

THE NEW NATIONAL OCEAN SCIENCES ACCELERATOR MASS SPECTROMETER FACILITY AT WOODS HOLE OCEANOGRAPHIC INSTITUTION: PROGRESS AND FIRST RESULTS

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ABSTRACT. Start-up performance and first results of the new Woods Hole Accelerator Mass Spectrometer are discussed. Special attention is given to the hemispherical ionizer sputter source and the triple-isotope injector design.

INTRODUCTION

In 1988, the National Science Foundation funded a research proposal to establish an Ocean Sciences Accelerator Mass Spectrometer (AMS) facility at Woods Hole Oceanographic Institution (WHOI) (Jones *et al.* 1990). The 9300 sq. ft. AMS addition to the existing McLean Laboratory on WHOI's Quissett Campus was completed in May 1990. The building was designed to house the dual injector AMS machine as well as sample preparation, engineering laboratory and offices in an optimized configuration to fulfill the high sample throughput requirements set by major Ocean Sciences field programs, such as World Ocean Circulation Experiment (WOCE) and Global Ocean Flux Study (GOFS). In summer 1990, the assembly of the 3 MV tandem accelerator mass spectrometer was completed as a single injector machine and went through several months of factory testing at US-AMS Co. in Topsfield, Massachusetts. During that period, a few important design improvements were made as a consequence of early test results. Details of the machine design have been published elsewhere (Purser, Smick & Purser 1990; Litherland & Kilius 1990) and is discussed by K. H. Purser (1992). In September 1990, the AMS machine was disassembled and shipped to WHOI, where it took 1.5 months to reassemble and install the machine in its final location. First beam was obtained at WHOI in November 1990, and on 22 January 1991, the first ^{14}C signals were observed in the ionization chamber. While initial phase testing continued, the parts for the second injector were shipped in March 1991, and are presently being assembled.

In this contribution, we focus on the start-up performance of the AMS machine. McNichol *et al.* (1992) discuss some of the issues of sample preparation at our facility.

AMS PERFORMANCE

The cesium sputter ion source is a design based on work by Middleton (1976) and is similar to that built by the Genus Co. in Newburyport, Massachusetts, for Lawrence Livermore National Laboratory. The hemispherical ionizer, through the center of which the negative ion beam is extracted, is capable of producing intense extracted beam currents in routine operation (0.5–1 mA, measured at the exit of the source). By closely monitoring and regulating both the Cs reservoir and the transfer tube temperatures, the Cs vapor supply is stabilized. The graphite sputter targets (containing *ca.* 2 mg of carbon for analysis) are pressed into 2-mm-diameter holes in aluminum cartridges. A carousel holds 59 of these targets. The target changer is operated by DC servomotors

on the high-voltage deck. The sputtered negative ions emerge through the hole in the ionizer, and pass through an extraction gap, gaining a total energy of 40 keV. The acceptance half-angle is 10 mrad, limited by an aperture plate at the exit of the source.

A cylindrical einzel lens brings the beam to an object point for the “recombinator,” a low-energy mass separator for the simultaneous injection of the three carbon isotope beams. The Toronto group (Litherland and Kilius 1990) designed the arrangement of magnetic and electrostatic elements of the recombinator, and the beam line layout was verified with code RAYTRACE (Enge 1990). Although not the first application of a recombinator to AMS (Southon, Nelson & Vogel 1990), this one is simple to set up and align, because of the separated functions of the vertically focusing electric slot lenses and horizontally focusing dipole magnets. The horizontal separation of the three parallel beams in the mirror plane of the recombinator is 19 mm. An aperture plate at this location removes mass 15 (NH^-) and higher masses. There are no defining apertures for mass 12, 13 and 14. A chopper wheel was introduced at this location to attenuate the mass 12 beam by a factor of 85, which makes it approximately equal in intensity to the mass 13 beam. Faraday cups are inserted to measure the mass 12 in the mirror plane, and at the entrance lens to the accelerator. Insertable flags can block the mass 13 and 14 beams for testing purposes. Typical pre-attenuation currents in the mass 12 channel are 50 μA .

After recombination, the three carbon ion beams have a central ray divergence of no more than 3.4 mrad, well within the acceptance of the accelerator. The total current injected into the accelerator is typically *ca.* 1 μA . The terminal of the tandem accelerator is run at 2.5 MV, and has proven to be quite stable (drift < 1 kV) if the dew point of the SF_6 tank filling is held below -50°C (STP) and the tank pressure kept at 75 psig. The total ripple (RF plus AC) on the terminal is *ca.* 160 V, measured by a calibrated capacitive pickup near the terminal. At the image point of the 110° magnet the analyzed ^{12}C and ^{13}C currents (optimized for charge state 3^+) are *ca.* 0.5 μA . The stripper gas pressure is *ca.* 0.01 mbar as measured by a terminal Pirani gauge. The transmission in all three isotope channels has been compared by switching the mass 13 beam into the 3 isotope paths of the recombinator as well as the high-energy leg of the system. The agreement between the various Faraday cup readings was within 5%. The ^{14}C leg and the gas ionization detector underwent further studies to assess backgrounds. We allowed ^{14}N to enter the detector by slightly raising the terminal voltage to 2.54 MV to measure the energy separation of the isotopes in the detector. The separation between the ^{14}C peak and the ^{14}N peak was > 3 half-widths. Figure 1 shows a typical ^{14}C energy spectrum for an NBS Oxalic Acid I sample. Because of the good energy separation, it has not been necessary to run the detector in the $\Delta\text{E-E}$ mode, relying on the high efficiency of the background suppression measures in the high-energy leg of the system (Purser & Litherland 1990). At rates of *ca.* 80 s^{-1} , it took less than 10 min to acquire 40 k counts in the peak. The precision and stability of the $\delta^{13}\text{C}$ measurements within a run cycle is routinely *ca.* 1 part per thousand. First estimates from blank samples and dead graphite yield a machine background of *ca.* 0.07 pMC in the present state of the system. Table 1 lists all relevant system parameters that presently determine a standard tune-up of the machine.

We have made a series of pre-acceptance runs on samples, prepared in our own laboratory, to assess various performance factors of both hardware and software. The overall system stability has been excellent, allowing extended periods of uninterrupted running. At present, the system operates in a semi-automatic mode: after an initial manual tune-up to a stable condition, the system is put into the automatic “analysis” mode. Up to 59 samples can be run in any order and with repetitions. The rates of the three carbon isotopes are monitored in acquisition periods of about 10 s, during which the ^{14}C pulse-height analyzer is gated while 10 independent readings of each of the ^{12}C and ^{13}C Faraday cup currents are recorded and averaged. The terminal voltage is continually monitored,

TABLE 1. System Parameters for Normal Tune-Up

Source vacuum	2E-6 mbar
Cs reservoir temperature	200°C
Cs transfer tube temperature	245°C
Extraction voltage	-31.5 kV
Target voltage	-7.2 kV
Target current	2.5 mA
Einzel lens voltage	-31.8 kV
X steerer voltage	-20.0 V
Y steerer voltage	47.5 V
Recombinator vacuum	1.6E-6 mbar
Recombinator magnets field	0.4 T
Slot lens #1 voltage	-19.7 kV
Y steerer #1 voltage	128.8 V
Slot lens #2 voltage	-19.4 kV
Y steerer #2 voltage	53.8 V
Q-snout vacuum	4E-7 mbar
Q-snout 2. electrode voltage	15.4 kV
Q-snout 3. electrode voltage	26.0 kV
Q-snout X steerer voltage	-6.0 V
Q-snout Y steerer voltage	0.0 V
Terminal voltage	2.5 MV
Stripper gas pressure (recirculated)	1E-2 mbar
Quadrupole vacuum	2E-7 mbar
Quadrupole #1/#3 voltage	-9.3 kV
Quadrupole #2/#4 voltage	7.8 kV
X steerer #1 voltage	156 V
Y steerer #1 voltage	-458 V
X steerer #2 voltage	-208 V
Y steerer #2 voltage	186 V
110° magnet field	1.24 T
33° deflector vacuum	1E-7 mbar
33° deflector (l) voltage	41.3 kV
33° deflector (r) voltage	-41.4 kV
90° magnet field	1.24 T
Detector isobutane pressure	17 mbar

and in case of a terminal shutdown, the data acquisition is interrupted until a background routine recovers the voltage to the prior setpoint. Figure 2 shows the preliminary results of a run cycle on three types of graphite targets, prepared from seafloor sediment CaCO₃ from Bahamas bank (40 pMC) (A), NBS Oxalic Acid I (B), and Buzzards Bay seawater (116 pMC) (C).

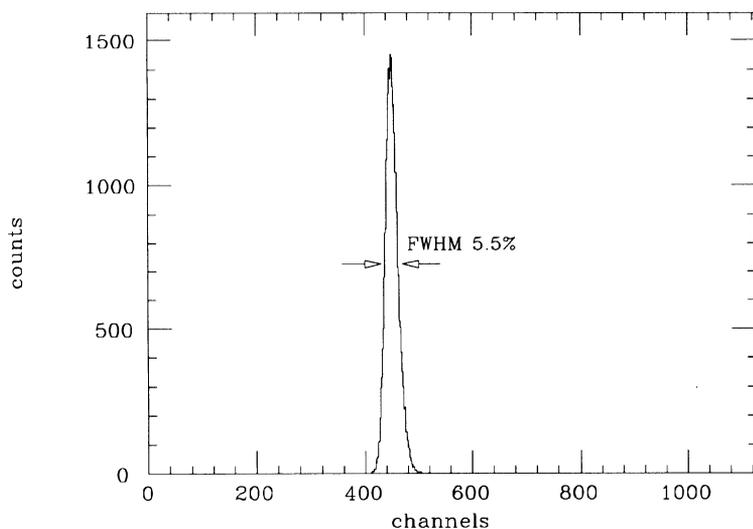


Fig. 1. Pulse-height spectrum (40 k counts) taken with the isobutane gas ionization detector for 10 MeV $^{14}\text{C}^{3+}$ ions from a graphite sputter target, prepared from NBS Oxalic Acid I. No lower or upper level discrimination was applied to the signals.

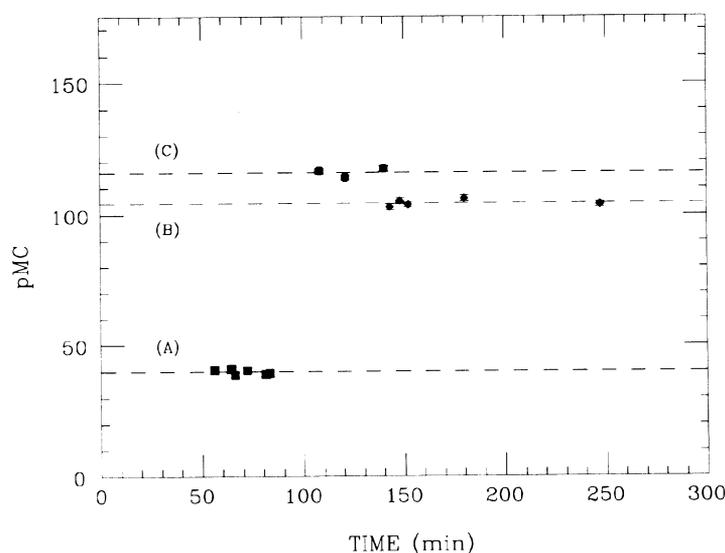


Fig. 2. ^{14}C levels measured during one AMS run cycle for three types of graphite samples, prepared from: (A) sediment CaCO_3 (Bahamas bank); (B) NBS Oxalic Acid I; (C) Buzzards Bay (Massachusetts) seawater. The data are normalized to the NBS Oxalic Acid I standard.

CONCLUSIONS

Even though the first test results are very encouraging, and lead us to believe that we will reach our goals in a timely manner, the real challenge still lies before us. Rigorous acceptance tests are planned for this summer, and high throughput rates have to be achieved on a day-to-day basis.

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REFERENCES

- Enge, H. A. 1990 "RAYTRACE" computer code.
- Jones, G. A., McNichol, A. P., von Reden, K. F. and Schneider, R. J. 1990 The National Ocean Sciences AMS Facility at Woods Hole Oceanographic Institution. *In* Yiou, F. and Raisbeck, G. M., eds., Proceedings of the 5th International Conference on Accelerator Mass Spectrometry. *Nuclear Instruments and Methods B52*: 278–284.
- Litherland, A. E. and Kilius, L. R. 1990 A recombinator for radiocarbon accelerator mass spectrometry. *In* Yiou, F. and Raisbeck, G. M., eds., Proceedings of the 5th International Conference on Accelerator Mass Spectrometry. *Nuclear Instruments and Methods B52*: 375–377.
- McNichol, A. P., Gagnon, A. R., Jones, G. A. and Osborne, E. A. 1992 Illumination of a black box: Analysis of gas composition during graphite target preparation. *Radiocarbon*, this issue.
- Middleton, R. 1976 A review of sputter negative ion sources. *IEEE Transactions of Nuclear Science NS-23*: 1098–1103.
- Purser, K. H. 1992 A high throughput ^{14}C accelerator mass spectrometer. *Radiocarbon*, this issue.
- Purser, K. H. and Litherland, A. E. 1990 The elimination of charge-changing backgrounds in an AMS radiocarbon system. *In* Yiou, F. and Raisbeck, G. M., eds., Proceedings of the 5th International Conference on Accelerator Mass Spectrometry. *Nuclear Instruments and Methods B52*: 424–427.
- Purser, K. H., Smick, T. H. and Purser, R. K. 1990 A precision ^{14}C accelerator mass spectrometer. *In* Yiou, F. and Raisbeck, G. M., eds., Proceedings of the 5th International Conference on Accelerator Mass Spectrometry. *Nuclear Instruments and Methods B52*: 263–268.
- Southon, J. R., Nelson, D. E. and Vogel, J. S. 1990 Injection systems for AMS: Simultaneous versus sequential. *In* Yiou, F. and Raisbeck, G. M., eds., Proceedings of the 5th International Conference on Accelerator Mass Spectrometry. *Nuclear Instruments and Methods B52*: 370–374.