

Initial tests of Accelerator Mass Spectrometry with the Argonne Gas-Filled Analyzer and the commissioning of the MONICA detector

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ABSTRACT

As the scope of Accelerator Mass Spectrometry (AMS) expands, there is an increased need to extend the capability of isobaric separation to the medium-heavy mass region. Existing AMS facilities are limited in their ability to separate radioactive nuclei in the $A = 100$ –200 range of interest from their neighboring stable isobars, as such measurements require higher energies than available in most facilities. ATLAS is one of the highest energy system used for AMS based experiments and has enabled isobaric discrimination for medium to heavy nuclides, notably via the Gas-Filled Magnet technique. A preparatory experiment performed in November, 2019, successfully demonstrated isobaric separation of ^{92}Zr - ^{92}Mo using the Argonne Gas-Filled Analyzer (AGFA) with high magnetic rigidity. Since that time, MONICA, an eight-anode ionization chamber that measures both energy loss and position with two sets of split anodes, has been developed to aid in AMS experiments at AGFA and has undergone four commissioning runs at the Nuclear Science Laboratory at the University of Notre Dame utilizing Si, Fe/Ni, and Mn beams. This report presents the AGFA AMS run (November 2019) and the subsequent commissioning runs of the MONICA detector, including preliminary measurements on the long-lived isotopes ^{39}Ar (268 y) and for the first time on ^{42}Ar (33 y).

1. Motivation and background

The utilization of the Argonne Tandem Linac Accelerator System (ATLAS) for Accelerator Mass Spectrometry (AMS) measurements offers a unique set of capabilities. Specifically, the combination of a positive ion source and the high energy per nucleon available from ATLAS allows for high sensitivity measurements of radioisotopes not currently possible at any AMS facility [1,2]. To further optimize the use of ATLAS for AMS measurements in the medium-heavy mass range where isobaric contamination is present, the use of the Argonne Gas-Filled Analyzer (AGFA) for AMS has been explored for the first time and a new detector, MONICA, was developed.

Previous AMS measurements at ATLAS have been performed with the Enge split-pole spectrograph operated in gas-filled mode [2,3]. At its focal plane, the Enge split-pole spectrograph had two detectors: a Parallel Grid Avalanche Counter, for the x- and y-position of entering particles, and an ionization chamber, for energy loss information. However, the Enge split-pole spectrograph has a magnetic rigidity limit of

0.7 Tm and, for heavier isotopes in the range of 100–200 nucleons, it is difficult to bend the beam at the high energies ATLAS provides.

Installed in 2017, the AGFA Spectrometer was developed at ANL for the separation of heavy reaction products up to and beyond the actinides [4]. AGFA has a high magnetic rigidity (2.5 Tm) which allows for the bending of heavy ions at high energies, opening up the possibility to study heavier isotopes using AMS at ATLAS. However, the current focal plane detectors on AGFA cannot give the detailed ion parameters needed to conduct such a measurement, so the commissioning and use of a new detector, described below, is required.

2. Preliminary AMS experiment at AGFA

An experimental run to demonstrate the feasibility of performing AMS using AGFA with minimal changes to the existing setup was conducted in November, 2019. Stable isobars ^{92}Mo and ^{92}Zr were

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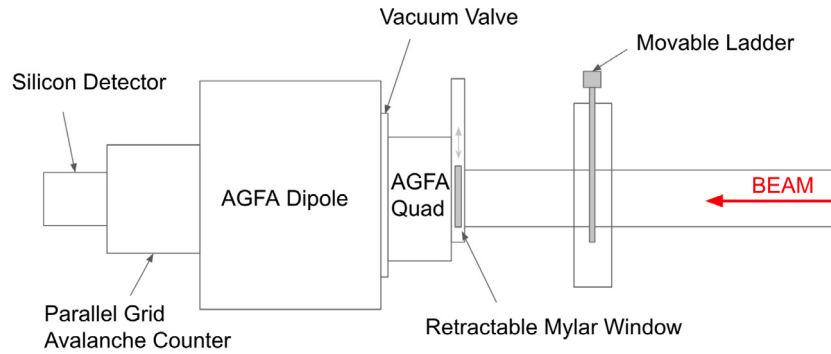


Fig. 1. A schematic of the setup of AGFA used for the initial experiment in 2019.

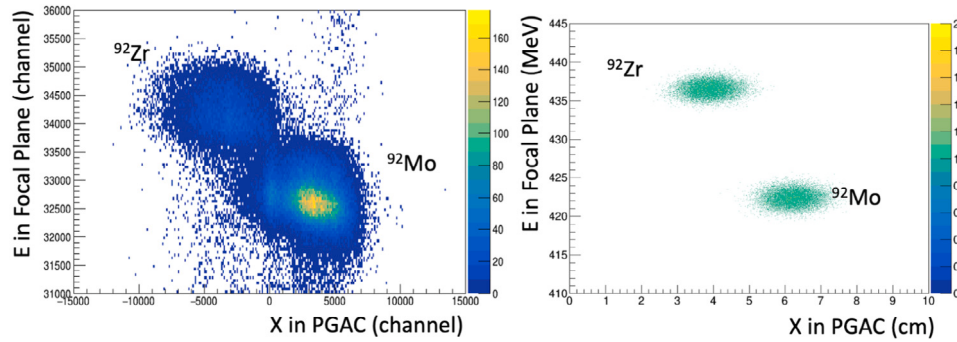


Fig. 2. Graphs showing the separation of ^{92}Mo and ^{92}Zr using AGFA at ANL. The graph on the left shows energy seen by the silicon detector on the focal plane verses the position recorded by the PGAC. The graph on the right shows the simulation of the ion transport through AGFA using experimental conditions.

selected for this experiment, as they could show the ability of AGFA to bend heavy isotopes and have a higher chance of displaying a clear separation between them due to their 2- Z difference. A cathode of enriched metallic ^{92}Mo and ^{92}Zr was mixed and used in the Electron Cyclotron Resonance (ECR) source, and the resulting beam, extracted as $^{92}\text{Mo}^{17+}$ and $^{92}\text{Zr}^{17+}$, was accelerated through ATLAS at an energy of 644 MeV.

As shown in Fig. 1, a movable target ladder was placed in front of AGFA, containing a Faraday Cup, a silicon detector, a scintillator, and a carbon foil stripper. The existing focal plane detector system, consisting of a Parallel Grid Avalanche Counter (PGAC) for position measurements and a silicon detector for energy measurements, was also used.

In the initial experiment, AGFA was filled with 7.8 torr N_2 with a magnetic rigidity of 1.14 Tm. With these settings, ^{92}Mo and ^{92}Zr were able to be clearly separated, both in position and energy, as seen in Fig. 2. This experiment provides evidence that AGFA can be used to separate two stable isobars, and supports a conclusion that AGFA can be used in future AMS experiments to separate a long-lived isotope from its isobaric contaminant.

There were, however, a few limiting factors to the efficacy of this first AMS run using AGFA. For one, the fragility of the window between the PGAC and AGFA limited the gas pressure in AGFA. Because of this window, the maximum operating pressure of the PGAC was 8 torr, which limited the pressure in AGFA to 7.8 torr. Based on simulations, however, the optimal pressure in the magnet for separation of ^{92}Mo and ^{92}Zr was around 9.5 torr. Additionally, the only energy discrimination tool in the setup was the focal plane silicon detector, which limited the identification of the isobars.

A detector that not only combines position and energy loss capabilities into one unit, but also allows for the sampling of two position/angle measurements will greatly improve the ability to identify rare isotopes using AGFA.

3. Design of MONICA

MONICA is a newly developed, position-sensitive, multi-anode ionization chamber. The detector (Fig. 3) was built at the Hebrew University of Jerusalem and was modeled from previous, successful designs [5, 6]. Several proof of principle and commissioning runs were performed at the Nuclear Science Laboratory (NSL) at the University of Notre Dame [7].

MONICA is comprised of a 24.6 cm $\times$ 20 cm $\times$ 25.6 cm chamber with a removable top. A 26 pin-HD feed-through mounted on the removable top passes signals from each of the eight anodes, the cathode, and the grid through the detector to a Mesytec MPR-16-L pre-amplifier. The cathode, anode, and grid plates are pictured in Fig. 3, with the bottom of the anode plate on the top. The anode and cathode plates are 9.7 cm apart with the frisch grid in between the two, positioned 1.83 cm from the anode plate. The support structure posts are made of Delrin[®] while the anode and cathode boards, as well as the frame for the Frisch grid, are made from printed circuit boards with gold-plated copper pads. The Frisch grid consists of gold-coated tungsten wires with 1 mm pitch and 20 μm diameter. Using three SHV feedthroughs, the anode is held at a positive voltage ($\sim +100$ –300 V), the cathode is kept at a negative voltage (~ -100 –300 V), and the grid is grounded to the spectrograph chamber. The bias voltages are isotope specific and vary depending on ion beam energy and detector gas pressure. A Mylar window mounted at the entrance is 8 cm height by 15 cm width, with thicknesses used ranging from 2 to 6 μm . The window has a support grid of longitudinal, stainless steel wires of 0.1 mm diameter and transverse, nylon wires of 0.2 mm diameter. This support grid can be biased via a resistor chain between the cathode and grid but, in tests not covered by this report, voltage applied to the support grid did not affect signals in any meaningful way.

The formatting of MONICA's anodes is seen in Fig. 4. The anode plate is segmented into six sections, the first five are 45 mm each,



Fig. 3. The top plate of the MONICA detector inverted on a tabletop, with the internal fixtures attached. The green plates are the cathode, Frisch grid, and anode from bottom to top and field cage electrodes surrounding the sides. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

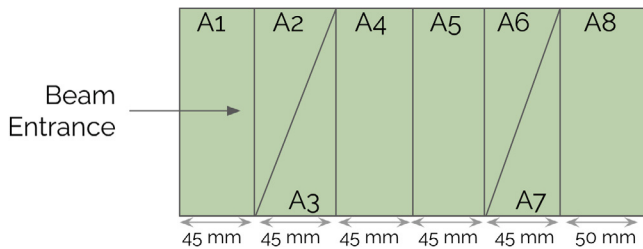


Fig. 4. Schematic of the anode segmentation into eight separate anodes, including two sets of split anodes. This anode design gives MONICA the ability to distinguish position as well as energy loss.

while the last is 50 mm. These lengths were selected on practical considerations for a variety of ions and energies. Along with four undivided segments (Anodes 1, 4, 5, and 8), two sections are split diagonally and comprise Anodes 2 and 3, and Anodes 6 and 7. The division of these segments into two equal triangles allows MONICA to gather information on both the energy loss and position of the particles as they move through the detector. Particles entering the center of the anodes will deposit approximately equal amounts of energies in A2 and A3 (or A6 and A7), while particles that are more toward one side will deposit more or less energy in the split anodes depending on their position. For example, a particle entering beam right will deposit more energy in A3 and A7 than it will A2 and A6. A summary of commissioning experiments done to affirm this ability and calibrate the position changes of beams entering the detector can be found in Section 4.1.1.

For use in future measurements, a movable shield was attached to the front of the detector. Comprised of two, 1 mm-thick Al plates tipped with thin, polished Ta strips to reduce slit scattering, this shield has the ability to symmetrically cut beam in the x -direction via a rotary cable operated from the spectrograph's exterior. This shield was not used in any measurements thus far, but can be used in future measurements.

4. Commissioning of MONICA

4.1. Commissioning of MONICA at the NSL

The MONICA detector was tested at the NSL at the University of Notre Dame throughout four separate experiments between September, 2020, and June, 2021. All runs utilized the Multi-Cathode Source of Negative Ions via Cesium Sputtering (MC-SNICS), the 10 MV FN Tandem Accelerator and one of two separate beamlines: the scattering chamber's beamline or the AMS beamline. See Ref. [7] for more information about lab functions at the NSL.

4.1.1. Proof of principle runs with MONICA inside scattering chamber

Two separate runs were performed with MONICA mounted inside a large scattering chamber. This chamber houses a vertically-mobile target ladder and three circular platforms, two of which rotate via cranks external to the chamber. The target ladder housed two collimators to help with beam tuning and focusing. MONICA was mounted on the middle platform and was able to be rotated in and out of the 0° position. A shield, used to block the MONICA window from scattering during tuning of the beam, and a silicon detector were placed on the upper rotating platform. A simple schematic of the setup on the rotating plates can be seen in Fig. 5.

The runs in the scattering chamber used a highly attenuated, 88 MeV $^{28}\text{Si}^{9+}$ beam tuned through a 5 mm collimator. The primary goal of these experiments was to test the position separation and identification using the two pairs of split anodes (A2/A3 and A6/A7). Fig. 6 shows the data accumulated from seven separate runs with the attenuated ^{28}Si beam. Between each run, the circular plate housing the detector was moved by approximately 2 degrees on either side of beam center until the beam was no longer going through the detector window. Each group shown in the figure was a separate run. Using this data, a full calibration of the position of the beam within MONICA between channel number and physical position was calculated. This experiment also verified that the split anodes can be used to track the position of the isotopes entering the window and their subsequent trajectory through the detector. A average energy resolution of 3.7%

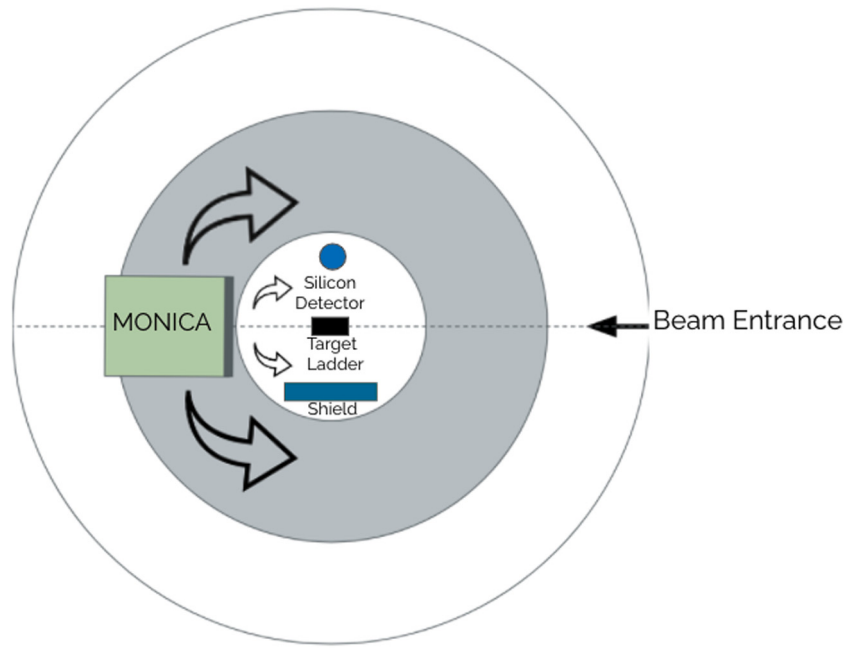


Fig. 5. Setup for commissioning experiments with MONICA using the large scattering chamber at the NSL. The two center plates are able to rotate separate from each other. MONICA was mounted on a stand on the middle circular plate, while the shield and the silicon detector were mounted on the top circular plate.

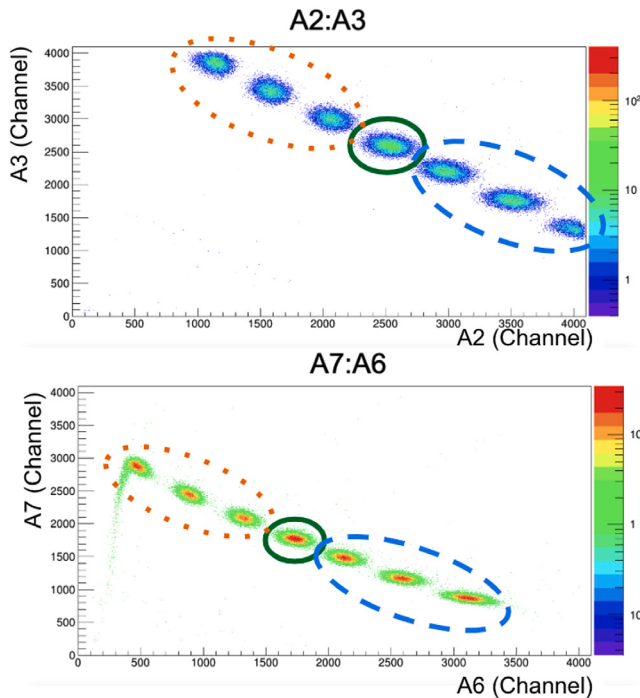


Fig. 6. Graphs display pairs of split anodes versus each other, showing a superposition of seven runs using a ^{28}Si beam to track the position of beam entry and trajectory through MONICA. The orange (dotted-line) circled runs are those entering to the right of beam center, the green (solid-line) circled run is that entering at beam center, and the blue (dashed-line) circled runs are those entering to the left of beam center.

was found for each full anode, and a resolution of 2.1% was determined for the cathode AC-coupled to the Frisch grid, which reflects the sum of energy losses across all anodes.

4.1.2. Commissioning of MONICA using the AMS beamline

Two commissioning runs were performed on the AMS beamline at the NSL using ^{28}Si , $^{53}\text{Mn}/\text{Cr}$, and $^{58}\text{Fe}/\text{Ni}$ beams, with the ^{28}Si beam acting as a diagnostic tool for the spectrograph. The AMS beamline contains additional quadrupoles and a Wien Filter to help focus the beam and rid it of isotopic contaminants. At the end of the beamline is a Browne-Buechner Spectrograph that can be used in vacuum or gas-filled mode. The MONICA detector was mounted inside the spectrograph.

The first goal of this test was to demonstrate MONICA's ability to separate the stable isobaric pair $^{58}\text{Ni}^{12+}$ and $^{58}\text{Fe}^{12+}$ using the GFM technique. With the spectrograph filled with 2.5 torr N_2 , MONICA filled with 15 torr Isobutane, and a 77 MeV beam, the isobaric pair were successfully separated in both position and energy, as seen in Fig. 7. The top two graphs show the separation in energy loss seen in Anodes 4 and 5, the middle two anodes in MONICA. The bottom left graph of the cathode's, coupled to the Frisch grid, energy signals show the total energy loss through the detector of the two isobars. Finally, the bottom right graph of A3 vs A2 shows the position separation of ^{58}Ni and ^{58}Fe after they go through the spectrograph.

The final commissioning experiment at the NSL was performed to conduct a preliminary AMS measurement on ^{53}Mn ($t_{1/2} = 3.74$ My), a rare isotope that had not been previously measured there. Samples of MnO_2 , obtained from A. Wallner (Helmholtz-Zentrum Dresden-Rossendorf (HZDR), Germany) and containing concentrations ranging from 10^{-8} to 10^{-10} , was mixed 2:1 $\text{MnO}_2:\text{Ag}$ and placed in a copper cathode in MC-SNICS to create the resulting 120 MeV beam.

Fig. 8 shows the separation of ^{53}Mn from its contaminant isobar ^{53}Cr in position via the second set of split anodes A6 vs A7. This experiment exhibited that a measurement of ^{53}Mn is possible. More information on beam creation, this measurement and further developments of AMS with ^{53}Mn at the NSL can be found in T. Bailey, et al. [8].

4.2. First use of MONICA at ANL: Preliminary AMS measurements of ^{39}Ar and ^{42}Ar using MONICA with the Enge split-pole spectrograph at ATLAS

MONICA was installed in the Enge split-pole spectrograph at ANL for use in an experiment dedicated to the detection of the long-lived

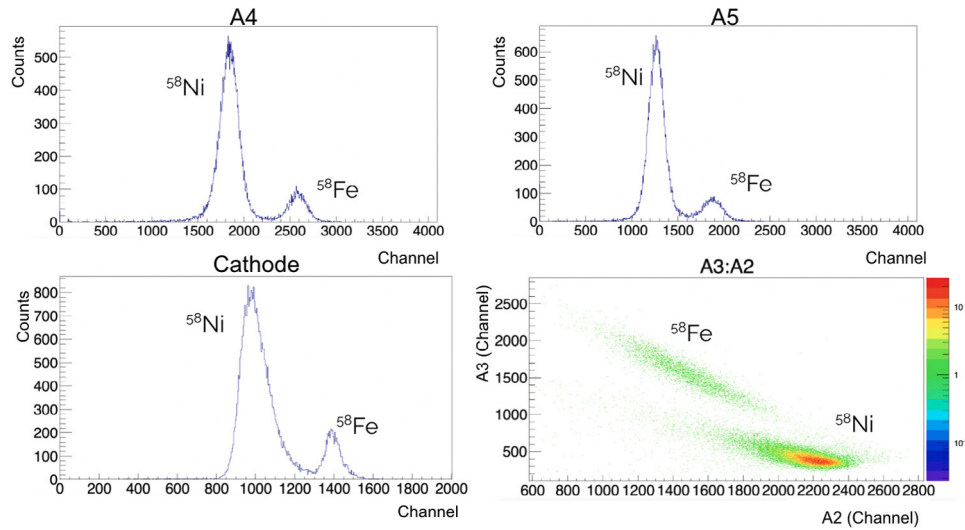


Fig. 7. Graphs displaying the separation of stable isobars ^{58}Ni and ^{58}Fe after utilizing the AMS beamline and spectrograph in gas-filled mode.

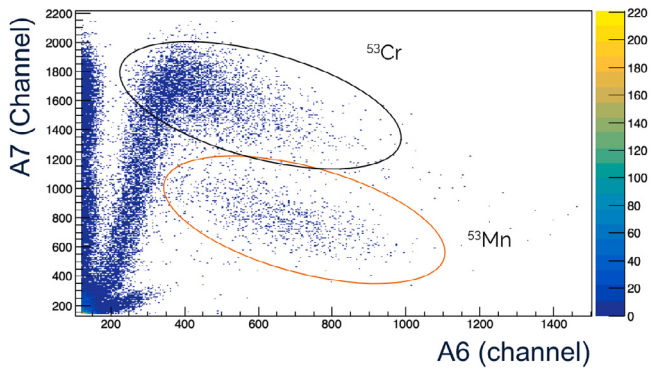


Fig. 8. A graph of the separation of ^{53}Mn from its contaminant isobar ^{53}Cr in position shown by the second set of split anodes A6 vs A7.

isotope ^{39}Ar ($t_{1/2} = 268\text{y}$) and, for the first time, the detection of ^{42}Ar ($t_{1/2} = 33\text{y}$). This experiment is a preparation study for future activation experiments on ^{40}Ar planned at the National Ignition Facility (NIF), aimed at studying the neutron-induced reaction $^{40}\text{Ar}(n,2n)^{39}\text{Ar}$ and possibly the $^{40}\text{Ar}(2n, \gamma)^{42}\text{Ar}$ reaction in a high-density stellar-like plasma [9].

^{39}Ar samples were prepared via the following neutron-induced reactions: $^{38}\text{Ar}(n, \gamma)^{39}\text{Ar}$ with thermal neutrons from the Soreq Israeli Research Reactor-1 (IRR1) at Soreq Nuclear Research Center (NRC) and $^{40}\text{Ar}(n,2n)^{39}\text{Ar}$, a fast-neutron reaction with 14 MeV neutrons from the DT generator at Technical University Dresden located at HZDR. The ^{42}Ar sample was produced by two-neutron capture on ^{40}Ar at the high-flux nuclear reactor at Institut Laue-Langevin (ILL) in Grenoble, France in an eight day irradiation in a 0.77 cm^3 high purity quartz sample at a nominal thermal neutron flux of $1 \times 10^{15}\text{ n cm}^{-2}\text{ s}^{-1}$. The IRR1 and ILL gas samples were diluted with ^{nat}Ar to reach a target isotopic abundance in the 10^{-12} range and a volume of the order of 50 cm^3 STP.

The $^{39,42}\text{Ar}$ nuclides were detected by injecting positive 8+ ions [2] from a newly-commissioned electron cyclotron resonance ion source (ECR-III) [10] and accelerated to 5.5 MeV/u by the ATLAS superconducting linear accelerator. Following the technique used in [11], the ions were dispersed in the Enge split-pole spectrograph, filled with 10 torr N_2 and identified using MONICA, filled with 25 torr CF_4 . The detection of ^{39}Ar and of ^{42}Ar , the latter for the first time as an AMS nuclide, are illustrated in Fig. 9, with spectra depicting the samples

from Soreq NRC, HZDR and ILL, respectively. Using the Soreq NRC sample of known $^{39}\text{Ar}/^{40}\text{Ar}$ abundance based on the $^{38}\text{Ar}(n, \gamma)^{39}\text{Ar}$ cross section [11,12] and the dilution factor as a reference, the preliminary isotopic abundances determined for the HZDR and ILL samples are $^{39}\text{Ar}/^{40}\text{Ar} = 4.3(6) \times 10^{-13}$ and $^{42}\text{Ar}/^{40}\text{Ar} = 2.4(3) \times 10^{-12}$, respectively. The $^{39}\text{Ar}/^{40}\text{Ar}$ ratio was determined relative to the mean of $^{40}\text{Ar}^{8+}$ and $^{38}\text{Ar}^{8+}$ beam intensities measured in a Faraday cup before the Enge split-pole spectrograph and normalization to the Soreq NRC sample.

Such a technique is not available for the $^{42}\text{Ar}/^{40}\text{Ar}$ ratio, and an alternate method was used, described here for its relevance to AMS techniques. We mixed a small amount ($\sim 1\%$) of enriched ^{21}Ne gas into the ^{42}Ar sample and optimized throughout the accelerator a beam of $^{21}\text{Ne}^{4+}$ ion-optically identical and acting as a “carrier” beam for the rare $^{42}\text{Ar}^{8+}$ ions. The $^{21}\text{Ne}^{4+}$ beam transmission, taken as identical to that of $^{42}\text{Ar}^{8+}$, was determined by charge current reading in the injection cup and in the Faraday cup in front of the spectrograph (0.11 enA) and was 59(1)%. The mixed beam was then admitted to the gas-filled Enge split-pole spectrograph which deflected the ^{21}Ne beam out of MONICA acceptance.

A small count rate (~ 20 cps) of scattered ^{21}Ne ions is observed in the detector well separated from ^{42}Ar (Fig. 9(e)). The count rate of ^{42}Ar ions measured in MONICA (0.110(4) cps) with the carrier beam tuning is consistent with that measured following the usual pilot beam optimization (0.114(6) cps) before the ^{21}Ne admixture (Fig. 9(d)). The final $^{42}\text{Ar}/^{40}\text{Ar}$ abundance ratio, quoted above, is obtained by normalizing the ^{42}Ar count rate in MONICA, corrected for the 59% transmission from the injection cup, to the $^{40}\text{Ar}^{8+}$ charge current measured in the same injection cup.

5. Future plans

AGFA was shown to provide sufficient position and energy separation despite experimental limitations, proving the feasibility of taking an AMS measurement using that magnet. Tests at the NSL using the scattering chamber and the Browne-Buechner Spectrograph showed MONICA’s functionality and capability to show the separation of isobars via energy and position. The preliminary AMS measurement of $^{39,42}\text{Ar}$ furthered the conclusion of the commissioning results, showing that MONICA could be used to separate a rare isobar from its contaminants. These findings all bode well for the future of AGFA in AMS experiments.

In the coming months, a stand will be machined that will allow MONICA to sit on the focal plane of AGFA. This stand will be tested and MONICA will then be used in an AMS measurement on that machine.

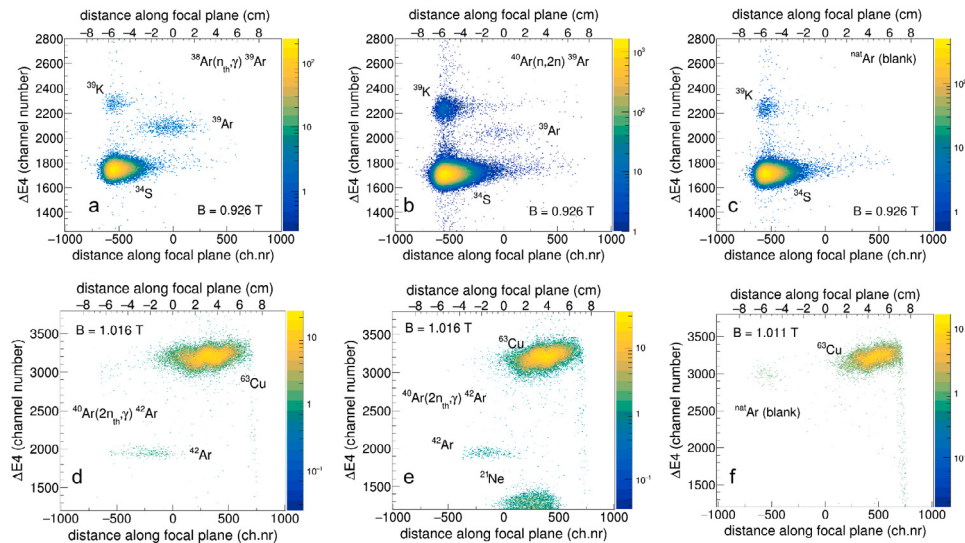


Fig. 9. (top) Identification spectra for ^{39}Ar produced by (a) $^{38}\text{Ar}(n, \gamma)^{39}\text{Ar}$ and (b) $^{40}\text{Ar}(n, 2n)^{39}\text{Ar}$ at a neutron energy of 14 MeV. (c) shows the spectra of a blank at the same settings as (a) and (b). (bottom) Identification spectra for ^{42}Ar produced by slow, two-neutron capture $^{40}\text{Ar}(2n, \gamma)^{42}\text{Ar}$. Spectra in (d) was taken with the pure ILL sample, spectra in (e) was taken with the mixed ILL + 1% ^{21}Ne sample (see text), and spectra in (f) was taken with a blank. The ^{63}Cu group (~ 10 cps) originates from $^{63}\text{Cu}^{12+}$ ions, degenerate in $\frac{m}{q}$ with $^{42}\text{Ar}^{8+}$, and likely due to ion source materials.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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