Preparation and measurement of an ^{37}Ar source for liquid xenon detector calibration

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Abstract

We present the preparation and measurement of the radioactive isotope ^{37}Ar, which was produced using thermal neutrons from a reactor, as a calibration source for liquid xenon time projection chambers. ^{37}Ar is a low-energy calibration source with a half-life of 35.01 days, making it suitable for calibration in the low-energy region of liquid xenon dark-matter experiments. The radioactive isotope ^{37}Ar was produced by irradiating ^{36}Ar with thermal neutrons. It was subsequently measured in a gaseous xenon time projection chamber (GXe TPC) to validate its radioactivity. Our results demonstrate that ^{37}Ar is an effective and viable calibration source, offering precise calibration capabilities in the low-energy domain of xenon-based detectors.

Full Text

Preamble

Preparation and measurement of a ^{37}Ar source for liquid xenon detector calibration

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We present the preparation and measurement of the radioactive isotope ^{37}Ar, produced using thermal neutrons from a reactor, as a calibration source for

liquid xenon time projection chambers. ^{37}Ar is a low-energy calibration source with a half-life of 35.01 days, making it suitable for calibration in the low-energy region of liquid xenon dark matter experiments. The radioactive isotope ^{37}Ar was produced by irradiating ^{36}Ar with thermal neutrons. It was subsequently measured in a gaseous xenon time projection chamber (GXe TPC) to validate its radioactivity. Our results demonstrate that ^{37}Ar is an effective and viable calibration source, offering precise calibration capabilities in the low-energy domain of xenon-based detectors.

Keywords: ^{37}Ar, Gaseous Xenon detector, Low-energy, Calibration source

Introduction

Xenon is an exceptional medium for particle detection due to its high density, large atomic mass, and excellent scintillation properties. The dual-phase xenon time projection chamber leverages these superior properties and is extensively utilized in dark matter searches [1–6], neutrino detection [7–11], and related experiments. It operates primarily through precise reconstruction of scintillation signals (S1) and ionization signals (S2) generated by particles depositing energy in liquid xenon (LXe). Scintillation photons detected by photomultiplier tubes (PMTs) generate a pulse signal referred to as S1. Ionization electrons, under the influence of an extraction electric field, drift into the gaseous xenon phase and emit secondary scintillation light through the electroluminescence process, recorded as S2.

The spatial coordinates of an event are reconstructed from the patterns of S1 and S2, with photoelectron counts proportional to the signal's energy magnitude. Geometric variations and inhomogeneous distributions of the electric field and light collection efficiency influence the detector response, leading to significant position dependence of S1 and S2 signal intensities. This not only reduces precision in energy and three-dimensional position reconstruction but also weakens the ability to distinguish between nuclear and electronic recoil events [12]. Therefore, it is essential to use a calibration source that can uniformly distribute throughout the LXe and yield mono-energetic signals to calibrate the detector response.

The ^{37}Ar gaseous source has emerged as an ideal calibration source due to its uniform mixing properties with xenon. The radioactive isotope ^{37}Ar, with a half-life of 35.01 days, can decay to ^{37}Cl and neutrinos [13] through electron capture. During this process, the atomic nucleus captures an electron from the K, L, or M shell. The resulting vacancies are filled by outer electrons, accompanied by emission of X-rays or Auger electrons. The total energy deposition corresponds to the binding energies of each shell: 2.82 keV (K-shell), 0.27 keV (L-shell), and 0.01 keV (M-shell), with decay branch ratios of 90.2%, 8.7%, and 1.1%, respectively [14–17]. The energy depositions from the K and L shells are close to the energy threshold of LXe dark matter detectors, making ^{37}Ar an ideal calibration source. Furthermore, ^{37}Ar can be removed using a cryogenic distilla-

tion tower similar to that used for ^{85}Kr [18], improving its potential application in detector calibration.

Production of ^{37}Ar has long been of interest due to its potential applications in low-background detection and fundamental nuclear research. In the atmosphere, the primary source of ^{37}Ar is the reaction of fast neutrons produced by cosmic rays: ^{40}Ar(n,4n)^{37}Ar [19]. Despite ^{40}Ar constituting up to 99.60% of natural argon, cross-section effects result in low ^{37}Ar yield, accompanied by production of numerous other radioactive isotopes, particularly the long-lived ^{39}Ar, which is highly undesirable. Another production method involves irradiating ^{40}Ca in calcium oxide (CaO) with fast neutrons [20]. This approach has been commonly used due to its high yield [21]. However, extracting ^{37}Ar from CaO requires preparing the target material in powdered form, and the ^{37}Ar gas must subsequently be distilled at high temperatures within a sealed container. This high-temperature distillation process imposes stringent requirements on technology and equipment. Moreover, powdered CaO may be carried along with the gas into the xenon detector, causing contamination. Impurities such as radon, which are co-distilled with ^{37}Ar, can also interfere with low-background experiments.

Thermal neutron irradiation of ^{36}Ar is an effective technique for preparing the radioactive isotope ^{37}Ar. Although the reaction cross-section for ^{36}Ar(n,γ)^{37}Ar is lower than that for ^{40}Ca(n,α)^{37}Ar, the preparation of target material is simpler and the range of products is more limited. This method is particularly suitable for high-sensitivity, low-background experiments such as those used in dark matter detection.

We performed a detailed simulation program based on Geant4 to identify various nuclei expected to be produced after irradiation. In particular, considering the complexity of the reactor neutron source's energy distribution, we needed to avoid producing by-products such as ^{39}Ar that would generate low-energy electronic recoil background in large-scale LXe detectors and would be difficult to remove. Since ^{37}Ar gas can be distributed in gaseous xenon at room temperature, we adopted a GXe TPC to measure ^{37}Ar radioactivity.

The structure of this paper is as follows: Section II describes in detail the preparation of ^{37}Ar, including simulation and feasibility assessment; Section III presents the measurement results of the ^{37}Ar activity through operation and analysis of the gaseous xenon detector.

II. Preparation of ^{37}Ar Calibration Source

A. Experimental Setup and Principles

The target isotope ^{37}Ar was produced by irradiating high-purity (99.935%) ^{36}Ar with thermal neutrons. This process involved sealing ^{36}Ar in a precisely specified quartz ampoule with a diameter of 1 cm, length of 4 cm, and wall thickness of 1 mm. The relative pressure of the package was negative. ^{37}Ar is produced

via neutron capture by ^{36}Ar. The reactor neutron source [22] generated a thermal neutron flux of 1.5$\times$10^{13}$ n/(cm^2 · s), with an irradiation duration of 2.17 hours. Additionally, due to intrinsic properties of the neutron source, an accompanying epithermal neutron flux of 6.25$\times$10^{11}$ n/cm^2/s was present. The uncertainty in neutron flux measurements was estimated at 5%.

Sealing the quartz ampoule was a critical step in the experiment. The melt-seal technique was used: as illustrated in Fig. 1 [Figure 1: see original paper], we used liquid nitrogen on the bottom side of the quartz ampoule to create a low-temperature environment for enrichment of ^{36}Ar, while the other side was sealed using a high-temperature hydrogen torch. This method ensured the airtightness and structural integrity of the seal. Fig. 2 [Figure 2: see original paper] shows the quartz ampoule in its pre- and post-neutron irradiation states. The transformation to a dark purple color is hypothesized to result from microscopic structural and chemical alterations induced by irradiation. Neutron irradiation is known to catalyze formation of color centers within the silicon dioxide matrix. These color centers introduce new energy levels within the electron bandgap, leading to photothermal absorption. The superposition of various absorption bands creates absorption maxima, which impart a tinting effect on the vitreous material [23, 24].

Following irradiation, the quartz ampoule was placed within a pressure transfer apparatus, as indicated by the red arrow in Fig. 3 [Figure 3: see original paper]. The apparatus shown in Fig. 3 is used for precise recovery of all gases generated after irradiation. The process begins with evacuating the apparatus to achieve vacuum, eliminating extraneous atmospheric influences. Release of trapped gas is achieved by applying pressure to the ampoule placed in the vacuum chamber via the pressure transfer apparatus with a maximum capacity of 100 N. The gas then diffuses and homogenizes within the system, allowing controlled and quantified extraction according to experimental requirements, ensuring both accuracy and integrity of the sample.

Based on simulation results (see Sec. II B), the yields and activities of nuclides such as ^{37}Ar and ^{39}Ar can be determined. Furthermore, the "burn-up" effect [25] was evaluated, which refers to potential reaction of newly formed nuclides with neutrons to produce other particles. Calculations indicate that the "burn-up" effect is negligible under our experimental conditions.

B. Thermal Neutron Irradiation Simulation

^{39}Ar is devastating for dark matter search experiments, making mitigation of background signals essential. To precisely identify nuclides generated during ^{37}Ar production and particularly exclude those with extended half-lives that are difficult to eliminate once introduced into the detector, we performed a detailed simulation experiment. The purpose was to emulate actual irradiation conditions and evaluate probabilities of producing other potential nuclides. We established the following simulation parameters.

Based on reactor neutron flux, a simulation was performed ensuring the thermal neutron proportion was maintained at 24/25, with the remaining fraction being epithermal neutrons. All neutrons were introduced randomly from the side to simulate natural variability of neutron incidence. To enhance yield of isotopes other than ^{37}Ar, particularly to amplify reactions with low probabilities, we increased the proportion of isotopes other than ^{36}Ar (the target nucleus for ^{37}Ar production) during simulation. When statistically analyzing results, we adjusted proportions to reflect actual yields, effectively scaling back the amplified ratios. Table 1 presents the composition and mass fractions of all gases before actual irradiation. This approach allows more accurate assessment of nuclide production during irradiation, ensuring detector sensitivity to dark matter signals is not compromised by presence of long-lived background isotopes.

Our simulation, informed by data presented in Table 2, provided cross sections of thermal neutron irradiation reactions and half-lives of selected argon isotopes [25]. This table enumerates cross sections associated with the (n, γ) process, with particular emphasis on ^{37}Ar, which uniquely possesses combined cross-sections for two distinct processes: σ(n, p)+σ(n, α) = (2040\$±\$340) barn. Table 3 extends this analysis to encompass all potential nuclides and their respective yields generated at a simulated pressure of 0.1 bar within the ampoule. It is evident that, in addition to ^{37}Ar, production probability of other nuclides is extremely low.

During simulation, specific attention was directed toward two nuclides, ^{29}Si and ^{41}Ar. Although ^{29}Si exhibits comparatively elevated yield, it is derived from neutron irradiation of ^{28}Si present in the quartz and is not expected to enter the gas source. In contrast, ^{41}Ar, despite its certain yield, has a half-life of merely 109.61 min, indicating rapid decay. Furthermore, presence of ^{39}Ar, if uniformly mixed with xenon within the detector, would pose a challenge for removal, significantly increasing the detector's background level. Simulation results substantiate our rationale for proceeding with subsequent experimental endeavors.

C. The Gaseous Xenon Time Projection Chamber

Before injecting the ^{37}Ar calibration source into ton-level detectors, we inject it into a GXe TPC to validate its performance. The detector operates with gaseous xenon at room temperature. Xenon is chosen as the detection medium due to its pivotal role in dual-phase time projection chambers (LXe TPCs) used in dark matter and neutrino experiments such as PandaX-4T, XENONnT, LZ and others. GXe TPCs provide several notable advantages for this work. First, GXe TPCs avoid operational complexities associated with cryogenics and slow control systems. Second, they feature lower detection thresholds and reduced background compared to LXe TPCs, as background is dominated by gamma rays and cosmic muons. Additionally, both argon and xenon, as members of the same group in the periodic table, exist in gaseous phase at room temperature, enabling uniform distribution within the detector. This uniformity is advanta-

geous for measuring calibration source activity and facilitating verification of activity estimations. Although gaseous xenon emits fewer photons compared to liquid xenon, leading to reduced efficiency in detecting S1-S2 paired events, S2-only analysis can estimate the decay rate with high detection efficiency.

The schematic diagram of the GXe TPC used in this measurement is shown in the top panel of Fig. 4 [Figure 4: see original paper]. This TPC serves as a prototype detector for the RELICS experiment [11]. The TPC is mounted inside a double-wall cryostat to provide thermal insulation and structural support. It is equipped with 14 Hamamatsu R8520-406 PMTs, compactly placed on the top and bottom of the TPC and optimized for high VUV photon detection efficiency. These PMTs operate at a working voltage of -800 V. Each array comprises seven PMTs in a regular hexagonal pattern, positioned above and below the drift region. The TPC walls are constructed of Teflon, which has excellent VUV reflectivity, enhancing light collection efficiency. This arrangement provides relatively high light collection efficiency and improves spatial resolution of detected events.

The bottom panel of Fig. 4 shows the operational principle of the GXe TPC for detecting ^{37}Ar decays. ^{37}Ar decays produce scintillation photons and ionization electrons in GXe. Scintillation photons are detected directly by PMTs as S1 signals. Ionization electrons drift under the electric field toward the proportional luminescence region, where they emit secondary scintillation light (S2). Top and bottom arrays of photomultiplier tubes capture S1 and S2 signals, enabling precise event reconstruction including energy and three-dimensional position.

The detector system integrates various subsystems including cryogenic, gas purification, data acquisition, and recycling equipment. The TPC operates at approximately 170 kPa pressure, with gaseous xenon continuously circulated through a hot getter system for purification. This process removes electronegative impurities such as oxygen and water, which may absorb scintillation light and ionization electrons, reducing detection and identification efficiency of ^{37}Ar decays. The electron drift region is defined by electrodes including the anode, gate, cathode, and five shaping rings, which establish a uniform electric field for electron drift and conversion of electrons to proportional scintillation photons. The anode is maintained at +1200 V to amplify S2 signals, while the gate, cathode, and screen are set to -1800 V, -2400 V, and -800 V, respectively. This voltage configuration ensures stable operation, minimizes risk of electrical breakdown, and provides suitable conditions for seamless calibration source injection during detector operation while minimizing impact on xenon gas purity.

Activity of the injected source is calculated based on volumetric relationships among the pipeline (including the cryostat containing the GXe TPC), storage container, and drift region of the TPC, assuming uniform distribution of ^{37}Ar. Detailed information about volumes within the injection system is provided in Table 4.

The ^{37}Ar source is introduced through multiple injections. The circulation pipe

enclosed by valves V1, V2 and V3 is defined as a dilution volume for source injection. Each injection is performed through several steps. First, the dilution volume is pumped to vacuum. The ^{37}Ar is then introduced to the dilution volume by opening V1. Consequently, 11% of the total source is introduced to the dilution volume and will be injected into the circulation. The source is then uniformly distributed into the system with a total volume of 28 L. As the drift region of the TPC is only 181 mL, another dilution factor of 0.6% is introduced. As a result, only 0.07% of total radioactivity is measurable in the GXe TPC.

III. Measurement of ^{37}Ar Radioactivity within the GXe TPC

A. Injection of the ^{37}Ar Source

The ^{37}Ar source is stored in a stainless steel container with 500 mL volume. A dedicated pipeline was developed to allow controlled introduction of a fixed portion of the ^{37}Ar source into the gaseous xenon detector system. A simplified diagram illustrating the injection and gas recycling route is shown in Fig. 5 [Figure 5: see original paper]. This dosing system is designed to allow precise control over source injection.

B. Data Acquisition and Signal Processing

To achieve high detection efficiency of low-energy signals from the ^{37}Ar source, all waveforms from PMTs are digitized using CAEN V1725 digitizers, which employ dynamic acquisition window (DPP-DAW) firmware for self-triggering readout. Digitized raw data are stored on a server, while subsequent event reconstruction and analysis are performed on dedicated analysis servers. Data acquisition was carried out over an 8-hour period both before and after injection of the ^{37}Ar source, allowing background subtraction. A software package was developed to process data acquired from each PMT and group them into peaks. A peak is defined as a waveform featuring two or more PMT signals within 300 ns. Scintillation and ionization signals from interactions with energy depositions in the GXe TPC, including decays of ^{37}Ar, produce peaks in the data.

The area of a peak is proportional to the number of photons detected by PMTs and is expressed in units of photoelectrons (PE) as calibrated by single photon counting with an LED. S1 peaks, induced by scintillation photons produced by direct excitation of Xe atoms or by recombination of electron and ion pairs from ionization, have a narrow temporal distribution with typical spread below 200 ns. S2 peaks, induced by electroluminescence of electrons drifting in GXe at strong electric fields (notably between gate and anode electrodes), have a wider temporal distribution with typical spread above 200 ns. The time spread of a peak is characterized by the leading time, defined as the time interval between the 0% to 50% percentile of the waveform area. The relative peak area distribution on PMT arrays depends on light collection efficiency of each PMT and is used to reconstruct interaction position. For S2 peaks induced

by interactions in the drift region, horizontal distribution is reconstructed from area distribution pattern on the top PMT array. S2 peaks can also be produced above the anode or below the cathode since the detector is operated in GXe mode. The area fraction of top (AFT), defined as the ratio of area recorded by top PMTs to total area, is distinguishable for S2 peaks produced in the drift region versus those below the cathode or above the anode.

The distribution of peaks in area and leading time space is shown in Fig. 6 [Figure 6: see original paper]. Peaks collected before and after injection of the ^{37}Ar source are shown in top and bottom panels of Fig. 6, respectively. Pulses with leading time above the dashed red line and area greater than 100 PE are attributed to beta or gamma interactions within the drift region of the GXe TPC. Pulses with area of 20 PE and leading time of 700 ns characterize S2 produced by single electrons drifting between gate and anode. Pulses with area below 500 PE and leading time below the dashed red line correspond to S1 signals.

Additional populations appear after injection of the ^{37}Ar source: signals with area around 2000 PE correspond to S2 from K-shell ^{37}Ar electron capture events in the drift region; pulses with area around 200 PE correspond to S2 from L-shell ^{37}Ar electron capture events in the drift region; pulses with area below 10 PE and leading time below the dashed red line correspond to S1 from K-shell ^{37}Ar electron capture events. Identification is based on the known energy spectrum of ^{37}Ar, in which K-shell and L-shell electron capture lines are at approximately 2.8 keV and 0.27 keV, respectively. Given the W-value of gaseous xenon (about 22.0 eV) and single-electron gain of approximately 20 PE, the expected S2 area for K-shell events is around 2000 PE, and L-shell contribution is roughly one-tenth of that, matching well with observed populations.

This study focuses on signals corresponding to ^{37}Ar K-shell decay events occurring within the drift region. Events detected outside this region are classified as background events. To suppress these background events, properties such as light collection efficiency distribution and electron transport processes must be understood. Since these have not been thoroughly simulated and photon detection efficiency of PMTs remains insufficiently understood, these factors introduce constraints in accurate signal analysis. Consequently, a data-driven analysis approach is used to reduce background and estimate activity of the ^{37}Ar source, compensating for lack of comprehensive detector simulations and allowing evaluation of ^{37}Ar source activity.

The analysis focuses on S2 signals, represented by regions above the red dashed lines in Fig. 6. Accurately determining ^{37}Ar activity requires meticulous data selection to minimize background noise impact. As shown in Fig. 7 [Figure 7: see original paper], three different types of background noise were identified and removed.

First, events occurring between anode and gate exhibit positive correlation between S2 area and leading time. These events are located in the lower region of

distribution shown in top panel of Fig. 7, indicating relationship between event timing and background signal intensity. Second, when photomultiplier is set to -800 V with positive anode voltage, ionized electrons generated by high-energy events can drift toward the anode under influence of electric field between anode and top PMT array. This drift results in peaks with larger area proportion on top PMTs. Similarly, events occurring between cathode and screen tend to produce signals with smaller area fraction on top. Furthermore, some pulses exhibit reduced light collection efficiency in specific regions, appearing on left side of distribution in top panel of Fig. 7. To correct for this bias, a Crystal Ball model is employed to describe this phenomenon and fit signal counts.

These background events are effectively removed by selecting waveforms based on their area fraction of top (AFT) and leading time. Distribution of AFT for events at fixed area in drift region is described by a skew-Gaussian to determine cut acceptance. The boundary corresponding to selection efficiencies of 2.5% and 97.5% is determined to be (0.627, 0.703). Events occurring between anode and gate have similar area fraction of top as signal events but are characterized by shorter leading times compared with events in drift region. Peaks with leading times shorter than approximately 1030 ns are excluded in this measurement, resulting in selection efficiency of 99%.

^{37}Ar K-shell Activity Estimate After peak selection, the magnitude distribution of area was obtained. The selected S2 spectrum from ^{37}Ar K-shell decay was analyzed using Gaussian and Crystal Ball distributions to determine event rates, as shown in Fig. 8 [Figure 8: see original paper]. The Crystal Ball distribution was selected because it provides more accurate representation of the spectrum, particularly accounting for effects of low photon detection efficiencies in certain regions of the projection chamber. The Crystal Ball function combines a Gaussian core with power-law tail, offering flexibility to model asymmetric features observed in the spectrum. Mathematically, it is expressed as:

$$f(x;\alpha,n,\bar{x},\sigma) = A\exp[-(x-\bar{x})^2/(2\sigma^2)] \text{ for } (x-\bar{x})/\sigma > -\alpha$$
$$f(x;\alpha,n,\bar{x},\sigma) = B\,[C - (x-\bar{x})/\sigma]^{-n} \text{ for } (x-\bar{x})/\sigma \leq -\alpha$$

where α determines the point at which Gaussian transitions into power-law tail; n indicates steepness of power-law tail; A and B are normalization constants ensuring continuity and smoothness at transition point.

The fit using Crystal Ball distribution yielded observed activity of approximately 14.96 Bq. Considering K-shell decays constitute 90.2% of all ^{37}Ar decays, and factoring in selection efficiency of 94.0% achieved through area fraction of top (AFT) and leading time cuts, total activity within drift region is estimated at $17.646 \pm 0.025(\text{stat.}) \pm 0.007(\text{sys.})$ Bq. This activity level is well-suited for calibrating liquid xenon dark matter detectors such as PandaX-4T and XENONnT.

IV. Summary

This study successfully synthesized the radioactive isotope ^{37}Ar using a reactor-derived thermal neutron source. With a half-life of 35.01 days, ^{37}Ar is particularly valuable for calibrating LXe TPCs in low-energy regions. The isotope was produced by irradiating high-purity ^{36}Ar with thermal neutrons in a quartz ampoule. Geant4 simulations were used to predict product types and activities, ensuring minimal production of long-lived isotopes such as ^{39}Ar.

The prepared ^{37}Ar source was injected into a GXe TPC for preliminary measurements. Upon injection, a notable increase in peak counts around 2000 PE was recorded, confirming successful synthesis and deployment of the source. A data-driven analysis approach was applied to reduce background noise and focus on S2 signals of ^{37}Ar K-shell decay. The activity of ^{37}Ar K-shell decay was measured to be approximately 14.96 Bq. The conversion of ^{36}Ar to ^{37}Ar via neutron activation is a critical factor in determining expected activity levels. Inaccurate estimation of initial ^{36}Ar content can lead to errors in calculating decay rates and activities of ^{37}Ar, highlighting the importance of precise control of argon content during preparation. To mitigate this issue, thorough review of the gas sealing process, particularly impact of temperature distribution during fusion sealing, could identify procedural errors that might contribute to underestimation.

In conclusion, this study successfully prepared and measured activity of ^{37}Ar, demonstrating its feasibility as a calibration source for low-energy dark matter searches in LXe TPCs. These findings establish a solid foundation for future applications in detector calibration and dark matter research.

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Note: Figure translations are in progress. See original paper for figures.