

## A NEW 1MV AMS FACILITY AT KIGAM

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**ABSTRACT.** A 1MV AMS was installed in KIGAM (Korea Institute of Geoscience and Mineral Resources). After 4 months of installation, the AMS started normal operation from January 2008. This multi-element AMS was developed by HVEE to measure  $^{14}\text{C}$ ,  $^{10}\text{Be}$ , and  $^{26}\text{Al}$ . The results of an acceptance test demonstrate that this machine is capable of routine  $^{14}\text{C}$  age dating and of measurements of other radioisotopes in terms of accuracy and precision as well as the background level. After installation, an investigation aimed at determining the stable operating conditions was conducted, and background levels were determined to be as low as  $10^{-15}$  for  $^{14}\text{C}$  and  $10^{-14}$  for  $^{10}\text{Be}$  and  $^{26}\text{Al}$ .

## INTRODUCTION

Many reports concerning the development of low-energy accelerator mass spectrometry (AMS) systems have been published over the last decade by several research groups following the lead of the ETH group (Suter et al. 1997; Synal et al. 2000; Müller et al. 2008; Hughey et al. 2000; Klein et al. 2006, 2007). Several compact AMS machines with energies lower than 1 MV are in operation around the world, and their feasible performance levels have been reported. KIGAM (Korea Institute of Geoscience and Mineral Resources) has installed a Cockcroft-Walton type 1MV compact AMS facility (4110Bo-AMS-3) at its geological and environmental analysis center. A 1MV AMS is a balanced system because it is compact with a much lower running cost and lower manpower requirements compared to 3 MV or higher energy AMS systems. However, it demonstrates acceptable performance not only for  $^{14}\text{C}$  measurements but also for the measurement of other isotopes such as  $^{10}\text{Be}$  and  $^{27}\text{Al}$ , whereas 500kV AMS are currently used only for the purpose of  $^{14}\text{C}$  measurements. This second AMS machine in Korea is intended to be dedicated to geological and environmental research. This machine is the second “operating” 1MV AMS system from HVEE (High Voltage Engineering Europa, B.V., Amersfoort, the Netherlands) after one installed in Spain (Chamizo et al. 2008). Recently, an additional 1MV AMS started operation at the Norwegian University of Science and Technology. It is the second AMS manufactured, but the third machine installed.

The project to install the AMS system at the KIGAM had been planned since 2003 and actually began with a contract with HVEE for a 1MV multi-element AMS system in 2005. The installation of the machine started in July 2007, and the acceptance test was completed at the end of October 2007. The results of the acceptance tests for  $^{14}\text{C}$ ,  $^{10}\text{Be}$ , and  $^{27}\text{Al}$  isotopes and the  $^{14}\text{C}$  dating performance are demonstrated in this work.

## DESCRIPTION OF THE AMS SYSTEM

As the details of the HVEE 1MV AMS layout have been published elsewhere (Klein et al. 2006, 2007; Chamizo et al. 2008), only a brief description will be given here. The layout of the AMS laboratory is shown in Figure 1. A sputter source with a 50-target carousel (SO110/50) is provided. This ion source has been slightly upgraded recently by HVEE; hence, it has a slightly different internal structure compared to the first machine in Spain. Isotope selection measurements can be done using a biased injector magnet (bouncer), as developed for multi-element measurements and operated at a frequency of 100 Hz, and a maximum voltage of 3 kV. Argon gas is employed for charge

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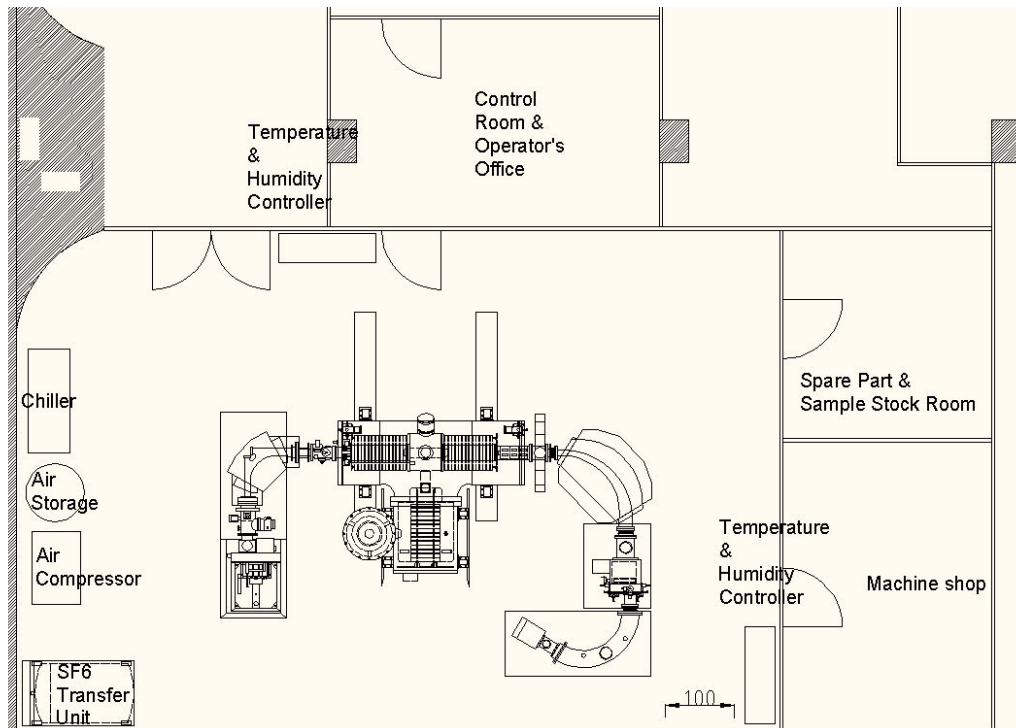


Figure 1 Layout of the 1 MV AMS machine of KIGAM. A large window is mounted on the wall between the accelerator room and the control room, so that operator can see the AMS machine during the operation. Distances in the figure are given in centimeters.

stripping at the accelerator. Two off-axis Faraday cups are provided for stable isotope measurements after an analyzing magnet. One is fixed for  $^{12}\text{C}$  measurements and the other is movable for  $^{13}\text{C}$ ,  $^9\text{Be}$ , and  $^{27}\text{Al}$  measurements. An electrical spherical analyzer (ESA) is used to separate ions with different energies from radioisotopes of interest. For Be measurements, a  $\text{Si}_3\text{N}_4$  absorber foil with a thickness of 150 nm is used to create an energy difference between the  $^{10}\text{Be}$  and the  $^{10}\text{B}$ . The radioisotope detector system is composed of a gas-filled ionization chamber with 2 anodes for  $\Delta E$ - $E_{\text{res}}$  measurement. The isobutane gas pressure of the detector is 7–8.5 mbar, and a 75-nm-thick  $\text{Si}_3\text{N}_4$  entrance window separates the detector gas from a beam line vacuum. The data collection system is composed of a 2-parameter multichannel analyzer (MCA) from FAST ComTec (FAST ComTec Communication Technology GmbH, Grünwalder Weg 28A, 82041 Oberhaching, Germany). The specifications of the machine are summarized in Table 1.

Table 1 Specifications of the 1MV AMS system of KIGAM.

Dimension	$4.2 \times 6.2$ m
Ion source	100 $\mu\text{A}$ max
Pre-acceleration voltage	35 kV
Bouncer	3 kV, 100 Hz
Terminal voltage and upcharge current	1 MV, 2 mA
Analyzing magnet	$90^\circ$ , 63 MeV amu, max current 300 A
Electrical spherical analyzer (ESA)	$120^\circ$ , 60 kV
Counting time	20 min (typical case for $^{14}\text{C}$ )
Typical $^{14}\text{C}/^{12}\text{C}$ background including reduction	$3 \times 10^{-15}$

## RESULTS AND DISCUSSION

### Acceptance Test and Optimization of the Measurement Conditions

#### Carbon

An acceptance test for  $^{14}\text{C}$  was done using 4 samples of NIST OXII and 1 standard sample of IAEA C1 (Le Clercq et al. 1998) as a blank sample. All samples were prepared at KIGAM. A description of the sample preparation process is published in these proceedings (Hong et al. 2010). A typical beam current of  $^{12}\text{C}$  at a Cs temperature of 80 °C was 10  $\mu\text{A}$ . It was possible to obtain 100  $\mu\text{A}$  at a Cs temperature of 100 °C. However, it is not necessary yet to reduce the measurement time because the accelerator has a larger throughput than that of sample preparation process. It is known that  $\text{C}^{2+}$  ions have the highest transmission rate at the terminal voltage of 1 MV. Furthermore,  $^{13}\text{CH}^{2+}$  molecules are not stable (Jacob et al. 2000). The maximum transmission between pre- and post-accelerator Faraday cups was 50% for  $^{12}\text{C}^{2+}$  with a stripper gas pressure of  $1.5 \times 10^{-2}$  Torr. This is very close to an earlier result obtained by Suter et al. (2000). However, when 2+ ions are used for the 1MV  $^{14}\text{C}$  analysis, interference by the  $^7\text{Li}_2$  molecular ions is severe in some cases. Hence, this forced the stripper pressure to be increased to  $2.1 \times 10^{-2}$  Torr to destroy the Li molecules. With this stripper gas condition, the beam transmission slightly decreased.

The ESA voltage was very carefully adjusted to avoid any Li contribution to the  $^{14}\text{C}$  counts. Figure 2 shows the relationship between the ESA voltage and the carbon and lithium count rates. The ESA ranges for the maximum count rate of these ions were slightly different. The  $^{14}\text{C}/\text{Li}$  ratio increased rapidly at an ESA voltage of ~55.2 kV. Additionally, the outer side of a slit in the front of the ESA was closed until the Li peak disappeared. The gas pressure in the ionization detector was also optimized to improve the mass resolution. With all of these efforts, Li molecular ions could be successfully suppressed without a loss of  $^{14}\text{C}$  ions as shown in Figure 3a, and a reasonable background could be obtained.

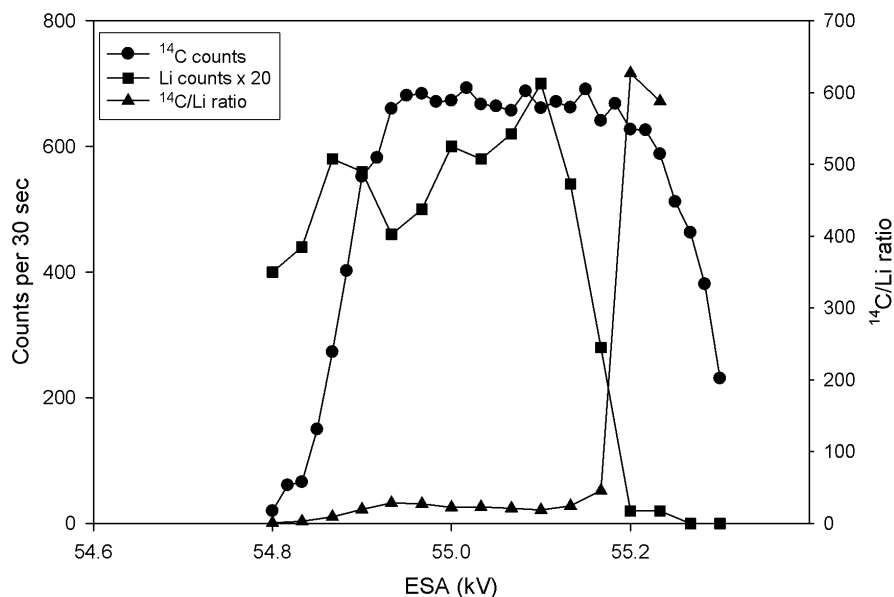


Figure 2 Dependencies of the  $^{14}\text{C}$  and Li counts on the ESA voltage after slit adjustment in front of the ESA. The Li curve was plotted with a scale multiplied by 20. Setting the ESA voltage higher than 55.2 kV effectively suppresses the Li counts.

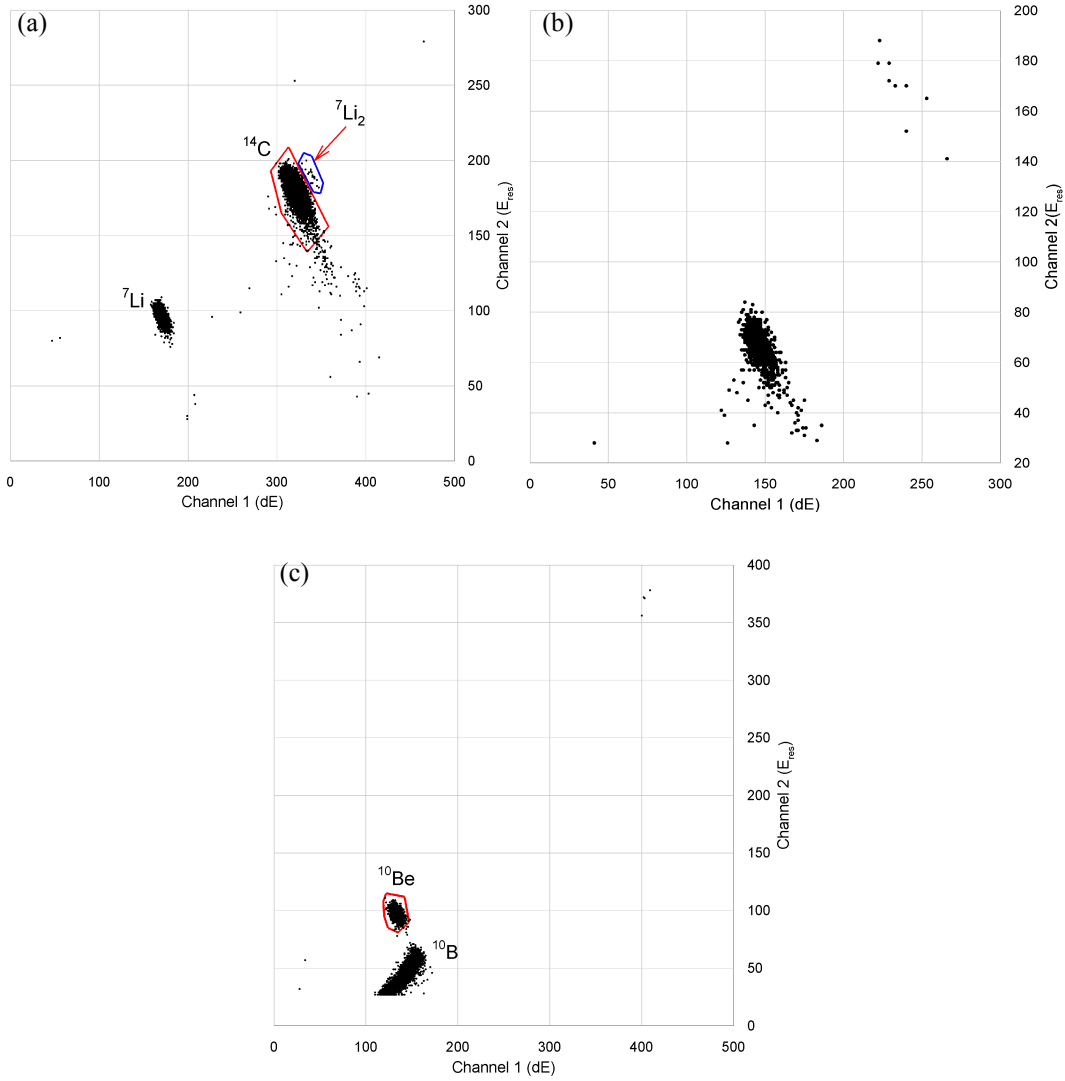


Figure 3 Typical spectra showing counts as a function of initial energy loss (dE) and residual energy ( $E_{res}$ ) from the 1MV AMS of (a)  $^{14}\text{C}$ , (b)  $^{26}\text{Al}$ , and (c)  $^{10}\text{Be}$  after optimization of the counting conditions.

As shown in Table 2, the  $^{14}\text{C}/^{12}\text{C}$  ratio of oxalic acid II was measured to be  $1.34 \times 10^{-12}$ , which is a smaller value than  $1.40 \times 10^{-12}$  as obtained by Chamizo et al. (2008) and  $1.56 \times 10^{-12}$  obtained by Liu et al. (2007). However, this does not affect age dating because it is not an absolute measurement. Each sample was counted 3 times for 24 min in a process that was divided into 48 blocks of 30-s counting. The total number of  $^{14}\text{C}$  counts for the 72 min was approximately 120,000 counts; thus, the statistical error was 2.9%. The standard deviation between 4 samples was 2.6%. The mean of  $^{13}\text{C}/^{12}\text{C}$  for 4 samples was measured to be  $1.04 \times 10^{-2}$ , with a standard deviation of 0.93%.

At the beginning of the test, the background was fairly high as a consequence of Li molecule interference. Eventually, the background value was reduced to  $2.34 \times 10^{-15}$  in routine operations by optimizing the beam optics and careful selection of an Fe catalyst with a low Li content. This back-

Table 2 Results of acceptance tests for  $^{14}\text{C}$ ,  $^{10}\text{Be}$ , and  $^{26}\text{Al}$ .

Element	Sample	Mean <sup>a</sup>	SD <sup>b</sup> (%)	Efficiency <sup>c</sup> (%)	$^{13}\text{C}/^{12}\text{C}$	SD of $^{13}\text{C}$ (%)	Background sample	Background
C	OXII	1.34E-12	2.59	95.7	1.04E-2	0.93	IAEA C1	2.34E-15
Be	ICN (5-1) (2.71E-11)	1.75E-12	8.98	6.46			Reagent for ICP-MS	1.71E-14
Al	ICN (4-1) (7.44E-11)	6.17E-11	9.66	82.9			Reagent for ICP-MS	2.72E-14

<sup>a</sup>Mean of the measured values of 4 samples of carbon and 3 samples for other isotopes.

<sup>b</sup>Relative standard deviations of 4 samples of carbon and 3 samples of other isotopes.

<sup>c</sup>Efficiency was calculated using the measured isotope ratio and consensus ratio of the samples. This value therefore includes the detector intrinsic efficiency, fractionation of the sample preparation, beam transportation, and fractionation of the complete AMS system.

ground value includes the machine background and reduction background. Sputtering for more than 5 min to clean the surface is necessary to obtain a low blank value.

### Aluminum

An ICN standard sample set having various activities was supplied by the University of Arizona, and an ICN 4-1 sample in the set was used for the Al acceptance test. The consensus value of the sample supplied by the manufacturer was  $7.44 \times 10^{-11}$ . For a background sample, a reagent for ICP-MS was used. Though a typical target holder material is aluminum, target holders made of copper were used for the  $^{26}\text{Al}$  measurement. A target was composed of a mixture of ICN 4-1  $\text{Al}_2\text{O}_3$  powder and Ag powder of identical weights. One area of difficulty in Al measurements is the low beam current. A typical beam current for  $^{27}\text{Al}^-$  from  $\text{Al}_2\text{O}_3$  is only 200 nA at a Cs temperature of 85 °C, which gives a count rate of approximately 10 cps at the detector. This is in good agreement with the results of Fifield et al. (2007). The  $\text{AlO}^-$  beam current was lower than the  $\text{Al}^-$  beam. However, a typical counting time of 450 s was enough to obtain a statistical error of 15%. If higher precision is necessary, the counting time should be extended to 30 min or longer.

The terminal voltage for Al measurement was limited to 700 kV since the analyzing magnet field could not sufficiently bend  $\text{Al}^{1+}$  ions with higher energy. At that terminal voltage, the magnet current was ~293 A, while the maximum current of the magnet power supply was 300 A. However, the cooling capacity for the magnet and its power supply was sufficient and the magnet was therefore stable during long-term operation at its maximum current. Beam transmission of the  $^{27}\text{Al}^{1+}$  ion at a terminal voltage of 700 kV and a stripper pressure of  $1.7 \times 10^{-2}$  Torr was found to be 30.1% between pre- and post-accelerator Faraday cups. The stripper pressure was selected only on the basis of stripping efficiency because there was no interference of the molecular beam for the  $^{26}\text{Al}$  measurement, as shown in Figure 3b. A movable Faraday cup at the post-accelerator site was used to measure  $^{27}\text{Al}$ .

Three ICN 4-1 samples were measured for the acceptance test. The mean value of the  $^{26}\text{Al}/^{27}\text{Al}$  ratio was  $6.17 \times 10^{-11}$ . The counting efficiency was smaller than that of carbon, as shown in Table 2. The standard deviation was 9.66%, and the blank value was  $2.72 \times 10^{-14}$ . The blank value of  $^{26}\text{Al}$  was higher than the  $^{10}\text{Be}$  blank value because the beam current was low to the point that a few counts led to a high  $^{26}\text{Al}/^{27}\text{Al}$  ratio. These are preliminary results, and further study of  $^{26}\text{Al}$  measurements including an improvement of the beam current is needed.

### Beryllium

ICN standards samples supplied from the University of Arizona were used for the acceptance test for beryllium. For the blank samples, the same samples used in the Al measurements were used.

$\text{BeO}^-$  ions were extracted from the BeO material, as this material can create higher beam current compared to other materials such as  $\text{BeF}_2$  or  $\text{BaBeF}_4$  (Müller et al. 2008). For the Be measurements, a sufficient beam current of  $\text{BeO}^-$  could not be obtained with a target mixed with Ag. After several trials with different materials, it was found that Nb powder created a good beam current in our ion source. A typical  $\text{BeO}^-$  beam was 2–3  $\mu\text{A}$  with a Nb and BeO mixture with a mass ratio of 1:1. This is higher than the result with the Ag mixed sample as well as the result of Grajcar et al. (2007).

The terminal voltage for the Be measurement was set to 1000 kV, which is the maximum voltage of this AMS. A stripper range from  $1.2 \times 10^{-2}$  to  $2.1 \times 10^{-2}$  Torr was tested, and the transmission of  $\text{BeO}^-$  at the pre-accelerator Faraday cup and  $\text{Be}^{1+}$  at the post-accelerator Faraday cup remained at 47%. To observe the transmission change, it may be necessary for a wider range to be tested.

The difficulty of the  $^{10}\text{Be}$  measurement is associated with the interference of isobar  $^{10}\text{B}$ , which has to be suppressed at approximately 7–10 orders of magnitude to reach a background level of  $10^{-14}$  (Müller et al. 2008). Several techniques have been developed to suppress  $^{10}\text{B}$  ions in  $^{10}\text{Be}$  ions (Kutschera et al. 1980; Zhao et al. 2004). The passive absorber foil method was adopted for the present AMS system to suppress  $^{10}\text{B}$ . A  $\text{Si}_3\text{N}_4$  foil with a thickness of 150 nm was used in the front of the ESA, and very precise ESA tuning was carried out to separate the  $^{10}\text{Be}$  and  $^{10}\text{B}$  peaks very clearly, as shown in Figure 3c.

Four ICN 5-1 samples, for which the consensus value of the  $^{10}\text{Be}/^9\text{Be}$  ratio was  $2.71 \times 10^{-11}$ , were measured for the acceptance test. The total counting time was 1800 s for each sample. Every sample was measured 3 times and every measurement was divided into 20 blocks of 30 s. The mean ratio was measured to be  $1.75 \times 10^{-12}$  with a standard deviation of 8.98%. The counting efficiency of  $^{10}\text{Be}$  was extremely low compared to  $^{14}\text{C}$  and  $^{26}\text{Al}$  since the charge states of many  $^{10}\text{Be}$  ions change and the beam becomes spread out because of the straggling effect while passing through the absorber foil.

A total of 9  $^{10}\text{Be}$  counts were measured for a blank sample for 5451 s, and the collected charge of  $^9\text{Be}$  was  $1.37 \times 10^{-5}$  C. Thus, the measured value of the  $^{10}\text{Be}/^9\text{Be}$  ratio was  $1.05 \times 10^{-15}$ . Using this and the counting efficiency, the  $^{10}\text{Be}/^9\text{Be}$  ratio of a blank could be calculated as  $1.71 \times 10^{-14}$ . This is a fairly good result for a small AMS machine. It is expected that the blank value could be reduced to  $10^{-15}$  range if a blank sample having a lower  $^{10}\text{Be}$  content is used.

### **Accuracy of $^{14}\text{C}$ Measurement**

Before the installation of the AMS machine, the sample treatment system for age dating was set up and the known samples treated in our laboratory were measured, with the help of the University of Arizona AMS laboratory, to check the sample preparation procedure. After installation, interlaboratory comparison tests were carried out to confirm the accuracy of the  $^{14}\text{C}$  measurements. Two wood and 2 soil samples were provided, and each sample was divided into 4 parts. All of these samples were completely unknown. Three parts of each sample were sent to 3 AMS laboratories to cross-check the dating results. The remaining parts were measured at the KIGAM AMS laboratory. Wood samples were treated using the traditional AAA (acid-alkali-acid) method. A humic acid fraction extraction method (Kristiansen et al. 2003) was adopted to prepare the soil samples. Combustion of the samples was performed with an elemental analyzer and they were reduced to graphite using Fe catalyst and H gas at 630 °C for 140 min. The typical reduction yield was 92%. The procedures used in this work are our routine methods for wood and soil samples.

Measurements consisted of 3 runs of sequential counting for each sample lasting 10 min. Each counting procedure was divided into 20 blocks of 30 s. This is also a routine counting condition. The

dating results from 4 laboratories agreed with each other in a wide range of ages from 3000 to 15,000 yr BP as summarized in Table 3. For the soil samples, it is considered that an older age may be reported from Oxford for Isa080052, as Oxford has a different preparation method from the other 3 laboratories. For OWd080041, which was the oldest sample in this test, the discrepancy between the laboratories was significant. Further investigation of this sample is needed.

Table 3 Results of interlaboratory comparison test between 4 AMS laboratories.

Sample	Type	KIGAM (BP)	Woods Hole (BP)	Kiel Univ. (BP)	Oxford Univ. (BP)
TSa070088	Soil	16,970 $\pm$ 200	16,600 $\pm$ 85	16,460 $\pm$ 70	16,970 $\pm$ 170 <sup>a</sup>
Isa080052	Soil	2940 $\pm$ 40	3050 $\pm$ 30	2875 $\pm$ 35	4665 $\pm$ 60 <sup>a</sup>
OWd080018	Wood	4330 $\pm$ 40	4510 $\pm$ 30	4360 $\pm$ 30	4346 $\pm$ 28
OWd080041	Wood	39,620 $\pm$ 530	44,600 $\pm$ 740	42,680 $\pm$ 660	34,440 $\pm$ 240

<sup>a</sup>Oxford measured the humin fraction of the soil, while KIGAM measured the humic fraction of the soil

The age dating accuracy was also checked by comparing the ages with dendrochronology results. <sup>14</sup>C ages of 8 sequential tree-ring samples with 10-yr steps were measured and compared with the tree-ring ages. Carbon ages from JS504B-2 to JS504B-9 were 910  $\pm$  50, 780  $\pm$  40, 820  $\pm$  40, 720  $\pm$  50, 680  $\pm$  40, 690  $\pm$  50, 670  $\pm$  40, and 640  $\pm$  40 BP. Since these ages are distributed in a large wiggle around 600–700 BP, it is difficult to match the carbon ages to calendar ages with normal correction. The results of the age set corrected with the wiggle-matching D\_Sequence function of OxCal v 3.10 (Bronk Ramsey 1995, 2001) are shown in Figure 4; this is in good agreement with the tree-ring ages within 30 yr.

Atmospheric data from Reimer et al (2004); OxCal v 3.10 Bronk Ramsey (2005); cub r:5 sd:12 prob usp[chron]

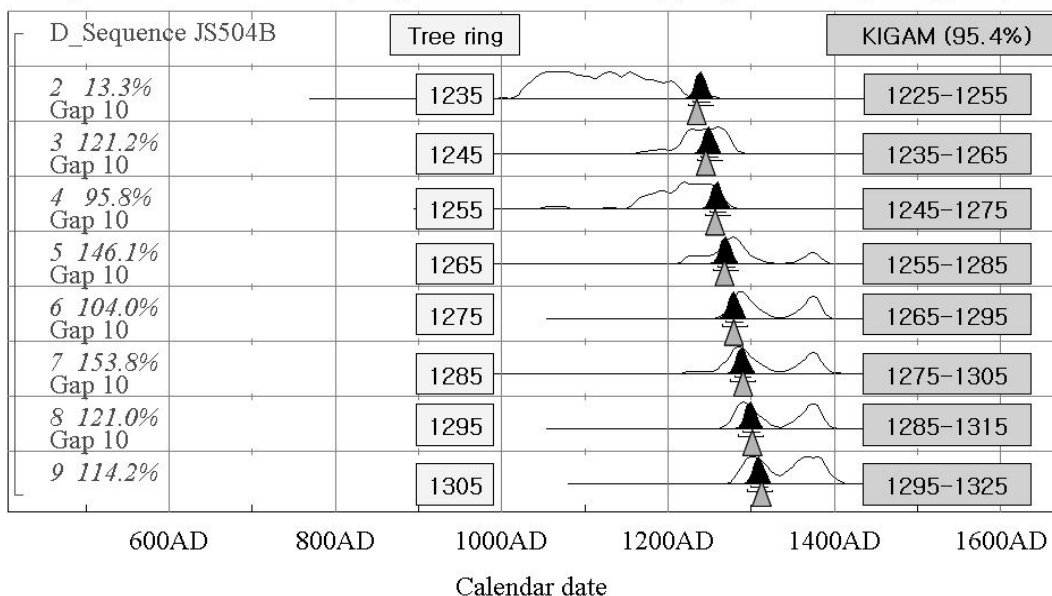


Figure 4 Comparison of tree-ring ages and carbon ages of sequential tree-ring samples. Carbon ages were obtained by wiggle-matching using OxCal 3.10 and the IntCal04 calibration curve data (Reimer et al. 2004).

## CONCLUSION

A 1MV AMS system for multi-element measurements at KIGAM was completely and successfully installed. The  $^{14}\text{C}$  and  $^{10}\text{Be}$  measurement conditions were tuned precisely, and preliminary measurements for  $^{26}\text{Al}$  isotope were carried out. The precision of a quantitative analysis of carbon was better than 3‰ and the background was found to be lower than  $3 \times 10^{-15}$  after suppressing the contribution of the Li molecules to the  $^{14}\text{C}$  counts. Li suppression was carried out by careful selection of Fe catalyst and precise tuning of the beam optics. It is expected that dating can be performed for samples up to 50,000 yr BP. The performance of the AMS system in an age dating was confirmed by inter-laboratory comparisons as well as by comparisons with tree-ring ages. We also contributed to the Fifth International Radiocarbon Intercomparison (VIRI), and are waiting for the results from the committee.

The precision of  $^{10}\text{Be}$  measurement was 8.98‰. However, in further study after the acceptance test for the  $^{10}\text{Be}$  measurement, the precision reached to a value better than 5‰. The  $^{10}\text{B}$  isobar was suppressed by a  $\text{Si}_3\text{N}_4$  foil with a homogeneous thickness of 150 nm. The background  $^{10}\text{Be}/^9\text{Be}$  ratio of  $1.71 \times 10^{-14}$  is sufficiently low to apply the system to environmental studies of sediment and rain water. However, it should be improved to at least  $5 \times 10^{-15}$  to study *in situ* cosmogenic nuclides in rocks. The Al measurement potential of the AMS machine was proved by the acceptance test. It is necessary to investigate and optimize the counting conditions.

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