UNIVERSITY OF ZÜRICH

Emanation Measurement System and GERDA Phase II Calibration

Author: Michael Miloradovic

Supervisors: Prof. Dr. Laura Baudis Giovanni Benato Manuel Walter

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Department of Physics UZH Faculty of Science

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Abstract

The GERDA experiment is based on a germanium detector array directly submerged in a liquid argon cryostat searching for the neutrinoless double beta decay of ⁷⁶Ge at the Laboratori Nazionali del Gran Sasso (LNGS). To ensure the purity of the liquid argon against gas emissions of the detector components, an emanation chamber has been built at the University of Zürich for emanation rate and composition analysis. Component materials such as Tetraphenyl-butadiene (TPB) coated Tetratex and Polytetrafluorethylen (PTFE) are studied with up to 10^{-13} mbar · 1/s rate sensitivity. Argon and xenon gas samples are measured down to 1 ppmv sensitivity.

The germanium diodes in the GERDA experiment are calibrated with ²²⁸Th sources immersed in liquid argon. The data analysis is performed using the Phase II calibration software. The entire quality cut sequence to discard non-physical and background events is studied on data sets from two Phase II integration runs. The rise time range of 200 to 3000 ns is found to be ideal. The trigger range quality cut parameters are optimised to 79.5–81.5 µs.

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Chapter 1

Introduction

According to the Standard Model of particle physics, the universe should be just radiation. The Big Bang would have produced an equal number of matter and anti-matter, eventually leading to the complete annihilation of the perfectly symmetrical early universe [1]. The observed matter dominance of today however is the remnant of a slight asymmetry introduced in its early stages. All the galaxies and stars descend from this small excess of matter – the baryon to photon ratio of $\eta = 6.19 \cdot 10^{-10}$ [2] from the Cosmic Microwave Background (CMB) serves as a relation of surviving to initial matter. The answer to the origin of this asymmetry and thus of our own existence could lie in the special nature of the neutrino [3]. It might be a Majorana fermion and therefore its own anti-particle [4]. This property could be responsible for all the observed phenomena as described in Sections 1.1–1.3. Hence, determining the nature of the neutrino is of fundamental importance in modern physics [5].

The Germanium Detector Array (GERDA) experiment, located underground at the Laboratori Nazionali del Gran Sasso (LNGS), searches for the neutrinoless double beta decay of ⁷⁶Ge with germanium diodes directly submerged in a liquid argon cryostat [6, 7]. The discovery of the decay would not only demonstrate lepton number violation, but also prove that the neutrino has a Majorana mass component [8]. Crucial information on the neutrino mass hierarchy and the absolute values of the neutrino masses could be gained as well. Chapter 2 describes the details of the GERDA experiment, the results from Phase I, as well as the upgrade to Phase II. The key factor of the experiment is the high sensitivity provided by its elaborate shielding and low component radioactivity [6]. Emanations from the constituent materials however could introduce contaminations into this clean environment. An Emanation Measurement System has been built and developed at the University of Zürich for the GERDA Collaboration to identify and measure the emitted impurities of several detector components. Chapter 3 consists of the description of the set-up, the development of the applied methods, and the results of the measurements.

The Phase II Calibration of the GERDA germanium detector array and the implemented software are explained in Chapter 4. The quality cuts and their parameters, that will be used for the calibration and the actual Phase II physics data, are investigated in this thesis on the basis of two full Phase II Integration runs.

1.1 Neutrinos and Oscillation

Neutrinos carry no electric or colour charge and thus do not interact electromagnetically or via the strong force. They are produced in interactions mediated by the weak force such as beta decays. Wolfgang Pauli predicted their existence in 1930 as completely new, undetected elementary particles to maintain the conservation of energy and momentum in the beta decay. [9]

In 1937, Ettore Majorana suggested that the neutrino could be its own antiparticle. The corresponding equation introduces a Majorana mass for the neutrino. In practice, all phenomena in experiments attributed to two related but different particles could thus be due to a single one with two chiralities. [4]

In recent years, the discovery of neutrino oscillations by a large number of different experiments has established the non-zero mass of the neutrinos [10, 11]. The mixing of the neutrino flavour states (ν_e , ν_μ , ν_τ) with the neutrino mass eigenstates (ν_1 , ν_2 , ν_3), in Equation 1.1, through the Pontecorvo-Maki-Nakagawa-Sakata (PMNS) matrix requires them to have a mass [12]. This explanation is an analogy to the Cabibbo-Kobayashi-Maskawa (CKM) matrix for the quark flavour mixing [13–15].

$$\begin{pmatrix} \nu_{e} \\ \nu_{\mu} \\ \nu_{\tau} \end{pmatrix} = \underbrace{\begin{pmatrix} U_{e1} & U_{e2} & U_{e3} \\ U_{\mu 1} & U_{\mu 2} & U_{\mu 3} \\ U_{\tau 1} & U_{\tau 2} & U_{\tau 3} \end{pmatrix}}_{\mathbf{U}_{\text{PMNS}}} \begin{pmatrix} \nu_{1} \\ \nu_{2} \\ \nu_{3} \end{pmatrix}$$
(1.1)

The PMNS matrix, given in Equation 1.2 with $c_{ij} = cos\theta_{ij}$ and $s_{ij} = sin\theta_{ij}$, is parametrised by the three mixing angles θ_{12} , θ_{23} , and θ_{13} . It also contains the Dirac phase factor δ and two Majorana phases α_1 and α_2 for the case of Majorana neutrinos. The Charge Conjugation Parity (CP) symmetry violating nature of these phases has been hypothesized, but not yet observed. [5]

$$\mathbf{U}_{\text{PMNS}} = \begin{pmatrix} c_{12}c_{13} & s_{12}c_{13} & s_{13}e^{-i\delta} \\ -s_{12}c_{23} - c_{12}s_{23}s_{13}e^{i\delta} & c_{12}c_{23} - s_{12}s_{23}s_{13}e^{i\delta} & s_{23}c_{13} \\ s_{12}s_{23} - c_{12}c_{23}s_{13}e^{i\delta} & -c_{12}s_{23} - s_{12}c_{23}s_{13}e^{i\delta} & c_{23}c_{13} \end{pmatrix} \begin{pmatrix} e^{i\alpha_{1}/2} & 0 & 0 \\ 0 & e^{i\alpha_{2}/2} & 0 \\ 0 & 0 & 1 \\ (1.2) \end{pmatrix}$$

Oscillation experiments measure the squared neutrino mass difference Δm_{ij} from the neutrino flavour change probability containing the PMNS matrix. They are however not able to distinguish between the Dirac or Majorana nature of the neutrinos and are not sensitive to the absolute neutrino mass scale. This leaves the problem of the neutrino mass hierarchy, i.e. whether the v_3 neutrino mass eigenstate is heavier or lighter than the other two. The normal hierarchy is given by $m_1 < m_2 << m_3$, whereas the inverted hierarchy ordering is $m_3 << m_1 < m_2$ [5].

1.2 Sterile Neutrinos and the See-saw Mechanism

To explain the occurrence of neutrino oscillations and masses, the Standard Model can be extended to include right-handed neutrinos. This avoids the introduction of a bare mass term and retains renormalisability. In parity violation experiments however, neutrinos were found to be exclusively left-handed [16, 17]. Right-handed neutrinos must therefore be *sterile* singlets and cannot interact weakly [18, 19]. With this extension, left-handed neutrinos gain a Dirac mass term directly from the Higgs mechanism in an analogy to the quark and charged lepton masses [20]. Mass is thereby generated by the Yukawa couplings to the

Higgs field interactions of the left-handed fermions with their right-handed chirals [19]. The result would be however on a similar mass scale as for other fermions and thus several orders of magnitude higher than the predicted three neutrino mass sum from cosmology [5].

A popular theory to explain the unusually low masses of the left-handed neutrinos is the see-saw mechanism. Sterile neutrinos can hold a bare mass term, such as the one predicted by Majorana, due to their weak isospin singlet nature. The left-handed pure states thus receive a Majorana component through mixing with the sterile neutrinos in addition to their Dirac mass term. The neutrino mass matrix formed by these Dirac (M_D) and Majorana (M_N) components is given by [19]:

$$\mathbf{M}_{\nu} = \begin{pmatrix} 0 & M_D \\ M_D^T & M_N \end{pmatrix}$$

$$\Rightarrow \lambda_+ \approx M_N, \quad \lambda_- \approx \frac{M_D^2}{M_N}.$$

$$(1.3)$$

Sterile neutrinos could be very heavy as their mass is completely unknown. In that case, one of the eigenvalues of the neutrino mass matrix (λ_+) would become approximately equal to the large Majorana mass component, while the other (λ_-) would be drastically reduced by it. As a result, the high mass of the sterile neutrinos could be responsible for the very low scale of the light neutrinos through mass mixing [15, 18, 19].

=

The extension of the Standard Model by heavy sterile neutrinos offers another advantage. In the CP violating decays of sterile neutrinos, leptons can be spontaneously generated. The increased number of leptons in comparison to anti-leptons results in a lepton asymmetry in the early universe. A conversion of leptogenesis into baryogensis could then lead to the observed baryon asymmetry, which in turn would be responsible for the universe as we know it [3, 19, 21].

In consequence, exploring the Majorana nature of the neutrinos could help reconcile major cosmological issues of modern physics and lead to the discovery of physics beyond the Standard Model of particle physics. The prime avenue to directly probe this fundamental characteristic and simultaneously obtain information on the absolute neutrino masses is the neutrinoless double beta decay [8].

1.3 Neutrinoless Double Beta Decay

The radioactive isotope ⁷⁶Ge contains 32 protons and 44 neutrons in its nucleus. In order to achieve a more optimal ratio of nucleons, neutrons would need to spontaneously decay into protons under the emission of an electron and an electron anti-neutrino to conserve charge and lepton number. In reality, the nucleus of ⁷⁶As, that would be attained by such a *beta decay*, has a lower binding energy than the original one. The even numbered protons and neutrons of ⁷⁶Ge make the nucleus more stable due to the spin-coupling than the odd-odd numbered ⁷⁶As. This interaction channel is thus energetically forbidden, as shown in Figure 1.1.



FIGURE 1.1: Mass parabola for the nuclei with atomic mass 76. A beta decay (red) from ⁷⁶Ge to ⁷⁶As cannot occur, because the binding energy of the daughter nucleus would be lower than the energy of the parent nucleus. Double beta decay (blue) to ⁷⁶Se is allowed.

A *double beta decay* to ⁷⁶Se is however energetically possible. Two neutrons are simultaneously converted to two protons, emitting two electrons and two electron anti-neutrinos. Figure 1.2a depicts the Feynmann diagram of the described interaction mediated by the weak force. As a higher order process, it has an extremely low decay rate. It is the rarest decay observed in laboratory experiments. The half-life of the double beta decay of ⁷⁶Ge is measured to be $T_{1/2}^{2\nu\beta\beta} = (1.926 \pm 0.094) \cdot 10^{21}$ yr [22].

The idea of a Majorana neutrino gives rise to an additional, theoretical decay channel. A neutrino that is its own anti-particle would allow for the annihilation of the two involved anti-neutrinos. In this *neutrinoless double beta decay*, shown in



FIGURE 1.2: Feynmann diagrams of the double beta decay (a) and the neutrinoless double beta decay (b). The simultaneous decay of two neutrinos into two protons emits – mediated by two charged weak gauge bosons – two electrons and two electron anti-neutrinos. In the neutrinoless case, the two Majorana neutrinos annihilate off-shell.

Figure 1.2b, the decaying nucleus would emit only two electrons back-to-back. The neutrinos are virtual. This interaction violates lepton number conservation by two units [8, 19].

The theoretical half-life for the neutrinoless double beta decay is given by [15]:

$$T_{1/2}^{0\nu\beta\beta} = \frac{1}{G^{0\nu}|M^{0\nu}|^2|m_{\beta\beta}|^2}$$
(1.4)

with the phase space integral $G^{0\nu} = 2.36 \cdot 10^{-15} yr^{-1}$ [23], the nuclear matrix element $|M^{0\nu}| = 2.8-5.2$ [24], and most importantly the effective Majorana mass $|m_{\beta\beta}|$. The neutrino masses of the mass eigenstates (ν_1 , ν_2 , ν_3) are therein connected to the elements of the PMNS lepton mixing matrix. The equation for light neutrino exchange takes the form of [15]

$$|m_{\beta\beta}| = |\sum_{i=1}^{3} U_{ei}^2 m_{\nu_i}|.$$
(1.5)

In this way, the observation of the neutrinoless double beta decay is a direct study of the absolute neutrino mass scale and the Majorana phases in the PMNS matrix. It would be an important step in finally determining the Dirac or Majorana nature of the neutrino. [8]

Chapter 2

The GERDA Experiment

The Germanium Detector Array (GERDA) experiment at the Laboratori Nazionali del Gran Sasso (LNGS) has been built by an international collaboration of 16 institutions for the high sensitivity search of the neutrinoless double beta decay of ⁷⁶Ge. It employs high-purity germanium diodes isotopically enriched in 76 Ge to > 86%, acting simultaneously as the detector and source material. The detection mechanism and the expected signal are described in Section 2.3. The germanium diodes are operated directly submerged in a 64 m³ high purity liquid argon cryostat of 4.2 m diameter. It provides the necessary operational temperature for the detectors as well as radioactive shielding. With the upgrade to Phase II (Section 2.2), a new liquid argon veto system is installed. It consists of a cylindrical volume with 2.2 m height and 0.5 m diameter located at the center of the cryostat. It contains a total of 16 photomultiplier tubes (PMTs) at its ends and a curtain of 810 fibers connected to Silicon photomultipliers (SiPMs). This hybrid veto system can detect wavelength shifted scintillation light produced by background events in the liquid argon. This is explained in detail in Section 2.5. Surrounding the cryostat, a 10 m diameter purified water tank suppresses γ radiation and absorbs neutrons. Cosmic muons are detected by a Cherenkov veto system using 66 PMTs installed inside the tank (Section 2.6). The experiment is located in an underground laboratory at LNGS, 1400 m below the surface to suppress cosmic rays. These shielding and veto layers ensure a substantial reduction of the background and the highest possible sensitivity for the experiment. Figure 2.1 depicts a schematic lateral view of the GERDA experiment. The instrument is described in detail in [6, 7, 25].



FIGURE 2.1: Schematic view of the GERDA experiment. The germanium detector array is lowered from a clean room into the liquid argon cryostat on strings via a lock and suspension system. A Cherenkov veto water tank surrounds the apparatus. Figure from [25].

2.1 Phase I Results

In GERDA Phase I, running from November 2011 to May 2013, 15 kg of semicoaxial and 3 kg of Broad Energy (BEGe) germanium detectors were used on four strings. With the innovative shielding system, a tenfold lower background than previous experiments was obtained: around $1 \cdot 10^{-2}$ cts/(keV · kg · yr) at Q_{ββ} for a total exposure of 21.6 kg · yr. With this progress, it was possible to achieve a new lower limit of $T_{1/2}^{0\nu\beta\beta} > 2.1 \cdot 10^{25}$ yr at a 90% C.L. [26] for the half-life of the neutrinoless double beta decay of ⁷⁶Ge, convincingly excluding a previous claim [27].

2.2 Phase II

For Phase II of the experiment, the active mass of germanium diodes is increased by an additional 20 kg of BEGe detectors on a total of seven strings. With their enhanced pulse shape discrimination (PSD) properties (Section 2.3) and the installation of the liquid argon veto system (Section 2.5), the background can be efficiently reduced by an order of magnitude. The goal is to achieve a median sensitivity of $T_{1/2}^{0\nu\beta\beta} \sim 1.5 \cdot 10^{26}$ yr for a total exposure of 100 kg · yr. Table 2.1 summarises the results of Phase I and the goals of Phase II [7, 25].

	Active mass	BI	Exposure	
Phase	[kg]	[cts/(keV·kg·yr)]	[kg·yr]	$T_{1/2}^{0\nu\beta\beta} [\rm yr]$
I (finished)	15	10^{-2}	21.6	$2.1 \cdot 10^{25} \text{ yr}$
II (expected)	35	10^{-3}	100	$1.4 \cdot 10^{26} \text{ yr}$

TABLE 2.1: GERDA Phase I results and Phase II goals in terms of active detector mass, background index, exposure, and the median sensitivity on the ⁷⁶Ge neutrinoless double beta decay half-life.

2.3 Particle Detection with Germanium Diodes

Charged particles and photons ionise the germanium crystals, producing charge carriers in the conduction band proportional to the incident energy. Under an applied reverse bias voltage on the semiconductor, the electrons and holes drift towards the electrodes. The resulting current can be measured [28].

A double beta decay produces two electrons, which can then be detected by the germanium diodes (Section 1.3). The neutrinos leave the detector without energy deposition. Depending on the decay channel, the measured energy sum of the two electrons appears differently in the spectrum. This is visualised in Figure 2.2. A continuum is observed for the case of two neutrinos additionally carrying away a variable amount of the total energy produced in the decay. If the neutrinos however annihilate off-shell, the maximum energy is fully distributed to the electrons. In the energy sum, the neutrinoless double beta decay would thus be measured as a sharp peak at the total reaction energy $Q_{\beta\beta}$. For ⁷⁶Ge, it is located at 2039 keV [6].

This peak is the signature that the GERDA experiment is searching for. The background in that region needs to be perfectly understood and minimised (Section 2.4). The germanium diodes play the parts of the sources – by being isotopically enriched in ⁷⁶Ge to > 86% – and the detectors simultaneously [7].



FIGURE 2.2: Theoretical spectrum of the summed energy of the detected electrons separating double beta decay (continuum, $2\nu 2\beta$) and neutrinoless double beta decay (peak, $0\nu 2\beta$). The maximum energy peak is only observed for the neutrinoless case and is located at $Q_{\beta\beta} = 2039$ keV.

Thermal excitations introduce electrons from the valence band into the conduction band, which can result in a *leakage current*. This effect needs to be minimised as it mimics a real signal [28]. In GERDA, the germanium diodes are thus kept at temperatures of around 86 K by the liquid argon in the cryostat. Their measurement signals are guided to radio-pure low-noise cryogenic pre-amplifiers that are located approximately 50 cm above the array. On the outside, they are passed into Flash ADC (FADC) channels, where they are converted for digital processing [6, 7, 25].

There are two types of high-purity germanium detectors employed in GERDA: Coaxial and the more advanced Broad Energy Germanium (BEGe) detectors. The first have a central hole almost as long as the detector height. The latter



FIGURE 2.3: A Broad Energy Germanium (BEGe) type detector is shown (left) side with three paths (blue, yellow, green) coming from interactions (circle) in the bulk volume. The produced holes (dashed coloured) drift towards the cathode (red disk) in characteristic trajectories. The electrons (continuous line) move towards the anode (surface of the cylinder, dotted). Charge and current time evolution (right) for the different paths are of identical shape, but differ in rise time. Figures from [29].

have a cylindrical shape with the positive high voltage applied on the anode, covering most of the outer surface. The cathode is a concentric are of size 1 mm² that collects the drifting holes produced in the interactions.

The left side of Figure 2.3 shows the schematic view of such a BEGe detector. Three trajectories are depicted that originate in the bulk volume. Their characteristic paths follow the applied electric field closely. Towards the end, close to the cathode, they all curve down in the same manner. An identical shape is thus observed for the time evolution of the induced signals, on the right of Figure 2.3. The charge and current pulses, which correspond to the three trajectories, are shown on the top and bottom respectively. Far away from the cathode, the induction is much more attenuated and the different drift paths differ only in a variation of their rise time depending on the drift length.

The resulting uniformity of the measured sharp signal peaks can be exploited with the help of Pulse Shape Discrimination (PSD). Single-Site Events (SSEs) – such as double beta decays – and Multiple-Site Events (MSEs), e.g. background γ -rays undergoing multiple Compton scattering inside the detector volume, can be distinguished. MSEs yield separated current peaks, while SSEs create only a single dominant one. This is illustrated in Figure 2.4. Due to this new design, BEGe detectors possess superior SSE to MSE discrimination properties and are thus the primary choice for additional active mass, providing a major background reduction (Section 2.4) [29, 30].



FIGURE 2.4: Illustration of event discrimination: double beta decay ($\beta\beta$) of ⁷⁶Ge are bulk Single-Site Events (SSEs). Single α/β decays are surface events. Multiple-Site Events (MSEs) can come from outside or inside the germanium and interact in multiple locations inside the detector. They can travel into another diode or the liquid argon and can be additionally identified in coincidence or with the produced scintillation light, respectively.

The new Phase II Strings – depicted in Figure 3.23 – each contain these germanium detectors, separated by Polytetrafluorethylen (PTFE) as an insulating spacer (Section 3.8.2). The strings can be lowered from a clean room above the experiment into the liquid argon cryostat via a lock and suspension system [7].

2.4 Experimental Background

All parts of the experiment in the vicinity of the detectors are made of low radioactivity materials by design. The surrounding rock, the purified water tank, and the high purity liquid argon cryostat all shield the experiment from external radiation. To guarantee the lowest possible background, in addition to passive suppression, all events need to be actively identified. To this end, the Cherenkov detector muon veto (Section 2.6), the liquid argon veto (Section 2.5), and the PSD for the germanium detector array are developed [7].

The two neutrino and neutrinoless double beta decays can be distinguished due to their characteristic distribution in the energy spectrum (2.2). All other detected events that deposit energy obscure the theoretical spectrum and are thus considered background that needs to be suppressed. Figure 2.5 shows the Phase I energy spectrum with the main individual contributions identified by their source [25].



FIGURE 2.5: Phase I energy spectrum identifying the background events measured in addition to the theoretical spectrum of Figure 2.2. The green line indicates the blinded window of $Q_{\beta\beta} \pm 20$ keV. The red horizontal bar represents the 200 keV window of the background index determination. Figure from [25].

The energy region below 500 keV is dominated by the beta decay of the long-lived cosmogenic isotope ³⁹Ar. Alpha and beta decays (²²⁶Ra, ⁴²K) are usually events that occur only on the detector surface as SSEs. In and above the continuum of the two neutrino double beta decay – which is dominant in the 500–1800 keV range – the decays of ²¹⁴Bi, ²⁰⁸Tl, ⁴⁰K, ⁴²K (and other isotopes) produce γ -lines. These external emissions of the daughter nuclei of the thorium and uranium decay chains can interact in multiple locations and are thus MSEs (Figure 2.4). The liquid argon veto (Section 2.5) can help in identifying these events if the particles travel outside the germanium at any point. Cosmogenically activated isotopes in germanium, such as ⁶⁰Co, can be detected as MSEs that originate inside the diode. Events that deposit energy in two or more germanium detectors can be vetoed on their coincidence with a quality cut. This is explained in Section 4.2 [25, 30].

Double beta decay events however are a very local phenomenon. In germanium, the range of an electron with 1 MeV kinetic energy is around 1 mm [31]. The two involved electrons thus deposit their energies in the bulk volume of the detector close to the original decay, which makes them SSEs. This is important for the final event identification in order to exclude any other types of events as background [25, 29, 30].



FIGURE 2.6: Representation of the energy windows used in the blind analysis of the GERDA Phase I energy spectra. The size of the blinded window (yellow) is 40 keV. The full energy range (blue) covers 100–7500 keV. Purple cutouts of size 10 keV are excluded due to known γ lines in the background model. Figure from [25].

Since a neutrinoless double beta decay is evidenced by a peak at $Q_{\beta\beta}$ of energy 2039 keV, the primary goal of the experiment is to achieve the lowest possible background particularly in that region. The GERDA Collaboration blinded a range of $Q_{\beta\beta} \pm 20$ keV in Phase I to ensure an unbiased analysis of the background before revealing the region of interest. Figure 2.6 shows a representation of these defined energy windows. The blinded window is displayed in yellow. The background outside the window was used to fix the calibration parameters, the quality cuts, and the background model [25].

Figure 2.7 depicts the best fit background model and the individual contributions in the region of 1930–2190 keV. The background index in the blinded window was estimated by an interpolation of the adjacent domains. This prediction for the background index inside the window was then successfully tested for consistency after unblinding two 15 keV broad sidebands. A region of $Q_{\beta\beta} \pm$ 5 keV remained blinded (the red region labelled ΔE in Figure 2.6) [25].

Similarly, the Phase II background model, quality cuts (Section 4.2), and analysis methods are to be investigated and set to their final values prior to unblinding. If they are carefully chosen, the best prediction for the number of expected events and thus the highest sensitivity for the final result can be achieved [25].



FIGURE 2.7: Phase I background best fit minimum model and individual contributions. The data inside the blinded window (UB, light grey) of size 40 keV is excluded from the fit. Figure from [25].

Section 4.2 concerns itself with the study of the most suitable parameters for the Phase II calibration quality cuts. In order to prevent any bias, the same quality cuts are to be applied for the calibration as for the actual physics data.

2.5 Liquid Argon Veto System

The high purity liquid argon, surrounding the germanium detector array, greatly decreases the γ -ray background from environmental radioactivity with a suppression factor on the order of 10^3 at $Q_{\beta\beta}$ [32]. The cryostat is additionally reinforced with an inner copper shield that reduces the amount of ⁴²K ions coming from ⁴²Ar decays [7].

In Phase II, a cylindrical volume of liquid argon inside the cryostat is used as an active veto. Figure 2.8 shows a schematic illustration of the veto surrounding the germanium detector array. Liquid argon produces scintillation light in response to energy depositions by γ -rays or electrons. The scintillation light wavelength of 128 nm is in the VUV range and thus under the threshold of the radiative transmissibility of PMT windows. Two copper shrouds lined with wavelength shifting reflector foils made from TPB coated Tetratex (Section 3.8.1) are installed around the top and bottom PMT array [33]. The scintillation light



FIGURE 2.8: Schematic view of the liquid argon veto surrounding the germanium detector array (middle) inside the cryostat. PMTs on the top and bottom detect the wavelength shifted scintillation light of background events. Figure from [7].

is thus converted to a higher wavelength before reaching the PMTs. Nine low radioactivity 3" PMTs detect light from the top and seven from the bottom of the array [25, 32, 33].

The central part of the cylinder features a curtain of 810 wavelength shifting fibers, also coated with TPB, that are connected to SiPMs for optimal light detection efficiency. Events observed by this hybrid system in coincidence with the germanium detectors can thus be vetoed. This reduces the background greatly, as external events and even a fraction of MSEs originating in the germanium traverse the liquid argon [25, 32].

2.6 Cherenkov Detector Muon Veto System

The mountains of Gran Sasso provide an overburden of 1400 m deep rock. This corresponds to around 3600 m of water equivalent shielding. The purified water around the experiment moderates and absorbs neutrons and suppresses external γ -radiation. High energy cosmic ray muons however, that arise from π and K decays in the atmosphere, can penetrate the rock and concrete surrounding the experiment and interact with the used materials. The muons traverse the tank with a velocity faster than the speed of light in water. The resulting Cherenkov radiation along their paths is then observed by 40 PMTs of 8″ diameter on the walls of the tank, 20 on the bottom, and 6 beneath the cryostat. Incoming muons from the top and neck of the cryostat are detected by a layer of plastic scintillators above the clean room. The purified water tank and the outside of the cryostat are lined with a wavelength shifting reflector foil that shifts the Cherenkov light from the UV into the optical range [6].



FIGURE 2.9: Inside the empty GERDA Cherenkov muon veto system water tank of 10 m diameter. 66 PMTs on the outer walls detect the Cherenkov light produced by incoming muons. The liquid argon cryostat is located in the center.

In conjunction with the liquid argon veto system, such events can be tagged as muons. The muon detection efficiency for Phase I with only the Cherenkov detector muon veto was around 98% [6]. Germanium detector events in coincidence can be rejected with a muon quality cut. This is described in Section 4.2.

Chapter 3

Emanation Measurement System

In the high sensitivity experiment GERDA, great care is taken to ensure a high purity of the liquid argon [7]. Emanations by the materials used inside introduce contaminations into this clean environment. Different materials have very disparate characteristic emissions and emanation rates that have to be accounted for. It is thus essential to identify the emitted impurities and to measure their emanation rates for any material in planned operation.

For this purpose, an Emanation Measurement System suited to the task has been built and developed at the University of Zürich for the GERDA Collaboration. Mass spectrometry analyses of GERDA component samples have been performed using the installed Residual Gas Analyzer (RGA). These samples include Tetraphenyl-butadiene (TPB) coated Tetratex and Polytetrafluorethylen (PTFE) after various cleaning methods. The system has also been developed to measure samples for other collaborations, such as XENON. PTFE, for example, plays a vital role as a component in the XENON1T experiment [34]. This thesis also includes an analysis of a xenon gas sample from a local xenon reservoir for liquid xenon experiments.

The physics of the mass spectrometer is explained in Section 3.1. The hardware set-up of the Emanation Measurement System is described in Section 3.2. Procedures for loading and filling as well as the different measurement methods for solid and gas samples can be found in Sections 3.3 and 3.4, respectively. A description of the software is given in Section 3.5. Then follows the characterisation of the RGA's built-in electron multiplier, the system volumes, and the blank chamber in Section 3.6. The measurements of an air argon mix and a xenon

sample are reported in Section 3.7. Section 3.8 is about the measurements of the solid samples: a TPB coated Tetratex sample and four PTFE samples treated by different cleaning methods. Finally, Section 3.9 comprises the conclusion and an outlook. The development of the Emanation Measurement System presented here has been achieved under the supervision of Manuel Walter. This chapter thus also includes results gathered in the framework of his PhD thesis [33].

3.1 Mass Spectrometry

The mass spectrometer of the system contains a hot-filament ionizer that creates electrons at a typical energy of 70 eV in a 2 mA emission current. Interactions of these electrons with gas molecules at a pressure lower than 10^{-3} mbar produce positive ions. The rate is relative to the type of molecule, the temperature, and most importantly the gas pressure. The ions are then guided and focused into a quadrupole mass filter, while the rest of the hot-filament electrons move through a source grid into a Bayard-Alpert (B-A) ionisation section, where they generate more ions in interactions with the gas. These ions then hit the collector wire of the B-A gauge, inducing a current proportional to the total gas pressure. Figure 3.1 illustrates both processes [35, 36].

The quadrupole mass filter is made of four rods of alumina and stainless steel with an applied radio frequency voltage. Only ions of a given mass-to-charge ratio can travel through the whole length of the filter, where they are focused on to a Faraday cup (also made from stainless steel and aluminium). The detected signal is thus proportional to the individual partial pressure of the original gas species corresponding to the chosen ion [35, 36].

The measured partial pressure is calibrated to N_2 and needs to be corrected for other gas types, depending on their relative ionisation gauge sensitivity k_r . Furthermore, fragmentation of a single species takes place during ionisation and results in multiple mass peaks with characteristic relative height. This is taken into account when calculating the partial pressures from a fragmentation peak. For both forms of corrections that are implemented into the software (Section 3.5), mainly correction specifications from the manufacturer Extorr are used [35, 37, 38].



FIGURE 3.1: A dual hot-filament ionizer (left: oblique close-up; right: the ionizer is on top of the mass filter, viewed from the side) creates electrons. In interactions with gas molecules, the electrons (red) produce ions (green) that are then guided downwards into a quadrupole mass filter (left: below and not displayed; right: pink). A Faraday cup (right: bottom) detects the filtered ions. The current is proportional to the initial partial pressure. Additional ions (blue) are produced and collected in the B-A gauge proportionally to the total pressure. Figures from [36].



FIGURE 3.2: An electron multiplier, located at the end of the mass filter, can convert the ion current to an amplified electron current before detection. Figure from [39].

3.1.1 Electron Multiplier

The ion current at the end of the mass filter can be converted to an amplified electron current via an electron multiplier located in front of the Faraday detector. This is shown in Figure 3.2. Such an amplified current can be gauged to a desired amplification factor and provides a higher detection sensitivity limit [35, 39].

3.2 Hardware Set-up

The hardware set-up of the Emanation Measurement System is depicted in Figure 3.3. It consists of a cylindrical stainless steel emanation chamber arranged as a cross and is capable of independent pumping of all components: One side is equipped with a CF100 flange for solid sample loading (Section 3.4.1) and a concentric all-metal VCR 1/4 inch valve (V_F) for gas sample filling (Section 3.3.1). The second side is connected by an all-metal CF40 valve (V_2) directly to a CF63 connection leading into a Pfeiffer Vacuum HiCube 80 Turbo vacuum pump stand. The turbo can be disabled for flushing purposes, as discussed in Section 3.4.1.

Through an all-metal CF40 valve (V₄) on the third side of the chamber, an MKS Instruments Granville-Phillips Series 390 Micro-Ion ATM pressure gauge can be connected for a local total pressure readout of the chamber. The data is displayed analogically, then fed to a computer and stored (Section 3.5). A wide range of pressures can be covered due to the combination of a Bayard-Alpert hot-filament (B-A) as an ionisation sensor, a pirani vacuum gauge as a heat-loss sensor, and a piezo vacuum gauge in form of a diaphragm. Figure 3.5 shows the various ranges of the sensors. The B-A hot-filament covers the lower ranges of pressure with a combined region between 10^{-3} to 10^{-2} mbar, leading into the region where the pirani vacuum gauge activates. Starting at around 10 mbar, the piezo vacuum gauge is responsible for the measurements at higher pressures. A *degas* function cleans out any molecular build-up on the surface of the ionizer through heat, generated by higher energy electron emissions of the source filament. The pressure gauge can be pumped independently by the turbo vacuum pump through an all-metal CF40 valve (V₃) [40].



FIGURE 3.3: Photograph of the Emanation Measurement System: Residual Gas Analyzer (RGA), pressure gauge (G) controlled by the black box on the right, cylindrical steel emanation chamber (Chamber) and four visible all-metal CF40 valves (V_{1-4}) connected to the turbo pump (Turbo) by a two-side CF63 cross. The gas bottles in the background can be connected to the VCR 1/4 inch filling valve (V_F) visible in green.

On the last side of the cylindrical emanation chamber, a Hositrad VML 14 gas regulating valve (V_R) allows for a precise regulation of the gas flow from the emanation chamber towards the Extorr Residual Gas Analyzer (RGA) XT200M. This is the core piece of the system. It contains a quadrupole mass spectrometer equipped with a thoriated iridium dual hot-filament that works as described in Section 3.1. A 1–200 amu/q mass range for partial pressure measurements down to around $5 \cdot 10^{-14}$ mbar with a variable scan speed of 0.1–1000 samples/s can thus be achieved by the system. The scan speed is the number of samples measured per second and therefore the inverse of the integration time in a charge measurement of a specific mass. The lower the scan speed, the lower the noise



FIGURE 3.4: Schematic view of the Emanation Measurement System: Residual Gas Analyzer (RGA), pressure gauge (G), turbo pump (Turbo), all-metal CF40 valves (V_{1-4}), VCR 1/4 inch filling valve (V_F), and gas regulating valve (V_R).



FIGURE 3.5: Granville-Phillips Series 390 Micro-Ion ATM pressure gauge sensor switching points: Bayard-Alpert hot-filament (B-A) as an ionisation sensor, a pirani vacuum gauge as a heat-loss sensor, and a piezo vacuum gauge in form of a diaphragm. Units of pressure: **Torr** = 1.33 mbar. Figure from [40].

level of a spectrum. An *AutoZero* functionality subtracts a per sample baseline measurement from the ion signals, eliminating any baseline offset and drift. The time for a complete spectrum is thereby increased and eventually reaches 10 hours for a full 1–200 amu/q measurement at a 0.1 samples/s scan speed. The built-in electron multiplier (Section 3.1) is gauged to an amplification factor of a 1000 and can be enabled for measurements that require the highest possible sensitivity. In Sections 3.7.1 and 3.7.2, such maximum sensitivity mass spectra are shown. [35]

A built-in thoriated iridium hot-filament B-A ionisation gauge and a pirani vacuum gauge offer the possibility to measure the local total pressure inside the RGA. The pirani gauge starts operation at atmospheric pressure level. The B-A hot-filament ionisation gauge then measures the total pressure in the region between 10^{-2} and 10^{-9} mbar. The total pressure measured by the RGA and the pressure gauge are consistent. The RGA also has a *degas* function to clean the ionizer from contaminations. The whole system has been baked to guarantee its purity. A schematic view of the set-up is depicted in Figure 3.4. [35]

3.3 Gas Sample Procedure

In gas sample measurements, the relative composition of the sample is the prime interest. The fractions are expressed in a *parts per million by volume fraction* (ppmv), which corresponds to a fractional value of 10^{-6} in relation to the main component. A procedure to achieve the highest possible sensitivity for gas samples has been developed and is explained in the following. The nomenclature of the schematic in Figure 3.4 is used in the description.

3.3.1 Gas Sample Filling Procedure

The gas bottle or container of a sample is connected to the filling valve V_F in the center of the CF100 flange on the insertion side. The filling tube of the gas bottle or the connection piece of the container needs to be pumped before the gas can be filled into the chamber to avoid contamination. After closing the valves V_1 , V_3 , V_4 , and the gas regulating valve V_R , a very high vacuum is reached in the RGA and the pressure gauge. The turbo pump is then disabled in an automatic

shut down to 0 rpm (1500 rpm at full power). Only the membrane pre-pump is still pumping and can withstand an insertion of atmospheric pressure gas. Upon opening the filling valve V_F , the full filling tube right up to the valve at the gas bottle itself or the connection piece of the container is pumped to vacuum. The turbo pump is then enabled again and starts up to its full power and pump capacity. After a minimum of half an hour, valves V_3 and V_4 can be opened to pump the emanations from the pressure gauge. This condition is maintained until most of the deposited moisture in the chamber is pumped out and the pressure gauge shows at least a comparable total pressure value to the one measured by the RGA. The valves V_1 and V_R can then be opened; the full system, including the connection at the filling valve, is now being pumped. Helium, one of the smallest gas molecules and completely inert, can then be sprayed on the connections of the filling valve V_F . Using a small scan range of 1 to 8 amu/q around helium (4 amu/q) in the RGA and an appropriate scan speed of 144 samples/s, leak tightness can be measured.

Once the system is again at high vacuum, the LabVIEW program that stores the pressure gauge readout history on a computer is enabled (Section 3.5 for more details). Next, the valves V_2 , V_3 , and V_R are closed. Since the pressure gauge is connected to the chamber at this stage, the total pressure can be read-out from its analogue display during the filling. A total pressure of approximately 1 mbar for the emanation chamber is the optimal fill amount. The pirani gauge correction factors that need to be taken into account are mostly linear in that region. Valve V_4 is closed off immediately afterwards, so the pressure gauge has no influence on the following measurements.

3.3.2 Gas Sample Measurement Method

Starting the data taking and storing of the RGA, a medium scan speed of 144 samples/s is initially chosen. This also determines the update speed of the RGA's total pressure readout. The gas regulating valve V_R is then opened a few turns to a marked position depending on the gas sample. Valve V_1 , connecting the RGA to the turbo pump, is kept fully open. This allows for a higher gas flow from the chamber through the RGA towards the pump. Hot-filament effects (Section 3.7.1.1) are thus circumvented. In such an equilibrium, a *constant* total pressure of $5 \cdot 10^{-6}$ mbar can be reached before any saturation effects occur (see

Section 3.4.2). Long, continuous scans with high sensitivity scan speeds, such as 0.1 samples/s, are therefore possible. They reduce the noise floor drastically. This procedure is hence named the *high sensitivity gas method*. With the addition of the built-in electron multiplier, the full potential of the RGA's sensitivity can be utilised. The measurements stay accurate for around six days with continuous pumping until there is not enough gas left in the chamber to reach the desired pressure in the RGA.

The calculation of gas fractions is done via a constant fit to the relative pressure of the gases. An example is given in Figure 3.6 for an air argon mix measurement sample (Section 3.7.1). Several corrections – including fragmentation [35, 37, 38], ionisation [35, 38], and electron multiplier (Section 3.6.3) – have to be taken into account in the process. This is achieved automatically on the software side of the programmed RGA data analysis program, explained in Section 3.5. In this case, a scan speed of 0.1 samples/s and enabled electron multiplier is used to measure the relative pressures with respect to the N₂ part, the main component of air. With this high sensitivity scan speed, each full measurement of the 1–100 amu/q mass range takes approximately five hours, while the whole measurement spans over 20 hours. The results for the air fractions and total mix fractions of this measurement and a verification of the high sensitivity gas method is shown in Section 3.7.1.

3.4 Solid Sample Procedure

The emanation rate is measured in units of mbar $\cdot 1/s$ and describes the characteristic rate of gases that originate from a solid sample. In order to measure these emanation rates, a certain procedure needs to be followed. The solid sample is to be inserted into the chamber with minimal additional contamination.

3.4.1 Solid Sample Loading Procedure

The LabVIEW program for storing the pressure gauge readout history is started. The gas regulating valve V_R , V_2 , and V_3 are closed and have to remain that way, such that the RGA is still pumped throughout the insertion of the solid sample. Argon is supplied by a bottle that is connected via a filling tube to the filling


FIGURE 3.6: Example of the *high sensitivity gas method* measurement on an air argon mix. A constant fit on the relative pressure with respect to the primary component (N_2 in this case) leads to the corresponding gas fraction. 0.1 samples/s scan speed, 1–100 amu/q mass range, total measurement time: 20 hours.

valve V_F . The inside of the chamber is then filled with argon slightly above atmospheric pressure to reduce the amount of moisture (water) introduced during the opening. The valves V_F and V_4 are then closed, sealing off the pressure gauge. The pumping time of the chamber is therefore greatly reduced as only the chamber volume itself is exposed to air at atmospheric pressure. The CF100 flange on the filling side of the emanation chamber can then be opened. During the whole loading procedure, disposable laboratory gloves have to be worn. The copper gasket needs to be kept clean if re-used. The solid sample is then inserted into the chamber. Afterwards, the CF100 flange is carefully closed again. During the opening and closing, the filling tube to the argon bottle does not have to be removed from the filling valve (V_F), speeding up the process.

 V_1 – connecting the RGA to the turbo pump – is then closed. The RGA remains at a very high vacuum throughout the procedure. The turbo pump is disabled in an automatic shut down. This shut down can be timed during the closing of the CF100 flange for minimal downtime. Only the membrane part of the vacuum pump stand is used to pump out the atmospheric pressure after opening V_2 . Five minutes later, the connection to the pressure gauge (V_4) is re-opened. The argon stored in that part of the system flushes through the chamber into the pump. The pressure gauge shows 4.5 mbar total pressure at the start of this mini-flush due to the pressure gauge volume. V_3 is opened; the chamber can be pumped from two sides more efficiently. A threefold argon flush is then carried out to remove gases, moisture and hydrocarbon depositions. Since all of the initially exposed system is made of stainless steel, the flushing is very effective. A flush involves filling up the chamber with argon during pumping, until 300 mbar is reached. As soon as the pressure drops down to 4.5 mbar again, the next flush is initiated. Argon is chosen as a flushing agent, because it can be observed as an independent peak in the mass spectrum at 40 amu/q. As an introduced contamination, it is therefore not superimposed on any common emanation substance. After the full flushing procedure, the turbo pump is restarted. All sections exposed to the flushing are then pumped until a high vaccuum is reached again. This takes around an hour. The remaining system, including the RGA, is then also pumped. The RGA is never exposed to the atmosphere or any contaminations at any point in time. A leak check with helium is then carried out. The RGA is set to a small scan range of 1 to 8 amu/q around helium and an appropriate scan speed of 144 samples/s. Helium is then sprayed on the CF100 flange to guarantee its leak tightness. Even a very small leak could be detected by the RGA in this manner. The Emanation Measurement System is then pumped to around 10^{-8} mbar (if attainable depending on the sample) before any measurements.

3.4.2 Solid Sample Measurement Methods

In order to determine the emanation rates for various gas species of a solid sample, a specific measurement procedure has been developed and verified (see Section 3.7.1.1). It is explained in the following.

3.4.2.1 Accumulation Method

After the loading procedure and a few hours of pumping, the RGA and the pressure gauge are to be sealed off from the chamber using the valves V_R and V_4 , respectively. When closing valve V_2 , is no longer pumped and the emanations from the solid sample can be accumulated inside for a higher sensitivity. This is called the *accumulation method*. Approximately 10 minutes before the desired level is reached, the RGA is employed to start data taking (Section 3.5.1). A

medium scan speed of 144 samples/s ensures the best mixture of speed and sensitivity for such a measurement. The electron multiplier is disabled throughout to avoid damaging the RGA when exposed to increased pressure. With 6 minutes left, valve V_1 – connecting the pump to the RGA – is also closed. This is the time it takes to fit and calculate the independent emanation rates of the RGA itself and the corresponding partial pressures p_{RGA} . The gas regulating valve (V_R) connecting the chamber to the RGA is then opened. Typically, the maximum total pressure in the RGA, which at the same time yields the highest level of sensitivity, is $5 \cdot 10^{-6}$ mbar. At higher pressures, saturation effects may occur. The emanation rate of a specific gas species can be calculated using the partial pressure p_{RGA} at the time of the opening of the gas regulating valve and the measured partial pressure p_{opened} afterwards. This removes any influence of the emanation rate of the RGA on the final result. The isothermal ideal gas law gives the total emanation as

$$p_{Emanation} \cdot V_{Chamber} = (V_{RGA} + V_{Chamber}) \cdot p_{opened} - V_{RGA} \cdot p_{RGA}.$$
(3.1)

The volumes of the RGA (V_{RGA}) and the emanation chamber ($V_{Chamber}$) are determined in Section 3.6.1.



FIGURE 3.7: Example of the *accumulation method* on a blank chamber measurement. Exponential fit (purple, zoomed in) to the CO₂ partial pressure curve (grey) for p_{opened} extrapolation. N₂/CO (black) visible as a downgoing example.

Figure 3.7 shows an example of the accumulation method carried out to calculate the emanation rate of CO_2 in the blank chamber (Section 3.6.4). The software for the RGA data readout and analysis is explained in Section 3.5. A linear fit is used to extrapolate the partial pressure p_{RGA} coming from the RGA at the time of opening the gas regulating valve. This causes a CO_2 increase and a subsequent maximum at scan 17. An exponential function is fitted to the CO_2 gas curve to extrapolate the value p_{opened} right after opening, including an estimation on the uncertainty. The decrease in partial pressure stems from a very slight getter pump effect of the RGA and the highly pumped system. The curve can also go down, with p_{opened} being thus lower (e.g. N_2/CO in Figure 3.7) depending on the specific local partial pressures in the chamber and the RGA arising from the respective local emanation rates.

The accumulation method is used for the emanation rate determination of a TPB coated Tetratex sample (Section 3.8.1) and the blank chamber measurement (Section 3.6.4). In Section 3.7.1.1, the accumulation method is furthermore verified using a gas sample with known composition.

3.4.2.2 Pressure Rise Method

For a solid sample with a much higher total emanation rate the procedure has to be slightly adapted. This is observed by a fast rise to the operational total pressure of $5 \cdot 10^{-6}$ mbar and beyond in only a few minutes. The accumulation of emanations inside the chamber is therefore not possible. Data taking is initiated with the RGA and the chamber still pumped (V₁ and V₂ open) and the leak valve fully open. When closing the valves V₁ and V₂, the partial pressures start to rise and traverse the optimal measurement region sufficiently long to later determine the emanation rates.

Figure 3.8 shows an example of this *pressure rise method* in the emanation rate calculation of N_2/CO from an ethanol cleaned PTFE sample (Section 3.8.2). The rise occurs after closing the valves to the turbo pump at around scan 15. A linear fit to the data points in that region of the partial pressure curve is used for the emanation rate calculation. The total pressure keeps ascending outside the zoomed window. At around $8 \cdot 10^{-6}$ mbar, above the measurement region, a clear saturation of the partial pressures is visible. It is therefore essential to measure inside the linear region of the partial pressures provided by the pressure



FIGURE 3.8: Example of the *pressure rise method* on the measurement of a PTFE sample cleaned with ethanol. A linear fit (red) to the N₂/CO linear region of the partial pressure curve (black) is shown. This is used for the calculation of the emanation rate. When the total pressure (brown) rises above around $8 \cdot 10^{-6}$ mbar, saturation is observed.

rise method. The RGA emanation rates and any getter effects are negligible for such a high total emanation rate.

3.5 Software System

The pressure gauge and the RGA are both connected to a computer in order to save their measured data with two different software programs. Governed by a LabVIEW program, the total pressure data of the pressure gauge is saved as a .LVM (LabVIEW Measurement) data file. The RGA's parameters and functions are controlled via the Extorr program *VacuumPlus*. It reads in the signal and displays the measured total pressure of the RGA as well as the current mass scan position. This is useful as a rough guideline when performing the measurement methods and helium leak checks (Sections 3.3.2 and 3.4.2). Data saving is done with a separate Extorr pipe writing program that stores the total and partial pressures for each mass point in a raw .XML (Extensible Markup Language) format. Headers for each full scan contain the relevant parameters such as scan range and scan speed. Measurement time and electron multiplier activity are however not saved. Future plans in this regard are discussed in Section 3.5.3.

3.5.1 RGA Data Analysis Software

A software system has been developed to display the saved raw RGA partial pressure data as full mass spectra and the subsequent evolution of specified component peaks for partial pressure, relative pressure, and total pressure. The analysis program is coded in C++ utilising CERN's ROOT classes. The creation of a class to store spectra and corresponding header informations from the .XML files read-in was done by Manuel Walter as part of his PhD thesis, while the peak identification and subsequent partial pressure evolution visualisation have been extended in the framework of this thesis. Additionally, fragmentation [35, 37, 38] and ionisation [35, 38] corrections of the gas species, that are automatically identified by their primary mass peaks, have been implemented into the program. These corrections (Section 3.1) are made according to Extorr specifications. The possibility of a simultaneous display of an arbitrary number of single full mass spectra has been added in form of a bash and terminal command option (-s) that includes the capability of electron multiplier correction (Section 3.6.3) where needed (-sEM). Examples of this are the mass spectra depicted in Section 3.7 and 3.8. There is also a fitting option (-f) for a chosen mass peak corresponding to a partial pressure curve, e.g. in Figure 3.7 and 3.8. A dedicated repository has been created and is maintained on the University of Zürich GERDA group github for easy deployment on any system.

3.5.2 Pressure Gauge Software

The pressure gauge raw data read-in from the .LVM and the visualisation of the total pressure graph have been also been programmed in C++ using ROOT classes as part of this thesis. Examples of the result are Figure 3.10 and 3.19. The program works by reading in the time and total pressure data through file stream functions, converting and storing them into ROOT class vectors. The graph is then displayed onto a canvas and can be saved from there. The program is accessible through the same github respository.

3.5.3 Software Outlook

In correspondence with the company Extorr, an interest in a time stamp inside the header of each .XML spectrum run, that is provided by the RGA, was expressed. At the time of writing, Extorr has now released and sent an update of their software featuring a date and time stamp in milliseconds. In the near future, the RGA data analysis program can thus be modified to reflect partial pressure evolution over units of time instead of scan number.

3.6 Characterisation

Prior to examining any gas or solid samples, the exact chamber volumes for the rate calculations have to be determined, the behaviour of the electron multiplier is investigated, and the blank chamber spectrum and emanation rates need to be studied.

3.6.1 Volume Determination

As mentioned in Section 3.4.2, the calculation of an emanation rate in units of mbar $\cdot 1/s$ from a partial pressure measurement requires the knowledge of all volumes of the Emanation Measurement System. The volume of the raw chamber, the valves V₂ and V₄, and the gas regulating valve have been determined from three-dimensional computer-aided drafting (3D CAD) object files of the technical drawings. They are displayed in Figure 3.9. V_{Chamber} is the combination of their volumes.

The volume V_{RGA} consists of the RGA, the volume on the closed side of valve V_1 , and the part of the opened gas regulating valve that is not included in V_C hamber. V_G includes the volume of the pressure gauge cross, the closed side of valve V_3 , and the opened valve V_4 . Both V_{RGA} and V_G have to be determined by experiment.

The pressure gauge is employed in this measurement with an accuracy of three digits for high pressures. In the range from around 10 mbar up to the region of interest around 1000 mbar, it operates in the gas type insensitive piezo vacuum



FIGURE 3.9: 3D models of the emanation chamber (left), an open (middle: purple volume) and closed valve (right: green volume) used for the volume determination of V_C hamber.



FIGURE 3.10: Volume determination of V_{RGA} and V_G by experiment utilising the pressure gauge. Region 1: 1000 mbar total pressure. Region 2: pumped to vacuum. Region 3: Pressure gauge is refilled with gas from the chamber alone leads to 887 mbar in and V_G . Opening the RGA to the constant gas amount, V_{RGA} can be found from region 4: 791 mbar.

gauge mode (Figure 3.5). All valves are closed except V₄, the connecting piece from the emanation chamber to the pressure gauge. Argon is filled into the chamber and the pressure gauge until 1000 mbar total pressure is reached. This corresponds to region 1 in Figure 3.10, which has been created from the digital pressure gauge readout (Section 3.5.2) during the experiment. Only the pressure gauge is then pumped by closing V₄ and opening V₃, which is made visible by the region 2 going down to 0 mbar. After some time, the gauge is refilled with gas from the chamber by closing first V₃ and then opening V₄. This corresponds to region 3. 887 mbar total pressure in both sections is reached from the previously stored amount coming from the emanation chamber alone. From this, V_G can be calculated. Afterwards, the gas regulating valve is fully opened and the gas can surge into the RGA volume. The resulting total pressure of 791 mbar in region 4 yields V_{RGA}. For both of these calculations, the isothermal ideal gas law is applied. Table 3.1 lists of the determined volumes of the Emanation Measurement System.

Component	Volume [1]
V _{Chamber}	2.228
V _{RGA}	0.305
V _G	0.284

TABLE 3.1: Volume determination results.

3.6.2 CO₂ and CO Reduction through Baking

In initial measurements, turning the gas regulating valve during measurements altered the emanation rates of CO_2 and CO visibly through a change of slope. A heating system was installed around the gas regulating valve for constant baking of 200 °C over two days (switched off at night). After baking, the CO and especially the CO_2 emanation rates of the blank chamber have been much lower. The amount of H_2 has increased as expected, as it is mostly emanated from the steel of the chamber, which was also exposed to heat during the baking. The total pressure has been lowered as well, allowing for a higher measurement sensitivity before any saturation effects occur (Section 3.4.2). Most importantly, turning the gas regulating valve has shown no influence on the blank emanation rates after the baking. Both the RGA and the pressure gauge have been degassed twice

right after baking to clean the ionizers as described in Section 3.2. Figure 3.11 depicts a measurement before and after baking.



FIGURE 3.11: Measurement before (a) and after (b) baking. The H_2 (red) rate is increased. The CO_2 (grey) rate is strongly decreased, the CO (black) rate and the total pressure (brown, uncorrected) curve are lower as well.

3.6.3 Electron Multiplier Characterisation

For high sensitivity measurements, the RGA is equipped with an electron multiplier that has been gauged to a factor 1000 for N_2 located at 28 amu/q. It can thus increase the ultimate sensitivity of the mass spectrometer. The physics of the electron multiplier is described in Section 3.1.1. For partial pressure measurements other than nitrogen, an electron multiplier correction factor needs to be calculated depending on the ion mass of the chosen gas. For this purpose, two separate measurements are performed: One for the blank chamber (Section 3.6.4) and one for a TPB coated Tetratex sample (Section 3.8.1). Five complete scans of the blank chamber without electron multiplication are taken for example while pumping. The electron multiplier is then enabled in the same run for eight scans and is finally disabled again for five more scans. This procedure can be seen in Figure 3.12. The total pressure remains constant throughout the measurement, because the B-A ionisation gauge is unaffected by the electron multiplier (Section 3.1.1).

The relation of the measured partial pressure averages over these scans for an enabled and a disabled electron multiplier gives the amplification factor depending on the ion mass. With the blank chamber and the TPB coated Tetratex measurements, data points in the range of 2–84 amu/q can be obtained. This allows for the creation of a correction curve that extends to even higher masses



FIGURE 3.12: The electron multiplier correction measurement for the blank chamber. From 0 to 5 scans, the electron multiplier is disabled while data taking. Between scan number 6 and 13, the electron multiplier is enabled and then disabled again from 14 onward. Displayed partial pressures in this figure are logically not electron multiplier corrected.



FIGURE 3.13: The amplification factor as a function of the mass from the combined blank chamber (as seen in Figure 3.12) and TPB coated Tetratex sample measurements. Exponential fits to the data points with (red) and without (blue) H_2 outliers. All electron multiplier corrections use the blue curve.

very well. Figure 3.13 depicts this electron multiplier correction curve as an exponential fit to the data points. The red curve shows that H_2 is an outlier and distorts the curve. It is possible that hydrogen is additionally released by the electron multiplier when enabled. In high sensitivity measurements, the H_2 emanations are dominated by the steel chamber (Sections 3.7 and 3.6.4) and thus not relevant in conjunction with the electron multiplier. Therefore, the blue curve – without H_2 – describes the other corrections better and well into the upper ranges. The equation of the blue correction curve with the parameters in Table 3.2 for all electron multiplier corrections is:

$$f(x) = e^{a+b \cdot x}.$$
(3.2)
Value Uncertainty
a 7.46 ± 3.68 \cdot 10^{-2}
b -0.02 ± 1.48 \cdot 10^{-3}

TABLE 3.2: Electron multiplier correction curve parameters and uncertainty.

3.6.4 Blank Chamber Characterisation

Figure 3.14 shows the high sensitivity mass spectrum of the blank chamber while pumping, amplified by a factor 1000 with the electron multiplier. In such a mass spectrum, the individual partial pressures for every measured ion mass-to-charge ratio are displayed. The dissociation of molecular ions forms fragments that are visible as a characteristic pattern in the mass spectrum. It is therefore possible to deduce the partial pressures of the original gases with the knowledge of all involved fragmentation patterns [35, 37].

The dominant peaks in descending order are H_2O (18 amu/q) from moisture, H_2 (2 amu/q) emanated from the stainless steel chamber, O (16 amu/q), N_2/CO (28 amu/q), and CO_2 (44 amu/q) as air residues and emanations from RGA components. Nitrogen and carbon-monoxide share the same mass peak at 28 amu/q and are as a consequence not easily distinguished. Cleaning and flushing procedures (Section 3.4.1) are thus usually performed using argon at an independent mass peak of 40 amu/q instead of nitrogen for better identification. The peak at 30 amu/q in the spectrum is composed of ethane (C_2H_6) and possibly shared with nitric oxide (NO) as a fragment of nitric acid (HNO₃), a vacuum

parts cleaning agent. All mass spectra representing partial pressure in relation to mass are corrected for electron multiplier effects, where necessary (Section 3.6.3). Fragmentation and ionisation effects are taken into account for all graphs that represent a partial pressure or relative pressure trend over scan numbers or time.



FIGURE 3.14: Blank chamber mass spectrum at 10 samples/s with a mass range of 1–46 amu/q. Electron multiplier corrected.

The calculation of the emanation rates of the different gases, arising from the blank chamber components, requires the knowledge of the involved volumes (Section 3.6.1), the measurement of the RGA emanations, and the determination of the chamber emanation peaks produced by the opening of the gas regulating valve. This is described in Section 3.4.2.

Nine full emanation rate measurements are performed for the blank chamber with the accumulation method. They show a variation in the measured emanation rates for different gas types over a total time of 957 minutes. In Figure 3.15 on the top, H_2O curves down fast to a constant value. CO_2 , below, on the other hand, shows a larger uncertainty around an average value. The H_2O adsorption to the surface of the chamber could eventually reach an equilibrium state with the pumping out of the water vapour. This could be responsible for the observed shape. The remaining amount of moisture is harder to get rid of. Gases, such as CO_2 however, might be stored inside the volumes of the system components and are thus more abundantly available. H_2 is emanated by the stainless steel walls, but also created in H_2O fragmentation and is therefore dependent on the



FIGURE 3.15: Blank chamber emanation rates for H_2O (top) and CO_2 (bottom) over nine independent measurements (blue triangles). H_2O depicts a trend-like behaviour, while CO_2 has a large uncertainty.

Gas	Mean $[10^{-12} \text{ mbar} \cdot l/s]$	$\sigma [10^{-12} \text{ mbar} \cdot l/s]$
H ₂	144.06	124.73
CO ₂	2.61	1.37
H ₂ O	0.73	0.26
N ₂ /CO	0.61	0.24

TABLE 3.3: Mean and standard deviation σ of the nine blank chamber emanation rate measurements of Figure 3.15.

moisture level. Regardless of the interpretation, the conservative approach is used to estimate the uncertainty. The mean and the standard deviation (σ) of all nine measurements for each gas type are summarised in Table 3.3. The low emanation rates of the chamber allow for up to 10^{-13} mbar · l/ rate sensitivity.

This is confirmed in Section 3.8.1 on the TPB coated Tetratex sample, where the blank chamber emanation rates are determined before and after the sample measurements. The total time of 957 minutes for the nine measurements here does not correspond to the pumping time in a direct blank chamber to solid sample comparison. The pumping time is the time passed between starting the pump down, after the chamber has been exposed to the atmosphere, and the accumulation phase of the measurement. The emanation rates in Table 3.3 are low, because the chamber has been pumped for several days. Variations of the emanation rates also depend on the last measured sample before the current one. The blank chamber emanation rates for comparison with the TPB coated Tetratex sample are measured at the same pumping time as the sample itself. This procedure should always be followed for samples with characteristic emanation rates only around an order of magnitude above the blank chamber rates. In this way, the background levels that undergo fluctuations can be judged accurately.

3.7 Gas Measurements

3.7.1 Air Argon Mix

A measurement with a known composition of gases not only provides a better understanding of the RGA behaviour in gas sample measurements, but also serves as a verification of all measurement and analysis methods. An air argon combination offers the possibility of comparison to an expected fraction of gases in dry air – nitrogen (N₂, fragmentation into N), oxygen (O₂, O), carbondioxide (CO₂, CO), as well as argon (Ar) and krypton (Kr) – while possessing a well-defined component peak at 40 amu/q for argon. In this air argon mix measurement, the main chamber is filled with argon 6.0 (> 99,9999% purity) from a gas bottle – following the procedure introduced in Section 3.3.1 – until the pressure gauge of the chamber displays 0.328 mbar. In that pressure regime, the pressure gauge employs its pirani gauge – as explained in Section 3.2 – which acts as a heat-loss sensor to detect the total pressure (Figure 3.5). Its detection is gas type dependent and pirani correction factors (Section 3.3.1) need to be taken into account in the later calculations. The bottle is then removed and a Drierite Laboratory Air and Gas drying unit is connected. 0.682 mbar dry air is added into the mix. The total amount of each component is therefore known.



FIGURE 3.16: Air argon mix mass spectrum (black) in comparison to a prior blank chamber spectrum (red) both using 0.1 samples/s scan speed with a mass range of 1–100 amu/q. Electron multiplier corrected. Total measurement time: 10 hours.

The mass spectrum of the air argon mix sample is depicted in Figure 3.16. It is taken with the high sensitivity gas method (Section 3.3.2) using a scan speed of 0.1 samples/s over a mass range of 1–100 amu/q. Alongside it is a blank chamber spectrum with the same settings. The electron multiplier is enabled for both spectra. The primary peaks are argon (40 amu/q, doubly-ionised at 20 amu/q) and air with its main components consisting of N₂/CO (28 amu/q), O₂ (32 amu/q), O (16 amu/q), N (14 amu/q), and CO₂ (44 amu/q). Krypton (84 amu/q), as expected only a small fraction of air, is also visible. H₂O (18 amu/q) and its fragment OH (17 amu/q) stem from the filling of the chamber with outside humid air even through the drying unit. A large part of the water however is moisture on the surfaces and accumulates in the RGA and chamber over time. It is harder to pump out than other gases and does not directly correspond to an input from the sample itself. This is visualised

by the red peaks from the background at 17–18 amu/q being rather close to the sample's in black. The shoulders on the left side of the highest peaks in the spectrum appear only for a very high sensitivity that minimises the noise floor. They are RGA artefacts, which do not compromise any measurements.

Gas fraction calculations are done using the measured mean relative pressures over time, as described in Section 3.3.2. Figure 3.6 is the relative pressure graph of the air argon mix taken in a measurement lasting 20 hours. Air fractions are determined in relation to N_2 as a primary component. This results in measured total and air gas fractions, as well as calculated expected fractions for both. They are given in ppmv relative to the total air amount and the total air argon mix, respectively. For argon, this expected fraction comes from the directly filled argon component and a percentage amount from the filled air. O_2 and Krypton however are assumed to arise only from the air addition. The obtained values are listed in Table 3.4.

	Total fraction		Air fr		
Gas	measured	expected	measured	expected	dev.
Ar	47.0%	45.5%	85.0%	82.7%	2.8%
O ₂	9.6%	11.5%	17.5%	20.9%	16.3%
Kr	0.7 ppmv	0.63 ppmv	1.27 ppmv	1.14 ppmv	11.4%

TABLE 3.4: Air argon mix gas fractions.

The absolute percentage deviation of the measured fractions from the expected is on average 10.4% as an estimate of the measurement uncertainty. Similarly, by fragmentation pattern calculation, the N₂ peak has a 10.0% uncertainty on its air fraction recalculation: 70.2% measured compared to 78.0% expected. These results establish the high sensitivity gas method as accurate down to around 1 ppmv for gas samples. This depends of course on the fragmentation pattern and ionisation correction of the chosen gas species.

3.7.1.1 Solid Sample Measurement Method Verification

All gas sample measurements of the air argon mix and the xenon sample (Section 3.7.2) are performed using the high sensitivity gas method to provide stable conditions for relative pressure readings. But the solid sample measurement techniques (Section 3.4.2) – applied in the measurements of the blank chamber,

the TPB coated TTX sample (Section 3.8.1), and the PTFE samples (Section 3.8.2) – can also be tested and verified with the known composition of the air argon mix.



FIGURE 3.17: Air argon mix measurement with the accumulation method. RGA emanation over 7 minutes (left, before peak) and gas regulating valve opening (start of peak). O_2 (blue) stays low without a peak. The scan speed is 144 samples/s.

Figure 3.17 demonstrates the accumulation method in conjunction with the air argon mix. It provides a stable fit for the RGA emanation that is then subtracted to obtain the real emanation rates. As expected from the filled composition, argon and N₂ are dominant. H₂ and H₂O, mostly emanated from the chamber and RGA, experience a dip in partial pressure when opening the gas regulating valve due to a wallpapering effect. The residual gases located on the surfaces are immediately coated over by several monolayers of argon and nitrogen, effectively keeping them from reaching the RGA. Due to the nature of the method, a much higher scan speed of 144 samples/s is required and therefore trace components such as CO₂ or krypton cannot be measured. The resulting argon gas fractions, listed in Table 3.5, can be recalculated from the relation to the N₂ as the primary air component.

	Total fraction		Air fra		
Gas	measured	expected	measured	expected	dev.
Ar	54.9%	45.5%	99.8%	82.7%	20.7%

TABLE 3.5: Air argon mix gas fractions with accumulation method.

All major gas components can be measured with the accumulation method except for O_2 . It stays at an almost background level. This phenomenon also appears for the blank chamber and the TPB coated TTX sample and is only encountered where the emanation rates are very low. The pressure rise method on the other hand – used for high emanation rates such as the PTFE samples – and the high sensitivity gas method – with its high gas flow through the RGA – give very accurate O_2 readings.



FIGURE 3.18: The hot-filament effect on the O_2 (blue) is visible at around scan number 144, where a strong decrease happens. CO_2 (grey) and C (magenta) rise up at that same time. The time required for one scan is approximately 36 minutes.

The hot-filament of an ion gauge could promote reactions with the oxygen and could lead to chemical compounds with different mass. The O_2 peak at 32 amu/q is thereby drastically reduced. After all the other air argon mix measurements, this possibility is studied in the following in a dedicated measurement. In the prior ones, the pressure gauge indicated the total pressure of 1 mbar during the filling and was then closed off from the emanation chamber via valve V_4 in order to remove any additional influences. This pressure was sufficient for the various tests with approximately six days of constant RGA pumping when performing the high sensitivity gas method. 12 days after the initial filling of the air argon mix, the emanation chamber was pumped through valve V_2 and closed off again. The 1 mbar of total pressure stored in the pressure gauge section is then released into the blank chamber resulting in a total pressure of 0.12 mbar. Data are taken

through the night with decreasing pressures and a scan speed of 1 sample/s. Now, valve V_4 remains open to investigate the impact of the pressure gauge on the residual gases. Figure 3.18 shows that around scan number 144, the O_2 partial pressure starts decreasing strongly, while the CO_2 rises up. This could be caused by the ion gauge of the pressure gauge turning on and enabling its hot-filament.



FIGURE 3.19: The pressure gauge readout curve of the Figure 3.18 measurement shows the intended total pressure decline. At 1066 seconds, the pressure gauge sensor switch happens.

The scan number 144 of Figure 3.18 corresponds to the time 1066 seconds of Figure 3.19, the total pressure curve of the pressure gauge during the measurement. The start of the O_2 increase thus coincides with the time where the total pressure curve experiences an apparent trend change and becomes less *fuzzy*, suggesting a sensor switch of the pressure gauge. This happens when the total pressure goes into the low 10^{-2} mbar region. Figure 3.5, introduced in Section 3.2, indicates a sensor switch of the pressure gauge starting in that same region of around 10^{-2} mbar. The then activated ionisation sensor is a B-A hot-filament that seems to strongly reduce the amount of oxygen detected. The dual hot-filament or the B-A hot-filament of the RGA could also be capable of the same effect, especially if the emanated amount is low in the first place. For much higher emanations however, where the pressure rise method is used, the O_2 amount can be measured very well (Section 3.8.2). The expected argon total fraction in Table 3.5 would be higher for an RGA volume devoid of O_2 . This

would reduce the deviation of the measured fractions relative to the expected ones down to 7%.

Molecules that are not fragments of O_2 or CO_2 are unaffected by the hot-filament phenomenon (Figure 3.17). It is therefore possible to determine the main component gas fractions of a gas sample with the same measurement technique as the one for a solid sample, serving as a verification of the method (Table 3.5). For very accurate gas fractions, including N_2 , O_2 , argon, and even trace amounts such as krypton, the high sensitivity gas method is recommended.

3.7.2 Xenon Sample

With the expertise from the sample with known composition, a xenon sample with unknown impurities is analysed. A small container with the sample inside is available. The filling procedure explained in Section 3.3.1 then needs to be followed.



FIGURE 3.20: Xenon sample mass spectrum (black) with a mass range of 1-200 amu/q in comparison to a blank chamber spectrum (red) taken just before with a range of 1-100 amu/q. The scan speed is 0.1 samples/s for both. Electron multiplier correction is applied. Total measurement time: 15 hours.

The high sensitivity gas method with 0.1 samples/s scan speed and electron multiplication is exploited to achieve the mass spectrum of the xenon sample depicted in Figure 3.20. With the full range of 1-200 amu/q, all the constituent

gases can be seen at a glance. The main fragmentation peaks arise from the primary component, the xenon. The biggest peaks are located in descending order at 132, 129, 131, 134, 136, 130, and 128 amu/q. The doubly-ionised peaks of the xenon are found at 66, 67, 68, 65, 64, and 60 amu/q.

Peaks that are not derived from the xenon are also visible, these include: H_2O (18 amu/q), OH (17 amu/q), N_2/CO (28 amu/q), N (14 amu/q), O_2 (32 amu/q), O (16 amu/q), CO_2 (44 amu/q), and argon (40 amu/q). Ethane (C_2H_6), possibly shared with nitric oxide (NO), constitutes the 30 amu/q mass peak, as explained in Section 3.6.4. The shoulders at around 110 amu/q and 60 amu/q are RGA artefacts that appear only for the highest sensitivity.

Evidently, there are impurities from gases not inherent to the xenon. These impurities stem from a leak to a local xenon reservoir, used for liquid xenon experiments, from which this xenon sample has been subsequently taken for study. The measured gas impurities of the xenon sample expressed in ppmv are shown in Table 3.6 in relation to the product specifications of the supplier Carbagas for the initial > 99.999% purity xenon. The argon and nitrogen Carbagas purity specifications are denoted as *Typical Properties* and thus only estimations. A measurement of the oxygen residue as a component of air – calculated from the direct O_2/N_2 ratio – is feasible even at a ppmv level.

	Total f	raction	Air fra	action	
Gas	measured	specification	measured	expected	dev.
H ₂ O	234.3 ppmv	<1 ppmv			
N ₂ /CO	73.4 ppmv	<2 ppmv			
O ₂	35.8 ppmv	<1 ppmv	28.4%	20.9%	35.9%
Ar	3.4 ppmv	<1 ppmv			

TABLE 3.6: Xenon sample gas fractions.

Clearly, the impurities are mostly components of air (N_2 , O_2 , Ar) and possibly moisture (H_2O). Most of the measured water however originates from the chamber itself. In Figure 3.20, the red peak at 18 amu/q of the blank chamber has almost the same height as the black one of the xenon sample.

A sample of the xenon has been sent to Carbagas for flushing and an analysis of the O₂ content only. Their laboratory results have attained a total fraction of

34.9 ppmv for O_2 in good agreement with the measurement results presented here.

3.8 Measured Solid Samples

3.8.1 TPB coated Tetratex Sample

In the GERDA Phase II liquid argon veto, the wavelength shifting reflector foils – described in Section 2.5 – are wrapped around two copper cylinders. Their location and function inside the experiment are illustrated in Figure 2.8. A Tetraphenyl-butadiene (TPB) coated Tetratex sample of size 362 cm² of this wavelength shifting reflector foil is studied on its characteristic emanations. The foil itself was developed by Manuel Walter as part of his PhD thesis [33]. This section includes results gathered with the Emanation Measurement System in the framework of his thesis on the foil's application for GERDA. The photographs in Figure 3.21 show how the TPB coated Tetratex, exposed to UV light, shifts the reflected wavelength into the visible spectrum.



FIGURE 3.21: Photographs of TPB coated Tetratex inside a prototype copper shroud (left) and a sample sheet (right). Both are illuminated by UV light and reflect blue light back. Normal light appearance: white. Pictures from [33].

The TPB coated Tetratex sample sheet is expected to have relatively low emanation rates and determining the exact blank chamber emanation rates right before and after the measurements is a good basis for later comparison. The sample is then inserted following the procedure described in Section 3.4.1. The mass spectrum of the sample depicted in Figure 3.22.



FIGURE 3.22: TPB coated Tetratex mass spectrum (black) with a mass range of 1-200 amu/q and main peaks labelled with the dominant components (blue). Electron multiplier corrected. Pumping time: 2 hours.

The most prominent peaks are H₂O, H₂, N₂/CO, and CO₂. The spectrum is very complex with descending hydrocarbon chains throughout the mass range: A tall peak at 41 amu/q, smaller fragmentations around 39 amu/q and 26–29 amu/q are characteristic of Butene (C₄H₈). Polyethylene (C₂H₄) is identified by three peaks descending down from 28 amu/q, 27 amu/q to 26 amu/q. It is a remnant of the polyethylene foil the TPB coated Tetratex was transported in. A residue of the solvent dichloromethane (CH₂Cl) is visible, which possesses a major peak at 49 amu/q, a minor one at 51 amu/q, and a descending line after its mass of 85 amu/q. In a reaction of the polyethylene foil with the solvent dichloromethane, large chlorinated hydrocarbons have been created: e.g. chloroeicosane (C₂OH₄₁Cl), which then fragments to peaks at 57 amu/q, 55 amu/q, 71 amu/q, 69 amu/q, and 91 amu/q.

Most importantly, the TPB coating as Tetraphenyl-butadiene ($C_{28}H_{22}$) is found to not detach from the wavelength shifting Tetratex reflector foil during a week of measurements, exhibited by extremely low and incoherent 165 amu/q and 167 amu/q peaks.

The emanation rate measurements of the TPB coated Tetratex are performed four times using the accumulation method (Section 3.4.2) for different pumping times since sample insertion. Around an hour of accumulation time is used in each iteration. The last two measurements are done specifically for CO_2 , N_2/CO , and

dichloromethane (CH₂Cl). Table 3.7 lists the emanation rates for the TPB coated Tetratex sample in direct comparison (not subtracted) with the blank chamber emanation rates taken prior and afterwards with the same pumping time.

$[10^{-12} \text{ mbar} \cdot l/s]$	Blank prior	TF	'B coated	d Tetrate	ex	Blank after
Pumping [min]	1317	1304	3244	4689	8686	1320
CO ₂	34	< 317	< 408	<434	< 189	97
N ₂ /CO	15.9	<47	<216	<117	< 52	13.0
H ₂ O	6.3	11.1	6.8			3.9
Ar	1.24	1.09	0.39			1.04
CH_2Cl_2	< 0.23	12.0	9.2	8.7	7.0	< 0.16
C_2H_4	< 0.23	0.50	0.61			< 0.16
C_4H_8	< 0.23	1.31	0.48			< 0.16
C ₂ nH ₄ nCl	< 0.23	< 0.34	< 0.27	< 0.30	< 0.17	< 0.16

TABLE 3.7: TPB coated Tetratex emanation rates in comparison to blank chamber emanation rates beforehand and afterwards. Pumping time in minutes since sample insertion. Sample size: 362 cm².

The CO₂ and N₂/CO emanation rates reach values more than an order of magnitude higher in comparison to the blank chamber. They increase at first for longer pumping times until 4689 minutes. In the last measurement, they are however decreasing again, which suggests an additional source for these gases during the measurement. The hot-filaments of the RGA could be responsible for the variation, as described in Section 3.7.1.1. The emanation rates are thus presented as upper bounds with an estimated detection limit of $5 \cdot 10^{-10}$ mbar. Dichloromethane (CH₂Cl), exclusively coming from the sample, exhibits an expected decrease in emanation rate the longer the sample is in vacuum. Trace amounts of polyethylene (C₂H₄) and butene (C₄H₈) are detectable, but not substantial. The collective emanation rates of the TPB coated Tetratex are therefore extremely low and well-suited for the GERDA experiment.

3.8.2 PTFE Samples

The high degree of light reflectivity and its low mass make Polytetrafluoroethylene (PTFE) an ideal holder material. Light is reflected almost completely, preserving all information for the photo detectors [34]. Figure 3.23 shows a GERDA Phase II string. The holders insulating the germanium diodes on every Phase II string are made of PTFE and so are the PMT holders of the liquid argon veto above and below the germanium detector array. In the XENON1T experiment, PTFE comes into operation as a cylinder construct that bounds the active liquid xenon in the Time Projection Chamber (TPC) and as an insulating spacer between PMTs [34]. The results will therefore be useful across experiments. To this end, the PTFE samples are measured and analysed on their emanations for three different cleaning methods: ethanol (C_2H_5OH), acetone (C_3H_6O), and nitric acid (HNO₃). These are all possible treatments before installation into the detector strings.



FIGURE 3.23: GERDA Phase II string with its small PTFE holder pieces (white), insulating the Germanium diodes (silver).

Four PTFE samples, all cut from one block, were milled to equal dimensions of 148 mm $^{+0.1\,mm}_{-0\,mm}$ by 69 mm $^{+0.1\,mm}_{-0\,mm}$ by 19 mm $^{+0.1\,mm}_{-0\,mm}$ corresponding to a surface

of 286.7 cm². Each of the PTFE pieces is then treated individually with one of the three cleaning methods at maximum a day before the corresponding measurement. The fourth piece serves as a verification sample and is also cleaned with ethanol after all other PTFE and TPB coated Tetratex measurements are over – in chronological order: PTFE ethanol, PTFE acetone, PTFE nitric acid, TPB coated Tetratex (Section 3.8.1), fourth verification sample PTFE ethanol. The ethanol and acetone cleaning procedures are as follows:

- 1. The PTFE and the beaker are wiped with the solvent (either ethanol or acetone) using Kimwipes.
- 2. The beaker is filled with 850ml of the solvent.
- 3. The PTFE is then placed inside and the beaker is covered with aluminium foil.
- 4. The beaker is treated in an ultrasonic bath filled with water for 15 minutes.
- 5. Steps 1 to 4 are repeated with fresh solvent while flipping the PTFE such that the other surface touches the bottom of the beaker.
- 6. The beaker is placed in the ultrasonic bath again. This time for 10 minutes.
- 7. The PTFE piece is removed and the solvent is left to drain from the PTFE for 30 seconds.
- 8. The sample is then packed in aluminium foil as dust protection.
- 9. It is left to dry for 10 minutes.
- 10. The PTFE piece is flipped over, packed into fresh aluminium foil, and left to dry for another five minutes.
- 11. It is then packed into clean aluminium foil and placed in a sealable bag.

Whereas ethanol and acetone cleaning procedures are the standard in both experiments, the nitric acid etching recipe was created specifically for GERDA PTFE pieces in close contact with the germanium diodes as a ²²²Rn reduction and ²²⁶Ra removal tool. The nitric acid preparations are more complex and described in the following:

1. The PTFE piece is degreased by immersion into acetone and a subsequent ultrasonic bath of 15 minutes.

- 2. Because nitric acid in contact with acetone can lead to an explosion, the acetone has to be thoroughly removed: The PTFE is rinsed multiple times with de-ionized water and put into an ultrasonic bath filled with de-ionized water for five minutes.
- 3. The PTFE piece is then immersed in a 5% (by mass) nitric acid (HNO₃) solution.
- 4. In this solution, the sample is treated using an ultrasonic bath for 15 minutes and then kept in the nitric acid for 1 hour 45 minutes.
- 5. After the acid treatment, the sample is rinsed several times with de-ionized water.
- 6. The sample is put into an ultrasonic bath with de-ionised water for five minutes.
- 7. The sample is wiped with Kimwipes and dried in air for 20 minutes.
- 8. The PTFE piece is flipped over, packed into fresh aluminium foil, and left to dry for another five minutes.
- 9. It is then packed into clean aluminium foil and placed in a sealable bag.

After each cleaning procedure, the respective sample is inserted into the chamber using the solid sample loading procedure described in Section 3.4.1.

3.8.2.1 Ethanol Cleaned PTFE and Verification Sample

After around an hour of pumping since the insertion of the ethanol (C_2H_5OH) cleaned PTFE sample, the mass spectrum is taken with a mass range of 1–100 amu/q, electron multiplication, and a high sensitivity scan speed of 10 samples/s. It is depicted in Figure 3.24 on top of a blank chamber spectrum with the same settings.

A large increase of the peaks associated to air (N₂ at 28 amu/q, O₂ at 32 amu/q, CO₂ at 44 amu/q, and Ar at 40 amu/q) in comparison to the blank chamber is observed. The argon peak at 40 amu/q is even higher than usual, originating from the threefold argon flushes after sample insertion (Section 3.4.1). Additional H₂O (18 amu/q) seems to be introduced by the sample into the chamber. A peak at 31 amu/q appears; it is the solvent ethanol (C₂H₅OH).



FIGURE 3.24: Ethanol cleaned PTFE sample mass spectrum (red) with a mass range of 1–48 amu/q in comparison to a blank chamber spectrum (black) taken just before insertion. 10 samples/s scan speed. Electron multiplier corrected. Pumping time around 1 hour.

The chamber is kept connected to the vacuum turbo pump while continuously taking mass spectra. The resulting *pump down curve* in Figure 3.25 reveals partial (top) and relative pressure (bottom) trends for the different gases over a whole night of pumping.

Ethanol (C₂H₅OH), in Figure 3.25 on the top, is visible at the start, but disappears into the noise. It is efficiently removed from the chamber. The argon abundance due to the flushes goes down fast on the partial pressure graph, while on the relative pressure one, it converges over the course of the night. From fragmentation calculations, the 28 amu/q peak is found to be dominated by the N₂ component. Air is apparently stored within the porous PTFE when the sample is exposed to it in the cleaning procedure. The ethanol, argon, and moisture however are located mostly on or close to the surface of the sample, exhibited by their similar partial pressure curve shapes. H₂O (light blue) curves down from being the second highest constituent and eventually dips below O₂ (blue) in Figure 3.25 on the top. The surface occurrence explains the faster removal by pumping, while the main air components – such as nitrogen, oxygen, and carbon-dioxide – persist longer. They have been absorbed deep into the PTFE bulk.



FIGURE 3.25: Ethanol (C_2H_5OH) cleaned PTFE sample pump down curve displaying partial (top) and relative pressure (bottom) trends for various gas components. N (black), O (blue), and OH (light blue) as fragments are always the lower corresponding coloured line. 100 scans correspond to around 5 hours 30 minutes.

The emanation rates of the ethanol cleaned PTFE sample are then measured with the pressure rise method explained in Section 3.4.2. Figure 3.8 shows the technique applied to a measurement of this sample. The results are listed in Table 3.8 beside the blank chamber emanation rates determined beforehand with up to 10^{-13} mbar $\cdot 1/s$ accuracy. The verification sample measurements, which follow the exact same procedure as the initial ethanol cleaned PTFE sample, are included as well. This ensures the accuracy of the solid sample measurement methods and the Emanation Measurement System as a whole.

[mbar·l/s]	Blank	Ethanol cleaned PTFE			
Pumping [min]	various	3900	5500	5590	6970
N ₂ /CO	$6.11 \cdot 10^{-13}$	$4.74\cdot 10^{-7}$	$4.44\cdot10^{-7}$	$4.35\cdot 10^{-7}$	$4.49\cdot 10^{-7}$
CO ₂	$2.61\cdot 10^{-12}$	$5.63\cdot 10^{-9}$	$3.76\cdot 10^{-9}$	$3.98\cdot 10^{-9}$	$2.47\cdot 10^{-9}$
O ₂	-	$1.37\cdot 10^{-8}$	$1.20\cdot10^{-8}$	$1.10\cdot 10^{-8}$	$1.01\cdot 10^{-8}$
0	$3.35\cdot 10^{-13}$	$1.11\cdot 10^{-9}$	$9.83 \cdot 10^{-10}$	$8.59\cdot 10^{-10}$	$6.19 \cdot 10^{-10}$
Ar	-	$1.40 \cdot 10^{-8}$	$1.33\cdot 10^{-8}$	$1.25\cdot 10^{-8}$	$1.24\cdot 10^{-8}$

TABLE 3.8: PTFE ethanol emanation rates for various pumping times since sample insertion in comparison to prior blank chamber emanation rate averages. Verification sample in **bold**. Sample surface: 286.7 cm².

The PTFE sample increases the emanation rates inside the chamber dramatically: N_2 is the dominant component and is increased by around a factor 10^6 in comparison to the blank chamber rate. The CO₂ emanation rate is approximately a thousand times higher. The blank chamber emanation is negligible in contrast. A steady decline of all emanation rates is observed over longer pumping times since sample insertion.

Figures 3.26 and 3.27 depict this emanation rate evolution of the gases N_2 , CO_2 , O_2 , and Ar for all measurements – including the times 6970, 8300, 10000, and 14100 minutes that have also been determined with the verification sample. For N_2 (Figure 3.26, top) – the dominant constituent – the verification measurements agree very closely with the earlier ones. Argon exhibits a similar behaviour. The CO_2 graph (Figure 3.26, bottom) follows an exponential shape. The discrepancy of the data points at 6970 minutes pumping time gives an estimate on the uncertainty of the measurement. An exponential fit through the data leads to the parameters in Table 3.9 for the function



FIGURE 3.26: Ethanol cleaned PTFE sample emanation rates over pumping time since sample insertion for N_2 (top) and CO_2 (bottom). Initial sample (blue) and verification sample (red) combined.



FIGURE 3.27: Ethanol cleaned PTFE sample emanation rates over pumping time since sample insertion for O_2 (top) and Ar (bottom). Initial sample (blue) and verification sample (red) combined.

	f(x) = e	$^{a+b\cdot x}$.
	Value	Uncertainty
а	-18.56	$\pm 3.94 \cdot 10^{-2}$
b	$-1.46\cdot10^{-4}$	\pm 1.41 \cdot 10 ⁻⁵

TABLE 3.9: Exponential fit parameters through the data points of Figure 3.26 on the bottom.

A constant relative uncertainty is assumed, considering the drastic increase of the measured emanation by the PTFE sample compared to the blank chamber (Table 3.8). The deviations from the fitted exponential function are then divided by the measured values. The standard deviation of these relative deviations is 8.2%, which is then individually multiplied by the respective measured values. This leads to an estimation on the actual deviations, signified by the error bars in Figure 3.28. The exponential fit then performed through these error bars leads to the parameters in Table 3.10 for the Equation 3.3.

	Value	Uncertainty
a	-18.70	$\pm 9.44\cdot 10^{-2}$
b	$-1.30 \cdot 10^{-4}$	\pm 1.17 \cdot 10 ⁻⁵

TABLE 3.10: Exponential fit parameters through the error bars of Figure 3.28.

These parameters are compatible with the initial ones in Table 3.9. This uncertainty estimation is used in Section 3.8.2.3 to calculate the significance of the observed phenomena.



FIGURE 3.28: Ethanol cleaned PTFE sample emanation rate uncertainty estimation for CO_2 from an exponential fit (red line) to the data points, that have been measured in two separate stages (initial and verification sample).

3.8.2.2 Acetone Cleaned PTFE Sample

The acetone (C_3H_6O) cleaned PTFE sample measurements are performed using a similar procedure. Figure 3.29 shows the mass spectrum of the PTFE Sample cleaned with acetone taken one hour after insertion with a mass range of 1–100 amu/q, electron multiplication, and a high sensitivity scan speed of 10 samples/s. For direct comparison, the ethanol cleaned PTFE spectrum is superimposed using the same settings. The spectrum is very similar to that of the PTFE ethanol sample, but the CO₂ peak at 44 amu/q is slightly elevated. At 58 amu/q and in the surrounding peaks, aceton and its fragmentations appear.

The pump down curve of the acetone cleaned PTFE sample is shown in Figure 3.30. The pump down trends observed with the ethanol cleaned sample are visible for PTFE acetone as well. For the case of H₂O they are even more pronounced. OH follows the H₂O shape closely. Water fragmentation is therefore the main contributor to OH as opposed to ethanol or acetone. When comparing Figures 3.25 and 3.30, acetone as a solvent seems to persist longer than ethanol. It is harder to get rid of in the pump down, although the total amount is not significant.



FIGURE 3.29: Acetone cleaned PTFE sample mass spectrum (black) with a mass range of 1–100 amu/q in comparison to the ethanol cleaned PTFE sample spectrum (red). 10 samples/s scan speed and electron multiplier corrected. Pumping time: 1 hour.



FIGURE 3.30: Acetone (C_3H_6O) cleaned PTFE sample pump down curve displaying partial pressures for various components. N (black), O (blue), and OH (light blue) as fragments are always the lower corresponding coloured line. 100 scans correspond to around 5 hours 30 minutes.
Table 3.11 lists the emanation rate measurements of the acetone cleaned PTFE sample in direct comparison to the ethanol cleaned one. The results are very close to each other except for the CO_2 emanation rate, which is around twice as large for the PTFE acetone sample.

[mbar·l/s]	Ethanol PTFE	Acetone PTFE
Pumping [min]	3900	3600
N ₂ /CO	$4.74 \cdot 10^{-7}$	$4.65 \cdot 10^{-7}$
CO ₂	$5.63\cdot 10^{-9}$	$9.18\cdot10^{-9}$
O ₂	$1.37\cdot10^{-8}$	$1.51\cdot 10^{-8}$
0	$1.11\cdot 10^{-9}$	$1.54\cdot 10^{-9}$
Ar	$1.40\cdot 10^{-8}$	$1.40\cdot10^{-8}$

TABLE 3.11: PTFE acetone emanation rates in comparison to the PTFE ethanol emanation rates at a similar pumping time. Sample surface: 286.7 cm².

3.8.2.3 Nitric Acid Cleaned PTFE Sample

The mass spectra of the nitric acid (HNO₃) and the ethanol cleaned PTFE samples at matched pumping times are superimposed in Figure 3.31. A mass range of 1–100 amu/q, electron multiplication, and a high sensitivity scan speed of 10 samples/s is chosen. For nitric acid additional peaks below 60 amu/q appear. The acetone peak at 58 amu/q and its fragments at 57 amu/q and 56 amu/q are also clearly visible and even higher than for the acetone measurement. To its left, it is flanked by a typical hydrocarbon chain as a descending group of peaks. Nitric acid and its fragmentations, e.g. at 46 amu/q, are also noticeable. The CO₂ peak at 44 amu/q is increased as well.

In the pump down curve of the PTFE nitric acid sample, depicted in Figure 3.32, acetone is present in high abundance: at the start, it is around two orders of magnitude higher than even for the acetone cleaned sample. This is caused by the nitric acid preparation that involves an aceton ultrasonic bath (Section 3.8.2). After a weekend of pumping, the acetone (C_3H_6O) goes down significantly. The nitric acid (HNO₃) seems to persist longer than the other solvents.

A comparison between the PTFE nitric acid sample and the ethanol cleaned one, at equal times of pumping, reveals similar emanation rates for N_2 and argon. Less O_2 emanation is detected. Most interestingly, the CO₂ emanation of



FIGURE 3.31: Nitric acid cleaned PTFE sample mass spectrum (black) with a mass range of 1–100 amu/q in comparison to the ethanol cleaned PTFE sample spectrum (red). 10 samples/s scan speed and electron multiplier corrected. Pumping time: 1 hour.



FIGURE 3.32: Nitric acid (HNO₃) cleaned PTFE sample pump down curve displaying partial pressures for various components. N (black), O (blue), and OH (light blue) as fragments are always the lower corresponding coloured line. 100 scans correspond to around 5 hours 30 minutes.

the nitric acid cleaned sample has an emanation rate around five times higher than the one for the ethanol method. This phenomenon is observed across all measurements and pumping times, as seen in Table 3.12.

[mbar·l/s]	Ethanol PTFE	Nitric Acid PTFE	Ethanol PTFE	Nitric Acid PTFE
Pumping [min]	3900	3600	5590	5590
N ₂ /CO	$4.74\cdot10^{-7}$	$5.37 \cdot 10^{-7}$	$4.35\cdot10^{-7}$	$4.94\cdot 10^{-7}$
CO ₂	$5.63\cdot 10^{-9}$	$3.47 \cdot 10^{-8}$	$3.98\cdot10^{-9}$	$1.75\cdot 10^{-8}$
O ₂	$1.37\cdot 10^{-8}$	$8.43\cdot10^{-9}$	$1.10\cdot10^{-8}$	$4.78\cdot 10^{-9}$
0	$1.11\cdot 10^{-9}$	$2.73 \cdot 10^{-9}$	$8.59 \cdot 10^{-10}$	$1.69 \cdot 10^{-9}$
Ar	$1.40\cdot 10^{-8}$	$1.77 \cdot 10^{-8}$	$1.25\cdot 10^{-8}$	$1.20 \cdot 10^{-8}$

TABLE 3.12: PTFE nitric acid emanation rates in comparison to PTFE ethanol for various pumping times since sample insertion. Verification sample in **bold**. Sample surface: 286.7 cm².

Using the estimated uncertainty of Section 3.8.2.1 on the CO₂ emanation rate, the significance of the observed CO₂ abundance in the nitric acid measurements in relation to the ethanol cleaned sample can be calculated. In this way, statistical fluctuations can be ruled out. With 62 σ and 42 σ significance for 3900 minutes and 5590 minutes pumping times, respectively, the difference in the applied PTFE cleaning method is clearly responsible for the elevated CO₂ emanation.

A probable cause for this observed effect is the reaction of acetone (C_3H_6O) with nitric acid (HNO₃), which results in not only more CO₂ and H₂O, but also C_2H_4O – also called *vinyl alcohol* (or *ethenol*). This vinyl alcohol possesses a mass of 44 amu/q and thus contributes directly to the same mass peak as CO₂.

3.8.2.4 Discussion

Ethanol as a solvent for PTFE is the most clean procedure of the three for general application. It provides the cleanest mass spectra in comparison to acetone or nitric acid methods. Ethanol as a solvent is efficiently removed by vacuum pumping over a shorter period of time. It then provides the lowest emanation rates, especially for CO_2 . The cleaning procedure of a PTFE sample with ethanol is much less elaborate and time-consuming than the one for the nitric acid method. The nitric acid etching procedure is a ²²⁶Ra removal tool to reduce

²²²Rn emanation from PTFE and is best used where this is valued over the drawbacks – higher emanation rates and procedure complexity – found here.

3.9 Conclusion and Outlook

The Emanation Measurement System's capability of solid sample emanation rate determination with up to 10^{-13} mbar \cdot l/s background accuracy has been demonstrated here. Component fractions of gas samples can be measured with up to 1 ppmv accuracy. The involved procedures have been established with a dedicated verification sample and an air argon gas sample of known mixture. Compositional analysis using mass spectra have been performed in a range of 1–200 amu/q over extended periods of time including the simultaneous observation of the development of all constituent species.

A xenon sample has been analysed over the full mass range and the present impurities have been identified and fractionally quantified. A TPB coated Tetratex sample of the wavelength shifting reflector foil used in the GERDA Phase II liquid argon veto has been studied. The low emanation rates, which are dominated by CO_2 with only an order of magnitude above the background, makes it an ideal component material for GERDA. The TPB coating has been observed to not detach from the Tetratex during a week of measurements.

PTFE is an integral component of GERDA and XENON1T and is implemented as reflectors, as insulating spacers, and as PMT holders. Four PTFE samples with ethanol, acetone, and nitric acid cleaning methods have thus been investigated and compared. N₂ as the prevalent emanation gas is found to be emanated around $4.5 \cdot 10^{-7}$ mbar $\cdot 1/s$ (3900 minutes pumping time since sample insertion) for all cleaning methods, while the CO₂ rate is shown to be highly dependant on the cleaning method. Ethanol cleaned PTFE exhibits the lowest CO₂ emanation rate at $5.6 \cdot 10^{-9}$ mbar $\cdot 1/s$ (3900 minutes pumping time) – the corresponding PTFE nitric acid rate is around five times higher – while also providing the cleanest mass spectra. Ethanol is thus the recommended general PTFE cleaning solvent.

The detailed knowledge provided by the Emanation Measurement System of any impurities introduced by component materials can benefit not only current, but future high sensitivity experiments as well.

Chapter 4

GERDA Phase II Calibration

The GERDA germanium detectors are calibrated with the help of three ²²⁸Th sources. They can be lowered into the liquid argon cryostat in the vicinity of the detectors using a Source Insertion System (SIS), which was developed and is maintained by the GERDA group of the University of Zürich [33, 41, 42]. The observed characteristic γ -ray spectrum of the sources exhibits peaks well-known in literature [43]. Matching the individual peaks to their corresponding literature energy values leads to a full range calibration curve. With its help, the ADC values on the measured spectra can be assigned to energies in units of eV.

The GERDA Phase II calibration software [44] is designed to perform this automatically. Relevant peaks in the calibration data are found and assigned to corresponding energy line literature values by the program. After identification, a full range calibration curve is created as explained in Section 4.1.

Beforehand, non-physical and background events are to be rejected with the help of *quality cuts* integrated into the software. The correct parameters for the individual quality cuts are investigated in this thesis on the basis of Phase II Integration run data to ensure the optimal selection of events. This is described in Section 4.2.

4.1 **GERDA Phase II Calibration Software**

The GERDA Phase II calibration software has been developed by Giovanni Benato as part of his PhD thesis [44]. The code is available in the GERDA Advanced Data Analysis (gerda-ADA) package, developed and maintained by the GERDA Collaboration. The visualisations of energy, trigger and rise time spectra – such as Figure 4.1 and onward – have been programmed as part of this thesis. All relevant parameters can be given to the calibration executable directly in the command line as options. These include a detector parameter file that defines the name, mass, active volume, and dead layer of each individual detector. For the Phase II detectors, a single file in the .JSON (JavaScript Object Notation) format defines these parameters. An additional detector settings .JSON file assigns the channel and high voltage used on each detector during the corresponding run. Since this varies for every run, the matching file can be supplied independently. The sources and their activity are recorded in the source settings file. The quality cuts .JSON file contains the choice of cuts and the parameter range in which these cuts are executed. Section 4.2 expands on this comprehensively. Lastly, a file list allows the read-in of all chosen runs as Tier2 .ROOT files at the same time.

The Tier2 level is the third step of the GERDA multi-tier data structure. Starting with the raw germanium and PMT data in the Tier0, the data are converted to ROOT format using MGDO [45] and GELATIO [46] software packages. This leads to the Tier1 rank. Results of the digital signal processing and analysis are stored in Tier2 .ROOT files [46].

After reading-in the input parameters and the chosen Tier2 files, the quality cuts are applied, discarding events which do not fullfill the desired requirements (Section 4.2). The program then searches for peaks using the peak finder function of the advanced spectra processing ROOT class TSpectrum. First, the prominent 2615 keV full energy peak is identified. It then serves as a relation for the other peaks that are compared to a list of literature values from NuDat 2.6 [43]. The calibration and resolution curves are then fitted in an energy range of 0–3000 keV and written into .ROOT files. The quality cuts and calibration, that are based on Tier2 files, therefore result in Tier3 files containing calibrated energy and quality cut flags [46].

4.2 Quality Cuts for GERDA Phase II

For an optimal energy calibration, event selection is performed through dedicated quality cuts. Non-physical and background events (Section 2.4) can thus be rejected before any peak fitting or advanced data analysis is applied, enhancing the accuracy of the final calibration.

The quality cuts can be either based on an event tag (e.g. pulser events), on the selection of events with a variable within a range or on a characterisation of multiple detectors together (e.g. coincidences). They are specified in their .JSON file, along with the corresponding range parameters, in the same order they are applied and can be enabled and disabled individually. For the study presented here, the exclusion of events is executed in six stages: pulser, muon, coincidence, single trigger, trigger range, and rise time range quality cuts.

First, pulser events are discarded. As described in Section 2.3, the germanium diodes are kept on a high voltage while their signals go into pre-amplifiers that are connected to a Flash ADC. Pulser events arise from a pulser signal fed through these pre-amplifiers. Their purpose is to ensure the stability of the pre-amplifiers and the whole electronics chain with a known signal during physics runs.

Figure 4.1 depicts the ²²⁸Th energy spectrum of the germanium diode GD02B collected during the Phase II Integration runs. The run contains 14 677 243 initial events using one Phase II string – eight BEGe detectors and eight channels. The signature is dominated by γ -ray emissions above an energy threshold from its daughter nuclei ²⁰⁸Tl and ²¹²Bi. The main peaks in the signature above the Compton continuum are listed in Table 4.1 from left to right. The leftmost peak below the shoulder, the 511 keV line, is partially suppressed due to the selected energy threshold. ¹³⁷Cs at 662 keV comes from a known contamination of the source material. The double escape peak (DEP) at 1593 eV is located in the middle of the plateau. The single escape peak (SEP), before the Compton edge, arises from the last large peak of ²⁰⁸Tl at 2615 keV. The escape peaks are created by photons that leave the detector without depositing their full energy. Between the Compton edge and the full energy peak, multiple Compton scattering events take place.



FIGURE 4.1: Energy spectrum of the ²²⁸Th sources (blue) with the quality cut kIsPulser applied. The identified pulser peak (red) is located at around 6850. This is taken with the germanium detector GD02B at 3000 V as part of the Phase II Integration run 150425. Energy in arbitrary units.

Energy [keV]	511	662	727	861	1079	1593	1621	2104	2615
Identification	²⁰⁸ Tl	¹³⁷ Cs	²¹² Bi	²⁰⁸ Tl	²¹² Bi	DEP	²¹² Bi	SEP	²⁰⁸ Tl

TABLE 4.1: ²²⁸Th source spectrum energy lines for the daughter nuclei. The double (DEP) and single escape peak (SEP) stem from the full energy peak of 208 Tl at 2615 keV.

The pulser is located higher in energy and can be identified and isolated easily with the option *kIsPulser*. From here on, the pulser events are removed from the spectra with *kNoPulser*. 11932 pulser events and 21843 pulser events are found for the respective runs, as listed in Table 4.2 [28, 43].

Run	Initial Events	Surviving Events	Pulser Events
150425	14677243	14 665 311	11 932
150528	8 850 647	8 828 804	21 843

TABLE 4.2: Pulser quality cut: initial number of events, surviving events, and pulser events.

Depending on the detector and the applied high voltage, the pulser is located at a different energy, which can be much higher for the case of germanium diodes 4/C, 1/D, and GD79C. Detectors GD35B, GD61C, and GD91C however look

almost identical to the one of GD02B (Figure 4.1). The ²²⁸Th signature itself is very similar for all of these detectors.

A peculiar spectrum is observed however for GD91B in Figure 4.2, exhibiting no real peaks except for the correctly identified pulser. But even that one is much broader than expected. The spectrum seems to be smeared out.



FIGURE 4.2: Energy spectrum of the ²²⁸Th sources (blue) with the quality cut kIsPulser applied. The identified broad pulser peak (red) is located at around 16 200. This is taken with the germanium detector GD91B at 2200 V as part of the Phase II Integration run 150425.

The diode showed leakage current and could not be used at the full operational voltage of 3000-3500 V, but had to be used at 2700 V, which leads to incomplete depletion. This explains the broadening of the physical peaks. The pulser should however remain sharp. The wideness of the pulser implies an additional technical problem of the contacts, cables or the pre-amplifier.

Next, muon events that come from cosmic rays are identified in the Cherenkov veto system employing the purified water tank around the cryostat, as described in Section 2.6. If the PMTs detect Cherenkov light in coincidence with events in germanium, these events can be classified as background and are consequently cut. With the option *kIsMuon* they can be selected, while *kNoMuon* is used to reject them. Table 4.3 shows that for run 150528 no muons were identified. The muon veto was turned off for that particular run and thus all events survive

the muon quality cut. 19 muon events are excluded for run 150425, where the

Cherenkov muon veto was fully working.

Run	Initial Events	Surviving Events	Muon Events
150425	14677243	14677224	19
150528	8850647	8 850 647	C

TABLE 4.3: Muon quality cut: initial number of events, surviving events, and muon events.

Coincidence events arise from sequential interactions detected in multiple detector channels simultaneously – such as Multiple-Site Events (MSE) discussed in Section 2.4. During calibration, they can be generated by the sources through e.g. γ -ray emissions undergoing multiple Compton scattering. Coincidence events might thus induce low energy tails on the peaks and should be excluded with *kNoCoincidence*.



FIGURE 4.3: Energy spectrum of the 228 Th sources plus pulser before (blue) and after (red) applying the quality cuts kNoPulser, kNoMuon, and kNoCoincidence. It is taken with the germanium detector 4/C at 3500 V as part of the Phase II Integration run 150425.

Figure 4.3 depicts the energy spectrum with the quality cuts pulser, muon, and coincidence applied. Since the energy of a coincidence event is deposited in multiple diodes, they are found and cut mostly in the lower region of a measured detector energy spectrum. Some events above and below the pulser peak are

rejected as well. The pulser quality cut gets rid of the peak at 17 000. So far, the coincidence events make up the largest part of dismissed events, as seen in Table 4.4 with *only* that quality cut applied.

Run	Initial Events	Surviving Events	Coincidence Events
150425	14677243	14 279 873	397 370
150528	8 850 647	8 547 843	302 804

TABLE 4.4: Coincidence quality cut: initial number of events, surviving events, and coincidence events.

The single trigger quality cut is a rejection of pile-up events. They are produced by two successive events that take place with a time delay shorter than the waveform length (164 µs for GERDA). Using a pseudo-Gaussian filter, the deposited energies of the pile-up events are summed up and cannot be discriminated. The quality cut command to reject them is *kSingleTrigger*. Evidently, many low energy events are excluded in the spectra of Figure 4.4. Pile-up events occur all over the spectrum, but are best seen in this valley below the energy threshold. On the bottom graph of Figure 4.4, a distribution of low energy events can be seen for detector GD91C. The shape indicates that they are of non-physical nature. It curves down again before reaching the energy threshold instead of progressively ascending towards the Compton continuum. The single trigger quality cut efficiently removes these noise events. As presented by Table 4.5, pile-up events make up around 0.01% to 0.1% of the total events for these respective runs. With a higher source activity, these values should increase further and the quality cut becomes even more important.

Run	Initial Events	Surviving Events	Pile-up Events
150425	14677243	14 662 371	14 872
150528	8 850 647	8 849 764	883

TABLE 4.5: Single trigger quality cut: initial number of events, surviving events, and pile-up events.

The trigger range quality cut makes use of two input parameters. If the preamplified signal of a germanium detector exceeds a certain threshold, a trigger activates, enabling the storage of the signal during a set microseconds timeframe. This range is defined directly through the *kTriggerInRange* command in the quality cuts JSON file. The Phase I values were 79.5 µs for the minimum and



FIGURE 4.4: Energy spectrum in the range from 0 to around 2000 keV of the ²²⁸Th sources before (blue) and after (red) applying only the quality cut kSingleTrigger. A lot of low energy events are rejected. The top graph is taken with germanium detector GD79C at 3500 V, the bottom with GD91C at 2700 V as part of the Phase II Integration run 150425.

82 µs for the maximum. For these values, Figure 4.5 depicts more rejected events in the lower and upper energy region, while Table 4.6 lists the total amount excluded. It is necessary to use it in conjunction with the coincidence quality cut, since an event could have a fitting trigger range in only one channel during a coincidence event. The pulser peak at around 8000 is also completely rejected by the coincidence cut.



FIGURE 4.5: Energy spectrum of the 228 Th sources and pulser before (blue) and after (red) applying only the quality cuts kNoCoincidence and kTriggerInRange with parameters 79.5–82 µs. This is taken with the germanium detector GD35B at 4000 V as part of the Phase II Integration run 150425.

Run	Initial Events	Surviving Events	Out of Range
150425	14677243	14254608	25 265
150528	8 850 647	8 543 224	4619

TABLE 4.6: Trigger range 79.5–82 µs with prior coincidence quality cut: initial number of events and surviving events. Out of range events for trigger range cut alone.

The rise time range quality cut is used to exclude events outside a certain range of the signal time evolution. The option *kRiseTimeInRange* has to be specified with arguments for a minimum and maximum in nanoseconds. The different interaction locations inside a germanium diode each have a specific drift path of the produced charge carriers, which results in a characteristic rise time of the signal [29, 30]. The expected germanium detector signal and the variation of

rise time are explained in detail in Section 2.3. An event with a rise time below around 200 ns is either non-physical or is due to an interaction point extremely close to the cathode with undesirable signal shape and amplified current peak [29]. Events with a rise time above 3000 ns are normally characterized by an energy underestimation and lead to the presence of low energy tails on the peaks. Figure 4.6 shows thus cut events not only in the low energy region, but also across the whole spectrum. Many events are cut in this last step, as displayed in Table 4.7.



FIGURE 4.6: Energy spectrum of the ²²⁸Th sources before (blue) and after (red) applying only the quality cuts kNoCoincidence and kRiseTimeInRange with parameters 200–3000 ns. This is taken with the germanium detector 1/D at 3500 V as part of the Phase II Integration run 150425.

Run	Initial Events	Surviving Events	Out of Range
150425	14677243	14 091 111	188762
150528	8 850 647	8 079 197	468 646

TABLE 4.7: Rise time range 200–3000 ns with prior coincidence quality cut: initial number of events and surviving events. Out of range events for rise time range cut alone.

In the case of diode GD91B a large fraction of events is rejected by the rise time cut. This is due to the low applied voltage of 2200 V, which induces a strong charge collection inefficiency. The reason is best studied on the actual rise time spectrum of the detector. Figure 4.7 for detectors 4/C and GD91B in



FIGURE 4.7: Rise time spectrum before (blue) and after (red) applying all quality cuts taken with the germanium detectors 4/C (top) at 3500 V and GD91B at 2700 V (bottom) as part of the Phase II Integration run 150425.



FIGURE 4.8: Rise time spectrum before (blue) and after (red) applying all quality cuts taken with the germanium detectors 4/C (top) at 3500 V and GD91B (bottom) at 2200 V as part of the Phase II Integration run 150528.

run 150425 and the top part of Figure 4.8 for 4/C in run 150528 exhibit expected and very similar distributions. The rise time spectrum of GD91B (Figure 4.8, bottom) however is anomalous. Most of the events are found very far outside the set range. This is further evidence of a technical problem introduced between the two runs beyond just the decrease of voltage. All other detectors depict analogous spectra to the one of detector 4/C. The rise time range parameters of 200–3000 ns are thus found to be a good judgement of physically relevant events.

The trigger range window set at 79.5–82 μ s can also be analysed again on the basis of the trigger time spectra. Figures 4.9 and 4.10 show this for the detectors 4/C and GD61C with the coincidence and trigger range quality cuts applied. All detectors in the two runs behave very similarly. In the spectra, the 82 μ s maximum of the trigger range seems to be set slightly too high in relation to the observed trigger distribution. The maximum can be improved to 81.5 μ s and thus visible outliers that survive the coincidence quality cut – such as in Figure 4.9 on the bottom – can be removed as well. Three events for run 150528 and six events for run 150425 can be additionally excluded. These Integration runs have been measured with Phase I sources. Due to the higher activity of the Phase II calibration sources, the number of these further cut events will be up to one order of magnitude higher. With higher statistics, stringent cuts become even more relevant.

4.3 Conclusion and Outlook

The entire quality cut sequence to discard non-physical and background events in two full Integration runs is summarised in Tables 4.8 and 4.9. They include the rejected events for each individual cut as well as the total surviving fraction at each stage. In contrast to Tables 4.2–4.7, not just single or paired cuts are applied to the data, but the full progression is followed. The concluding energy spectrum after executing all quality cuts is depicted in Figure 4.11.

The quality cuts work very well, providing a total event exclusion of 5–9% for these Integration runs and source activity (Tables 4.8 and 4.9). In the spectrum view presented here, the best parameters can be found easily. A rise time range of 200 to 3000 ns has proven to be an ideal window of time for the best event selection. The trigger range parameters however can be optimised from



FIGURE 4.9: Trigger time spectrum before (blue) and after (red) applying kNo-Coincidence and kTriggerInRange with parameters 79500–82000 ns. This is taken with the germanium detectors 4/C (top) at 3500 V and GD61C at 4000 V (bottom) as part of the Phase II Integration run 150425.



FIGURE 4.10: Trigger time spectrum before (blue) and after (red) applying kNoCoincidence and kTriggerInRange with parameters 79500–82000 ns. This is taken with the germanium detectors 4/C (top) at 3500 V and GD61C at 4000 V (bottom) as part of the Phase II Integration run 150528.

a maximum of 82 μ s to a range of 79.5–81.5 μ s, rejecting additional events. The higher activity of the Phase II sources will benefit from the better constraints even more.

The same quality cuts are to be used for the actual Phase II physics data as for the calibration to prevent the introduction of any bias. Since the quality cut parameters need to be fixed before unblinding the window around $Q_{\beta\beta}$ (Section 2.4), predicting the most suitable values is essential to ensure the highest possible sensitivity. A stricter upper limit of 81.5 µs on the trigger range quality cut is the resulting proposition to the GERDA Collaboration. In the same manner as presented here, these parameters can be further optimised with additional Phase II data in the future.



FIGURE 4.11: Energy spectrum of the 228 Th sources plus pulser before (blue) and after (red) applying all quality cuts. This is taken with the germanium detector GD35B at 4000 V as part of the Phase II Integration run 150425.

Quality Cuts	Last Cut Events	Resulting Events	Total Fraction
Initial Events	-	14 677 243	100.00%
Pulser	11 932	14665311	99.92%
Pulser & Muon	19	14 665 292	99.92%
Above & Coincidence	385 419	14 279 873	97.29%
Above & SingleTrigger	14 636	14265237	97.19%
Above & Trigger Range	10630	14254607	97.17%
Above & Rise Time Range	177 890	14076717	95.91%

TABLE 4.8: Integration run 150425 quality cut sequence with last cut events for each added quality cut and total surviving fraction at each stage.

Quality Cuts	Last Cut Events	Resulting Events	Total Fraction
Initial Events	-	8 850 647	100.00%
Pulser	21 843	8828804	99.75%
Pulser & Muon	0	8828804	99.75%
Above & Coincidence	280 961	8 547 843	96.58%
Above & SingleTrigger	871	8 546 972	96.57%
Above & Trigger Range	3748	8 543 224	96.53%
Above & Rise Time Range	465 184	8 078 040	91.27%

TABLE 4.9: Integration run 150528 quality cut sequence with last cut events for each added quality cut and total surviving fraction at each stage.

Chapter 5

Conclusion and Outlook

Neutrinoless double beta decay is a compelling avenue to investigate the Majorana nature of the neutrino, the neutrino mass hierarchy, and the absolute mass scale. Searching for this decay, the GERDA experiment – based on a germanium detector array directly submerged in a liquid argon cryostat – aims to achieve the lowest background and the highest sensitivity possible.

An Emanation Chamber System has been built at the University of Zürich for emanation rate and composition analysis of GERDA component materials. Different measurement methods have been developed to study solid and gas samples. The blank chamber has been characterised to understand the background for each measurement. In verification measurements with a known air argon mix composition, the Emanation System has been shown to be reliable and accurate. Argon and xenon gas samples are measured to 1 ppmv sensitivity. The emanation rates of all solid samples can be studied with up to 10^{-13} mbar \cdot l/s sensitivity. A sample of Tetraphenyl-butadiene (TPB) coated Tetratex – the wavelength shifting reflector foil material used in GERDA - has been investigated and compared to the blank chamber, at equal pumping times since sample insertion, before and after the measurements. As a result, TPB is found to not detach from the Tetratex foil during a week of measurements. Three cleaning methods – ethanol, acetone, and nitric acid – on Polytetrafluorethylen (PTFE) samples have been analysed. A fourth ethanol cleaned sample has been used to verify the observed effects. Ethanol is the best cleaning method with the least amount of emanations. The Emanation Measurement System will also be a great asset for the component analysis of future high sensitivity experiments.

The GERDA Phase II calibration software quality cuts have been studied on the basis of two full Phase II Integration runs (150425 and 150528) in six stages: pulser, muon, coincidence, single trigger, trigger range, and rise time range quality cuts. A rise time range of 200 to 3000 ns is shown to be the best time window for event selection. The trigger range parameters however are found to reject additional background events if optimised to a more stringent upper limit of 81.5 µs. The concluding proposition to the GERDA Collaboration is a trigger range quality cut parameter setting of 79.5–81.5 µs.

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