2.5). Signals created by photoelectric absorption have a sharp energy peak, while in Compton scattering the energy transfer from photon to electron  $E_e$  depends on the scattering angle  $\phi$  and can be described by:

$$E_e(\phi) = E_\nu - E'_\nu(\phi) = E_\nu \left(1 - \frac{1}{1 + \frac{E_\nu}{m_e c^2}(1 - \cos\phi)}\right)$$
(2.2.1)

with  $E_{\nu}$  being the photon energy. Only full energy signals, where the photon loses all its energy, either due to multiple Compton interactions or photoelectric absorption within the detector medium are of interest for PET. Increasing the number of full energy signals could be achieved by scaling up the detector volume. Though this would improve the detection efficiency, the spatial resolution would deteriorate. A good way to improve the detection efficiency is by increasing the cross section of photoelectric absorption,  $\sigma_p$ . For high energetic photons  $\sigma_p$  scales with  $Z^5$ , while the cross section for Compton scattering  $\sigma_c$  scales with Z, making high Z materials desirable for PET detectors. Most commonly used are scintillation block detectors (figure 2.7) with LSO-<sup>2</sup> or LYSO-<sup>3</sup> crystals. High energy photons excite electrons within the detector medium, either by Compton scattering or photoelectric absorption. These electrons lose most of their energy due to inelastic scattering and excite further electrons into higher energy bands. When these electrons transition into lower energy bands they emit light that can be detected by photo multipliers (PMTs) that are optically coupled to the scintillation crystals. LSO- and LYSO- crystals have the disadvantage that photons at  $511 \,\mathrm{keV}$  have a 70%probability of Compton scattering within the detector [KL08]. For larger crystals the probability of multiple interactions within the medium increases, therby improving the number of full energy interactions. The image quality highly depends on the accurate reconstruction of the interaction point within the detector. In the case of the detector

<sup>&</sup>lt;sup>2</sup>Lutetium Oxyorthosilicate

<sup>&</sup>lt;sup>3</sup>Lutetium-Yttrium Oxyorthosilicate



Figure 2.5 –  $^{137}$ Cs spectrum of the source used in all measurements in this work, measured with Ge-detector.

depicted in figure 2.7 the reconstruction of x and y position is achieved by segmenting the scintillator crystal into a 2D array. Reflective coating between the crystal segments ensures that the light is guided towards the PMTs in the segment where it was created. The light produced in one segment is detected with different sensitivities by all PMTs and the position can be reconstructed by comparing the signal heights A,B,C and D measured by all four PMTs [CR86]:

$$R_x = \frac{A+B}{A+B+C+D}$$
$$R_y = \frac{A+C}{A+C+D}.$$

This type of reconstruction is susceptible to statistical noise, depending on the number of photons detected. Specially at high count rates, as expected in PET, multiple gammas can interact in several crystals, and produce a signal that looks like a single  $\gamma$  at a wrong position. In addition to good spatial and energy resolution PET-detectors need good



**Figure 2.6** – LOR vs probability distribution, allows for better discrimination between true and false signals. Two annihilation processes take place at the same time. Only three of the photons are detected. In (a) all three detectors respond within the coincidence time window, resulting in two LORs. If both lines are added to the data one of them will contribute as noise. In (b) only one of the probability distributions has its center within the subject, allowing to disregard the other.

timing resolution for the measurement of coincident photons. Timing resolutions in the region of a few 100 ps can also be used for time of flight (TOF) measurements, here the arrival time difference between two photons from the same annihilation process is measured. This allows to determine a probability distribution for the actual position along the LOR where the decay happened instead of a line of response (figure 2.6). Determining a probability distribution delivers additional input to the 3D reconstruction and allows for a better discrimination between false and true signals hence improving the NECR.



**Figure 2.7** – Block detector. Light generated in the segmented scintillation crystals is measured by the PMTs A-D. From the ratio of the signal amplitude the x- and y- position is reconstructed. For better visibility all scintillator segments above the diagonal between PMT B and C are hidden in this drawing.

# 3 Liquid Ionization Chamber Detector

Liquid ionization chamber detectors compared to noble gas filled ones have the advantage of a higher density, which is decreases the attenuation length. Xenon TPCs (Time Projection Chambers) where both scintillation light and ionization signals are measured are shown to have a good energy resolution in the order of 10% for 511 keV photons  $[ABK^+09]$ . Timing resolution in the sub-nanosecond regime for such a detector are shown in  $[GAM^+05]$ . Due to the small lateral electron diffusion in liquid xenon  $[ABD^+05]$ a 3D spacial resolutions in the sub millimeter regime is possible [SCP<sup>+</sup>02], [ACG<sup>+</sup>08]. One disadvantage of a liquid filled Xenon TPC for PET is the small photo absorption probability for 511 keV photons of 24% [YRT<sup>+</sup>14]. The low boiling point of 165 K also requires cryogenic technologies to be built into the detector, resulting in higher cost and an overall more complex and error-prone system. Having a room temperature liquid with similar electric properties can reduce the intricacy of a such a detector thus lessen cost and complexity of the system. Organometallic liquids like TMS (Tetra Methyl Silane) were shown to have characteristics that are desirable for a detector medium [EFH<sup>+</sup>99]. But due to its low charge number the photo electric efficiency with a value of 0.04% a detector build with TMS is unsuited for PET-applications. A possible candidate with a high photo fraction of 47% for 511 keV photons is Tri-Methyl-Bismuth (TMBi), an organometallic liquid that consists of a Bismuth atom suspended between three methyl groups ( $C_3H_9Bi$ ). A new detector setup proposed by scientists at CEA Sarclay [YRT<sup>+</sup>14] making use of TMBi as detector material is described in the following sections.

### 3.1 CaLIPSO

CaLIPSO (Calorimetre Liquide Ionisation, Position Scintillation Organometallique), is a novel PET-detector principle that uses TMBi as the active detector material [YRT<sup>+</sup>14]. The CaLIPSO detector is planned to have 2 readout channels, a fast optical channel for the timing signal and a slower electrical channel for x-y position reconstruction and energy determination.

When a charged particle passes through a dielectric medium at speeds greater than the phase velocity of light within this medium electromagnetic radiation is emitted (Cherenkov radiation). Photo absorption of 511 keV photons create electrons with high velocities in TMBi, the Cherenkov radiation emitted from those is detected by PMTs and used as a timing signal (figure 3.1). Due to inelastic scattering of the photo electron further molecules are ionized. The created electrons then drift due to an applied electrical field towards a pixelized anode. A Frisch-grid separates signals created by the slow moving positively charged ions from the signals of the electrons. The distribution of induced charge signals in the anode pixels allows to reconstruct energy and x-y position of the interaction. From the difference in the timing signal of the Cherenkov light and arrival time of the charge pulses the z-coordinate of the interaction within the detector can be calculated. This allows for a 3D reconstruction of the interaction point within the detector with millimeter resolution [KJS<sup>+</sup>18].



Figure 3.1 – CaLIPSO detector. Photo electrons from the 511 keV photons propagate faster than light within the TMBi creating Cherenkov radiation which is detected by the PMTs providing the timing signal. The electrons loose energy due to inelastic scattering within the detector medium and ionize further molecules. These electrons are then drifted past the Frisch grid towards a pixelized anode where a charge signal is measured. From the pixel distribution the x- and y- position is reconstructed. The timing signal from the PMTs and charge signal is used to reconstruct the z-position of the interaction. (graphic redrawn from  $[FTM^+18]$ )

## 3.2 Onsager Theory

Highly energetic photons can ionize molecules: The photo electron, separated from its mother molecule, loses energy to the surrounding molecules due to multiple inelastic scatterings until it reaches thermal equilibrium. In 1938 Onsager derived a theory that describes the probability for a electron-ion pair to recombine under the influence of an electric field and Brownian motion. In absence of other forces than the Coulomb force this probability is proportional to the reciprocal of the Boltzmann factor  $k_b$  [Ons38]. Figure 3.2 depicts the situation: The potential energy of an electron-ion pair separated by a distance r within a dielectric medium with the dielectric constant  $\epsilon_r$  and an applied electric field E is given by:

$$U(r) = -\frac{e_0^2}{4\pi\epsilon_0\epsilon_r r} - e_0 E\cos\theta \qquad (3.2.1)$$

here  $\theta$  denotes the angle between to the electric field direction and a vector pointing from the ion towards the electron (see figure 3.2). In a medium with the temperature Ta critical distance  $r_c$  is defined as the distance where the coulomb energy is equal to the thermal energy of the medium:

$$r_c = \frac{e_0^2}{4\pi\epsilon_0\epsilon_r k_b T}.$$
 (3.2.2)

For separations of  $r \gg r_c$  diffusion outweighs the coulomb attraction and the electronion pair escapes the mutual attraction. For  $r < r_c$  the coulomb attraction leads to recombination.

The probability  $p(r, \theta, E, T)$  that the electron-ion pair escapes initial recombination was presented by Pai and Enck [Pai75]:

$$p(r,\theta,E,T) = \exp(-A)\exp(-B)\sum_{n=0}^{\infty}\sum_{m=0}^{\infty}\frac{A^m}{m!}\frac{B^{m+n}}{(m+n)!}$$
(3.2.3)



Figure 3.2 – Onsager problem: Electron and ion are separated by as distance r.  $\theta$  is the angle that spans between the vector pointing from the ion to the electron and a vector parallel to the electric field.

with

$$A = \frac{e_0^2}{4\pi\epsilon_0\epsilon_r k_B T r} \qquad \text{and} \qquad B = \left(\frac{e_0 E r}{2k_B T}\right) (1 + \cos\theta) \qquad (3.2.4)$$

the first terms of equation 3.2.3 can be written as:

$$p(r,\theta,E,T) = \exp\left(-\frac{r_c}{r}\right) \left[1 + \frac{1}{2!} \left(\frac{e_0}{k_B T}\right) r_c E + \frac{1}{3!} \left(\frac{e_0}{k_B T}\right)^2 r_c \left(\frac{r_c}{2} - r\right) E^2 + (3.2.5) \frac{1}{4!} \left(\frac{e_0}{k_B T}\right)^3 r_c \left(\frac{r_c}{2} - r_c r + \frac{r_c^2}{6}\right) E^3 + \dots\right]$$
(3.2.6)

Mozumder [Moz74] derived a series that made it no longer necessary to calculate the infinite sums in Onsager's theory and gave an escape probability of:

$$p(r, \theta, E, T) = 1 - S_{-} \exp\left[-\eta - \zeta(1+z)\right]$$
(3.2.7)

with:

$$\eta = \frac{r_c}{r}$$
 ,  $\zeta = \frac{e_o E r}{2k_B T}$  and  $z = \cos \theta$  (3.2.8)

 $S_{-}$  is the expansion of  $\exp[\eta + \zeta(1+z)]$ , containing only negative powers of r:

$$S_{-} = \exp[\eta + \zeta(1+z)](1-P)$$
(3.2.9)

Averaging P over a random distribution of  $\cos\theta$  Mozumder calculated the escape probability to be:

$$\bar{P} = 1 - (2\zeta)^{-1} \sum_{k=0}^{\infty} A_k(\eta) A_k(2\zeta).$$
(3.2.10)

With the coefficients

$$A_k(\eta) = 1 - \exp(-\eta) \left( 1 + \eta + \frac{\eta^2}{2!} + \dots + \frac{\eta^k}{k!} \right)$$
(3.2.11)

the escape probability in the limit of low electric field strength E becomes:

$$\bar{P} = (1 + \eta\zeta) \exp(\eta) = e^{-r_c r} \left( 1 + \frac{e_0^3}{8\pi\epsilon_0\epsilon_r k_B^2 T^2} E \right).$$
(3.2.12)

# 4 Experimental Setup

Building an organometallic liquid based detector requires a good understanding of the electric properties of the detection material. Therefore a purification and measurement system has been developed and tested with TMS. The setup needs to meet high demands in terms of clean handling of the liquid, hence only components that meet ultra high vacuum requirements are used. The system can be split into two main parts. The purification system and the detector. The purification system is used to clean the liquids and test purification methods. The detector is utilized in two modes:

- **current mode**: The current mode is used to determine the free ion yield of the liquid, by measuring the current induced by ionizing radiation at a given electric field strength.
- pulse mode: Energy spectra are measured and compared to a simulated spectra.

## 4.1 Purification System

The transport of free electrons in the detector requires liquids with very high purity. Electronegative contaminations like  $O_2$  have to be on levels below 1 ppm [Gru93]. Achieving those purities and retaining them places high demands on the materials and purification techniques used. Only stainless steel and ceramics that satisfy Ultra-High-Vacuum (UHV) requirements are used in the purification system and detector (figure 4.1). To reduce the contamination with residual gases adsorbed on the walls, all systems are baked at temperatures of  $T \geq 200$  °C and pumped to a vacuum better than  $10^{-7}$  mbar. This is achieved by using a combination of a dry fore-pump, a turbomolecular pump (TMP)

and a cold trap<sup>1</sup>. After bakeout the system pressure is measured to be better than  $5 \times 10^{-9}$  mbar (sensitivity of the pressure gauge used).

<sup>&</sup>lt;sup>1</sup>A vacuum vessel than can be cooled using liquid nitrogen, located in front of the TMP, most gases condense in this trap. This protects the mechanical pumps from contact with those gases.



Figure 4.1 – Experimental setup. Samples can be filled into vessel (a). A mixture of ethanol and liquid nitrogen can be used to cool the container below temperatures of -99 °C (meltingpoint of TMS). Using a combination of a cold trap, TMP and backing pump the whole system can be evacuated. Transfer of liquids is achieved by differential vapor pressure, created by regulating the temperature of two double walled vessels (c) (detailed description in figure 4.3). Molecular sieves (b) are used to remove impurities from the sample. A residual gas analyzer (d) can be used to examine the samples. The detector (e) is located directly under the right double walled vessel an can be filled by opening the valve below the container.





**Figure 4.2** – *LabVIEW* interface, showing the gas system. Multiple *Swagelog* valves (green) can be used to alter the path the gas is pumped through.

The main focus of the purification system lies in the clean handling of the liquids used. Only high quality *Swagelog* valves and stainless steel components are used in the setup. Two *Lauda Eco RE420* cooling/warming thermostats are used to control the temperature of two double walled containers (figure 4.3). Warming up one and cooling down the other container results in differential vapor pressures allowing to cleanly pump between the containers. Each tank is equipped with a *MKS Type 626 Baratron* manometer, that monitors the pressure. The pump path is controlled using different valves. Figure 4.2 shows the gas system as displayed in the *Lab VIEW* interface that is used to monitor the system.

#### 4.2.1 Purification Protocol

Handling the liquids requires to follow strict protocols to reduce the risk of contaminating the liquid. Especially the transfer into the purification bench and the cleaning process pose a high risk of contamination. For the measurements with TMS the following protocols were developed:

**Transfer** The TMS sample used in all measurements is provided by *Sigmaaldrich* with a purity better than 99.99%. It is delivered in a small glass flask with a protective argon atmosphere on top. Argon is used because it is an inert gas that can be removed by freezing the sample and pumping away the gaseous argon. In order to transfer the sample into the purification setup a vacuum steel vessel is baked out and sealed using valve. Both sample and vessel are than opened under a protective Argon atmosphere within a glove box, and the liquid TMS is filled to the vessel which is then sealed again.

**Freeze Pump and Thaw** The argon atmosphere and other residual gases are removed by freezing the liquid to a temperature below the melting point of TMS at -99 °C and pumping away the gas layer above the frozen TMS. After pumping for five minutes using the cold trap and a TMP, the sample is warmed to a temperature of -80 °C, is thawed and than is frozen and pumped again. This process is repeated five times.

Molecular Sieves A similar purification protocol as used in the KASCADE project  $[EFH^+99]$  is applied. The sample is pumped through different molecular sieves that are used to clean the liquid 4.3. Molecular sieves are porous materials with uniform pore sizes, that filter via adsorption on their large surfaces. Due to their small pore sizes large molecules like TMS can not enter the pores while smaller molecules like water can. This increases the effective surface area seen by smaller molecules and reduces the speed with which those molecules can pass through the filter drastically. This is especially effective with polar molecules that have a higher tendency to get adsorbed on surfaces. Prior to usage, the sieves are heated and pumped using the TMP to a vacuum better than  $10^{-6}$  mbar at 390 °C sieve temperature. The TMS sample used in this measurement is filtered first by a 4 Å Na-Al-silicate sieve with a pore size of 0.4 nm, reducing the number of impurities such as oxygen, nitrogen, carbon dioxide and straight chain hydro

carbons. After this the sample is filtered by a 5 Å sieve, removing larger impurities upto a molecular size of 0.5 nm. Filtering smaller impurities first reduces the risk of them entering the larger pores in the 5 Å sieve and detach already adsorbed impurities. After purification the detector is filled with the liquid and first measurements to determine the free ion yield are conducted.



**Figure 4.3** – Filter with molecular sieves. The sample is pumped from the red to the blue container. Two *Lauda Eco RE420* cooling thermostats are used to pump tempered oil through the outer shell of two double walled containers. The inner volume is connected to the vacuum system where two pressure gauges monitor the vapor pressure. By warming up the red container while cooling the blue a differential vapor pressure is created. The liquid evaporated in the red container passes through the molecular sieve (white) and than condenses in the blue container. Small impurities can enter the pores in the molecular sieve and can be adsorbed. Pumping from top to bottom in the molecular sieve reduces the risk of gas bubbles passing through the powder without getting filtered properly.

## 4.3 Detector



**Figure 4.4** – CAD drawing of the detector. HV is applied on the cathode (red). A guard ring (blue) reduces inhomogeneities in the electric field from the boundaries of the detector plates. The signal is measured at the anode (green). Anode and guard ring are on the same electric potential. All components used satisfy UHV requirements. A detailed technical drawing can be found in appendix I.1.

The number of free charge carriers generated per 100 eV energy deposited  $G_{fi}$  within the active detector medium, called  $G_{fi}$ , is an important characteristic. Since a greater number of electrons generated corresponds to a larger signal and better energy resolution in a future PET-detector, quantifying this number is important when deciding which liquid to use in a future detector. In order to benchmark the detector, first test measurements with TMS have been performed. A way to determine the  $G_{fi}$  value of a liquid is using it as a dielectric medium in between anode and cathode of a capacitor and generate free charge carriers in the liquid by introducing ionizing radiation from a radioactive source. Measuring the current between both detector plates and dividing by the elementary charge e allows to determine the number of free charge carriers detected. In combination with a calculation of the deposited energy within the liquid the free ion yield can be

determined (see section 5.3).

#### 4.3.1 Detector Setup

Building a detector that allows for a precise measurement of the liquids electric properties places high demands on the electronics. Only low noise electronics in combination with filters are used for the detector. To reduce the noise the detector is electrically isolated from the purification system and shielded by a Faraday cage. The anode of the detector (figure 4.4) is composed of two concentric parts, the anode-disk (green) and the guard ring (blue), both sit on the same electric potential. Measuring signals only on the anode disk and disregarding those of the guard ring simplifies the description of the detector. Since inhomogeneities in the electric field from the boundaries of the cathode (red) only lead to the guard ring, resulting in a highly homogeneous electric field between anode disk and cathode (see figure 4.5). Additionally the segmented set up confines the active volume to the region between anode disk and cathode, allowing for a more precise simulation of the active medium.

#### 4.3.2 Electronics

#### 4.3.2.1 High Voltage

The high voltage is generated by an *iseg 224M* high voltage supply with a peak to peak ripple smaller than  $2 \text{ mV}_{ss}$  [ise18]. The power supply is remotely controlled by a *LabVIEW* program to allow for automated measurements at different voltages. The HV is smoothed by a filter box (figure 4.6) and than transferred to the HV-feed through leading to the cathode.

#### 4.3.2.2 Current Measurements

Signals from the anode are passed through a SHV-feed-through to a protective circuit consisting of a 220 pF capacitor and a 1 G $\Omega$  resistor in parallel, protecting the measurement electronics from large current spikes. The current is amplified using a *DDPCA-300 Sub Femto Ampere Current Amplifier* with a maximum gain of  $10^{13} \frac{V}{A}$  and a peak to peak



**Figure 4.5** – Simulation of the electric field within the detector. The colors of anode disk (green), guard ring (blue) and cathode (red) correspond to the colors of the CAD-drawing in figure 4.4. Field lines from the boundary of the cathode only lead to the guard ring. The field lines between anode disk and cathode are mainly homogeneous.

noise of  $\leq 0.4$  fA [FEM19]. The amplifier converts the current into an equivalent voltage which is then measured by a NI-9205 module with a residual gain error of 115 ppm of reading [Nat17]. A *LabVIEW* program checks in 0.5 s intervals if the voltage measured is between 0.5 V and 9.5 V and automatically adjusts the gain of the current amplifier to ensure all measurements are at the optimal settings. The data is then transmitted via Ethernet to a computer where it is stored for later analysis.

#### 4.3.2.3 Pulse Measurements

Since in actual PET applications the measurement of individual events is required, a series of pulse measurements are conducted. For this the current amplifier is replaced with a combination of charge sensitive preamplifier and a C.A.E.N N968 spectroscopy amplifier. The signals are then digitized using a Ortec 926 ADCAM MCB. The preamplifier has to fulfill two main tasks: matching the impedance of the detector cell to the subsequent



**Figure 4.6** – Filter box used for current measurements. High voltage is applied to the left of the box. A series of low pass filters smooth the HV. The voltage is then fed to the cathode via a high voltage vacuum feed through.

electronics in order to reduce the load on the detector and amplifying the relatively low signals produced in the active medium. The one used in this setup was build by the electronics workshop of the institute based on a design by Manfred Schrader [Sch90] and stands out due to a very high signal to noise ratio. It is a charge sensitive preamplifier, meaning that it produces an output voltage proportional to the charge input. A charge calibration of the acquisition system is implemented to accurately scale the acquired data.

#### **Charge Calibration**

Figure 4.7 shows a schematic overview of the setup that is used to conduct a charge calibration. The calibration is done for the entire acquisition system, consisting of preamplifier, spectral amplifier and  $ADC^2$ .

Mono energetic  $\gamma$  radiation from a <sup>241</sup>Am source is used to produce electric pulses with a narrow charge distribution in a Si-diode. Knowing the energy of the ionizing radiation, and measuring the peak position within the spectrum allows to calibrate the system to a natural constant. From the energy of the ionizing radiation the produced charge can be calculated knowing that it takes an average of 3.63 eV to generate an electron ion pair in Silicon. The spectrum measured using the <sup>241</sup>Am source is shown in figure 4.8. To obtain the peak positions from the spectrum the data is fitted using a sum over 5 Gaussian distributions:

<sup>&</sup>lt;sup>2</sup>Analog to Digital Converter

Peak number	1	2	3	4	5
Energy [keV]	13.9	17.9	free parameter	26.4	59.5

**Table 4.1** –  $\gamma$ -Energies expected in the <sup>241</sup>Am spectrum. [ESC+98]

$$f = b + \sum_{i=1}^{5} A_i \cdot \exp\left(\frac{-(K - K_0(E_i, \text{slope, offset}))^2}{\sigma_i^2}\right)$$
(4.3.1)

The center of each Gauss is defined by:

$$K_0(E_i, \text{slope}, \text{offset}) = \text{slope} \cdot E_i + \text{offset}$$
 (4.3.2)

Equation 4.3.1 was parameterized in a way that all peaks expected within the spectrum are on a straight line, with the energies  $E_1, E_2, E_4, E_5$  being fix parameters obtained from table 4.1. Using this form directly yields a channel to energy conversion of the detector system defined by the fit parameters slope and offset.



Figure 4.7 – Setup used for charge calibration. A  $^{241}$ Am source generates gamma radiation. The radiation generates a charge that is measured by system of preamplifier, spectroscopy amplifier and a MCB.



Figure 4.8 – Measured  $^{241}\mathrm{Am}$  energy spectrum, measured with an Si-pin diode. Spectral amplifier amplification 100.

The energy to channel conversion with an amplification a can be calculated from the fit parameters:

$$K'(E_{keV}, a) = E_{keV} \cdot \frac{\text{slope}}{100} \cdot a + \text{offset}$$
(4.3.3)

The factor 100 is due to the measurement settings with an amplification of 100 at the spectral amplifier. a has to be adjusted to the amplification of the spectral amplifier used in later measurements. Correcting measurements by subtracting the offset from the fit allows to simplify  $K'(E_{keV}, a)$  to:

$$K(E_{keV}, a) = E_{keV} \cdot \frac{\text{slope}}{100}a \tag{4.3.4}$$

Knowing it takes an average of  $p = 3.63 \times 10^{-3} \text{ keV}$  to create an electron-hole pair in a silicon detector allows to convert the channel number in equation 4.3.4 to an equivalent charge:

$$K(Q, a) = \frac{\text{slope}}{100} \frac{Q}{e} \cdot 3.63 \times 10^{-3} \,\text{keV} \cdot a \tag{4.3.5}$$

The generated charge in organometallic liquids is defined by the free ion yield  $G_{fi}$ :

$$Q = e \cdot E_{100eV} \cdot G_{fi} = 10e \cdot E_{keV} \cdot G_{fi} \tag{4.3.6}$$

the factor 10 is added to account for the  $G_{fi}$  is defined as the number of charge carriers per 100 eV deposited energy. Substituting Q from equation 4.3.5 with 4.3.6 and using the fit results from figure 4.8 gives the charge to channel conversion:

$$K(E,a) = 2.36 \,\frac{\text{channel}}{\text{keV}} \cdot 3.63 \times 10^{-3} \,\text{keV} \cdot E_{keV} \cdot G_{fi} \cdot a \tag{4.3.7}$$

$$= 8.572 \times 10^{-3} \cdot E_{keV} \cdot G_{fi} \cdot a \tag{4.3.8}$$

#### 4.3.3 Systematic Effects

#### 4.3.3.1 Active Volume

Measurements of the free ion yield require a detailed simulation of the deposited energy per time within the detector. It is therefore of great importance to know the dimensions of the volume contributing to the signal. To determine the distance between anode and cathode two separate measurements were done. First, during assembly, a distance plate of  $d_d = (2.8 \pm 0.1)$  mm was placed between anode and cathode and later removed. Additionally a CT-scan of the detector was made. As seen in figure 4.9a the cathode is tilted by an angle of  $\alpha = 1.2^{\circ}$  to the cathode plane. The sag of the cathode is estimated to be 1.4 mm. The tilt and sag of the cathode is implemented into a *Comsol* simulation. From this simulation the Maxwell capacitance between anode and cathode is obtained




(a) CT-scan of detector, made by Klaus (b) 3D comsol simulation of detector. Schäfers.

Figure 4.9 – Detector CT-Scan and Simulation. The CT-Scan shows a tilt of  $1.2^{\circ}$  between the electrodes, from this tilt a sag of 1.4 mm is calculated and implemented into a *Comsol* simulation of the detector.

and than compared to the measured capacitance.

**Measured Capacitance** The capacitance between anode and cathode in vacuum is determined by ramping up the voltage applied to the cathode from  $V_0 = 0$  V to  $V_1 = 500$  V with a slope of  $V_s = 2$  Vs<sup>-1</sup> (see figure 4.10). The background corrected data is fitted with a function of shape:

$$I = I_0 + A \cdot \sin(2\pi f t + \phi_0) \tag{4.3.9}$$

from the fit the current  $I_0 = (3.07 \pm 0.05)$  pA and its error is obtained. Using this value the capacitance C can be calculated to:

$$C = \frac{Q}{U} = \frac{250 \,\mathrm{s} \cdot (3.07 \pm 0.05) \,\mathrm{pA}}{500 \,\mathrm{V}} = (1.53 \pm 0.03) \,\mathrm{pF} \tag{4.3.10}$$

**Simulated Capacitance** The *Comsol* simulation shows a linear behavior between capacitance and plate distance (figure 4.11) which can be described by:

$$d_c = -1.74 \,\frac{\rm mm}{\rm pF} \cdot C + 5.50 \,\rm mm \tag{4.3.11}$$

$$= (2.84 \pm 0.09) \,\mathrm{mm} \tag{4.3.12}$$

Within the error both measurements of the distance are in agreement. With the anode disk radius r = 12.5 mm the detector volume V can be calculated to be:

$$V = 2\pi r \cdot d_d = (1394 \pm 44) \,\mathrm{mm}^3 \tag{4.3.13}$$

#### 4.3.3.2 Source Activity

The source activity is determined using a Activimeter ISOMED 2010 and measured to be  $(1.03 \pm 0.05)$  MBq

#### 4.3.3.3 Source Position

The source is mounted on an L-shaped arm that can be moved using a stepper motor (figure 4.12). Two end switches define the "in" and "out" position of the source ensuring a reproducible source position in between measurements. The Stepper motor is mounted on a linear translation table, that allows to move the source parallel to the detector and position the source perpendicular to the center<sup>3</sup> of the detector. Using the translation stage the source is moved perpendicular to the detector plates and the induced current is measured. The translation table is fixed at the position where the maximal current is measured.

 $<sup>^{3}\</sup>mathrm{equal}$  distance between anode and cathode, measured from the center of the anode disk perpendicular to the anodes surface



**Figure 4.10** – Measured current in vacuum while ramping up the voltage applied at the cathode from 0 V to 500 V. Top panel shows the raw measurement data. The background is subtracted from the data and the absolute value is displayed in the middle panel. A function that accounts for the oscillation from the control electronics of the HV-supply is fitted to the data. From the fit the current and its error is obtained. The lower panel shows the measured current minus the fitted oscillation.



Figure 4.11 – Plate distance vs. capacitance. The simulation shows a linear behavior.



Figure 4.12 – The source can be moved using a stepper motor. Two end switches define reproducible positions of the source. In the "out" position (red) the distance between source and detector center is increased by a factor of  $\approx 15$  compared to the "in" end position (green), and a lead brick blocks the direct view from the source to the detector. At the "in" position the source, located at the tip of an iron rod, is positioned  $(3.2 \pm 0.1)$  cm from the center of the detector. A linear translation table (yellow) allows to accurately position the source perpendicular to the center of the detector.

# 5 Measurements with TMS

According to Onsagers theory the probability of generating free electron ion pairs in an insulating liquid depends on three variables. For high electric field strengths this probability is characterized by a linear function that can be described by the applied electrical field E, temperature T and the relative permittivity  $\epsilon_r$ . In this chapter first measurements with TMS are conducted. Section 5.1 shows a method to determine the  $\epsilon_r$  of TMS. Using this value the expected slope in Onsagers theory can be calculated. The calculated slope is then compared to a measured value in section 5.3 by measuring the free ion yield as a function of the applied electric field with a constant detector temperature and fitting a modified version of Onsagers theory to the data. In section 5.4 the fitted function is used to calculate an energy calibration of the detector and compare the measured energy spectrum in pulse mode to a simulation of the system.

## 5.1 Permittivity

By filling the detector with TMS, the Coulomb forces of charges located on the opposing plates of the detector are reduced, resulting in a higher capacity of the detector. Measuring this change allows to determine the relative permittivity  $\epsilon_r$  of the material. Comparing the capacity of the detector in vacuum to the one filled with TMS gives a direct handle on  $\epsilon_r$  (eq. 5.1.3). In order to measure the permittivity of TMS the voltage applied to the cathode of the detector is ramped up with  $2 \text{ Vs}^{-1}$  to a voltage  $U_m = 500 \text{ V}$  (fig. 5.1). From the current measured at the anode disk the stored charge can be determined. The capacity of the detector in vacuum and filled with TMS  $C_{VAC}$  and  $C_{TMS}$  respectively can be written as:

$$C_{VAC} = \epsilon_0 \frac{A}{d} = \frac{I_{VAC} \cdot t_m}{U_m} = \frac{Q_{VAC}}{U}$$
(5.1.1)

$$C_{TMS} = \epsilon_0 \epsilon_r \frac{A}{d} = \frac{I_{TMS} \cdot t_m}{U_m}$$
(5.1.2)

Using the currents and their errors obtained from the fits in figures 5.1 and 4.10  $\epsilon_r$  is measured to be:

$$\epsilon_r = \frac{C_{TMS}}{C_{VAC}} = \frac{I_{TMS}}{I_{VAC}} = \frac{(5.89 \pm 0.05) \,\mathrm{pA}}{(3.07 \pm 0.05) \,\mathrm{pA}} = 1.92 \pm 0.03 \tag{5.1.3}$$

The measured value for  $\epsilon_r$  is in complete agreement with the literature value of  $\epsilon_r = 1.92$  [EK84].



**Figure 5.1** – Measured current with TMS filled detector while ramping up the voltage applied at the cathode from 0 V to 500 V. Top panel shows the raw measurement data. The background is subtracted from the data and the absolute value is displayed in the middle panel. A function that accounts for the oscillation from the control electronics of the HV-supply is fitted to the data. From the fit the current and its error is obtained. The lower panel shows the measured current minus the fitted oscillation.

## 5.2 Simulations

In order to determine the number of free charge carriers generated per deposited energy a  $GATE^1$  Monte Carlo simulation (MC) of the detector and the source is performed by B. Gerke. The simulation provides the number of interactions per time n within the active medium of the detector. Each interaction corresponds to an energy transfer  $E_i$ from photon to electron and a position  $z_i$  perpendicular to the detector planes. From the rate spectrum (figure 5.2) the deposited energy per second  $P_S$  can be calculated to:

$$P_S = \sum_{i}^{n} E_i = 162 \,\mathrm{MeV} \cdot \mathrm{s}^{-1} \tag{5.2.1}$$

<sup>&</sup>lt;sup>1</sup>"GATE is an advanced opensource software developed by the international OpenGATE collaboration and dedicated to numerical simulations in medical imaging and radiotherapy." http: //www.opengatecollaboration.org



Figure 5.2 – Simulation of the energy spectrum induced by a  $^{137}$ Cs source in the detector filled with TMS.

## 5.3 Free Ion Yield

The free ion-yield  $G_{fi}$  is defined as the number of free ion-electron pairs produced per 100 eV deposited energy in a medium. A possible way to determine  $G_{fi}$  is to measure the current I induced by ionizing radiation. Multiplying I with 100 and dividing by the product of deposited energy per time  $P_S$  and the elementary charge e,  $G_{fi}$  can be calculated to

$$G_{fi} = \frac{I}{e} \frac{100}{P_S}.$$
 (5.3.1)

Measuring the  $G_{fi}$  value as a function of the electric field strength (figure 5.4) allows to incorporate the measurements into the Onsager theory.

#### 5.3.1 Background Correction

The current induced by the source is expected to be in the range of a few 10 fA. Drifts in the background can deteriorate the accuracy of the measurements. In order to reduce the uncertainty generated by those multiple measurements with and without source are performed. Figure 5.3 shows an example of measurements at U = 4000 V. The upper part of the figure shows currents for 13 measurements with(blue) and 14 measurements without source(green) over a total time span of 15 min. Linear fits to the background current  $I = m_i t + I_{0i}$  right before and after each measurement with source are calculated. This is done for each time the source is positioned at the detector. The linear background is then subtracted from the measurements. A histogram (right panel) of the corrected data for background and measurement with source is generated and than fitted using a Gaussian distribution of shape:

$$g = A \exp\left(\frac{-(I - I_0)^2}{2\sigma^2}\right).$$
 (5.3.2)

from those fits the center for the background  $I_{0b}$  and the measurement points with source  $I_{0s}$  are obtained. Resulting in a measured radiation introduced current of:

$$I(U) = I_{0s} - I_{0b} (5.3.3)$$

#### 5.3.2 Zero Free Ion Yield

Expressing the separation probability in equation 3.2.12 in terms of the number of free charge carrier pairs generated per 100 eV deposited energy gives:

$$G'_{fi}(E,T,\epsilon_r) = G^0_{fi} \left( 1 + \frac{e_0^3}{8\pi\epsilon_0\epsilon_r k_B^2 T^2} E \right) = G^0_{fi}(1+\alpha E)$$
(5.3.4)

here  $G_{fi}^0$  is the free ion yield at 0 electric field strength. Using  $\epsilon_r$  and its error from equation 5.1.3, and the detector temperature of  $T_D = (291 \pm 1)$  K,  $\alpha$  is calculated to be:



**Figure 5.3** – Background correction for current measurement at 4000V. Top panel shows the measurement data and linear fits to the background. In the main panel the background corrected currents are displayed. On the right panel a histogram of the background corrected data, fitted with a Gaussian distribution is displayed. The current is obtained from the Gaussian fit.

$$\alpha(T, \epsilon_r) = \frac{e_0^3}{8\pi\epsilon_0\epsilon_r k_B^2 T^2} = (5.97 \pm 0.102) \cdot 10^{-5} \,\mathrm{cm} \cdot \mathrm{kV}^{-1}$$
(5.3.5)

Onsager theory suggest a linear behavior between electric field strength and free ion yield. Since the measurements show an exponential decrease towards lower field strengths equation 3.2.12 is modified to account for this behavior. It is assumed, that for low electric field strengths e.g. the probability of recombining with an ion of a different ionization event increases due to the larger drift time, resulting in a decreasing current. This reduction can be described by an exponential decay that depends on the position where the ionization occurred. The electron is created at a distance d to the anode. The probability of measuring the electron is written as:



**Figure 5.4** – Free ion yield vs. the electric field strength. The data is fitted with eq. 5.3.9. Data below 400 V is excluded from the fit, because the function did not sufficiently describe them.

$$P = \int_0^h \frac{1}{h} \exp\left(\frac{-t_d}{\tau}\right) dd = \frac{1}{h} \int_0^h \exp\left(-\frac{d}{\mu E \tau}\right) dd$$
(5.3.6)

With the drift time  $t_d$ , the plate distance h, the electron life time  $\tau$  and the electron velocity  $\mu$ . Since radiation takes place from the side and the source is positioned half way between both plates, it is assumed, that ionization takes place evenly distributed between 0 and h. With  $x = \frac{-d}{\mu E \tau}$  equation 5.3.6 can be written as:

$$P = \frac{1}{h} \int_0^{\frac{h}{\mu E \tau}} e^x \frac{\mathrm{d}x}{-\mu E \tau} = \left[\frac{1}{h\mu E \tau} e^x\right]_{\frac{-h}{\mu E \tau}}^0 = \frac{\mu E \tau}{h} \left(1 - \exp\left(\frac{-h}{\mu E \tau}\right)\right)$$
(5.3.7)

with  $t_h = \frac{h}{\mu E}$ , P can be expressed:

parameter	value
au	$(5.99 \pm 0.90) \cdot 10^{-6} \mathrm{s}$
$G_{fi}^0$	$(0.54 \pm 0.01) \frac{e^{-}}{100 eV}$
$\alpha$	$3.79 \pm 0.20 \cdot 10^{-6} \mathrm{cm} \cdot \mathrm{kV}^{-1}$

Table 5.1 – Fit values from figure 5.4

$$P = \frac{\tau}{t_h} \left( 1 - \exp\left(\frac{t_h}{-\tau}\right) \right) = \frac{\tau \mu E}{h} \left( 1 - \exp\left(\frac{h}{-\tau \mu E}\right) \right)$$
(5.3.8)

Multiplying  $P(E, \tau)$  with  $G'_{fi}(E, G^0_{fi}, \alpha)$  yields a form that can describe the behavior at lower electric field strengths:

$$G_{fi}(E,\tau,G_{fi}^{0},\alpha) = G'_{fi} \cdot P = G_{fi}^{0} \cdot (1+\alpha E) \cdot \frac{\tau}{t_h} \left(1 - \exp\left(1 - \frac{-t_h}{\tau}\right)\right)$$
(5.3.9)

Fitting the data from figure 5.4 with equation 5.3.9 using a  $\chi^2$  function and *iminuit*<sup>2</sup> yields the fit parameters from table 5.1. The fit value for  $G_{fi}^0$  is in good agreement with most literature values (see table 5.2). The fit value for  $\alpha$  and the calcutated value from eq. 5.3.5 differ by a factor of 1.5.

#### 5.3.3 Conclusion

Measuring the radiation induced current by alternating between measurements with and without source is a good tool in reducing the error introduced by background drifts. By fitting a Gaussian distribution to the histogram of the background corrected currents the statistical error is reduced. Eq. 5.3.9 describes the data well and the fitted  $G_{fi}^0$  value of  $(0.54 \pm 0.03) e^-/100 eV$  is in agreement with most of the literature (table 5.2). Excluding data below 400 V lowered the reduced  $\chi^2$  from 1.8 to 1.1 and changed the obtained values

<sup>&</sup>lt;sup>2</sup>"iminuit is a Python interface to the MINUIT C++ package. It can be used as a general robust function minimisation method, but is most commonly used for likelihood fits of models to data, and to get model parameter error estimates from likelihood profile analysis." https://iminuit. readthedocs.io/en/latest/

for  $G_{fi}^0$  from 0.5 to 0.54 and for  $\alpha$  from  $4.6 \times 10^{-6} \text{ cm} \cdot \text{kV}^{-1}$  to  $3.8 \times 10^{-6} \text{ cm} \cdot \text{kV}^{-1}$ . The cuts were made because a correct description of the curve is important for the energy calibration done in the next chapter. Comparing the measured  $G_{fi}$  values of this work to the  $G_{fi}$  of other publications (figure 5.5) shows that the measurements that are in best agreement with those of this work (Shinsaka et al. 1993) suggests a  $G_{fi}^0$  value of  $0.47 \text{ e}^-/100 \text{ eV}$ . The difference in the  $G_{fi}^0$  value rises due the different parameterization of the Onsager theory that is used to fit the data. Since the measurements presented in this work is difficult.



**Figure 5.5** – Free ion yield digitized from the publications cited in table 5.2 compared to the measurement in this work. Specially the measurement of Engler et al. 1993 shows good overlap with the data of this work, the constant shift can be explained due to the uncertainty of the source activity. The original plots can be found in appendix I.2-I.5.

paper	$G_{fi}^0$	reference
Lopez et al., 1988	$0.51\pm0.05$	[LMD88]
Engler et al., 1993	$0.61\pm0.01$	[EKV93]
Shinsaka et al., 1993	0.47	$[SNE^{+}93]$
Hara et al., 1997	$0.52\pm0.01$	$[HOH^+98]$
Faradeche et al., 2018	$0.52\pm0.03$	$[FTM^+18]$
This work	$0.54\pm0.03$	

**Table 5.2** – Comparison of  $G_{fi}^0$  value with values from the literature. Within the error margins this work is in agreement with all except the values from [EKV93] and [SNE<sup>+</sup>93]. Figure 5.5 shows the digitized measurements from the cited papers

### 5.4 Pulse Measurements

In addition to the charge calibration described in section 4.3 a good understanding of the signal generation in the detector is required to understand the spectra measured.

#### 5.4.1 Simulated Spectra

Two types of corrections are applied to the events generated in the GATE Monte Carlo simulation, a position dependent correction, that describes the signal amplitude in dependence of the point of interaction within the detector and a Gaussian broadening to account for electronic noise within the measurement setup.

**Position dependent correction** The energy stored in the electric field between anode and cathode of the detector E depends on the applied voltage U and the capacity C of the detector and can be described by:

$$E = \frac{1}{2}CU^2$$
 (5.4.1)

As seen in figure 5.6 negative charge carriers like electrons drift to the anode, while the positive ions drift to the cathode. This drift induces a charge on both capacitor plates which reduces stored energy, resulting in a measurable signal. Compared to the electron velocity  $v^-$ , the drift velocity  $v^+$  of ions in liquids is fairly small ( $\frac{v^-}{v^+} \approx 1000$  [Gru93]).



Figure 5.6 – Electron ion pair is separated due to the electric field.

Figure 5.7 depicts the signal rise due to electron and ion. The signal rise induced by the ion drift is so slow that it can be neglected within the integration time frame, making the electron drift the only contributor to the measured signal. Not measuring the ion signal causes the signal amplitude to be highly dependent on the point where the electron is created. Electrons that start their drift close to the cathode generate a full signal, while those created at the anode do not produce a measurable signal. The energy change of the capacitor due to the drift of charges can be described by the field strength  $\vec{E}$ , the charge q(t) and the drift distance  $d\vec{x}$ :

$$d\left(\frac{1}{2}CU^{2}\right) = d\left(\frac{1}{2}\frac{Q^{2}}{C}\right) = q(t)\vec{E}\,d\vec{x}$$
(5.4.2)

The time dependent charge signal can be described by the Heavyside step function  $\theta(t)$  which is 0 for  $\theta < 0$  and 1 for  $\theta \ge 1$ .:

$$q(t) = -e(\theta(t) - \theta(t - t_0))$$
(5.4.3)

with a drift time  $t = t_0$  and d being the distance between both plates:

$$t_0^- = \frac{d - x_0}{v^-} \tag{5.4.4}$$

$$t_0^+ = \frac{x_0}{v^+}.\tag{5.4.5}$$

for  $v^- \gg v^+$  the induced charge within the integration time frame becomes



**Figure 5.7** – Signal as generated by electron and ion with velocities  $v^-$  and  $v^+$ . For illustration purposes the velocities of electron and ion in this plot are of the same order. In most liquids the electrons are a lot faster than the ions  $\frac{v^-}{v^+} \approx 1000$ . Due to its higher velocity in the medium the signal generated by the electron rises faster. The total charge is just measured if the signal from both electron and ion is considered. The amplitude of each signal depends on the position where electron and ion get separated between the detector plates.

$$\frac{Q \,\mathrm{d}Q}{C} = \begin{cases} -e|\vec{E}|v^- \,\mathrm{d}t & \text{for } 0 \le t \le t_0\\ 0 & \text{else} \end{cases}.$$
(5.4.6)

With  $\frac{Q}{C} = U = |\vec{E}|d$  equation 5.4.1 can be written as:

$$dQ = \begin{cases} -e\frac{v^{-}}{d}dt & \text{for } 0 \le t \le t_0\\ 0 & \text{else} \end{cases}.$$
 (5.4.7)

and integration leads to:

$$Q = -e\frac{v^{-}}{d}t_{0} = -e\frac{v^{-}}{d}\frac{d-x_{0}}{v^{-}} = -e\frac{d-x_{0}}{d} = -e\left(1-\frac{x_{0}}{d}\right).$$
(5.4.8)

Since in the measured spectrum ionization takes place within the entire detector volume this position dependent term has to be taken into account. Figure 5.8c shows how this correction term influences the spectrum of a mono energetic peak at 661 keV. A detector with this design does not provide correct energies for individual events, which is crucial for a PET-detector. A possible way to fix this problem is by adding a Frisch-grid as depicted in figure 3.1 directly in front of the anode. The Frisch-grid separates the ion signal from the electron signal. Electrons created within volume in front of the grid drift towards the anode and pass through the Frisch-grid. Now all electrons that pass through the grid have a defined start point, resulting in a position independent charge signal at the anode. The position of the grid should be chosen in a manner that the volume between grid and cathode is much larger than the volume between anode and Frisch-grid, since ionization in this volume only contributes as noise. A small distance between Frisch-grid and anode also results in a shorter signal with large amplitude, producing a sharper signal peak. To account for electric noise a Gaussian shaped uncertainty is convoluted to the simulation (see figure 5.8b). All corrections are applied to the unbinned data of the MC. Using the Onsager parameterization from Eq. 5.3.9, the fit parameters from table 5.1 and the pre amplifier calibration from 4.3.8 an energy to channel conversion for each voltage is



(a) simulated signal with sharp energy peak at (b) Gaussian uncertainty convoluted with (a) 611 keV



(c) position dependent energy contribution, z position is taken from simulation

(d) convolution of (b) and (c)

Figure 5.8 – This figure shows the corrections that are applied to the simulated data in order for it to be comparable to the measured data.

calculated and then applied to each event with energy  $E_i$  from the MC:

$$K'_{i} = 8.572 \,\frac{\text{channel}}{\text{keV}} \cdot 10^{-3} \cdot E_{i} \cdot G_{fi}\left(\frac{U}{d}, \tau, G_{fi}, \alpha\right) \cdot a \tag{5.4.9}$$

Here U is the applied voltage and d = 0.282 cm is the plate distance. All measurements are acquired with a spectral amplifier amplification of a = 1000. The channels are then corrected to account for the position dependent signal contribution:

$$K_i^* = K_i' \cdot \left(1 - \frac{z_i}{d}\right) \tag{5.4.10}$$

where  $z_i$  is the distance to the cathode. In the last step a Gaussian uncertainty is applied to the data by adding  $r_i$ , a random number generated with a Gaussian normal distribution of width  $\sigma$  as the probability density function:

$$K_i = K_i^* + r_i(\sigma = 192) \tag{5.4.11}$$

 $\sigma = 192$  is obtained from the measured width of the pulser peak. The data is then binned with a binwidth of 100 channels and compared to the measurement at the given voltage.

#### 5.4.2 Measured Spectra

Spectra at different voltages were measured, one example of the raw data is shown in figure 5.9. Similar to the current measurements, the spectra are taken by alternating measurements with and without source. Each spectrum is computed from the sum of 30 2 min spectra totaling to a measurement time of 1 h each for measurements with source and background. A *Canberra 807* pulser is used to generate pulse with  $\approx 2.2 \text{ mV}$  amplitude at  $\omega_p = 50 \text{ Hz}$  that are fed to the test input of the preamplifier. From fitting a Gaussian distribution:



**Figure 5.9** – Measured pulse spectrum at 4000 V. Top panel shows the measured background spectrum. The middle panel shows the spectrum measured with the <sup>137</sup>Cs source. Both spectra consist of 30 measurements, each 2 min of acquisition time, alternating between measurements with and without source. Top and middle panel show the sum over all background and source measurements at the given voltage. A pulser with 50 Hz repetition frequency gives a voltage pulse on the test input of the preamplifier. From a Gaussian fit to the pulser peak a dead time correction can be determined and is used to correct the measured rates. The measured rate from the measurement background rate is subtracted from the measured rate with source, giving the background corrected spectrum at the bottom.



Figure 5.10 – The measured spectrum at 4000 V together with simulated spectrum. For energies above 280 keV the measured and the simulated spectrum are in good agreement. At lower energies the simulation provides a slightly a higher count rate.

$$G = b + A \cdot \exp\left(-\left(\frac{x - x_0}{2\sigma}\right)^2\right) \tag{5.4.12}$$

to the pulser peak the area A is determined. The spectra are then converted to background corrected rate by dividing through the area A and multiplying with the pulser frequency times the binwidth. The measured data is then compared to the simulated spectra. Figure 5.10 shows an example spectrum measured at 4000 V. The corrected simulation and the measurement show good agreement for channels above 1800. Towards lower channel numbers simulation and measurement display a small deviation. This behavior can be observed at all spectra that are measured with a cathode voltage above 800 V (appendix I.6-I.10). This effect might be caused by an uncertainty in the cutoff threshold of the DAQ. This uncertainty could effect the efficiency with which the data is collected towards lower channels.

### 5.4.3 Conclusion

Using the fit to the current measurements and a charge calibrated preamplifier, a voltage dependent energy calibration for the detector was calculated. Spectra at different voltages were measured and compared to the converted simulated spectrum. The slight deviation between simulated and measured spectra towards lower channel numbers might be caused by a reduced collection efficiency towards lower channel numbers, caused by an uncertainty in the cutoff threshold. Repeating the measurements with a lower amplification of the spectral amplifier could reduce the noise and thus lower the uncertainty in the cutoff threshold.

# 6 Summary and Outlook

This work is about the development and testing of a purification and measurement system for organometallic liquids for the use as a PET detector. The main focus in the development of the purification system was put on the purification of the liquids used and implementing techniques developed by former experiments. Due to many changes in the setup during development, data obtained before and after purification is not comparable, hence no data is presented. A slow control system that allows to monitor important parameters of the system and controls the data acquisition was developed and used for all measurements. Automating the measurement system allowed for reproducible measurements with high statics. First measurements with TMS were conducted and comparison to literature shows a good understanding of the data. Measurements using TMBi are planned in the near future and will profit from the knowledge gained during the measurements with TMS. Future detectors that are planned to have a segmented anode and a Frisch-grid are currently under development and are planned to be tested soon.

# I Appendix

I.1 Detector CAD



 $\label{eq:Figure I.1} \textbf{Figure I.1} - \textbf{Technical drawing of the detector cell.}$ 

# I.2 Plots from Literature



**Figure I.3** – [SNE+93]



Figure I.5 - [FTM<sup>+</sup>18]

# I.3 Pulse Measurements



Figure I.6 – Spectra measured between 100 V and 800 V



Figure I.7 – Spectra measured between 900 V and 1600 V



Figure I.8 – Spectra measured between 1700 V and 2400 V



Figure I.9 – Spectra measured between 2500 V and 3200 V



Figure I.10 – Spectra measured between 3300 V and 4000 V

# II Plagiatserklärung der / des Studierenden

Hiermit versichere ich, dass die vorliegende Arbeit über Set Up of a Purification and Measurement System for Organometallic Liquids for a Future PET Detector and Test Measurements with Tetramethylsilaneselbstständig verfasst worden ist, dass keine anderen Quellen und Hilfsmittel als die angegebenen benutzt worden sind und dass die Stellen der Arbeit, die anderen Werken – auch elektronischen Medien – dem Wortlaut oder Sinn nach entnommenen wurden, auf jeden Fall unter Angabe der Quelle als Entlehnung kenntlich gemacht worden sind.

Münster, April 30, 2019

Ich erkläre mich mit einem Abgleich der Arbeit mit anderen Texten zwecks Auffindung von Übereinstimmungen sowie mit einer zu diesem Zweck vorzunehmenden Speicherung der Arbeit in eine Datenbank einverstanden.

Münster, April 30, 2019

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