New Results for Single Stage Low Energy Carbon AMS

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ABSTRACT. A new configuration of the NEC single stage, low energy carbon AMS system (patent pending) has been built and tested. The injector includes two 40-sample ion sources, electrostatic and magnetic analysis, and fast sequential injection. The gas stripper, analyzing magnet, electrostatic analyzer, and detector are on an open air 250 kV deck. Both 12C and 13C currents are measured on the deck after the stripper, and an SSB detector is used for 14C counting. Injected 12C and 13C currents are also measured. Automated controls follow a user-specified run list for unattended operation. Initial test results show precision for 14C/12C ratios of better than 5 per mil, and backgrounds for unprocessed graphite of less than 0.005 x modern. We will report final results for precision, background, and throughput and discuss related design features.

1. Introduction

For ^{14}C accelerator mass spectrometry (AMS), milligram-size graphite samples are loaded into an ion source, which produces negative ion beams of ^{12}C, ^{13}C, and ^{14}C ions and of molecules such as, ^{13}CH and $^{12}CH_2$, from the sample material by sputtering. After mass analysis, acceleration, molecular dissociation, and magnetic and/or electrostatic filtering, the ^{12}C and ^{13}C beams are measured in Faraday cups, and the ^{14}C ions are counted in a surface barrier or gas filled detector to determine the ^{14}C concentration in each sample.

Historically, ^{14}C AMS systems have used tandem electrostatic accelerators operating at about 2.5 MV or higher. Tandems accelerate the negative ions from the ion source to a positively charged terminal, where the ions pass through a “stripper” (usually a long, narrow tube filled with argon gas at a few millitorr pressure) that strips electrons from the ions and dissociates molecules. The positively charged ions from the stripper accelerate a second time, back to ground potential. Typical tandem accelerators are housed in steel tanks containing SF_6 gas at 5-6 atmospheres pressure for electrical insulation.

The use of negative ions and the stripper eliminate otherwise overwhelming interferences, making tandems well suited for AMS of carbon and other elements. For ^{14}C AMS, since nitrogen does not form a stable negative ion, this primary mass-14 interference is eliminated, and dissociation in the stripper eliminates molecular isobars.

Voltages less than about 2.5 MV were thought to be inadequate for dissociating molecular background interferences until the ETH Zurich group demonstrated low background carbon AMS with 0.5 MV tandem accelerators [1-2]. Since then, eight similar commercial 0.5 MV AMS systems are in use, being installed, or in manufacture. These “compact” systems meet the needs for applications requiring routine precision of about 3‰ and carbon backgrounds below 0.2% modern (50 ka, 0.027 dpm^{14}C/gmC).

Some emerging AMS applications require less stringent performance of about 5‰ precision with 0.5% modern (0.068 dpm^{14}C/gmC) carbon backgrounds. Based on experiments with the compact systems that demonstrated such performance at voltages to below 300 kV, National Electrostatics Corp. (NEC) developed the Single Stage Accelerator Mass Spectrometer (SSAMS, patent pending) as an alternative to the tandem-based design.

2. Single Stage AMS (SSAMS) System Design

Unlike tandems, SSAMS uses an air insulated deck in place of the high pressure SF_6 insulated accelerator, and SSAMS does not have a second acceleration stage. The beam filtering and measuring components are located after the stripper on the deck. This avoids an interference seen in tandem systems where molecular breakup fragments can change charge during the second acceleration and make their way to the ^{14}C detector.

A 300 keV prototype SSAMS proved feasibility [3], but performance was inconsistent. The simplified test design measured ^{12}C before acceleration and accelerated only the mass-14 beam. Results were good if the system was properly tuned and did not drift, but these factors were unknown until the measurement was nearly completed. In addition, the 300 keV deck accommodated a 40 sample ion source, but not a 134 sample source.

The commercial SSAMS design (Figure 1) reverses the prototype's layout. The ion sources and injection beam line are at ground potential, and the stripper, analyzing magnet, offset Faraday cups to measure ^{12}C and ^{13}C beam currents, an electrostatic spherical analyzer (ESA) to filter the mass-14 beam, and a surface barrier detector are mounted on a 250 kV deck. The first SSAMS sold (Figure 2), for the Radiocarbon Laboratory at the Geobiosphere Science Center in Lund, Sweden, has two 40 sample MC-SNICS ion sources. Other SSAMS's have been ordered with 134 sample MC-SNICS ion sources.

SSAMS uses sequential isotope injection with the vacuum chamber of the injection magnet biased to preset voltages in sequence for injection and acceleration of each isotope. Running at 10 Hz, ^{12}C is injected for about 150 microseconds per cycle, ^{13}C for about 300 microseconds per cycle, and ^{14}C the rest of the time (about 99% of each cycle).

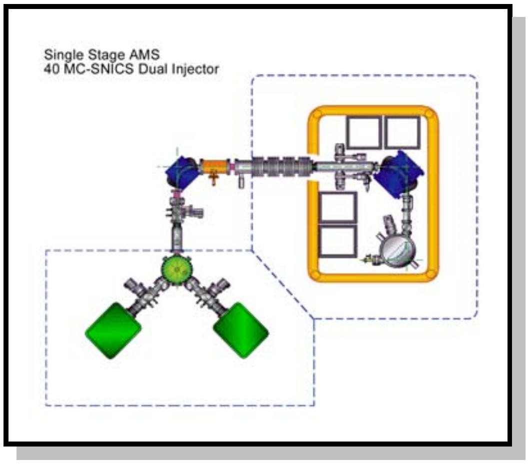

Figure 1. The first commercial Single Stage AMS system is equipped with dual 40 sample ion sources and a fast sequential injector. Mass 12, 13, and 14 beams are measured on the 250 kV deck after acceleration, stripping, and filtering.

Injection beam line components include a 45 degree, rotatable ESA to switch between ion sources; Y-steerers, one of which is automatically cycled to preset values for each isotope during the injection sequence; an injection magnet; an offset Faraday cup to measure injected ^{12}C and ^{13}C currents; and an Einzel lens to focus the beams through the NEC GP acceleration tube and the molecule dissociating gas stripper.

The tapered stripper tube is about 46 cm long. Two 250 l/sec turbo molecular pumps differentially pump the stripper housing. Identical pumps are on each ion source and the beam lines. An ion gauge on the stripper's inner pumping chamber provides accurate stripper gas pressure readings. A beam profile monitor before the injection magnet facilitates beam tuning, as do Faraday cups by each source, the stripper, and the detector. A 4 kVA isolation transformer powers beam line, control, and data acquisition components on the deck.

Figure 2 SSAMS system for the Radiocarbon Laboratory at the Geobiosphere Science Center, Lund, Sweden, during factory tests. The larger (blue) 300kV deck for the prototype tests is in the background to the left of the SSAMS.

System control is by NEC's *AccelNET* control software, running on Linux. It has monitor displayed controls and assignable knobs and meters. It automatically cycles through multiple measurements on each cathode in the order and for times the operator specifies in a run list. It updates gated detector spectrum displays during data acquisition; displays and logs ratios and parameter values for each run on each cathode; and logs ^{14}C events and ^{12}C and ^{13}C beam currents for each injection cycle. The $^{13}C/^{12}C$ ratio indicates system stability during measurements and yields delta 13C correction factors for unknowns. Detector data acquisition is on only while ^{14}C is being injected. The operating parameter database simplifies start up.

Off line analysis software plots run ratios, calculates rare isotope ratios and uncertainties for cathodes from the runs on each cathode and for samples from the cathode results, subtracts background, applies delta 13C corrections, and outputs results in percent modern carbon, conventional radiocarbon age, and specific density (dpm^{14}C /gmC, and attomole^{14}C /mgC). Chi squared analysis determines if the scatter of ratios is consistent with their counting statistics. Detailed “per cycle” plots of run data and repeat off line data reduction are included.

3. Performance and Comparisons

The following results are preliminary to the extent that they may be improved, because time and other constraints prevented comprehensive final testing. Despite the limitations, performance improved, especially backgrounds, as set up, design, and operating values came together over a period of about 3 weeks. By shipment, the SSAMS system surpassed the design goals of 5‰ precision with 0.5% modern (0.068 dpm^{14}C/gmC) carbon backgrounds.

At 284 keV (40 keV injection, 244 kV deck bias), $^{14}C/^{12}C$ and $^{14}C/^{13}C$ precisions were better than 5‰ (for example, see Table 1), and ratio scatter was consistent with counting statistics.

Backgrounds for dead graphite normalized to OxII or OxI were better than 0.4% modern (44.3 ka, 0.054 dpm^{14}C /gmC) and sometimes near 0.25% modern (48 ka, 0.034 dpm^{14}C /gmC).

TABLE 1. Initial Testing - Precision for 3 OxI Cathodes at 284 keV - 01 July 2004

Cathode Position	Ave ^{12}C (µA)	Ave ^{13}C (nA)	^{14}C Counts	^{14}C/^{12}C	^{14}C/^{13}C
3	9.318	101.9	56,708	8.1256e-13	7.4289e-11
7	9.312	101.4	56,802	8.1445e-13	7.4756e-11
15	9.636	105.7	58,674	8.1300-13	7.4096e-11
Mean				8.1334e-13	7.4380e-11
Relative Standard Deviation				0.12%	0.46%

As expected, beam transmission (mid-30% range) was below the stripping yield. SSAMS efficiency (^{14}C counts per Coulomb of ^{12}C) was 78% to 87% of the efficiency of the compact 0.5 MV AMS systems, which is an improvement from 73% for the prototype. These are both probably due to beam loss from increased scattering in the stripper at low energies.

Good vacuum in the SSAMS is important, because its shorter beam lines provide less resolution for filtering interferences than in the compact and 3 MV systems. An extra pumping baffle between the stripper and analyzing magnet improved the vacuum in the analysis beam line and reduced ratio scatter.

The amounts by which component settings can vary without affecting results (“flat tops”) in the SSAMS system are comfortably wide, similar to those seen with the compact systems.

A comparison of operation at 284 keV and 244 keV (204 kV deck bias) informs against going to lower energies. At 244 keV, the scatter of rare isotope ratios was unacceptable. For ANU sucrose, the SDOM of cathode ratio scatter was 0.55% for 0.15% counting statistics, and for “dead” commercial graphite the SDOM was twice as high as the 2.5% statistical limit. The measured rare isotope ratios for the “dead” graphite were 40% to 50% higher, which suggests that interferences from inadequate dissociation and scattering or charge exchange in the analysis beam line are unacceptably worse at the lower energy.

Compared to the compact 0.5 MV tandem AMS system, the SSAMS requires about 40% less floor space and is priced about 27% lower. SSAMS uses about 7.5 kVA in normal operation.

4. Conclusions

The open air single stage SSAMS is capable of routine use for applications requiring modest precision in the 4‰ to 5‰ range with backgrounds below 0.5% modern for carbon. This includes measuring samples that are originally in or diluted down to or just below the 1% modern (~0.1 dpm^{14}C /gmC) range. Sample throughput is sufficient, for example, for unattended measurement of a wheel of 40 samples to a precision of 5‰ (for modern samples) during an overnight shift.

Installation of the first SSAMS, in Lund, Sweden, was completed in less than 3 weeks, and final acceptance tests have just begun as this is being written. Long term operation will indicate ultimate background limits and routine performance levels.

References

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