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Accelerator mass spectrometry: Is the future bigger or smaller?

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Abstract

Since its inception in the late 1970s, accelerator mass spectrometry has become a powerful tool for measurement of trace amounts of natural radionuclides. In this paper, we review recent advancements in AMS and discuss future directions of this powerful technique. We highlight some recent developments, including the introduction of smaller accelerators, novel detection systems and the development of new analytical capabilities. We believe that the future prospects for AMS measurements are practically unlimited and that AMS has a vital role to play in the exploration of space, where radionuclide measurements are a key factor in the understanding of processes on other planets.

Keywords: accelerator mass spectrometry; cosmogenic radionuclides; radiocarbon dating

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1. Introduction

Accelerator mass spectrometry (AMS) combines the fundamental features of a mass spectrometer with a medium-energy accelerator. This method allows mass spectrometry to be done at MeV energies, where molecular interferences can be eliminated and standard nuclear physics particle detection methods can be employed. This approach was first taken in 1977 by several laboratories working independently at 4 different institutions [1–3].

The first AMS experiments used a variety of machines, including large accelerators (~ 10 MV) and cyclotrons. By 1980, the interest in AMS had grown rapidly and the General Ionex Corporation began to manufacture small accelerators (~ 2.5 MV) designed specifically for AMS. Today, the smaller machines (<5 MV) dominate the field, although a number of large accelerators (>5 MV) remain at the forefront of AMS research. Most new AMS instruments are currently manufactured by High Voltage Engineering Europe (HVEE) and National Electrostatics Corporation (NEC) to measure particular isotopes.

For earth science applications, AMS is primarily concerned with the measurement of cosmogenic radionuclides. These isotopes are produced when cosmic rays impinge on the atmosphere and the Earth's surface. Nuclear reactions producing cosmogenic radionuclides involve primary and secondary particles of varying energies. These nuclides are most useful for the dating of surface processes on Earth. The example of ^{14}C, which is produced by secondary neutrons in the upper atmosphere, is perhaps the most well-known (Fig. 1).

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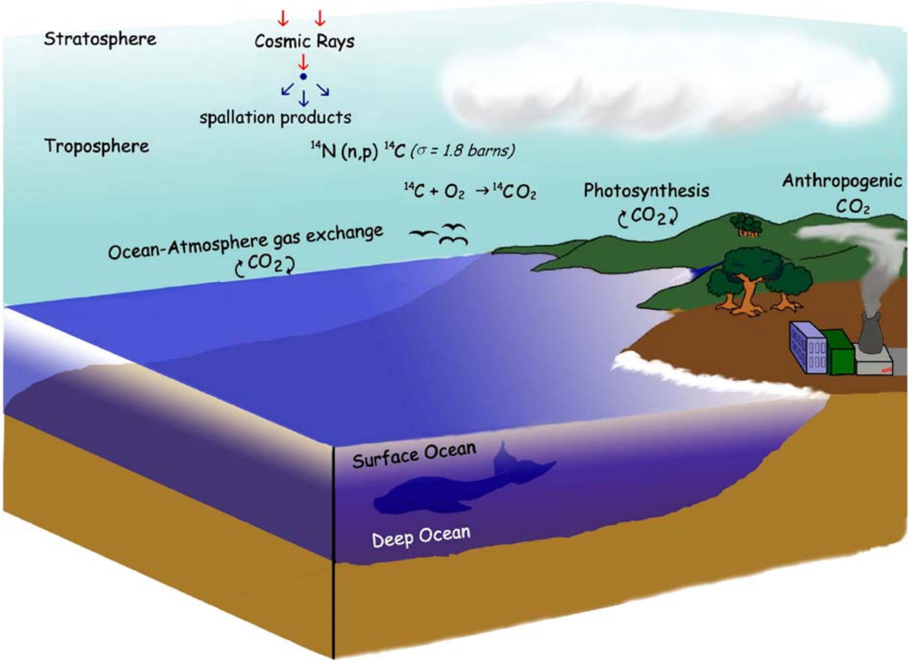


Fig. 1. Production of ^{14}C in the atmosphere, showing the production of ^{14}C by the action of cosmic rays on the upper atmosphere. (Figure courtesy of Tanya Burr.)

Measurement of the radioactive decay of ^{14}C as a method of dating was described by Arnold and Libby [4], for which Willard F. Libby received the Nobel Prize in 1960. Libby used decay counting to measure the amount of radiocarbon in a sample and the method required grams of carbon for a precise measurement. As the science progressed, this size requirement became a significant limitation and efforts to reduce the requisite sample size began. Atom counting with mass spectrometry was attempted but conventional mass spectrometers are limited by poor background performance due to the high abundance sensitivity required, and isobaric and molecular interferences which are significant at the concentration levels of natural radionuclides. Anbar [5] showed that conventional mass spectrometry could not achieve the sensitivity necessary for radiocarbon dating. The problem was solved by combining a mass spectrometer with an accelerator, and applying mass filtering techniques common to high energy nuclear physics. The size requirement for an AMS radiocarbon measurement immediately reduced the amount of sample required by a factor of 10^3 to 10^4.

AMS had an immediate impact when it became a viable technique. The First Conference on Radiocarbon Dating with Accelerators (1978) explored the potential of the new method with applications from a wide range of scientific disciplines, which were presented by key scientists at that time. These included: archaeology (Haynes [6]), geology (Rubin [7]), oceanography (Broecker and Peng [8]), paleoclimatology (LaMarche [9]), atmospheric sciences (Currie [10]) and medicine (Kielson and Waterhouse [11]). The promise of expanding and refining the radiocarbon calibration was also emphasized as this was a cornerstone of the radiocarbon dating method (Damon et al. [12] and Stuiver [13]). The potential for measuring a whole new group of cosmogenic isotopes, apart from ^{14}C, was also explored (Arnold [14]).

It is interesting to look back at some of the proposed applications discussed at the first AMS meeting, which have since provided fruitful results in the intervening years. These include: (1) the dating of hundreds of archaeological sites which were previously undatable, (2) the dating of tiny samples such as rare manuscripts, insects, seeds, or individual organic molecules, (3) the analysis of individual fractions in heterogeneous sediments and rocks, (4) the dating of individual varved layers in lake and ocean sediment cores, (5) the dating of geologic hazards such as earthquakes and volcanic eruptions, (6) the dating of individual foraminifera for paleoclimate studies, (7) the analysis of meteorites to determine their cosmic ray histories,

(8) the measurement of radiocarbon in atmospheric particulates, (9) the measurement of multiple cosmogenic isotopes from single samples, and (10) the use of radiocarbon as a biomedical tracer.

All of these ideas led to productive avenues of AMS research and continue to add to our understanding of the earth sciences. It is the intention of this review to track the progress of a number of these questions, familiar to the authors, and consider future directions of AMS research. We do not attempt to chronicle the development of the field as a whole, and the reader is referred to the reviews of Tuniz et al. [15] and Fifield [16] for a broader treatment of the growth of the field.

Not all of the ideas presented at the first AMS conference have met with immediate success. Among these were the concepts of a CO_2 ion source for every lab, the extension of the radiocarbon timescale to 80 or 90 ka BP, and the routine dating of polar ice with ^{14}C. These goals offered unexpected obstacles which remain the object of current studies, as discussed below.

1.1. The fate of cyclotrons

In 1977, Muller [3] followed up on earlier observations of Alvarez and Cornog [17] that a cyclotron could be used to select individual isotopes and discussed the use of a cyclotron for AMS. This method was adopted by Raisbeck et al. [18] and used successfully for ^{10}Be measurements. Unfortunately, cyclotrons are not predisposed to AMS work, since an important part of AMS measurement is the ability to switch between isotopes and between samples in a convenient manner. Chen et al. [19] at Shanghai pioneered the use of a mini-cyclotron for AMS work (Fig. 2). They were able to measure ^{14}C at natural levels but the inherent advantages in the design of electrostatic particle accelerators for AMS have made them the accelerator of choice.

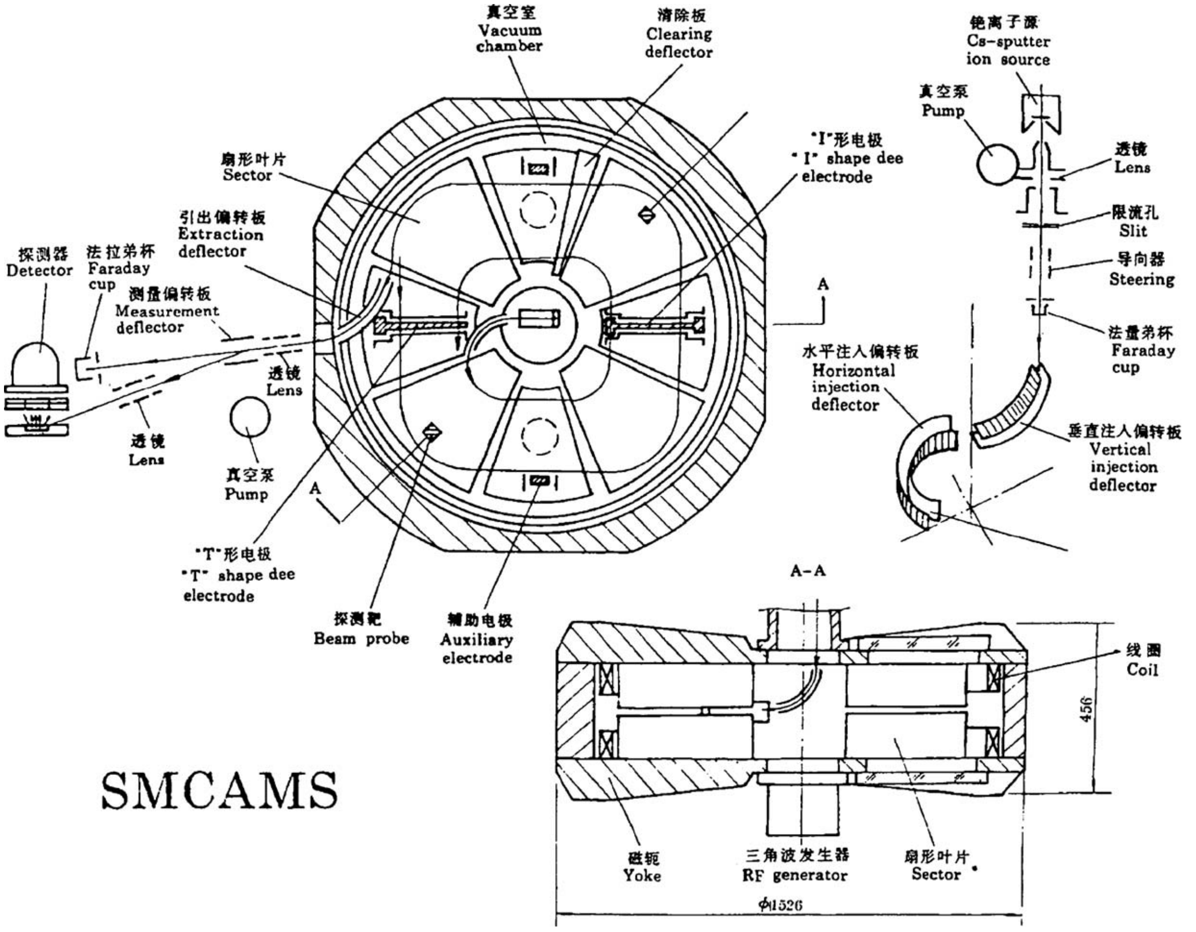


Fig. 2. Diagram of the mini-cyclotron system at the Shanghai Institute of Nuclear Research [19].

1.2. Electrostatic AMS

As noted above, the successful demonstration of the detection of ^{14}C using AMS was first published in 1977 [1–3]. Scientists at Toronto, Rochester and McMaster Universities showed that by combining a tandem accelerator with a mass spectrometer, one could successfully destroy molecular ions and ^{14}C could be resolved from all other nuclides. This method was soon demonstrated to be applicable to many other rare long-lived nuclides, including ^{10}Be, ^{36}Cl, ^{41}Ca and ^{129}I [15]. Later work has expanded these applications to include nuclides of about 20 elements. By 1982, dedicated AMS machines were installed at Oxford, Arizona, Gif-sur-Yvette, Nagoya and Toronto; and a number of multi-use machines at ETH-Zürich, Utrecht and elsewhere were dedicating significant beam time to the effort as well.

The first purpose-built machines relied on the design of Purser et al. [20] or the redesign of existing Van de Graaff accelerator systems. Many of the older machines which contributed significantly to early AMS research have been decommissioned, such as Chalk River, Rochester, Pennsylvania, McMaster, University of Washington, to name several. A number of other machines were moved, such as the relocation of the Rutgers tandem accelerator to the Australian Nuclear Science and Technology Organisation and the transfer of the University of Washington accelerators to the Lawrence Livermore National Laboratory. The field has grown continuously since that time, with over 52 purpose-built AMS machines operating in 2005, and numerous orders for new machines pending.

2. AMS radiocarbon methods

Radiocarbon dating using Accelerator Mass Spectrometry (AMS) differs from the decay counting methods in that the amount of ^{14}C in the sample is measured directly, rather than waiting for individual radioactive decay events. This makes the technique 1000 to 10,000 times more sensitive than decay counting. This sensitivity is achieved by accelerating sample atoms as ions to high energies using a particle accelerator, and using nuclear particle detection techniques. A photograph of the 3 MV AMS machine at the University of Arizona is shown in Fig. 3. For a detailed description of the theory and operation of an AMS, the reader is referred to Tuniz et al. [15] and Fifield [16]. Later in this paper, we will refer to the injection side of the machine, where negative ions are produced as the "low-energy" side and the part beyond the accelerator as the "high-energy" end (see Fig. 3). Today, an external precision of about ±0.35% in ^{14}C content, or ±30 yr in uncalibrated radiocarbon age is possible on a single 0.5-mg-sized sample target in 20 min of measurement time. Samples as small as 100 μg or less have been successfully dated to about ±80 yr BP and even smaller samples have been measured for special experiments. With longer counting times or when several targets are measured, we can reduce the single target error (by $\sqrt{N}$, where N is the number of targets) to about 0.2%, or better than ±20 yr in radiocarbon age [21,22].

In the case of longer-lived radionuclides such as ^{26}Al, ^{10}Be, ^{36}Cl, ^{41}Ca and ^{129}I, which were very difficult to measure using counting techniques, AMS has made

Fig. 3. Photograph of the high-energy beam line and detector system of the University of Arizona 3 MV Pelletron accelerator mass spectrometer.

measurements of small amount of these radionuclides routine [16].

3. A trend to smaller machines

Even as late as 1998, the comprehensive AMS summary of Tuniz et al. [15] did not pay much attention to the idea that smaller machines might become commonplace. However, this has become a reality over the last several years, starting with a suggestion by Purser [23] that this might be feasible, based on earlier studies by Lee at Toronto [24]. The first detailed report of Hughey et al. [25] was followed by the studies of Suter et al. [26]. The latter studies resulted in an original design of a 0.5 MV machine constructed at ETH-Zürich in cooperation with NEC.

3.1. A 0.5–1 MV tandem

The first proposed design for a <1 MV AMS was that of Hughey et al. [25], who reported on the design of a compact 1 MV machine for biomedical work at the AMS-7 Conference in Tucson, in 1996. This machine was further described in Hughey et al. [27].

Working about the same time, Suter et al. [26] discussed the possibilities of a 0.5 to 1 MV AMS which would operate in the 1+ or 2+ charge state. Suter et al. [26] laid the groundwork for this development with studies showing that molecular interferences could be destroyed with a higher stripper gas pressure than previously used and that 3 MeV Li^{2+} could be separated from $^{14}C^{2+}$ in the E–ΔE gas detector. Most investigators had considered the 2+ charge state impractical due to the presence of $(Li_2)^{2-}$, which occurs naturally in Cs in the ion source. Later, Suter et al. [28] reported on the design of a prototype 0.5 MV machine which would use the 1+ charge state. Although the machine could operate in other than the 1+ charge state, due to transmission considerations most of the discussion on these small machines has involved the 1+ charge state. Because a higher stripper-gas pressure, with a gas thickness of 2 $\mu g/cm^2$, is needed to ensure destruction of CH+ ions [24], Suter et al. [28] note that the maximum transmission is ~40%. It is interesting to note that operation in the 2+ charge state would be ideal close to 1 MeV [29].

Several 0.5 MV machines (Fig. 4) have now been built and are operational at the University of Poznan, University of Georgia and University of California-Irvine, with additional machines under construction. In 2002, there were discussions about the possibility of going to even lower terminal voltages, perhaps as low as 200 kV, which would eliminate the need for a costly accelerator. This is feasible from a careful study of the stripping yields at these energies. A prototype design of this machine is currently under construction in Zürich [30]. However, there are a number of practical limitations to very small machines, specifically that they are really only useful for studies of some nuclides which do not have serious molecular interferences, e.g. ^{14}C, ^{129}I and perhaps ^{26}Al. A second limitation is that the control of the stripper-gas pressure is delicate and essential. In the 1+ charge state, molecular ions can exist, but all small molecular ions are unstable at greater than 1+. Hence, the assumption of higher energy AMS operating conditions, that molecular ions are destroyed, cannot be taken for granted at low energy. High stripper gas pressure is required to destroy molecular ions and this may lead to other consequences, such as unwanted charge-exchange under poor vacuum conditions (e.g. Roberts [31]). Southon et al. [32] have also reported on the technical difficulties of these operating conditions.

Gracjar et al. [33] reported on initial studies on ^{10}Be at energies of about 600 kV on the terminal. The ^{10}Be was stripped to the 2+ charge state giving a total energy of 1.43 MeV. A stack of carbon foils was placed between the high-energy magnet and the electrostatic analyzer. Isobar separation at 1.7 to 3 MV indicated that this method was feasible and could suppress interfering B counts by up to 5 orders of magnitude. Fifield et al. [34] have discussed the detection of Pu isotopes using AMS at 300 kV on the terminal. Their measurements were made with the small 500 kV NEC machine at Zürich. These authors also showed that although transmission peaked for 3+ ions with a small stripper thickness of 0.2 $\mu g/cm^2$, a 2 $\mu g/cm^2$ foil was required to assure molecular dissociation into the 1+ and 2+ atomic species. Fifield et al. were able to obtain as much as 15% transmission into the 3+ charge state of Th.

3.2. Even smaller machines

Synal et al. [30,35] discussed some test experiments on a 200 kV machine which eliminated both the accelerator tank and also the accelerator tubes. The device designed by Synal et al. consists of two gap lenses and a stripper tube. The potential is generated by a commercial power supply. The device, of course, must be placed in the mass spectrometer (which makes it much larger). Synal et al. [35] reported a background of 0.4 to 1.3×10^{-14} could be obtained with the instrument, suggesting that it could be useful for radiocarbon dating.

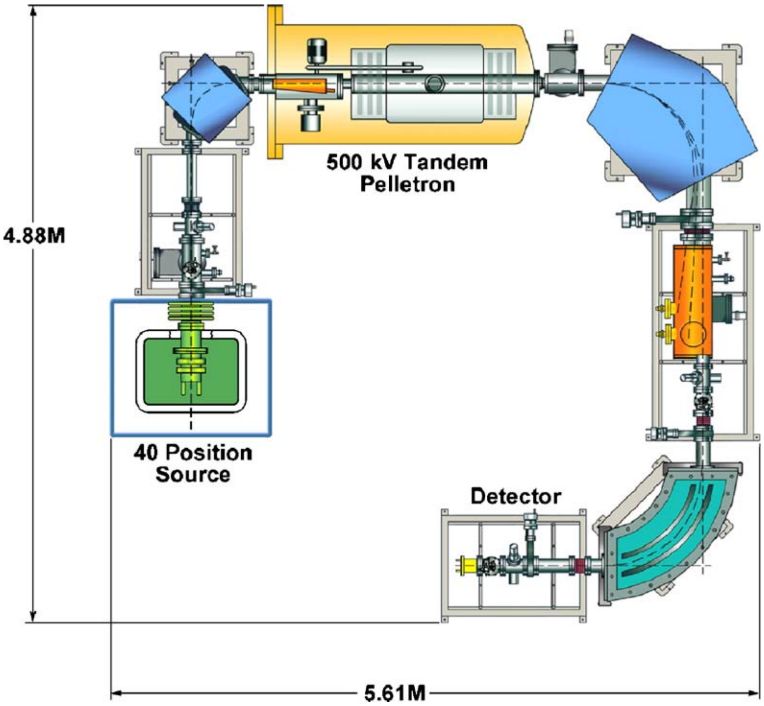


Fig. 4. Diagram of the NEC Lawrence Livermore 0.5 MV accelerator mass spectrometer. Courtesy of National Electrostatics Corporation.

In 2003, Schroeder et al. [36] discussed the design of another machine, which has since been installed at the University of Lund and includes a 300 kV accelerator with a standard accelerator tube section, but without any gas tank (Fig. 5). The gas tank could be eliminated since the high voltage can be sustained in air. This is described as a "single stage AMS", although it still has several stages. This device operates at 250 kV by floating the high-energy part of the machine at 250 kV with a commercial power supply, a section of accelerator tube

Fig. 5. Photograph of the 0.25 MV single-stage accelerator mass spectrometer, developed for the University of Lund. Courtesy of National Electrostatics Corporation.

in air provides the acceleration (see Fig. 6) [37]. The stripper is then at ground potential and the high-energy mass spectrometry is performed following the stripper without any further acceleration. This interesting design is foreseen as a useful tool for biomedical applications as well as other radiocarbon studies. A modification of this instrument reversed the original design so that the low-energy part of the machine was at high (negative) voltage and The University of Lund group report that this arrangement ([37]) gives backgrounds similar to the 0.5 MV machines discussed previously.

4. Ion sources

Improvements to the design of AMS machines have focused on the ion source, as this component is critical to the overall stability of the instrument. As noted previously, the development of a CO_2 ion source has been a goal of AMS since the beginning. This is because such an ion source could greatly simplify target preparation. Currently, the most common type of ion source is a Cs sputtering ion source which uses graphite as the preferred target material for radiocarbon analyses. A complete description of negative-ion sources and different target compounds is given by Middleton et al. [38]. Cs sputtering ion sources can be problematic. Careful maintenance is needed to prevent Cs buildup and the deposition of oxides must be removed at frequent intervals. Some newer designs of Cs sputter ion sources address this difficulty with easy access to the source to facilitate cleaning. Cs sputtering ion sources have proved reliable and the high ion currents (50–200 μA C^-) they produce make them the preferred technology. Future improvements to our understanding of Cs sputtering and to target chemistry will no doubt allow us to optimize the ionization efficiency of these devices.

The Oxford group pioneered the development of a gas ion source. In their design [39], CO_2 was diffused through a Ta or Ti frit and the gas ionized using the sputtering-type design already mentioned. In a different approach, Kim et al. [40] discussed the operation of a microwave plasma source, which produces positive ions. The positive ions must pass through a charge-exchange canal, containing Mg vapor, to produce negative ions for AMS. This charge-exchange process reduces transmission efficiency somewhat and introduces additional complexity to the beam optics of the machine.

4.1. AMS source improvements for ^{10}Be analysis

Zhao et al. [41] at the University of Toronto have worked on improvements to ^{10}Be measurements using BeF_2 instead of BeO as the target material. The authors demonstrated that isobaric interference from ^{10}B is much reduced in BeF_2 targets. BeF_2 has also been tested at the ETH-Zürich laboratory and initial results there

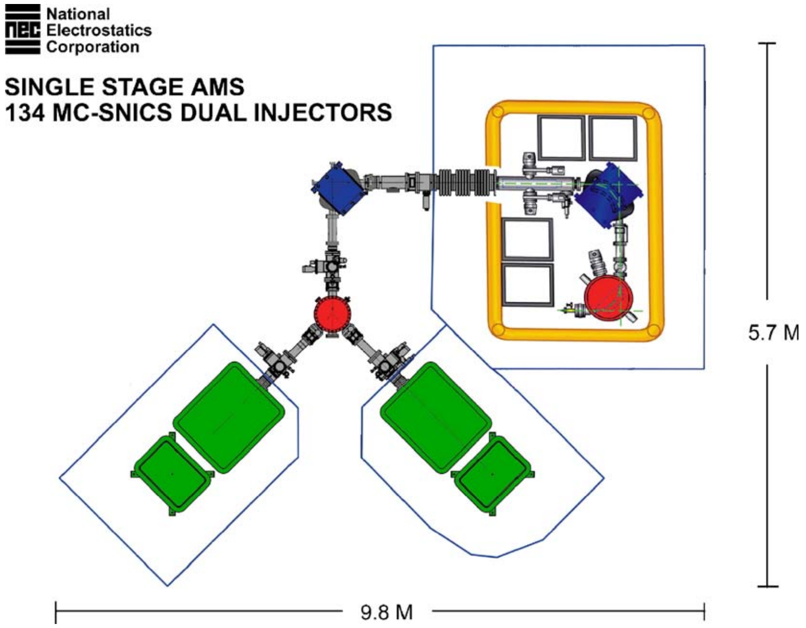


Fig. 6. Diagram of the 0.25 MV single-stage accelerator mass spectrometer at the University of Lund. Courtesy of National Electrostatics Corporation.

indicate that BeF_2 may make ^{10}Be measurements possible on a 0.5–1 MV tandem accelerator [42]. The use of fluorides and other novel target materials should improve ionization efficiencies [38] for Be as well as for other radionuclides. Currently, low beam currents hamper the analysis of ^{26}Al and ^{10}Be; emphasizing the need to improve our understanding of the ionization process during sputtering.

5. Specialized detection systems

Conventional detection systems use Si surface-barrier detectors, gas ionization detectors, or time-of-flight detectors. Si surface barrier detectors are ideal for nuclides with minimal isobaric interferences. For measurement of Be, Cl and heavier nuclides, gas detectors are used where the other isobar can be separated from the isobar of choice, by slowing down the ions preferentially in a hydrocarbon gas, such as isobutane or methane. The electrons emitted by the slowing ions are picked up by electrodes, allowing one to obtain measurements of the rate of energy loss (ΔE) as well as the residual energy (E) collected in the final stage of the detector. Although these devices usually have 2–3 stages, NEC has developed a 5-stage model and Wacker et al. [43] reportedly have designed an optimized 7-stage detector for ^{99}Tc. An alternative design uses an electric field in the direction of the ion motion to measure the rate of energy loss and total energy [44]. Time-of-flight detectors are often used for heavy ions. This allows the separation of ions of different mass remaining in the ion beam at the detector. In this type of system, the ion passes through a foil which generates an electron shower, counted as the “start” pulse and a second foil as a “stop” detector, and some labs have used a surface-barrier detector as the “stop” detector. The chief disadvantage of such detectors is that the mass resolution may be limited. This type of detector was used for early ^{129}I measurements [45]. However, measurements using a beam line with a 77° spherical electrostatic analyzer indicate that TOF is unnecessary for ^{129}I, provided there is sufficient resolution in the electrostatic elements of the AMS [46].

Another improvement has been the development of very thin silicon nitride Si_3N_4 windows (see www.silson.com) which can be used for ultrathin windows of gas detectors, allowing for improved energy resolution, especially important at low energies [34]. Other innovations for detector systems include an X-ray detector which uses characteristic X-rays for certain heavy elements, such as nickel and iron [47,48].

Another type of detector under development by the Vienna group [49] is a calorimetric low temperature detector (LTD). This type of detector uses deposited phonon energy (lattice-vibrational quanta), which produce an increase in the temperature of the detector dielectric material. Since nearly all the deposited energy ends up as heat, this type of detector has advantages for detection of higher-energy and mass particles. These detectors consist of small sapphire crystals with superconducting Al “thermometers”. For heavy ions measured at high energy, LTDs demonstrate exceptional energy resolution ($\Delta E/E$) as compared with surface barrier or gas ionization detectors.

6. Improvements to chemical processing

6.1. Radiocarbon

There have been a number of improvements to the conventional chemical pretreatment protocols for radiocarbon samples over the last several years. The simplest pretreatment is the acid–alkali–acid protocol for organic materials and acid etching for carbonates. Although this methodology is still widely used for many samples, it has long been recognized that more complex chemical pretreatment is required for many kinds of samples [50].

6.1.1. Compound-specific dating

The idea that a gas chromatograph could be coupled with an AMS, in the same manner as a gas-chromatograph and a conventional mass spectrometer (GC-IRMS) is compelling [51]. The ability to be able to measure discrete stable-isotope values for specific compounds has revolutionized GC-MS measurements. In the same fashion, a GC-AMS instrument would pave the way for major advancements in the field of radiocarbon.

To our knowledge, the best work proving the potential of this method is the work of Pearson et al. [52], who separated 31 different biomarker lipid compounds from marine sediments from the Santa Barbara and Santa Monica basins, California. In this approach, multiple collections of material run through gas chromatography columns were required to collect enough material for dating.

There are several technical difficulties which must be overcome in order to make GC-AMS measurements. Chief among these are the small amounts of gas which can be injected through a gas chromatograph, and the transient nature of GC pulses. Schneider et al. [53] have reported a flow of 200 μl

CO_2 per minute (100 μg C) can produce 20 μA to 60 μA of C^-, but these values would be much reduced in a real GC situation. We should also consider that the GC pulses are generally less than a minute long, perhaps 10 s. We also note that 20–60 ion source current is equivalent to ~2.4 to 7.2 ng C actually being measured by the ion source. We can contrast this performance with a 100 μg sample of C converted to graphite which produces 20–30 μA for 10–20 min at much higher count rates.

In any case, we expect that the likely size of sample, after separation of the different compounds, would be more likely between ~1 and 10 μg C. We presume that if flow and ionization efficiencies are the same, that this would be equivalent to 0.2 and 2 μA. Future developments in this field must focus on maximizing ion yield during the short transient time that the gas passes through the ionizing region of the ion source. This engineering goal would be well worth the effort, given the potential rewards of GC-AMS measurements. A notable example is the work of Ohkouchi et al. [54], who applied this technique in marine sediments.

Liberman et al. [55] discussed a different design based on a 1 MV AMS. Again, the goal of these workers is to use small AMS machines (0.5–1 MV on the terminal) for biomedical tracer work on ^{14}C and ^{3}H.

6.1.2. Selective combustion methods for radiocarbon dating

The importance of getting good dates on sediments has been championed by the late John Head [56]. Due to complications and the possibility of contamination, radiocarbon dates on low-carbon sediments can be problematical. In the past several years, there have been a number of significant improvements to sample pretreatment changes in this view.

6.1.2.1. Acid–base oxidation. Bird et al. [57,58] showed that oxidative acid treatment of charcoals could lead to improved dates, especially for samples in the 30–40 ka range. This methodology is important because it potentially allows us to extend the radiocarbon time-scale further back in time, with better contamination control [59] and because it allows us to further define what a “good” charcoal is for dating. This method has been successful in getting better agreement between radiocarbon measurements and other dating techniques for samples older than 30 ky. Some examples include radiocarbon ages from the 40 to 58 ky Border Cave, South Africa [58], and Devils Lair, Australia [60]. These results have not resolved archaeological questions surrounding the controversial Pedra Furada site however [61]. The oxidative acid approach has been followed up by others. Alon et al. [62] have focused on trying to characterize any remaining humic substances in cleaned charcoal samples using Raman spectroscopy. This method shows promise for verifying that humic contaminants have been removed.

6.2. Automation of sample preparation

Recently, many groups have focused on automation for routine sample preparation, to minimize repetitive tasks and maximize reproducibility of pretreatment steps. Pearson et al. [52] developed a semi-automatic sample preparation scheme. The Oxford group developed a semi-automatic method based on a CHN analyzer, but this gave some cross-talk in the measurements and good blanks were difficult to obtain. At the University of Arizona, we have fabricated an automated pretreatment device which feeds known quantities of reagents to pretreat bone samples. This allows us to standardize chemical pretreatment by removing subjective variables during sample preparation. Some laboratories have used robotic devices for repetitive tasks for ^{10}Be measurements in ice [63] and for graphite preparation [64]. Radiocarbon tracer studies require large data sets, and automated sample pretreatment will allow AMS laboratories to accommodate this need.

7. Novel and interesting applications

AMS applications are very numerous and we can only summarize a few highlights. AMS has revolutionized the field of radiocarbon and currently most radiocarbon measurements are made with AMS. Radiocarbon AMS has wide applicability to the earth and ocean sciences, archaeology, hydrology, atmospheric and pollution studies, art verification and even extraterrestrial studies. Measurements of the nuclides ^{10}Be, ^{26}Al, ^{36}Cl and others have become important tools in the growing field of “cosmogenic surface exposure dating”. ^{36}Cl also has an important role in hydrology.

Although the focus of this article is on the earth sciences, it should be noted that there is an important future role for AMS in the fields of biochemistry and biomedicine [15]. Indeed, entire AMS laboratories have been devoted to biomedical applications at York and Livermore [65,66]. One possible use of the 0.5 MV machines is the measurement of Tritium. The Rossendorf group has pioneered this work using a 0.5 MV instrument [67]. AMS measurement of T at natural levels is likely not possible due to the low number of atoms and difficulties in sample preparation. However, in an interesting application, Stan-Sion et al. [68] used AMS measurements to establish depth profiles of T in fusion reactor walls.

7.1. In situ cosmogenic nuclides in rocks

An important new area for dating is the use of cosmogenic radionuclides produced in situ at the surface of the Earth by interactions of cosmic radiation with the silicate in surface rocks [69]. Measurements of radionuclides produced in situ in the surfaces of rocks, soils and potentially, archaeological materials, at the Earth's surface have become a major and important use of AMS studies. This is particularly true for the nuclides ^{10}Be, ^{14}C, ^{26}Al and ^{36}Cl. These methods have been applied to changes in landscape evolution: weathering, sediment transport and soil development, retreat and advance of glaciers, tectonics, volcanic flows, meteorite impacts and other phenomena.

This method relies on the time of exposure of a sample near the surface of the Earth, where it will be exposed to significant cosmic radiation. Higher-altitude samples receive more exposure. A limitation of these methods is the need to be concerned about various spatial corrections, that is, the location of the sample as a function of latitude, altitude and partial shielding by surrounding geological features can affect the results. These applications have been summarized by Gosse and Phillips [69], as well as Cockburn and Summerfield [70].

Some of the first applications of in situ terrestrial cosmogenic nuclides (TCN) were by discussed by Lal and Arnold [71] and later by Nishiizumi et al. [72]. These authors studied the build-up of ^{10}Be and ^{26}Al in quartz from glacially polished rocks. There have been some spectacular examples of the use of this method for dating glacial moraines from many regions of the world. Schäfer et al. [73] used ^{10}Be, ^{26}Al and ^{21}Ne to study the limited evidence for glacial advances in Tibet. Other examples are the detailed work on Swiss alpine glaciers [74], New Zealand [75], and in Antarctica. Studies of surface-exposure dating of the Sirius Formation, Antarctica show an apparent continuous irradiation over the last 2 million years, using ^{10}Be and ^{26}Al [76]. Other studies have been used to determine erosion rates of glacially rounded bedrock and glacial erratics in Antarctica [77]. Stone [78] discussed differences in production rates in Antarctica, which he ascribed to possible differences in atmospheric pressure over this region.

Gosse et al. [79] used ^{10}Be dating on samples from the Wind River Range (Wyoming) to show that the last glacial maximum (Pinedale) was about 21.7 ka and the inner Titcomb basin moraines were of younger Dryas age. Similarly, Zreda and Phillips [80] established a ^{36}Cl chronology of the moraine sequences on the eastern side of the Sierra Nevada. The development of in situ ^{14}C is most important to future understanding of earth-surface processes. In a detailed paper, Lifton et al. [81] showed that in situ ^{14}C could be used to date the ages of the Bonneville shorelines in Utah and also showed the method was consistent with other dating estimates of these surfaces. It is also important to recognize the importance of muon reactions, which can produce cosmogenic products at a considerable depth in the Earth's surface [82–84].

Recently, a project has been proposed to the U.S. National Science Foundation and the European Union to produce the "baseline" information necessary to obtain precision dates using these methods. This program, called CRONUS, anticipates that if we can define production rates, scaling parameters and other components of the calculations, such as cross sections, then we have the possibility of obtaining ±5% measurements of age for in situ cosmogenic radionuclides. These methods will then select primary geologic calibration sites, to provide the "ground truth" studies for these measurements.

An important task in the in situ field is the improvement of standard sites and reference locations. Kubik et al. [85] have discussed the Köfels landslide in Austria as an example of a site which has the characteristics needed for "standard" locations. This site is a large landslide and can be cross-dated to the radiocarbon calibration curve using buried wood from the slide. The production rates for ^{10}Be and ^{26}Al at the site were determined to be 5.75 ± 0.24 ^{10}Be/yr/g SiO_2 and 37.4 ± 1.9 ^{26}Al/yr/g SiO_2 [85]. These are only some of the numerous examples of the use of terrestrial cosmogenic radionuclides which can be found in the literature.

7.2. Atmospheric and in situ radionuclides in ice

Raisbeck et al. [86] used the levels of ^{10}Be in polar ice to examine fluctuations in the cosmic-ray flux over the last 60 ka. These studies were followed later by McHargue et al. [87,88], who looked at ^{10}Be in rapidly deposited marine sediments. Ice cores have a long record of changes in atmospheric CO_2 and CH_4 content. Attempts to "date" this trapped ice have proved difficult [89]. Results obtained by Vander Kamp et al. [90]

suggest the CO_2 also contains a component of in situ produced ^{14}C. Some "heroic" experiments by Petrenko et al. [91] seek to try to determine the ^{14}C age of the trapped CO_2 and CH_4 components, by separating the trapped component from the in situ signal.

Lal et al. [92,93] have used the build-up of in situ ^{14}C to study the history of ice cores. The production of ^{14}C can be used to show that the accumulation of ice can be tracked using cosmogenic nuclides. Lal et al. [94] also showed that the production rates of ^{14}C over time can be studied, if the accumulation rate of the ice for a given site is well-known. In this work, they showed a peak in ^{14}C production during the period 9–10 ka, which is not well explained. This appears to be due to an excursion in production rate which is not well recorded by ^{10}Be in ice or atmospheric ^{14}C.

7.3. Extension of the radiocarbon calibration curve

There have been various attempts to extend the calibration curve beyond the limits imposed by the availability of tree rings. The most useful so far has been the record based on comparison of ^{14}C with U–Th in corals [95,96]. Since that time, there have been many improvements to the calibration. The most recent formal calibration was INTCAL 04. [97]. This calibration uses trees dated by dendrochronology to cover the period from the present back to about 12.5 ka. The calibration continues from 12.5 to 26 ka, using corals dated with the U–Th technique and varved marine sediments. An example of a portion of the calibration curve from 0 to 1950 AD is shown in Fig. 7.

Speleothems have been proposed as a potential calibration record on several occasions [98,99]. Beck et al. [100] produced an extensive record of ^{14}C vs. U–Th from a Bahamas speleothem. There are some questions about the reservoir corrections for speleothems, as there are indeed for marine varve records. At the current time, the best chronologies are still based on the coral and tree-ring records [101].

7.4. Stepped combustion for improved radiocarbon work on sediments

McGeehin et al. [102], drawing on the experience in the dating of pottery [103,104] proposed a stepped-combustion method in order to separate the organic fraction of sediments from more resistant carbon trapped in clays. This was based on the work on Scharpenseel and Becker-Heidemann [105] on the carbon components of soils. These authors had shown that the higher-temperature components derived from soils tended to be older and also associated with the clay fractions, particularly of soil B horizons. Delqué-Kolic [103] had used this idea to try to date pottery, and did some laboratory experiments to create pottery to demonstrate the usefulness of this technique. Results of McGeehin et al.'s work show that the lower-temperature (400 °C) combustion fractions of the humin material (left after acid and base pretreatments), and possibly the finer <63 μm humic material represent the best samples for measuring the age of sediments [102].

In recent experiments at Arizona, Haynes et al. [106] have compared the different approaches of wet oxidation and selective combustion. We conclude that these different approaches are an improvement, for certain kinds of samples.

7.5. Possible use of in situ radionuclides for archaeological samples

Partridge et al. [107] were able to use ^{10}Be exposure dating of sediment samples found in a cave to estimate the age of the oldest known Australopithecus fossil. This represents the beginning of a departure for in situ studies into archaeology, which has a rich potential. Although Partridge et al. dated the sediments associated with the human skeleton, and this raises the usual issues of archaeological association, the principle is important. At our laboratory, we have been investigating the possibility of using in situ ^{14}C to date Late Glacial artifacts. There are many technical and sampling problems inherent in the effort, but if successful the method could prove very valuable for archaeological studies.

Verri et al. [108] discuss the applicability of ^{10}Be to dating of flint tools from a Neolithic site in the Negev desert, Israel. These authors have used ^{10}Be to show that flint artifacts from Qesem Cave and Tabun Cave (Israel) may originate from surface flint or deep mining. It was not possible to determine the age expected to be approximately 300,000 yr old, due to the variable exposure of the flint material. However, the ^{10}Be measurements clearly indicated the difference between surface-collected and deep-quarried material, an important new application for ^{10}Be. Similarly, Ivy-Ochs et al. [109] considered the potential of using ^{10}Be and ^{26}Al for dating Middle Paleolithic artifacts from Egypt.

7.6. Nuclear verification

The proliferation of nuclear weapons technology has made the ability to trace small quantities of nuclides such as ^{236}U and ^{99}Tc very valuable. AMS has the potential of several applications to nuclear-safeguard

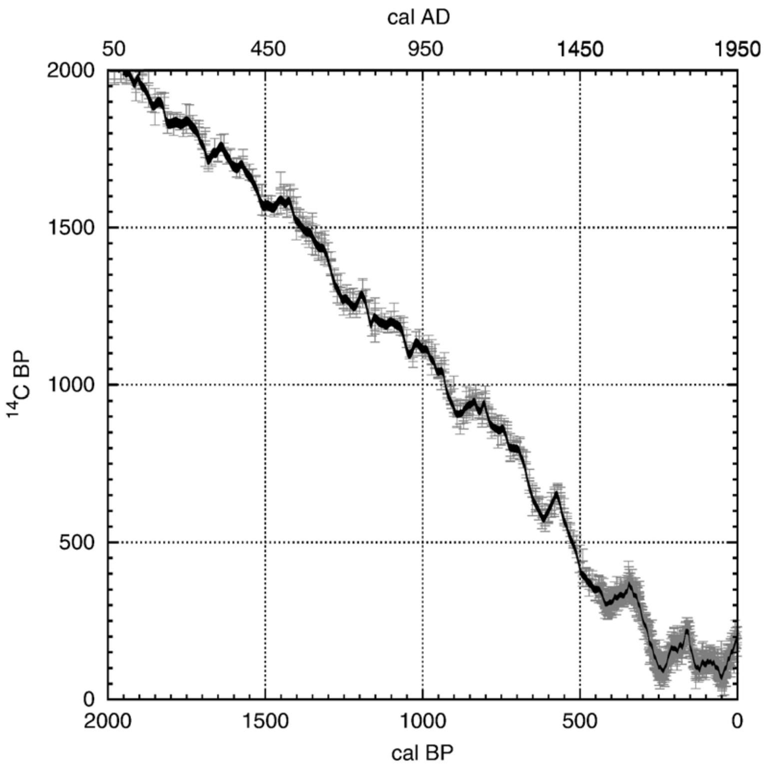


Fig. 7. The IntCal04 terrestrial radiocarbon calibration curve (1σ envelope) and data with 1σ error bars, for the periods 50 to 1950 AD. After Reimer et al. [97].

investigations. For example, Tims et al. [110] have studied Pu and Ra isotopes in soils by AMS at Canberra and others [111] have developed AMS measurements of ^{236}U for nuclear safeguards programs. Similarly, Marchetti et al. [112] have also reported on AMS studies of U and Pu isotopes, particularly ^{237}Np and ^{236}U, which are relevant to a variety of nuclear contamination, risk assessments and safeguards issues.

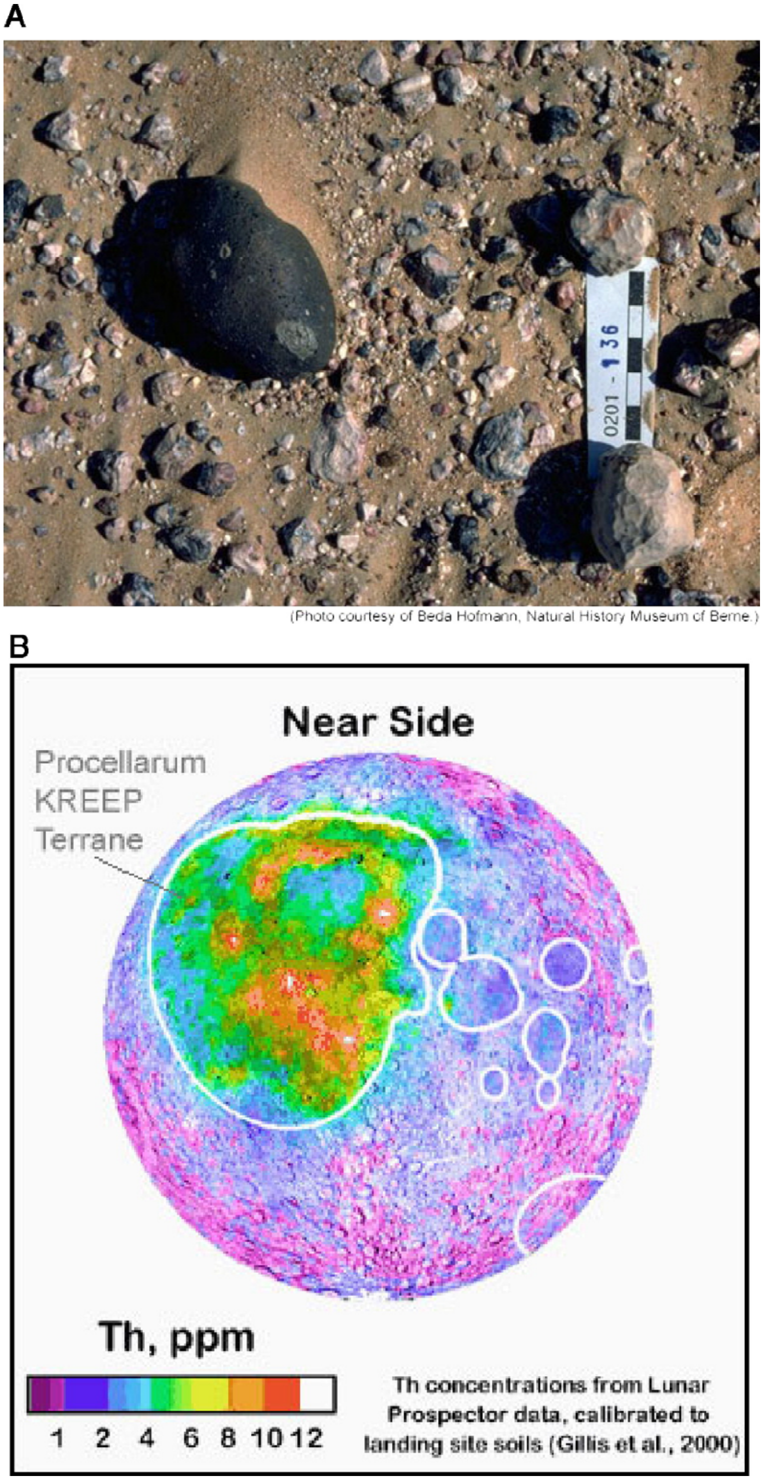
Another new application is ^{99}Tc, Wacker et al. [43] have developed new chemical and AMS methodologies for the measurement of ^{99}Tc. Although in the early stages, this has great potential as a tracer for nuclear waste, since ^{99}Tc does not occur naturally, except possibly by minor muon reactions in the subsurface. Skipperud et al. [113] discuss AMS measurements of Pu isotopes from the Yenisey and Ob estuaries in Siberia. The results show an increase in Pu and a decrease in the 240/239 ratio going upstream towards a nuclear installation.

8. Non-cosmogenic nuclides of geological interest

The extinct nuclide ^{182}Hf decays to ^{182}W. Vockenhuber et al. [114] proposed to use AMS on the VERA machine to study the daughter nuclide. AMS has always had the potential to be useful to the studies of the products of non-equilibration decays in the early solar system, but most of these studies have usually been easier by conventional mass spectrometry. Sie et al. [115] have proposed using AMS for Re/Os measurements and Winkler et al. [116] used AMS to study anthropogenic ^{244}Pu. There have been a number of attempts to measure natural ^{244}Pu which might originate from supernovae. AMS has potential for such measurements, with careful sample selection.

9. Extraterrestrial studies

There have been a number of uses of AMS ^{14}C, ^{10}Be, ^{26}Al and ^{36}Cl measurements for determining the terrestrial ages of meteorites [117,118]. The large number of meteorites recovered from Antarctica, as well as from desert regions, has made these measurements very useful to establish their terrestrial history. The exposure history of lunar samples gives us much information about the flux of galactic and solar cosmic radiation in the past. For example, it was recently demonstrated that a meteorite of lunar composition could be identified as coming from one particular region of the Moon (see Fig. 8), using a combination of cosmogenic nuclide studies and elemental analyses [119]. Several groups of scientists [120–122] have studied detailed depth profiles of various nuclides in lunar rocks and soil cores. Other more recent studies



(Photo courtesy of Beda Hofmann, Natural History Museum of Berne.)

Th concentrations from Lunar Prospector data, calibrated to landing site soils (Gillis et al., 2000)

Fig. 8. Lunar meteorite Sayh al Uhaymir 169 and its source location on the Moon (adapted from *Science* [119]).

have focused on the possibility of determining the radionuclides in cosmic dust samples and cosmic-dust magnetite [123].

10. Oceanography

AMS was instrumental in demonstrating the applicability of the "conveyor belt" model of the world's oceans [124]. One of the great applications of AMS dating using radiocarbon has been the World Ocean Circulation Experiment (WOCE). This grand experiment collected water samples from long transects across the oceans, at various depths. Over 12,000 measurements were performed using the Woods Hole AMS facility, which was originally installed for this purpose. A summary of their recent results is given by Key et al. [125]. This and earlier papers also attest to the excruciating detail and scope of the program, which would not have been possible without AMS. An example of the type of results which have been obtained is shown in Fig. 9. The profile of ^{14}C both latitudinally, longitudinally and with depth is now known for large sections of the world oceans, enhancing our understanding of their complex physical oceanography. A new area of studies is the separation of specific classes of compounds, such as alkenones [ref] which potentially will give new information on the geochronology of marine sediments.

10.1. ^{129}I studies in the ocean

^{129}I studies in the ocean were pioneered by Linas Kilius at Toronto [45]. Since then, ^{129}I has been found to be a ubiquitous tracer in the ocean, and one can follow the plume of ^{129}I emanating from European nuclear reprocessing plants throughout the world's oceans [126,127]. At Arizona, Biddulph [46] has demonstrated that the bomb-spike for ^{129}I migrates rapidly through the ocean and this is presumably by atmospheric transport. Lopez-Gutierrez et al. [128] have studied the distribution of ^{129}I from rainwaters.

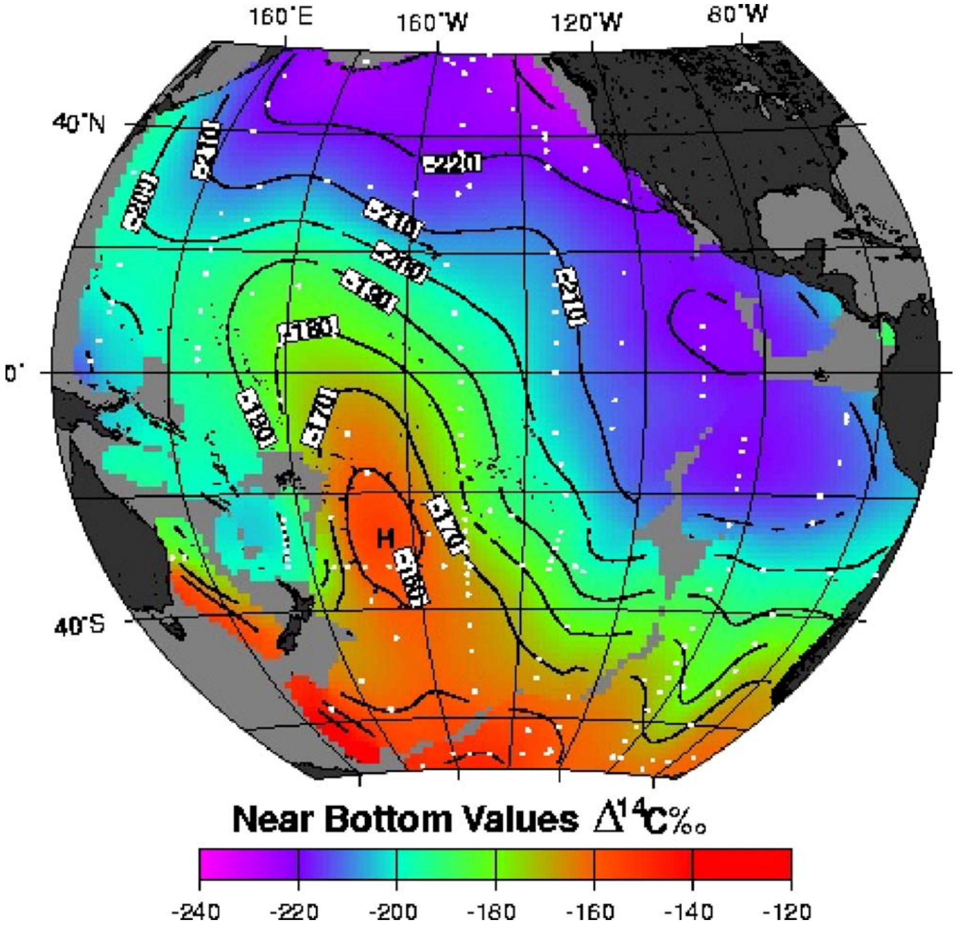


Fig. 9. Summary of measurements of 14C measurements in near-bottom waters from the Pacific Ocean as part of the World Ocean Circulation Experiment.

11. Paleoclimatic applications

An immense number of studies have used AMS radiocarbon and other radionuclide studies applied to paleoclimatic studies. Some recent studies are of interest, especially relevant to the question of the long-term periodic climate change. Recently, Turney et al. [129] demonstrated the long-term cyclicity of El Niño events on millennial time-scales. Rowe et al. [130] have contributed a large amount of work on the dating of Lake Titicaca. Charles et al. [131] demonstrate the importance of monsoon–tropical interactions. Millennial-scale climatic change was linked to ice-rafting events by Charles et al. [131] and Bond et al. [132] and also related to possible solar influences. Similar impressions of this type of periodicity are evident in other oceanic records [133,134] , lake sediments [135] and in records of periodicity in forest-fires [136]. Barber et al. [137] have generated a viable explanation for the widely observed 8200 yr cold event in the northern hemisphere. AMS dates were crucial to this study. The use of AMS in paleoclimate studies goes well beyond the scope of this paper, and we refer the reader to more general treatments for a broader discussion of the subject [15,16,138–140].

12. Exotic AMS applications using full ion stripping

A precise means of measuring ^{59}Ni, ^{63}Ni and ^{60}Fe is to use a very large accelerator. These measurements have been performed by fully stripping the ions of all electrons, using the very large AMS machines at Munich and in Canberra. Straume et al. [141] discussed ^{63}Ni as used for the measurement of Cu samples from Hiroshima, following earlier studies of ^{36}Cl. The purpose of these studies was to establish the epithermal and thermal neutron fluxes from the radionuclide records.

In a different application, Knie et al. [142] identified ^{60}Fe in manganese nodules from the Pacific ocean floor. They assert these few counts they measured are evidence of supernova-produced ^{60}Fe on the Earth. Schnabel et al. [143] have studied ^{59}Ni resulting from cosmic-ray interactions with iron meteorites and used this information for modeling the size of the Canyon Diablo iron meteorite, which produced Meteor Crater in Arizona.

13. Future possible uses of AMS

The development of much smaller AMS machines does not exclude the usefulness of larger machines particularly for the measurement of the heavier nuclides. However, their development means that for some radionuclides, especially ^{14}C, that an increasing number of smaller AMS laboratories will proliferate. These laboratories will be available to make the tens of thousands of radiocarbon measurements which are expected each year by the scientific community. The increasing compact size of these machines does not yet mean that we can have a “table-top” AMS device, though it is increasingly likely that some kind of truly compact device could be built for specialist purposes.

13.1. Where can we go?

It is unlikely that an AMS device would ever be made small enough to transport on a space craft, but it is not impossible. More appropriate is the discussion of measurements of the radionuclides on returned Martian samples, expected by about 2015. Radionuclides have taught us much about the Moon's surface. We know for example that it is a very stable surface, and we can say something about the integral cosmic-ray exposure on the moon. Similarly, we have learned about the exposure history and infall rate of meteorites from their radionuclide compositions. Refinement of AMS equipment and chemistry to deal with ultra-small samples of Martian atmospheric CO_2, ice and rock samples should be developed in advance of sample-return missions, to make of the opportunity.

For practical experiments, we will have to wait for returned samples of interest. Samples of the NASA Genesis mission returned in September 2004 (see Fig. 10), although unfortunately, since the return module crashed into the Utah desert, some studies may be compromised. This mission exposed various materials to irradiation by the solar wind [144] and will test the limits of AMS for detection of many nuclides; indeed this is even more so due to possible contamination due to the crash-landing of the return module. Scientists on this project will attempt to measure low levels of oxygen isotopes implanted into Si and radionuclides such as ^{10}Be and ^{26}Al, implanted into Si and other substrates.

Mars sample return is expected in the next decade. Perhaps we will eventually have “sample return” missions from other bodies, as well as Mars. Of particular importance will be the measurement of ^{14}C in the Martian atmosphere and other reservoirs. This is becoming increasingly important due to the recent evidence of water on Mars in the past. The best approach for Mars is sample return, which we expect to happen in the next decade. Returning atmospheric samples from Mars, surface sediments and carbonates, along with polar ice-cap material, would allow us to

Fig. 10. Artist's depiction of the GENESIS mission solar-wind measurement (NASA photo).

develop a good model of the “carbon cycle” and size of exchangeable reservoirs on Mars. AMS measurements of ^{14}C in these materials would allow us to establish a Martian carbon cycle. Jakosky et al. [145] have discussed the expected sizes of various reservoirs and using this information, we can devise a prototype Mars carbon cycle (see Fig. 11).

Information on Martian geomorphology could result from cosmogenic nuclide studies of surface features, analogous to those conducted on the Earth, and the much higher production rate on Mars would make this easier on Mars. Even further afield, there are other “carbon cycles” to be explored. For example, Lorenz et al. [146] discussed the possibility of radiocarbon measurements of atmospheric species from Titan.

14. Conclusions

AMS has brought about a great revolution in radionuclide measurements over the last ¼ century. New developments in both applications and methodology continue to make this technique of inestimable value to a wide range of studies. We could only touch on a few of these exciting topics in this paper, but we hope that the reader has received an overview which will be useful.

We believe we can predict that the following areas are likely to benefit from future developments in AMS within 5–10 yr:

1. Surface exposure dating and in situ cosmogenic dating will expand rapidly as a result of the concerted international effort to characterize production rates through the CRONUS program.
2. Gas ion sources for ^{14}C measurements will become a routine.
3. On-line methods such as attaching gas chromatography to an AMS and high-pressure liquid chromatography will allow on-line AMS measurements of very small samples with minimal sample processing. These methods will also allow for dating of specific compounds [147].
4. Paleoclimatic studies will be improved by a much better understanding of the oceanic system due to in-depth studies of ^{14}C in corals, speleothems and

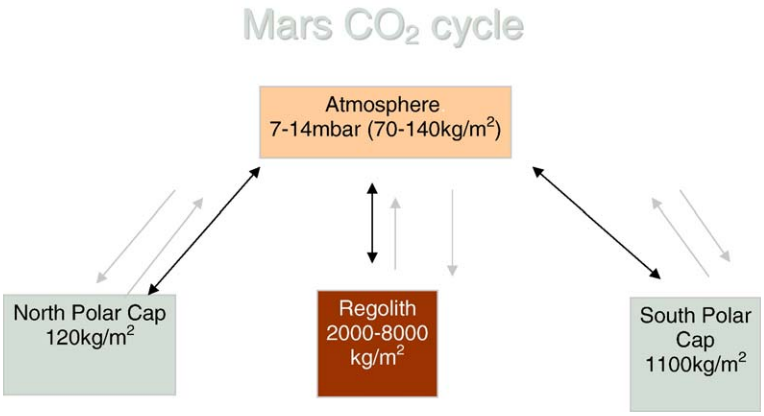


Fig. 11. Possible Martian carbon cycle using reservoir sizes estimated by Jakosky et al. [145]. Radiocarbon and other radionuclides will play a crucial role in determining the size of these reservoirs.

marine varved sediments. Further investigations of the driving forces for periodicities in the climatic record will also result.

5. Single-stage AMS using lower voltages and less cumbersome technology, such as the new Lund AMS, will mean a rapid expansion of the number of laboratories which perform AMS, at least for radiocarbon. This means that the larger facilities will have to concentrate on more difficult radionuclide measurements.

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