$^{39}\text{Ar}/\text{Ar}$ measurements using ultra-low background proportional counters

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Abstract

Age-dating groundwater and seawater using the $^{39}\text{Ar}/\text{Ar}$ ratio is an important tool to understand water mass-flow rates and mean residence time. Low-background proportional counters developed at Pacific Northwest National Laboratory use mixtures of argon and methane as counting gas. We demonstrate sensitivity to ^{39}Ar by comparing geological (ancient) argon recovered from a carbon dioxide gas well and commercial argon. The demonstrated sensitivity to the $^{39}\text{Ar}/\text{Ar}$ ratio is sufficient to date water masses as old as 1000 years.

1. Introduction

Argon-39 is an important tracer in environmental age dating of glaciers, oceans, and groundwater (Loosli 1983, Loosli and Purtschert 2005). The 269-year half-life of ^{39}Ar allows dating in the challenging age range between methods that are applicable for samples as old as ~50 years (^3H , $^3\text{H}/^3\text{He}$, ^{85}Kr , CFCs, SF_6) and methods applicable for samples older than 1000 years (^{14}C , ^{36}Cl , ^{81}Kr). This intermediate age range, 50 to 1000 years, is important for characterizing the flow of groundwater (Corcho Alvarado, Purtschert et al. 2007, Newman, Osenbrück et al. 2010, Visser, Broers et al. 2013). Argon-39 is almost exclusively produced by cosmic-ray interactions and production by man in the atomic age is negligible (Cennini, Cittolin et al. 1995). Additionally, the $^{39}\text{Ar}/\text{Ar}$ ratio is used so quantitative argon yields from the water are not required and details about the recharge process do not influence the dating results in contrast to other methods. This lends an inherent robustness to ^{39}Ar age dating and facilitates quantification of water migration and climate history (Corcho Alvarado, Leuenberger et al. 2011). Despite the utility of ^{39}Ar age dating, the required sensitivity for direct beta counting is challenging and the only laboratory to develop relevant sensitivities is the underground laboratory in Bern (Loosli, Moll et al. 1986). Sensitivity to ^{39}Ar

has been demonstrated using atom trap trace analysis and atomic mass spectroscopy (Jiang, Williams et al. 2011).

Ultra-low-background proportional counters (ULBPCs) have been developed at Pacific Northwest National Laboratory (PNNL) (Aalseth, Day et al. 2009). These counters are designed to measure radioactive isotopes from environmental samples including ^3H , ^{14}C , ^{37}Ar , and ^{39}Ar . After installation in an ultra-low-background counting system (ULBCS) at the PNNL Shallow Underground Laboratory, the ULBPCs have been shown to have a count rate of 234 counts per day (cpd) in the energy range 3-400 keV when filled with 7 atm of P-10 counting gas, 90% Ar-10% CH_4 (Seifert, Aalseth et al. 2012).

Atmospheric argon is predominantly composed of ^{40}Ar ; however, trace amounts of ^{39}Ar are found in the atmosphere. Argon-39 decays to ^{39}K via beta decay with a Q-value of 565 keV and a half-life of 269 years. The activity of atmospheric argon is 1.01 Bq/kg, corresponding to an abundance of 0.808 ppq (Benetti, Calaprice et al. 2007). The ULBPCs at PNNL have an internal volume of 100 mL and are designed to run with pressures up to 10 atm. Commercial P-10 gas (90% Ar, 10% CH_4) prepared with atmospheric-sourced argon thus provides 14.0 disintegrations per day of ^{39}Ar activity for each atmosphere of fill pressure at 297 K. With a 3-keV low-energy threshold and a 7-atm fill pressure, ULBPC efficiency for ^{39}Ar is estimated to be 85%. Thus the observed rate under these conditions with commercial P-10 gas of 234 cpd is expected to include 83.3 cpd from the ^{39}Ar activity, roughly one-third of the observed rate.

Argon-39 is created in the atmosphere by cosmic-ray interactions producing fast neutrons driving the $^{40}\text{Ar}(n,2n)^{39}\text{Ar}$ reaction. Although ^{39}Ar can be created underground through the reaction $^{39}\text{K}(n,p)^{39}\text{Ar}$ (Yokochi, Sturchio et al. 2012), gas sourced from an appropriate geological depth and composition has been found to be low in ^{39}Ar activity (Acosta-Kane, Acciarri et al. 2008). Extraction of low-activity argon from a carbon dioxide well in Colorado has recently been developed as a resource for rare-event searches such as direct detection of dark matter (Back, Calaprice et al. 2012). Experimental efforts have not measured any ^{39}Ar radioactivity in the argon from this CO_2 well, the best limit is currently 0.65% the radioactivity of atmospheric argon (Xu, Calaprice et al. 2012). This manuscript describes results from filling a ULBPC at PNNL with this geological argon.

In this work, ULBPC measurements were made on argon from two sources, one referred to as geologic argon and one referred to as atmospheric argon. The measurements were made with a single ULBPC. The geologic argon was extracted from a CO_2 gas stream from a southwestern Colorado well by a vacuum-swing pressure-absorption process. The extracted gas mixture, a mixture of helium, nitrogen and argon, was transported to the Fermi National Accelerator Laboratory and purified in a cryogenic distillation column (Back, Alexander et al. 2012). The atmospheric argon sample was commercial detector grade P10 gas with argon distilled from air.

Counting these argon samples permits measurement of ULBPC with and without atmospheric ^{39}Ar activity allowing a demonstration of the sensitivity for age dating using $^{39}\text{Ar}/\text{Ar}$ ratios. Additionally, the lower background levels observed with geologic argon are relevant for improving sensitivity for ^3H and ^{14}C samples prepared as methane, where geologic argon can be used for the remainder of the required count gas in an ULBPC.

2. Proportional Counter / Experimental Description

The ULBPC technology developed over the past several years by PNNL is the product of an effort to produce a low-background, physically robust, gas-proportional counter for low-level counting of gas samples. The ULBPC was designed to handle a wide range of gas volumes and has high beta detection efficiency. The materials chosen for the detector were selected for their radiopurity. High-purity electroformed copper offers low radioactivity along with good electrical and mechanical properties. The

ULBPC body is an electroformed copper cylinder roughly 9 inches in length having a ~0.1 L internal volume. PCTFE (polychlorotrifluoroethylene), was chosen for the gas seal and high-voltage insulation for its low gas permeability and reasonable measured radioactivity. The ULBPC anode wire is 0.001-inch-diameter niobium.

A shallow underground laboratory was recently constructed at PNNL in Richland, Washington (Aalseth, Bonicalzi et al. 2012). This facility, commissioned in late 2010, supports the dual objectives of performing ultra-sensitive measurements and providing ultra-clean materials and radiation detector research. The shallow underground facility has an effective depth of ~30 meters water-equivalent.

After the ULBPC was filled with a gas sample above ground, it was moved to the shallow underground laboratory and placed in the ULBCS. The ULBCS is designed with both active and passive shielding around the detectors (Seifert, Aalseth et al. 2012). The passive layers of shielding include approximately 3 inches of copper around the core measurement chamber that houses the detectors, 6 inches of lead around the copper, and an additional outer layer of 1 inch of 30%-borated polyethylene for neutron shielding. This passive shielding is surrounded by active cosmic-ray veto shielding in the form of 2-inch-thick polyvinyl toluene scintillator paddles. The signal path in the ULBCS cave has been optimized to reduce micro-discharge events (Mace, Aalseth et al. 2012).

The count gas composition loaded into the ULBPC detector was 90% Ar, 10% CH₄, with a partial pressure of 6.3 atm of argon and a total fill pressure of 7 atm. After the detector was installed in the ULBCS cave, an ²⁴¹Am source (~40 µCi) was placed on the detector and high-voltage bias applied. The high-voltage bias on the ULBPC detector was adjusted, near 3250 V for this type of load, such that the gamma-ray peak of 59.5 keV from the ²⁴¹Am source was centered at a specific channel (nominally channel 10,405 of 16384) to optimize the dynamic range for an energy range of 0-400 keV. The resulting calibration peak is shown in Figure 1. After energy calibration data were collected and the ²⁴¹Am source was removed, the detector counted the gas sample. The geologic argon was counted in two separate ULBPC fills, one for 77.7 days (live time of the detector) and one for 120 live days, in the ULBCS cave. The atmospheric argon was measured for 45.4 live days. All three fills were performed with the same proportional counter.

The system stability was found to be excellent over these time periods. Fluctuations in the counting rates were found to be consistent with the expected statistical fluctuations over the 100 day periods. The system calibration is now done weekly and the gain variation is less than 1% per week.

The data collected from the argon measurements were analyzed using pulse-shape discrimination to examine each digitized pulse in the data set (Aalseth, Day et al. 2012). The differential pulse-height spectrum in the energy range 0-400 keV is shown for one ULBPC fill in Figure 2. The geologic argon sample is shown as the blue trace (crosshatch fill). The atmospheric gas sample is shown as the black trace (no fill). The red trace (dotted fill) shown in Figure 2 is the difference between the atmospheric and geologic argon spectra. The integral difference between the atmospheric and geologic argon spectra over the range 3-400 keV is 69.6±2.7 cpd for the first fill and 71.0±2.6 cpd for the second fill. Both samples were consistent within the statistical errors and the combined measurement yields a rate of 70.3±1.9 cpd from ³⁹Ar in atmospheric argon in the ULBPCs.

3. Discussion

The data from the geologic argon show a reduction of 70.3±1.9 cpd. This is significantly lower than the expected ³⁹Ar count rate in the atmospheric argon of 83.3 cpd. Since the geologic argon has been demonstrated to have ³⁹Ar levels below the sensitivity of this measurement, this disagreement between the observed and expected rate suggests additional differences between the measurements.

Cosmic rays that escape detection in the ULBCS cosmic-ray veto are the predominant background, responsible for 70-95% of the background events not attributable to ^{39}Ar . The rate of events coincident with the cosmic-ray veto is observed to be 0.5% higher in the data taken with the atmospheric argon sample. Thus, a systematic uncertainty of 1% (1.6 cpd) is assigned to the background subtraction due to a change in rate. However, if this change in rate is due to an efficacy change in the cosmic ray veto, then the systematic error could be larger. The total rate of cosmic-ray coincident events is 10,000 cpd. If the 0.5% change in the veto-coincident rate is symptomatic of a change in veto efficacy, then the systematic effect could be as high as 30% (50 cpd). Here we assign a systematic error of 10 cpd to the cosmic-ray veto efficacy.

The events coincident with signals in the cosmic-ray veto are useful to constrain some systematic effects. The relative argon loads between the two samples can be estimated by measuring the energy deposition of through-going muons. Muons passing through the ULBPC loaded with the geological argon sample left $2.7 \pm 1.5\%$ less energy. Given the uncertainty in the energy discrepancy, the argon loading is treated as a 5% systematic error, 3.5 cpd. More careful accounting can reduce this below the statistical errors expected in ^{39}Ar dating.

^{85}Kr may be in commercially purified atmospheric argon in quantities that could effect the ^{39}Ar measurement (Benetti, Calaprice et al. 2007). The krypton contamination of the atmospheric argon used in this measurement was not quantified, so we assign a systematic error of 5% (3 cpd). The primary source of error due to krypton contamination is the possibility that the krypton removal efficiency fluctuates in the commercial distillation of air. Future measurements of the krypton levels in the atmospheric argon samples can limit this systematic effect.

The excess rate in the atmospheric argon is found to be 70.3 ± 1.9 (statistical) ± 18.1 (systematic) cpd and the largest systematic error is from the efficacy of the cosmic-ray veto. The deficit in the count rate demonstrates the ability to measure ^{39}Ar concentrations important for age dating samples up to 1,000 years old. The ^{39}Ar dating accuracy for various sample ages is shown in Figure 3. This demonstrated capability requires 630 mL of argon gas, which can be extracted from approximately 3 tons of water. For counting times of 100 days, these counters are able to screen samples of argon to 5% the atmospheric ^{39}Ar abundance. This screening capability is not sufficient for liquid argon dark matter experiments where a screening capability of 0.5% of the atmospheric abundance is desired. The residual background would need to be reduced to ~ 1 cpd to enable such a screening capability with the ULBPCs.

The residual background appears to be dominated by leakage through the cosmic-ray veto surrounding the ULBCS. The cosmic-ray veto does not cover the front face of the shielding and the residual backgrounds measured with this geologic argon sample match the spectrum of the veto-coincident events. A number of improvements could be implemented to reduce the backgrounds in the ULBPCs, including multi-wire proportional counters to afford cosmic-ray rejection within an individual detector and extending the cosmic-ray veto to cover the front face of the ULBPC shield.

Finally, residual activity in the materials of the ULBPCs may contribute a small number of events to the counting rate. The activation of the copper ULBPC housing is expected to result in ~ 0.3 events / (neutron / cm^2) and there is an ongoing effort to measure the neutron flux within the ULBPC shielding enclosure.

4. Summary and Outlook

Sensitivity to natural levels of ^{39}Ar has been demonstrated using a sample of geologic argon. A sample of argon known to have activity less than 0.5% of atmospheric argon, ~ 400 cpd per kilogram, was counted in a ULBPC at PNNL. The demonstrated background using this argon mixed with geological methane is

166±1.5 cpd in the 3-400 keV energy range. The ^{39}Ar signal from two samples of 7 atm P-10 mixture using atmospheric argon was demonstrated to be 70.3 ± 1.9 (stat) ± 18.1 (syst) cpd in the same energy range, using two independent geologic argon samples for background measurements. These results imply ^{39}Ar age dating with the ULBPCs at PNNL is feasible for samples as old as 1,200 years with 100-day counting times. This establishes PNNL as the second laboratory worldwide capable of such measurements with low-level counting for environmental transport applications.

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Figure 1. Calibration spectra from ^{241}Am source with a Gaussian fit to the 59.5 keV peak. The induced copper x-ray peak at 8.0 keV is also fit with a Gaussian. The resolution (FWHM) is 17% at 8.0 keV and 12% at 59.5 keV.

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Figure 2. The measured energy spectra for two argon samples are shown. The black (empty) histogram is from a 45.4 live day count of the atmospheric argon. The blue (hatched) spectrum is from a 77.7 live day count of the geologic argon. The red (dotted) histogram is the difference between the two spectra and is attributed to the ^{39}Ar in the atmospheric argon.

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Figure 3. The expected dating accuracies for samples of various ages using the demonstrated signal and background rates from this work are shown. The errors are the expected 1σ statistical errors in the age determination. Points are only graphed if the expected signal is 3σ above the demonstrated background.

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