Nuclear Instruments and Methods in Physics Research B 268 (2010) 799–803

Contents lists available at ScienceDirect

Nuclear Instruments and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb

Fluorides or hydrides? ^{41}Ca performance at VERA's 3-MV AMS facility

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ARTICLE INFO

Article history:
Available online 7 October 2009

Keywords:
^{41}Ca
CaF_2
CaH_2
CaF_3^-
CaH_3^-
AMS
ΔTOF
Ionization chamber
SiN

ABSTRACT

Recent improvements in isobaric suppression for medium-mass isotopes, e.g. ^{41}Ca, offer new possibilities for tandem accelerators with terminal voltages of 3 MV or lower; i.e. when dealing with particle energies $\leqslant$1 MeV/amu. In particular, detection of ^{41}Ca requires sufficient discrimination of the stable isobar ^{41}K. We explored the limits of ^{41}Ca detection at our 3-MV AMS facility by means of different types of particle detectors: The ΔTOF method, which is based on the different flight-time of isobars after passing a thick absorber foil. The second method makes use of a new type of compact ionization chamber: ^{41}K and ^{41}Ca are separated in energy due to their different energy loss in the detector entrance foil and the detector gas, which is measured via a segmented anode.

At VERA we measured ^{41}Ca/Ca ratios below 10^{-13} for commercial CaF_2 material serving as blank samples. CaH_2 sputter targets, with the extraction of CaH_3^-, yielded background ratios as low as ^{41}Ca/Ca = 1×10^{-15}. The typical measurement precision at VERA for ^{41}Ca measurements was between 2% and 5%. These results demonstrate that AMS facilities based on 3-MV tandems have reached the sensitivity level of larger AMS facilities for a wide range of applications, with the advantage of high overall efficiency and sample throughput.

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1. Introduction

The detection of the long-lived radionuclide ^{41}Ca ($t_{1/2}$ = (104 ± 5) kyr, [1]) with AMS became of considerable interest in the 1980's shortly after it was realized that ^{41}Ca might represent a possible tool for bone dating (radio-calcium dating, [2]). However, soon it turned out that natural ^{41}Ca/Ca isotope ratios show remarkable fluctuations, depending on the lithosphere-constitution, erosion rate, altitude and also shielding of the cosmic radiation by the earth's geomagnetic field. Differences up to two orders of magnitude were measured for samples collected at different regions around the world. Due to these local variations in the ^{41}Ca/Ca isotope ratio, radio-calcium dating could not be developed as a global dating tool. Recently, ^{41}Ca-AMS measurements are focused on studying cosmic-ray exposure history and terrestrial ages of meteorites [3], exploration of its sensitivity as a biological neutron-dosemeter [4], and as a means for tracer studies in various biomedical applications (see e.g. [5]).

Detection of ^{41}Ca requires sufficient discrimination of the stable isobar ^{41}K. Successful AMS measurements have shown that the use of CaF_2 and CaH_2 sputter targets with the extraction of CaF_3^- and CaH_3^-, respectively, gives the best results [2,6–13]. CaH_3^- is produced from CaH_2 powder, a material which is highly reactive in air due to its strong hygroscopic potential. Therefore, such a compound has to be handled and stored under inert atmospheric conditions (Ar). A drawback of this chemical form is its elaborate production process (see e.g. [14,15]. However, extracting CaH_3^--ions has the great advantage that KH_3 does not form stable negative ions [6] in significant amounts – if at all. The use of CaH_3^- allows therefore to measure ^{41}Ca/Ca isotope ratios down to natural levels, i.e. at 10^{-14} and $<10^{-15}$ [16,17,11,18]. At the time when ^{41}Ca was explored as a potential dating tool [1], it was exclusively CaH_2 which was utilized for the AMS measurements.

Higher ^{41}Ca/Ca isotope ratios can be found in other applications, e.g. the measurement of cosmogenic ^{41}Ca in meteorites, or for biomedical applications with enriched ^{41}Ca materials, where ratios well above 10^{-13} are common. In such cases, CaF_2 can be used. Indeed, when extracting CaF_3^--ions, KF_3 is significantly, although not completely reduced [13,18]. The second point, which makes CaF_2 as sputter material favorable, is the uncomplicated handling,

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doi:10.1016/j.nimb.2009.10.034

storage and the straightforward sample production: CaF_2 is easily produced by precipitation with hydrofluoric acid.

Until recently, ^{41}Ca measurements have been performed almost exclusively at AMS laboratories based on larger accelerators. Facilities based on 3-MV tandems or smaller ones were not able to discriminate between the isobaric and possible isotopic interferences to the rare ^{41}Ca signals. However, recent technical developments in particle detection mainly based on the use of very homogeneous absorber foils (Silicon nitride, SiN, Silson Ltd., UK, see also [19]) and on the development of a compact-type ionization chamber at ETH in Zurich [20,21] resulted in improved isobaric suppression also for medium-mass isotopes, like ^{36}Cl and ^{41}Ca. These findings offer new possibilities for accelerators with terminal voltages of 3 MV or lower; i.e. when dealing with particle energies $\leqslant$1 MeV/amu. In particular, the VERA facility consists of a high-energy spectrometer designed to allow low isotope ratio measurements (for more details see [22,23]): a high-resolution analyzing magnet followed by another high-resolving 90°-electrostatic deflector. In case of ^{41}Ca detection an additional switching magnet efficiently rejects ions of wrong charge state which underwent charge exchange due to collisions in the residual gas. The combination of these filters helps for low isotope ratio measurements, e.g. ^{41}Ca detection using CaF_2 and CaH_2 samples. First exploratory measurements at VERA have been reported previously [24]. In this paper, we will summarize the potential of a 3-MV AMS system for measurements of ^{41}Ca with both CaF_2 and CaH_2 sputter targets.

2. Measurement procedures using CaF_2

We used two different detector setups for our ^{41}Ca measurements – both are based on recent developments and technical improvements in particle detection [20–22,25]. We started our investigations on ^{41}Ca with CaF_2, because this chemical form is mostly used in AMS nowadays and CaF_2 allows a convenient handling. The isobar ^{41}K is still present in the beam, and requires isobaric suppression at the particle detector side. We used CaF_2 material prepared from $CaCO_3$ which was assumed to contain negligible amounts of ^{41}Ca. As reference material, CaF_2 powder was available with ^{41}Ca/Ca isotope ratios between 10^{-10} and 5×10^{-13} from TU Munich and from Kunihiko Nishiizumi [26].

CaF_2 powder material was mixed with Cu powder and pressed into Cu sample holders. For comparison, Ag powder was also used as mixing material but no improved performance was found. In order to obtain maximum separation of ^{41}K from ^{41}Ca, we pushed – for the first measurements – the tandem to terminal voltages up to 3.4 MV (see [22]). The charge state 4^+ was selected – it is a compromise between high particle energies needed for isobar separation, and a high charge-state yield. While the 3^+ charge state was found to give a higher yield (up to 18% of the injected beam was measured after the analyzing magnet), the yield for 4^+, typically 12%, was still acceptable. We used gas stripping with O_2 for all measurements on Ca. For higher charge states the yield drops roughly by a factor of two per charge state. With improved performance of ^{41}Ca detection we lowered the terminal voltage to 3 MV. In general, particle energies between 13.2 and 15.0 MV were available. Typical CaF_3^- currents injected into the tandem accelerator were of the order of 150 nA, with maxima up to 500 nA when pushing the ion source.

For particle detection, we first explored the ΔTOF technique [22,25], which is based on the combined information of time-of-flight (TOF, two micro channel-plates, 65 cm apart) and energy (E) measurement. A Bragg-type ionization chamber was used for the measurement of the residual particle energy. Close to the start detector a stack of SiN foils with an adjustable absorber thickness was placed. Isobars, after passing these thick and homogeneous silicon nitride membranes, will experience a different energy loss which results in a different TOF and energy signal.

Depending on the thickness of the absorber foils, we observed a particle transmission through the ΔTOF-setup between 10% and less than 1%, for a SiN thickness between 500 nm and 2000 nm, respectively. The low transmission is due to angular scattering in the absorber foil. We observed a strong dependence of the isobaric suppression factor on the terminal voltage, i.e. on the particle energy. ^{41}Ca measurements were performed at a terminal voltage of 3 MV (first tests have been performed at voltages up to 3.4 MV), which corresponds to particle energies of about 13 MeV for CaF_3^- injected. For SiN absorber foils with a thickness of 750, 1500 and 2250 nm their isobaric suppression factor (^{41}K events detected versus counts found in the ^{41}Ca integration bin for blank samples and corrected for the cutting edges) yielded values of 15, 50 and up to 100, respectively. The integration bin was set to accept between 20% and 50% of the registered ^{41}Ca signals. The corresponding particle transmission (fraction of 4^+-particles finally accepted as ^{41}Ca events) observed was between 7%, 2% and less than 1%, respectively. About 14% of the particles passing the start foil of the ΔTOF-setup could enter the final energy detector when using the thinner absorber foils (500–750 nm). We could increase the isobaric suppression factor to values significantly above 100 by adding more SiN absorber foils, however, at the cost of a strongly decreased particle transmission, with values well below 1%. Two different foils for the start signal of the TOF-setup, DLC and SiN foils were used for ΔTOF: the use of DLC as start foils introduced some low-energy tails [25]; however, replacement by SiN foils decreased the TOF efficiency significantly, only approx. 10% of the particles passing the foil gave detectable timing signals (for more details see [27]), therefore measurements were performed usually with DLC foils see Table 1.

Fig. 1 shows a comparison between measured isotope ratios of six ^{41}Ca reference materials and their nominal values. Also plotted is the isotope ratio for CaF_2-blank samples whose nominal value was arbitrarily set to 10^{-15}. In this case a detection efficiency of 7% was obtained (with an absorber thickness of 650 nm). Using the ΔTOF-setup, isotope ratios down to a few 10^{-13} could be measured. The reproducibility of the CaF_2^- measurements (i.e. the standard deviation of the mean for samples where their isotope ratios and uncertainties are not limited by counting statistics or background corrections) was in the range between 5% and 8%.

The development of a new compact-type ionization chamber at the ETH in Zurich [20,21] with a SiN foil as entrance window, provides a significantly improved energy resolution. We adopted this design at VERA [28]. With this detector, the isobars, ^{41}K and ^{41}Ca, are separated in energy due to their different energy loss in the detector entrance foil and the detector gas (ΔE/E-detector). Energy loss and residual energy are measured via a segmented anode. While the separation power between ^{41}Ca and ^{41}K was found to be similar for both setups, ΔTOF and ionization chamber, the particle detection efficiency of nearly 100% for this compact chamber clearly favors this detector. We found a particle transmission be-

Table 1
Characteristics of Ca beams from the two different sputter materials CaF_2 and CaH_2.

	CaH_2	CaF_2
Negative ion	CaH_3^-	CaF_3^-
Charge state	4^+	4^+
Particle energy	15 MeV	13 MeV
$^{40}CaH_3^-/^{40}CaF_3^-$	500 nA[a]	150 nA[b]
$^{40}Ca^{4+}$	540 nA	72 nA
Transmission	27%	12%

[a] Typical values, max. current 1500 nA.
[b] Max. current 450 nA.

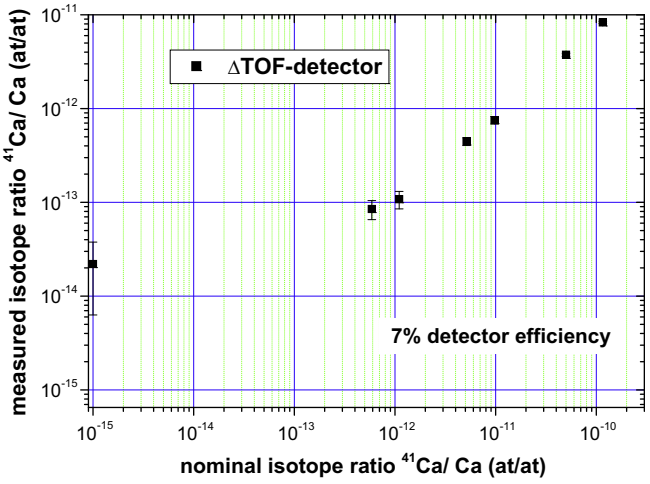

Fig. 1. Measured isotope ratios for various reference samples versus their nominal values and blank samples. The ΔTOF-setup was used for these CaF_2-samples with an absorber thickness of 650 nm.

tween 40% and 45% for this detector with cutting edges accepting only half of the ^{41}Ca events; i.e. more than 80% of the $^{41}Ca^{4+}$ ions gave a detector signal. The corresponding suppression factors were found to be about 200–400. This value can be compared with the corresponding suppression factor obtained under similar conditions for ^{36}Cl measurements, where, a factor of 1600 was observed (see [23,27]), however, for significantly higher particle energies. In contrast to ^{36}Cl, this suppression is sufficient for ^{41}Ca applications as discussed below due to significantly lower ^{41}K count rates compared to the ^{36}S count rates.

Spectra obtained with both types of detection setups are displayed in Fig. 2. The upper plot (a) shows the TOF-signal measured for a sample after passing through a 1000 nm SiN foil and for a flight-path of 65 cm. The right peak consists of ^{41}Ca counts and originates from a standard with an isotope ratio of 10^{-10}. The left peaks originate from ^{41}K counts (two CaF_2-samples, assumed to be low in ^{41}Ca). The lower plots (b) and (c) show a two-dimensional spectrum obtained with the $\Delta E/E$-detector. Displayed are the two energy-loss signals of the segmented anode. Fig. 2b represents signals from a CaF_2-blank sample. Fig. 2c shows the same spectrum for a standard with an isotope ratio ^{41}Ca/Ca of 10^{-11}. The rectangles indicate the bins typically applied for ^{41}K and ^{41}Ca counting (the cut is chosen such that 50% of the registered ^{41}Ca events were accepted). ^{41}Ca measurements on reference samples with high isotope ratios, performed with the compact ionization chamber, yielded a reproducibility between 2% and 5%.

Clearly, the compact ionization chamber outperforms the ΔTOF technique in its detection efficiency and in the reproducibility of the measurements. It allows measurements with detection efficiencies exceeding 50% (ten times that found for typical conditions for the ΔTOF technique) together with a ^{41}K suppression higher compared to the ΔTOF technique. In view of these advantages we now completely switched to the $\Delta E/E$ technique for ^{41}Ca measurements.

3. Measurement procedures using CaH_2

Commercially available CaH_2 material (Alfa Aesar) was used for our studies on the performance of CaH_2 as sputter targets. With that new material we obtained sufficient high ion currents and therefore an "inexhaustible" amount of test material. In addition, such material was exposed to reactor neutrons in order to produce ^{41}Ca via neutron capture on ^{40}Ca. In this way, samples with isotope ratios ^{41}Ca/Ca between 10^{-12} and 10^{-11} were produced directly as CaH_2 samples which allowed avoiding the chemical preparation of CaH_2 sputter material. These samples served as reference materials and were compared to unirradiated blank samples.

Sputter samples were produced under Ar atmosphere by pressing a mixture of CaH_2 and Ag powder (or Cu powder) into Cu sample holders. Our 40-sample target wheel was loaded into the ion source using a flexible glove bag, which was spilled with Ar gas as well. CaH_3^- was extracted from the source and injected into the tandem whose terminal voltage was chosen to 3.0 MV. Like for CaF_2-samples, the 4^+ charge state was selected, which corresponds to particle energies of 15 MeV. We measured a particle transmission of 27%, i.e. the fraction of particles injected compared to the analyzed ones at the high energy side. The transmission is about twice that for CaF_2 measurements, which is mainly due to the higher velocity of the Ca ions at the terminal stripper. Typical $^{40}CaH_3^-$ ion currents of 500 nA were measured for the commercial samples, with maximum currents exceeding 1.5 μA, when pushing the ion source. Besides ^{40}Ca, also the ^{42}Ca currents were measured in fast-switching mode, utilizing the offset Faraday-cups, at both, the low-energy and the high energy side.

We investigated the possibility of repeated use of such a material in a later beam-time. However, even when stored immediately after use under dry conditions, significant CaH_2 decay was visible and the negative-ion currents dropped to levels of a few nA. A well-conditioned Ar-filled box is therefore mandatory for storage and a possible re-usability of such samples.

Due to the substantially lower background of ^{41}K, blank isotope ratios in the range of ^{41}Ca/Ca = 1×10^{-15} were observed. Depending on the bin-size used for counting the ^{41}Ca signals, values between 6×10^{-16} and 1×10^{-15} can be obtained. Fig. 3 shows the results of AMS measurements of unirradiated blank samples. The compact ionization chamber was used for these measurements. Its isobaric separation was similar to the CaF_2 runs, however, with much less ^{41}K in the beam. Out of the 15 sputter samples, about half gave no ^{41}Ca count at all, the others very few counts. The reproducibility of CaH_2 measurements for reference samples was between 2% and 3%.

4. Applications

4.1. $^{40}Ca(n,\gamma)^{41}Ca$ at stellar temperatures

We irradiated CaF_2 material at Forschungszentrum Karlsruhe with neutrons to produce ^{41}Ca from ^{40}Ca via neutron capture [29,30]. The aim of this study was the measurement of the production of ^{41}Ca under stellar environments, which focuses on neutron energies in the keV range. A well-established neutron irradiation setup at Karlsruhe was utilized [30]. At this facility, stellar conditions can be simulated directly. A Maxwellian–Boltzmann – like neutron energy-distribution was produced, which corresponds to stellar conditions of a temperature of kT = 25 keV. After the neutron activation, the samples were analyzed at VERA for their ^{41}Ca/Ca isotope ratio. Those samples gave an isotope ratio of $(1.34 \pm 0.07) \times 10^{-11}$, a value comfortable high for our first quantitative Ca measurements.

4.2. Cosmogenic ^{41}Ca in a meteorite

We analyzed the ^{41}Ca content of the meteorite Rio Cuarto 001 [31], whose terrestrial age was measured to 410,000 years. In this case, isotope ratios of a few times 10^{-13} were expected. Again, we chose CaF_2 as sputter material for this measurements. Results obtained from four sputter cathodes are plotted in Fig. 4. The error bars include an additional systematic uncertainty from ^{41}K

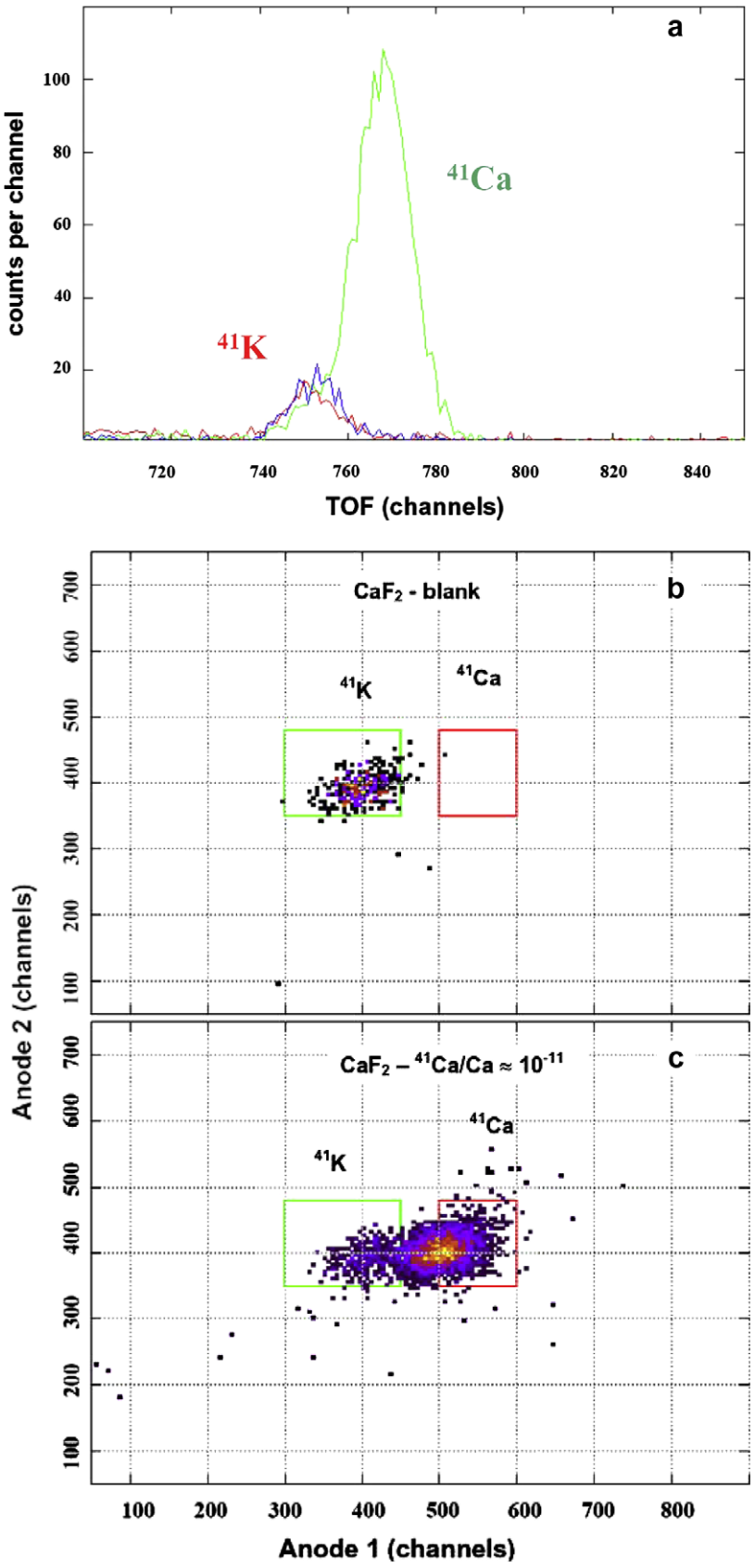

Fig. 2. Separation of ^{41}K from ^{41}Ca for CaF_2-samples. (a) depicts a spectrum obtained with the ΔTOF-setup. (b and c) shows the energy signals from the ionization chamber ($\Delta E/E$-detector) for a blank sample and a standard, respectively.

background. The mean value (line) and its uncertainty (dashed lines represent the standard deviation) are shown as lines. As indicated by the dashed lines, at these low isotope ratios, the final uncertainty is dominated by the uncertainty of the background.

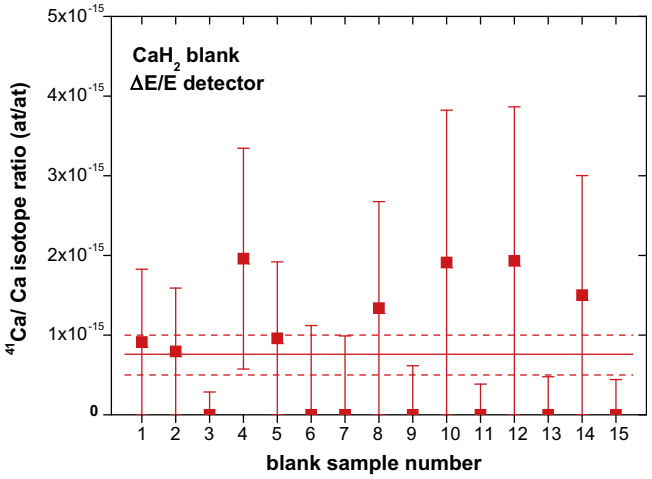

Fig. 3. Isotope ratios $^{41}Ca/Ca$ for a series of CaH_2 blank samples (Sigma Aldrich) measured with the ionization chamber. A mean value of $(8 \pm 2) \times 10^{-16}$ is found for a tight bin setting around the ^{41}Ca signal (see Fig. 2).

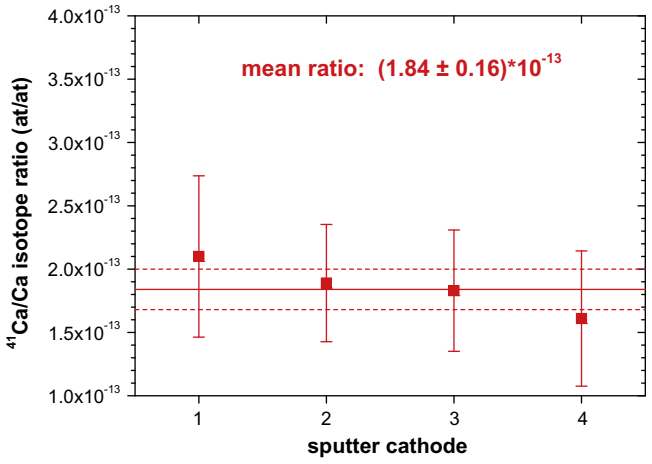

Fig. 4. Isotope ratios $^{41}Ca/Ca$ measured for a meteorite (Rio Cuarto 001 [31]). At 1×10^{-13}, error bars are dominated by systematic uncertainty from ^{41}K background correction (CaF_2-samples with $\Delta E/E$-detector).

The uncertainty of the isotope ratio of unprocessed commercially available CaF_2 material, however, was about a factor of two lower. We measured a "background" value of $^{41}Ca/Ca = (1.0 \pm 0.3) \times 10^{-13}$ for rock samples, and found $(0.50 \pm 0.15) \times 10^{-13}$ for unprocessed CaF_2.

5. Conclusions and summary

Utilizing the superior homogeneity of SiN absorber foils and the new development of a compact-type ionization chamber allows AMS facilities based on 3-MV tandems to extend their measurement capabilities to ^{41}Ca. At VERA, using CaF_2 as sputter material allows measurements of isotope ratios $^{41}Ca/Ca$ down to a few 10^{-13}, and we found background levels in the 10^{-14} range. Such a setup is capable for measurements in applications of biomedical research, nuclear astrophysics and meteorites. Utilizing CaH_2 samples, the low isobaric background allows to measure ^{41}Ca at natural concentrations, i.e. between 10^{-14} and low 10^{-15}. The drawback of elaborate sample production and delicate sample handling required for CaH_2 is somewhat balanced as new ion source designs allow a whole batch of sputter samples to be loaded in one step for its measurement. An advantage of smaller AMS facilities compared to laboratories based on larger accelerators is the higher particle transmission, which results also in a higher overall efficiency in Ca measurements.

Acknowledgement

We want to express our thank to Kuni Nishiizumi who provided various batches of AMS standards for AMS measurements at VERA.

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