

## DEVELOPMENT OF RADIOCARBON DATING IN CHINA OVER THE PAST 50 YEARS

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**ABSTRACT.** On the arrival of the 50th anniversary of *Radiocarbon*, we review important developments in radiocarbon dating in China during the past 50 years, especially concerning 3 aspects: sample standard and preparation, accelerator mass spectrometry (AMS) facilities, and <sup>14</sup>C applications. Specifically, these events are marked by the establishment of the Chinese sucrose charcoal standard in China; the development of small-sample dating in the Xi'an Laboratory of Loess and Quaternary Geology, Chinese Academy of Sciences (CAS); the progress of the AMS facilities in Beijing (China Institute of Atomic Energy and Beijing University); the innovation of the mini-cyclotron-based AMS at Shanghai Institute of Nuclear Research, CAS; the exploration of the Xia-Shang-Zhou chronology project in China; the establishment of the Xi'an multi-element AMS at the Xi'an-AMS Center; and the breakthrough in tracing the geomagnetic intensities and precipitation from <sup>10</sup>Be in Chinese loess at the Institute of Earth Environment, CAS.

### INTRODUCTION

A landmark in the history of radiocarbon dating in China was established in late 1965 with the dating of the first 4 samples using the <sup>14</sup>C method (Qiu and Cai 1962, 1972; Rudolph 1973). Since then, <sup>14</sup>C dating in China has developed rapidly and has acquired many important results. Some long-lasting unresolved academic difficulties in archaeology, such as Xia-Shang-Zhou chronology, have achieved their expected outcome in the first phase of dating. The <sup>14</sup>C results in geology have provided time bases for various geological fields over the past 50 kyr, including geological event comparison, sea-level and seashore changes, land-form growth, and paleoclimate change. For example, the systematic study on abrupt climate change of the East Asia Monsoon and its instability over the past 13 kyr has presented several new points of view (Zhou et al. 1992, 1996, 1997, 1999a,b, 2001, 2002, 2005). <sup>14</sup>C dating has thus been increasingly applied by Chinese historians and geologists.

In the 1970s, about 40 institutions engaged in work related to <sup>14</sup>C dating, with nearly 20 of these institutions establishing their own <sup>14</sup>C laboratories. As the number of <sup>14</sup>C laboratories increased, and to facilitate intercomparison and exchange among the labs, Chinese scientists decided in the winter of 1975 to establish the nationally agreed carbon standard—the Chinese sucrose charcoal standard (Qiu et al. 1982).

Before the 1990s, when China could not afford to have an AMS facility, most <sup>14</sup>C dating work in China was carried out either domestically by conventional liquid scintillation counting (LSC) or by sending samples to overseas laboratories for accelerator mass spectrometry (AMS) measurement. Therefore, the development of the small-sample (100–250 mg) device at Xi'an in the early 1990s overcame the difficult problem of small-sample dating in archaeology and geology (Zhou et al. 1994, 1995).

Owing to the scarcity of <sup>14</sup>C AMS facilities in China and the high cost of a conventional tandem-based AMS, the Shanghai mini-cyclotron-based AMS (SMCAMS) project was initiated in 1985. After 13 yr of persistent effort, the first <sup>14</sup>C result was obtained in 1993 and the first real sample was

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measured in 1998, which showed the “revival of the cyclotron AMS” as commented by G Raisbeck in his closing speech at the 6th International Accelerator Mass Spectrometry Conference (AMS-6).

The first AMS facility in China was the 13MV tandem AMS at the China Institute of Atomic Energy (Beijing). This is a part-time (only ~10%) AMS facility and its terminal voltage is too high to be used for  $^{14}\text{C}$  dating. The second AMS facility in Beijing University is too busy to meet the domestic  $^{14}\text{C}$ -dating measurement demands. The third AMS facility, in Shanghai, is an experimental device more suitable for biomedical applications. Furthermore, all 3 AMS facilities are located in eastern China, and no routine  $^{10}\text{Be}$  measurement was available. It was thus necessary to build a new AMS facility in western China. A new 3MV Tandatron-based multi-element ( $^{10}\text{Be}$ ,  $^{14}\text{C}$ ,  $^{26}\text{Al}$ , and  $^{129}\text{I}$ ) AMS facility was then imported in 2006 at the Xi'an-AMS Center. In the meantime, a 0.6MV NEC-made compact AMS was imported at Beijing University and a project to import a 6MV AMS has been planned at the China Institute of Atomic Energy.

While the  $^{14}\text{C}$  dating methods were being developed in China, trace research was also being carried out using  $^{10}\text{Be}$  in Chinese loess. Early on, the published research was exclusively concerned with paleoclimate. Within the last few years, however, authors have been engaging in tracing the geomagnetic intensities from  $^{10}\text{Be}$  in Chinese loess. Some new ideas and methods were put forward, and the first geomagnetic intensity curve was derived from loess  $^{10}\text{Be}$  for the past 80 kyr, which is in good agreement with the SINT-200 and NAPIS 75 curves.

On the arrival of the 50th anniversary of *Radiocarbon*, we, in this paper, discuss several important events in the development of  $^{14}\text{C}$  dating in China during the past 50 yr, concerning 3 aspects: sample standard and preparation, AMS facilities and  $^{14}\text{C}$  applications, as well as loess  $^{10}\text{Be}$  trace research.

#### ESTABLISHMENT OF THE CHINESE SUCROSE CHARCOAL STANDARD IN CHINA

As the number of  $^{14}\text{C}$  laboratories increased and the  $^{14}\text{C}$  dating application fields extended, and to improve the accuracy of  $^{14}\text{C}$  dating and the convenience of exchange and intercomparison among Chinese labs, scientists decided to establish the nationally agreed carbon standard, for which the Archaeology Institute of the Chinese Academy of the Social Sciences, the History Department of Beijing University, and the Guiyang Institute of the Earth Chemistry, Chinese Academy of Sciences were responsible. In September 1981, the “Chinese sucrose charcoal standard” was accepted at the first Chinese National  $^{14}\text{C}$  Conference (Qiu et al. 1982, 1983).

In order to determine the ideal material for the standard, charcoal, nut carbon, filter paper, and cellulose powder were tested. Finally, pure carbonized sucrose (sugar carbon) was selected as the standard matter. About 1000 kg of sucrose refined from raw sugar beets harvested in 1977 were bought, the analytic reagent sucrose was extracted, and the pure sugar was carbonized (Qiu et al. 1982, 1983). No volatile matter remained in the pure sugar. The weight ratio of their burned residue (dust powder) was <1‰, so the influence of radioactivity from the dust powder was lowered to a negligible level (Qiu et al. 1982, 1983). The adsorptive surface of the pure sugar was measured for the specific surface, resulting in ~60 m<sup>2</sup>/g, a little bigger than for the conventional matter. Such an adsorption can be removed by vacuuming under heating before burning; thus, the Chinese sugar carbon standard will not be affected by the radioactivity from the adsorbed gases (Qiu et al. 1982, 1983).

In order to precisely calibrate the  $^{14}\text{C}$ -specific radioactivity of the Chinese sucrose charcoal standard, wood (1847–1854), NBS oxalic acid, and ANU sucrose were chosen as standards. All samples were prepared in the same laboratory and adopted identical chemistry procedures in order to decrease the fractionation. The  $^{13}\text{C}$  was measured for each sample, which was used to correct the fractionation for the measured  $^{14}\text{C}$ -specific radioactivity (Qiu et al. 1982, 1983). The measured  $^{13}\text{C}$

of the sugar carbon is  $19.32 \pm 0.56\%$  relative to the international PDB standard, and the measured <sup>14</sup>C-specific radioactivity of the sugar carbon is  $\bar{R} = 1.362 \pm 0.002$  relative to the international modern carbon standard (Qiu et al. 1982, 1983). Table 1 lists the results of the Chinese sucrose charcoal standard measured in the 3 responsible laboratories (Qiu et al. 1982).

Table 1 Results of Chinese sucrose charcoal standard measurements in the 3 responsible laboratories (the Archaeology Institute, Chinese Academy of Social Sciences (CASS); the History Department of Beijing University; and the Guiyang Institute of Earth Chemistry, Chinese Academy of Sciences (CAS). This table was originally given in Qiu et al. (1982).

Measuring institute	Measured Chinese sucrose charcoal standard (average)	Measured international modern carbon standard (average)	Sugar carbon/modern carbon
Archaeology Institute of the CASS			
(1st measurement)	$12.227 \pm 0.030$	$8.985 \pm 0.017$	$1.3608 \pm 0.0042$
(2nd measurement)	$12.562 \pm 0.017$	$9.224 \pm 0.014$	$1.3619 \pm 0.0028$
(3rd measurement)	$9.115 \pm 0.022$		$1.3590 \pm 0.005$
History Department of Beijing University			
(1st measurement)	$12.363 \pm 0.021$	$9.077 \pm 0.010$	$1.3620 \pm 0.0028$
(2nd measurement)	$12.709 \pm 0.028$	$9.344 \pm 0.018$	$1.3600 \pm 0.004$
Guiyang Institute of Earth Chemistry, CAS		$10.027 \pm 0.026$	$1.3760 \pm 0.0076$

### SMALL-SAMPLE DATING IN XI'AN LABORATORY OF LOESS AND QUATERNARY GEOLOGY

#### The Small-Sample <sup>14</sup>C Dating Device

The establishment of a relatively accurate <sup>14</sup>C chronology for the well-defined loess-paleosol sequences in northern China is an important component of any investigation of global climate change over the past 30,000 yr. The carbon content of these sediments ranges between 1 and 2%. Intensive agriculture introduced a relatively high concentration of young water-soluble organic compounds, which are then adsorbed onto the clay component of the paleosols. Thus, it is advisable to subject the organic component of the paleosols to extensive pretreatment and chemical fractionation to obtain reliable <sup>14</sup>C ages (Head et al. 1989; Zhou et al. 1990, 1992). The final components for dating are usually too small for conventional liquid scintillation counting (LSC) (Polach et al. 1988). Because of the scarcity of <sup>14</sup>C AMS facilities in the 1990s in China, and the expense of obtaining AMS dates from overseas, the concept of upgrading an existing radiometric <sup>14</sup>C dating facility to handle carbon sample sizes between 25 and 250 mg was very attractive (Polach et al. 1988).

In the early 1990s, the Xi'an Laboratory of Loess and Quaternary Geology, CAS, developed a small-sample <sup>14</sup>C dating facility (Zhou et al. 1994, 1995) consisting of a Wallac 1220 Quantulus™ liquid scintillation spectrometer. We decided that using a full-scale benzene synthesis apparatus for samples of <250 mg carbon decreased the efficiency of preparation and yield of benzene. Thus, we designed a miniature system based on the procedures developed at the ANU Radiocarbon Dating Research Unit (Gupta and Polach 1985). The miniature benzene synthesis apparatus also enabled us to keep possible memory effects to a minimum. This line can produce ~0.3-mL benzene samples, which are then measured for <sup>14</sup>C activity using 0.3-mL Teflon® vials developed by Wallac Oy. The counting performance of the Quantulus spectrometer using 0.3-mL vials was evaluated, and a potential age limit of ~45,000 BP was obtained for samples containing up to 250 mg carbon. This method enabled us to determine the <sup>14</sup>C activity of samples containing 250 mg carbon with acceptable precision, even though the counting is slow. This dating facility fills the gap between large-sample (2.4–6 g carbon) and microsample (<1 mg carbon) handling to form a <sup>14</sup>C dating method sequence.

To test the accuracy of the technique, a series of previously dated samples was collected from the ANU Laboratory and the Xi'an Laboratory archives. The results of the cross-check indicated that the Xi'an Laboratory small-sample facility using liquid scintillation spectrometry can produce  $^{14}\text{C}$  ages with precision comparable to other counting techniques. Mike Barbetti, Director of the NWG Macintosh Centre for Quaternary Dating, Sydney University, commented when reviewing this work that "By establishing the technique at Xi'an Laboratory and demonstrating that it provides good quality results, the authors have made an important and worthwhile contribution to technology in radiocarbon dating."

### Results for the Fourth International Radiocarbon Intercomparison (FIRI)

In 2001, the Xi'an Laboratory of Loess and Quaternary Geology took part in the Fourth International Radiocarbon Intercomparison (FIRI) study as the one of the only participants from China. To guarantee the accuracy of the results, different kinds of samples were pretreated with corresponding physical and chemical procedures (Lu and Zhou 2003). After having been cleaned and crushed, the wood samples were put into the Soxhlet unit for leaching with a mixture of chloroform and ethanol (2:1), ethanol, and distilled water. After adding the  $\text{NaClO}_2$  to oxidize the samples under the acid conditions and using 10% NaOH to remove the alkaline components, the wood cellulose was extracted for dating. The residual of the modern malt plasm was crushed. We prepared twin samples for each sample: a graphite sample for AMS measurement and small samples (240 mg) for LSC. The graphite samples were measured at VERA, in Vienna (Priller et al. 2000), with a precision of <1% for most of the samples. The small samples were measured at this laboratory using a Quantulus 1220. During measurements (30 min each), 0.3-mL Teflon vials were used and ANU sucrose and calcite were taken as the standard and background sample, respectively. For comparing the measured results with the consensus values (Table 2) (Lu and Zhou 2003), the  $t$  statistic was well below 3.481 at the 95% confidence level, indicating that our results were extremely close.

Table 2 Comparison between results from the Xi'an Laboratory of Loess and Quaternary Geology and consensus values during the Fourth International Radiocarbon Intercomparison (FIRI).

Sample #	Consensus values	$t$ value		Carbon quantity (mg)	$t$ value	
		AMS	LSC			
A	45,868 yr	55,283 ± 9385 yr	1.01	>40,000 yr	0.2438	—
B	46,504 yr	54,854 ± 8040 yr	1.08	>40,000 yr	0.2441	—
C	18,132 yr	18,065 ± 100 yr	0.45	18,610 ± 360 yr	0.2131	1.76
D	4510 yr	4472 ± 45 yr	0.71	4510 ± 110 yr	0.2429	0.00
E	11,738 yr	11,761 ± 80 yr	0.08	11,660 ± 220 yr	0.2437	0.13
F	4482 yr	4500 ± 45 yr	0.16	4440 ± 140 yr	0.2454	0.09
G	110.55 pMC	111.46 ± 0.51 pMC	3.18	110.27 ± 1.45 pMC	0.2451	0.04
H	2229 yr	2174 ± 50 yr	1.21	2340 ± 130 yr	0.2435	0.73
I	4469 yr	4436 ± 40 yr	0.68	4580 ± 160 yr	0.2439	0.48
J	110.56 pMC	111.25 ± 0.49 pMC	1.98	109.83 ± 1.45 pMC	0.2429	0.25

### PROGRESS OF AMS FACILITIES IN BEIJING

#### China Institute of Atomic Energy, Beijing

The AMS machine at the China Institute of Atomic Energy is a HI-13 tandem-based accelerator, with which the mid-heavy nuclides (including  $^{10}\text{Be}$ ,  $^{26}\text{Al}$ ,  $^{36}\text{Cl}$ ,  $^{64}\text{Cu}$ ,  $^{79}\text{Se}$ ,  $^{126}\text{Sn}$  and  $^{129}\text{I}$ ) have been measured (Jiang et al. 1994a,b, 1997), and applications have concentrated on the fields of geo-



sciences, biomedical sciences, nuclear physics, and astrophysics (Jiang et al. 2000a,b). The typical parameters of the CIAE AMS used for measurements of <sup>10</sup>Be, <sup>26</sup>Al, <sup>36</sup>Cl, <sup>41</sup>Ca, <sup>79</sup>Se, and <sup>129</sup>I are listed in Table 3 (Chen JE et al. 2007). Different detecting techniques have been developed along with an ΔE-E ionization chamber for the detection of rare nuclides and the suppression of their isotope and isobar interferences, as indicated in Table 3. Among other things, an absorber of 15.3 mg/cm<sup>2</sup> Ni is added in front of the ΔE-E ionization chamber for <sup>10</sup>Be measurement; the TOF technique is adopted for isotope identification for the measurement of <sup>129</sup>I; and a projectile X-ray AMS (PX-AMS) system has been set up for suppression of isobar interference in <sup>79</sup>Se and <sup>64</sup>Cu measurements (He et al. 2000). The suppression factor of Br K counts is ~250 m, corresponding to a <sup>79</sup>Se/Se ratio of 3.6 × 10<sup>-9</sup>, which improved the sensitivity by more than 2 orders of magnitude compared with a single Au-Si detector. The overall detection efficiency was (7.0 ± 0.4) × 10<sup>4</sup> K counts per <sup>79</sup>Se ion (Guo et al. 2000a,b). Measurement techniques such as a gas-filled magnet and a gas-filled time-of-flight detector have also been studied (Jiang et al. 2000b). Recently, a project has been planned at CIAE to import a 6MV AMS for analyzing even heavier nuclides.

Table 3 Typical parameters of the CIAE AMS used for measurements of <sup>10</sup>Be, <sup>26</sup>Al, <sup>36</sup>Cl, <sup>41</sup>Ca, <sup>79</sup>Se, and <sup>129</sup>I nuclides (Chen JE et al. 2007).

Radioisotopes	Negative ions	Terminal voltage (MV)	Detection method	Sensitivity
<sup>10</sup> Be	BeO	8.4	absorber + ionization chamber	11 × 10 <sup>-14</sup>
<sup>26</sup> Al	Al, AlO	7.6	ionization chamber	11 × 10 <sup>-14 to -15</sup>
<sup>36</sup> Cl	Cl	8.0	ionization chamber	2 × 10 <sup>-15</sup>
<sup>41</sup> Ca	CaF <sub>3</sub> , CaH <sub>2</sub>	8.2	ionization chamber	3 × 10 <sup>-14</sup>
<sup>79</sup> Se	Se	8.2	projectile X-ray	2 × 10 <sup>-11</sup>
<sup>129</sup> I	I	8.0	TOF	11 × 10 <sup>-13</sup>

### Beijing University

The AMS facility at Beijing University (PKU-AMS) is an EN tandem-based accelerator mainly for <sup>14</sup>C, <sup>10</sup>Be, and <sup>26</sup>Al measurements (Chen JE et al. 1994). It was the only routine <sup>14</sup>C measurement facility for quite a long time in China. Since 1996, it has been upgraded to meet the requirements of the Xia-Shang-Zhou Chronology Project (Guo et al. 2000a,b; Liu et al. 2000; Chen JE et al. 2007). A new ion source, MC-SNICS, from NEC was installed, the injection system was reconstructed, and the alignment and vacuum of the beam line was improved on the PKUAMS, all of which led to a precision of 0.5% in the <sup>14</sup>C/<sup>13</sup>C ratio measurement, with a background corresponding to a <sup>14</sup>C age of 50 kyr, and an increase of the total transmission efficiency by 100% (Li et al. 2000; Li 2002). This facility played an important role in the completion of the Xia-Shang-Zhou Chronology Project.

In 2004, a NEC-manufactured compact AMS facility (Figure 1) was imported at Peking University (Liu KX et al. 2007; Chen JE et al. 2007). This AMS facility dedicated to <sup>14</sup>C measurements is a Pelletron-type accelerator with a maximum terminal voltage of 0.6MV and a NEC 40-sample multicathode SNICS ion source. The tested precision and reproducibility of the facility are better than 0.3% for modern samples and the machine background is 4 × 10<sup>-16</sup>, corresponding to a <sup>14</sup>C age of 65 kyr. Under a terminal voltage of 0.46 MV, an ion beam transmission of 43% has been reached. Since it is expected that this facility is capable of measuring 3000 samples per year, the original EN tandem-based AMS facility is now mainly used for measurements of <sup>10</sup>Be and heavier nuclides.

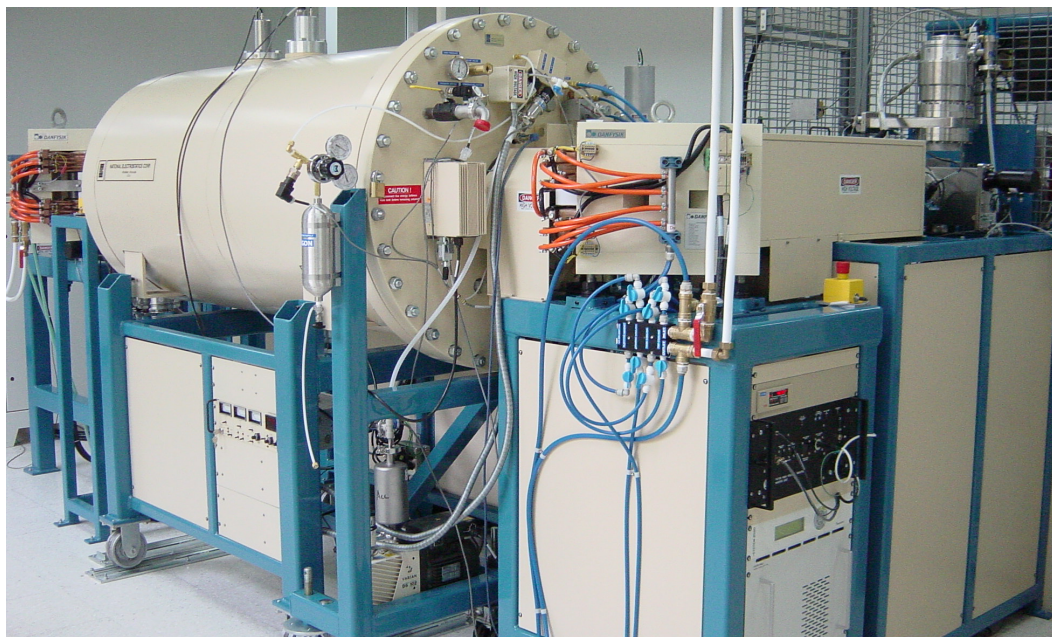


Figure 1 Photo of the compact AMS at Beijing University

## THE MINI-CYCLOTRON-BASED AMS AT THE SHANGHAI INSTITUTE OF NUCLEAR RESEARCH, CAS

### The SMCAMS Project

Since the early appearance of AMS, the AMS community has always been looking forward to small, even very small AMS facilities, because the existing tandem-based AMS machines were too sophisticated and expensive to be in widespread use.

In the 1950s, there was a fever to build small cyclotron mass spectrometers. However, all of them failed to get an abundance sensitivity greater than  $10^{-9}$  due to their trivial beam intensity (Clark 1984). After the re-introduction of the cyclotron AMS in 1977, a small cyclotron by the name “Cyclotrino” with a uniform magnet was initiated by the R A Muller group at Berkeley. Unfortunately, the Cyclotrino, after having been explored for 2 versions (Bertsch 1987; Friedman 1987; Welch 1987), failed to achieve acceptable  $^{14}\text{C}$  counting. It was said that the Cyclotrino had been moved to another laboratory of Berkeley for its third version, called CMS with a uniform permanent magnet (Young et al. 1993), on which no further report was seen.

Following the initiative of the Berkeley group, the mini-cyclotron with a non-uniform magnet was also submitted by the Maobai Chen group at Shanghai Institute of Nuclear Research, CAS, in late 1985 (Chen MB et al. 1987). After careful calculation and analysis, it was realized that the mini cyclotron as AMS could by no means be treated just as a conventional cyclotron, and a series of new ideas and unique technical measures were put forward (Chen MB et al. 1989a,b, 1990, 1995, 1996). The project SMCAMS (Shanghai Mini-Cyclotron-Based Accelerator Mass Spectrometer) was funded by the NSF of China and supported by the Chinese Academy of Sciences as a key project in early 1989. On 5 March 1993, the first  $^{14}\text{C}$  counting for a modern sample was successfully obtained at the new facility (Chen MB et al. 1994). It was not until 1998 that the first real  $^{14}\text{C}$  measurements were carried out (Chen MB et al. 2000), which comprised 7 archaeological samples some 2000–

3000 yr old and 2 geological samples that were nearly 30,000 yr old. An intercomparison of the measured results with results from the University of Arizona AMS and Beijing University AMS showed agreement within 1 to 2  $\sigma$  (Zhou et al. 2000). The <sup>14</sup>C counting rates for the standard samples made from Chinese sugar (1.36 times modern carbon) reached 20–25 cps on the mini-cyclotron AMS. This was really a breakthrough for small cyclotrons as AMS after have been explored for more than 1 decade (Figure 2). The technical measures that guaranteed the success of the SMCAMS are summarized in Table 4.

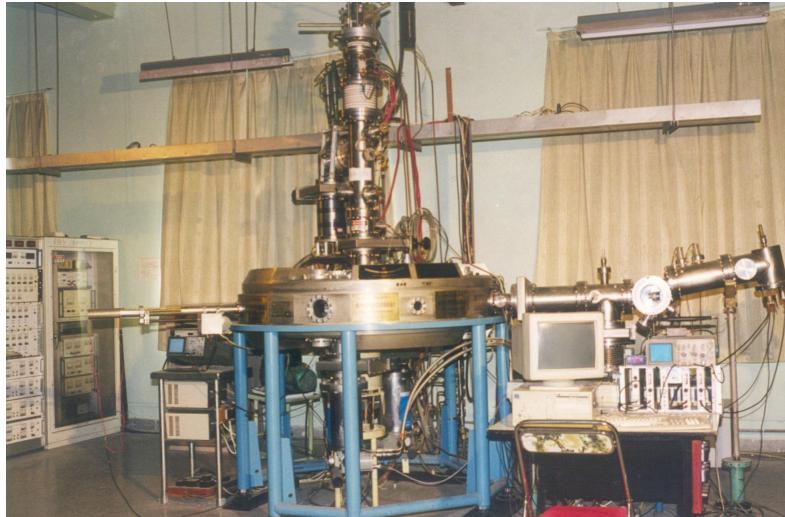


Figure 2 Photo of the Shanghai mini-cyclotron-based accelerator mass spectrometer (SMCAMS).

Table 4 Adopted technical measures that have guaranteed the success of the SMCAMS.

- (1) Adopting triangle wave acceleration voltage, rather than the usual sine wave voltage, for improving the particle acceptance of the mini-cyclotron under a high harmonic operation;
- (2) Constituting the differential dee electrodes with wedge shapes, rather than the usual dee dummy dee electrodes, for suitability of operation with high harmonic and for elimination of the phase convergence and divergence effect on particles;
- (3) Introducing the auxiliary electrodes and asymmetric electrodes with “T” and “I” shape varying in width for obtaining the particle turn spacing wide enough and the least particle phase convergence or divergence during injection and extraction;
- (4) Arranging a pair of spherical electrostatic injection deflectors, rather than the usual “mirror” or “spiral inflector,” for increasing the axial injection efficiency;
- (5) Designing a non-uniform magnet with high flutter and without trim coils, rather than a uniform magnet, for providing enough axial focusing and simplifying the axial injection;
- (6) Keeping the constant magnetic field while controlling the electrical parameters for sequential acceleration;
- (7) Choosing the larger magnet with low magnetic field, rather than the small magnet with high magnetic field, for enlarging the extraction turn spacing and setting the prohibitive zone to thoroughly suppress the background particles;
- (8) Combining the yoke of nickel-coated magnet with the acceleration vacuum chamber for simplicity;
- (9) Programming the ions to be analyzed around the equilibrium phase for fully making use of the powerful function of the triangular wave voltage;
- (10) Making up the dynode-microchannel plate detector, rather than the usual nuclear detector, for a low counting rate of low-energy radioisotope ions;
- (11) Also equipping the Cs sputter negative ion source with a multisample device for lowering the resolution demand and sequential measurement.

### Status of the SMCAMS

The SMCAMS facility keeps the advantages of both tandem- and cyclotron-based AMS. It possesses the capability of accelerating negative ions as in a tandem AMS. Furthermore, the negative ions can be directly extracted for measurement without the need of stripping. Hence, SMCAMS is the first negative heavy ion cyclotron in the world. If necessary, positive ions can also be analyzed. It also retains the function of resonance analysis of a cyclotron AMS. Moreover, the alternate acceleration can be carried out for dating applications without the need of changing the magnetic field; and primarily, it can be set up at any dating laboratory for utilitarian applications because of its low cost and operation charge, and with no expenditure of money for shielding or special buildings as a result of its small size, low energy (50 keV), low magnetic field (4 kG), and low power consumption (12 kW). Therefore, it was possible for such a mini-cyclotron AMS to achieve widespread use.

After the announcement of the successful SMCAMS results at the AMS-6 conference in 1993, there was a rush starting from 1994 to build compact tandem AMS with 0.5 to 1MV worldwide (Hughey et al. 1997; Mous et al. 1997; Suter et al. 1997). When SMCAMS made its first appearance, its advantage over the existing tandem-based AMS was its compact size and low cost for bioscience applications. However, after the success of the compact tandem-based AMS in the following years, the advantages of SMCAMS no longer exist. In addition, the main disadvantage of cyclotron-based AMS over tandem-based AMS is its non-flat top transmission due to the existence of the injection and extraction deflectors with narrow gaps in SMCAMS. Thus, it is impossible to make SMCAMS a commercial product, and the accelerator designers have had to use this existing SMCAMS by themselves for biomedical applications.

Since the completion of the SMCAMS in 1998, several hundred samples relating to archaeology, geology, and biomedicine have been measured on it with a measured precision around 1–3%. If the sample preparation system had been available in this laboratory, more samples would be measured on it. Some upgrades to the facility have been completed in the past years, including new pneumatic 24-cathode structures in the Cs sputter negative ion source, a new RS-485 bus-based control system, and a new sample pressing device (Liu YH et al. 2005). In order to extend the function of the SMCAMS, which is dedicated to  $^{14}\text{C}$  analysis at present, to  $^{26}\text{Al}$  analysis for biomedicine applications under the existing conditions, the possibility has been studied (Wang SL et al. 2007), of which the parameters including the turn number, harmonic number, and RF frequency were determined. The orbit programming and beam optics were calculated and the pretests to accelerate ions with mass numbers of 24, 25, 26, and 27 were carried out. Finally, the  $\text{Al}_2\text{O}_3$  sample with an isotope ratio of  $1.0 \times 10^{-10}$  was measured (Wang SL et al. 2008). The  $^{26}\text{Al}^-$  ions were detected and its frequency response curve shows the peak of  $^{26}\text{Al}^-$ , though very weak, is well separated from the most neighboring interfering molecular ions,  $^{25}\text{MgH}^-$ .

### THE XIA-SHANG-ZHOU CHRONOLOGY PROJECT

Chinese ancient civilization and its independent origin is one of 4 famous civilizations in human history, and has been lasting for nearly 5000 yr uninterrupted. However, the Chinese ancient chronology was established just after 841 BC and the Xia-Shang-Zhou dynasties, which have a special position in Chinese ancient history but yet have never been well confirmed by historians, which is really a pity for research of Chinese ancient history, and for world ancient history (Li et al. 2000; Li 2002).

In May 1996, the Xia-Shang-Zhou Chronology Project was commissioned in China as a national project. The Xia-Shang-Zhou Chronology Project is a multidisciplinary research program; its ultimate goal is to determine with an identifiable absolute chronology the location and time frame of the



Xia, Shang, and Western Zhou, the 3 earliest dynasties in Chinese history (Li et al. 2000; Li 2002). The Chronology Project has involved the collaboration of more than 200 specialists in the fields of archaeology, history, astronomy, and <sup>14</sup>C dating from about 30 institutes and universities. Prof Qiu SH, from the Archaeology Institute of the Chinese Academy of the Social Sciences, was one of the chief investigators of the project. Some 44 topics organized under 9 themes, each with explicit, realizable goals, were set up to implement the Chronology Project. After more than 4 yr of studies, a new chronological chart of the Three Dynasties was derived and disseminated in the autumn of 2000 (Li et al. 2000; Li 2002). The new chart offers the exact reigns of 9 late Shang kings and 10 Western Zhou kings, and a framework for the chronology of the early Shang and Xia. Some important data points of the chronology are the beginning of Xia and that of Shang are dated to ~2070 BC and ~1600 BC, respectively; the date King Pan Geng moved the Shang capital to Yin is estimated to be ~1300 BC; and the Zhou conquest of the Shang (i.e. the beginning of the Zhou Dynasty) is set at 1046 BC (Li et al. 2000). The project results were released in November 2000, completing the preset objective and paving the way for the further study of the origin of the Chinese civilization (Li et al. 2000; Li 2002).

The project correlated <sup>14</sup>C dating, archaeological dating methods, historical textual analysis, astronomy, and other interdisciplinary methods to achieve more greater temporal and geographic accuracy. Selecting of sequential (serial) samples and using LSC and AMS methods for <sup>14</sup>C dating, combined with wiggle matching of the tree rings, played an important role in the timescale calibration (Li et al. 2000; Li 2002). To meet the project's requirements for high-precision <sup>14</sup>C measurement, special research on the sample preparation and measurement methods were carried out and an upgrade of the existing PKUAMS facility was done from 1996 to 1998 (Guo et al. 2000a,b; Liu et al. 2000; Chen et al. 2007). Pretreatment procedures for the wood and bone samples were determined, which improved the sample preparation efficiency. Meanwhile, existing procedures to pretreat bone materials using filtration were implemented, and it was decided to use the gluten ingredients of bone materials for <sup>14</sup>C dating (Li et al. 2000; Li 2002). During the project, intercomparisons among different laboratories were repeatedly carried out using LSC and AMS methods for the various sequential samples from different areas over different periods, including the IAEA standard samples. The results agreed well within the error range (Li et al. 2000; Li 2002).

## **THE XI'AN MULTI-ELEMENT AMS AT THE XI'AN-AMS CENTER**

### **The Xi'an AMS Center**

With the support of the Chinese Ministry of Science and Technology, Chinese Academy of Sciences, and Chinese Ministry of Education, the Xi'an-AMS Center was formally established in July 2006, by the Institute of Earth Environment of the Chinese Academy of Sciences and Xi'an Jiaotong University. Its main equipment is a 3MV Tandatron-based multi-element AMS manufactured by HVEE (Figure 3). This new AMS Center is located on the Sci-Tech campus of Xi'an Jiaotong University, in southeastern Xi'an City. The facility housing, including an accelerator hall, utility rooms, and a 3-story sample preparation lab and office building, has a total floor space of 2500 m<sup>2</sup>. As an AMS lab in western China, we plan to measure <sup>10</sup>Be and <sup>14</sup>C in the near term, and expand the analytical work to include <sup>26</sup>Al, <sup>129</sup>I, and possibly others (such as <sup>41</sup>Ca) in the future. It is expected that the new facility will provide an important service to domestic and overseas researchers in earth environmental studies, archaeology, biomedical research, and possibly other fields (Zhou et al. 2007a,b).

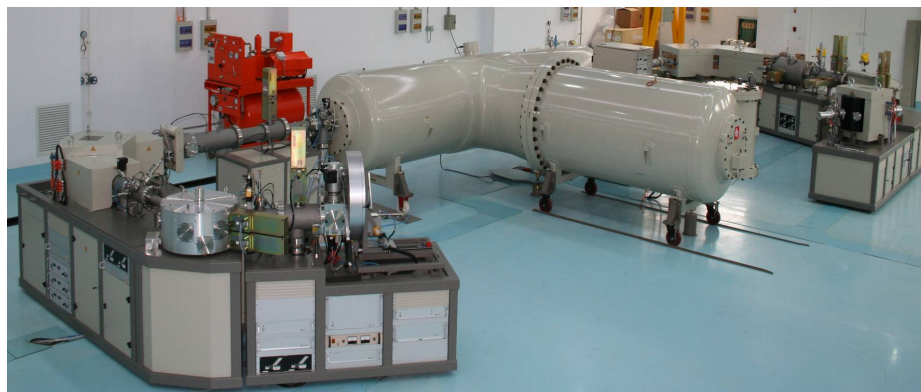


Figure 3 Photo of the Xi'an 3MV multi-element AMS (Xi'an-AMS Center)

### Status of the Xi'an-AMS Center

The AMS at the Xi'an-AMS Center is the fourth version of similar products using the bouncer technique (Gott dang and Mous 1997, 1999). Along with the 3 preceding systems at JAERI (Aramaki et al. 2000), Lecce (Calcagnile et al. 2004), and INFN (Mando, personal communication, 2004), this facility is a multi-element system with a single beam line dedicated to AMS applications. The major components of the Xi'an AMS are given in Table 1 of Zhou et al. (2006). The important features are listed in Table 5 (Mous et al. 1995, 1998; Gott dang and Mous 1997, 1999; Klein et al. 2004).

Table 5 Important technical features of the Xi'an multi-element AMS facility.

- (1) Ion source with grounded body makes for safe operation and easy maintenance;
- (2) Sequential injection system with fast cycling of 100 Hz minimizes the effect of changes in the system status, like ion source instabilities;
- (3) Beam blanking unit can precisely define the measuring time for different isotope ions;
- (4) Q-snout makes it possible to use low-energy injection of 35 keV;
- (5) Accelerator tubes with combined magnetic permanent and declined electrostatic field suppression of secondary electrons result in a very low X-ray radiation level outside the tank during operation;
- (6) Slit stabilization device automatically adjusts the terminal voltage to precisely position and measure the stable isotope ions in the Faraday cup;
- (7) An exceptionally uniform  $\text{Si}_3\text{N}_4$  foil realizes a near complete removal of  $^{10}\text{B}$ ;
- (8) The high dispersion of the  $65^\circ$  electrostatic analyzer separates the isobar  $^{10}\text{B}^{+3}$  ions far from the  $^{10}\text{Be}^{+3}$  ions ( $\sim 3.6$  cm) so as not to enter the detector;
- (9) The  $30^\circ$  magnet improves the precision of  $^{14}\text{C}$  and the minimum measurable sensitivity of other isotopes;
- (10) The high quality of the "flat top" transmission is shown by the good scan curves.

The Xi'an-AMS facility was installed in June 2005 and completed its final acceptance tests for 4 nuclides ( $^{10}\text{Be}$ ,  $^{14}\text{C}$ ,  $^{26}\text{Al}$ , and  $^{129}\text{I}$ ) in July 2006. The general results of 4 nuclides during the acceptance tests have been shown in Table 1 of previous papers (Zhou et al. 2007a,b). The acceptance tests have proven that a sequential injection-based AMS can reach the same high  $^{14}\text{C}$  precision as a simultaneous injection-based AMS (Liu L et al. 2007). The resulting background of  $3.65 \times 10^{-15}$  for the  $^{10}\text{Be}/^9\text{Be}$  ratio is comparable to that obtained by Raisbeck et al. (1987), which once again convinced the AMS community that high measurable  $^{10}\text{Be}$  sensitivity can also be reached on an AMS with a terminal voltage lower than 3 MV.



While the Xi'an-AMS Center was being constructed, <sup>14</sup>C, <sup>10</sup>Be, and <sup>129</sup>I sample preparation systems were designed and have now been established by the Center's scientists and engineers (Zhou et al. 2007b). Samples from ice cores (<sup>10</sup>Be), meteorites (<sup>10</sup>Be, <sup>26</sup>Al), bare rock (<sup>10</sup>Be, <sup>26</sup>Al), and loess (<sup>10</sup>Be) were prepared and measured. The background of the BeO reached  $1.7 \times 10^{-14}$ , while the measured precision of the <sup>129</sup>I was 1–1.6%. Based on the existing Zn-based <sup>14</sup>C preparation system, a new H<sub>2</sub>/Fe-based system was built, with an output of 8–16 samples per day. About 4500 samples have been measured so far on the Xi'an-AMS, with about half of the samples coming from external laboratories.

## **BREAKTHROUGH OF TRACING THE GEOMAGNETIC INTENSITIES FROM <sup>10</sup>Be IN CHINESE LOESS AT THE INSTITUTE OF EARTH ENVIRONMENT, CAS**

### **<sup>10</sup>Be in Tracing Geomagnetic Intensities**

Production rates of cosmogenic nuclides play a key role in their geophysical applications to tracing geomagnetic field intensities, because the shielding effect of the geomagnetic field that deflects the incident cosmic particles produces a strong correlation between geomagnetic intensity and the atmospheric cosmogenic nuclide production rate (Finkel and Suter 1993; Frank et al. 1997; Lal 1988; McHargue et al. 2000). Unlike the sedimentary <sup>14</sup>C isotope, which has shorter half-life and continuously exchanges with the atmosphere reservoir, the sedimentary <sup>10</sup>Be isotope is not retransferred into the atmosphere and can cover a longer period of time. Hence, the knowledge of past variations in <sup>10</sup>Be production rates is especially important for tracing paleogeomagnetic intensities. Numerous studies tracing the paleogeomagnetic field have shown that the <sup>10</sup>Be records from marine sediments and ice cores do generally reveal the effect of this geomagnetic modulation (Raisbeck et al. 1987; Guyodo and Valet 1996; Frank et al. 1997; McHargue et al. 2000). However, the <sup>10</sup>Be records in loess do not reflect such an effect at all due to the complexity of the origin and accumulation process of <sup>10</sup>Be in loess (Figure 4) (Shen et al. 1989; Zhou et al. 2007c,d). As a result, published studies on <sup>10</sup>Be records in loess had been exclusively concerned with paleoclimate research (Beer et al. 1993; Heller et al. 1993; Guo et al. 1996; Shen et al. 2000), where the effects of the <sup>10</sup>Be production rate in relation to the paleogeomagnetic field were not addressed.

Since 1) there is not a generally accepted record of paleogeomagnetic intensities available at present from either marine or ice records (Frank et al. 1997); 2) the wind-blown loess sequence in China is regarded as one of the most complete terrestrial records for paleoenvironmental studies (Pan et al. 2001), due to the fact that sedimentary <sup>10</sup>Be in loess spanned an entire Quaternary interval, much wider than those in marine sediments and ice cores; and 3) there are considerable uncertainties regarding the B/M polarity conversion (~780 kyr) (Zhu et al. 1994, 1998), the advantages of sedimentary loess <sup>10</sup>Be records might provide a new insight into clarifying the issue. Thus, the Weijian Zhou group at the Institute of Earth Environment, CAS, has been tracing the geomagnetic excursion events and paleointensities from high-resolution Chinese loess <sup>10</sup>Be records in order to prove that loess is able to reliably record the behavior of Earth's magnetic field.

### **Innovative Approach to Trace Geomagnetic Intensities from <sup>10</sup>Be in Loess**

Based on the high similarity between the <sup>10</sup>Be concentration curve and the magnetic susceptibility curve measured from the Luochuan loess in China during the past 80 kyr (Wu 2004), the author proposed an idea to separate the climate effect from the geomagnetic effect on the measured loess <sup>10</sup>Be record in order to trace paleogeomagnetic events by using the susceptibility as a proxy of <sup>10</sup>Be composition affected by climate factors (Zhou et al. 2007c). After sequentially investigating 3 analytical methods for the separation (Zhou et al. 2007c,d; Xian 2007; Xian et al. 2008), and especially with the discovery of the "mean value concept" in a monolinear regression of multivariable geological



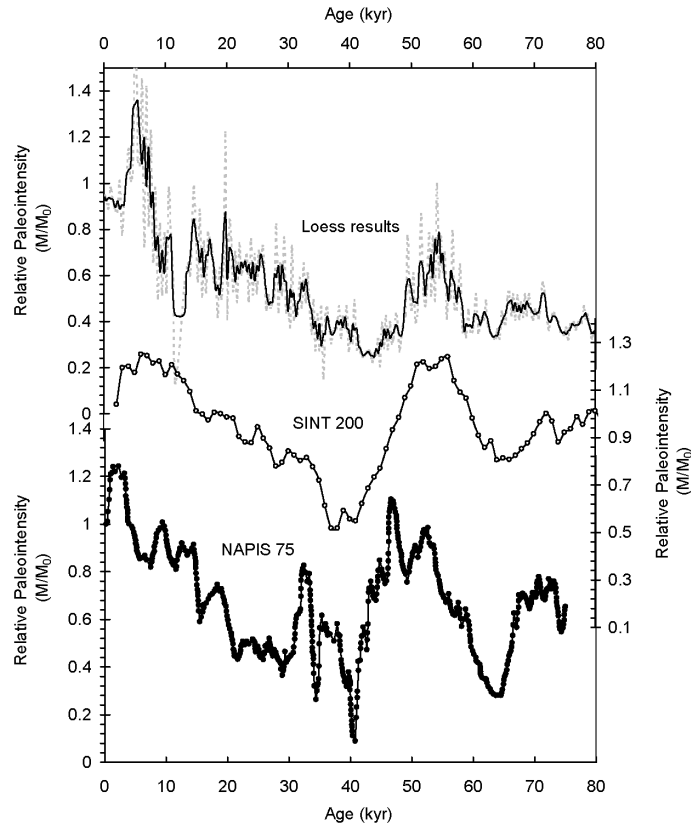


Figure 5 Curve of the first reconstructed paleogeomagnetic intensities for the past 80 kyr from <sup>10</sup>Be in Chinese Louchuan loess and its comparison with the famous SINT-200 and NAPIS75 curves.

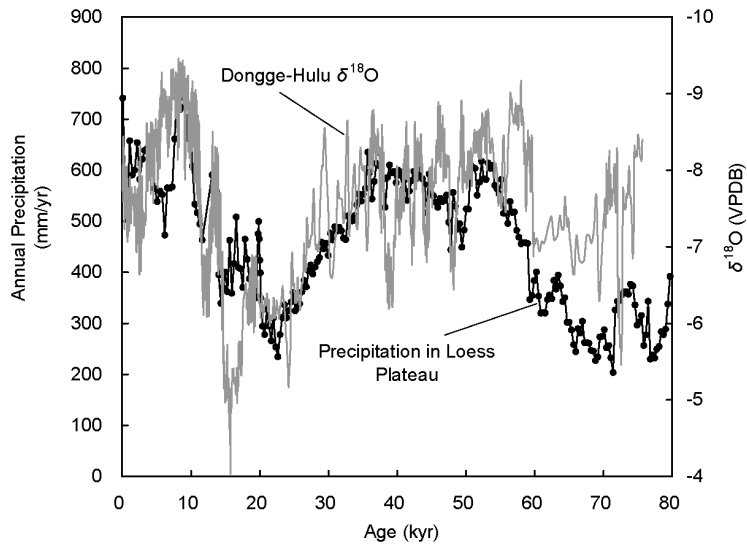


Figure 6 Curve of the reconstructed 80-kyr precipitation history over the loess plateau from <sup>10</sup>Be in Chinese Louchuan loess and its comparison with the speleothem δ<sup>18</sup>O-based records of paleo-Asian monsoon intensity from Dongge and Hulu caves.

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