Present status of BINP AMS

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The AMS is mainly dedicated for research in archaeology, geology, biomedical science and other fields by measurements of the ratio between carbon isotopes. The accelerator mass spectrometry is an ultra-sensitive method of isotopic analysis. The ratio between isotopes ¹⁴C (Radiocarbon) and ¹²C in samples can be less than 10⁻¹⁵. So, the counting of the individual atoms is used for detection of such low radiocarbon concentration.



Now the BINP AMS is a single Russian facility for radiocarbon analysis of samples by accelerator mass spectrometry.

At the end of 2017, the facility was officially registered as a "unique scientific installation" called "BINP AMS SB RAS". Now every scientific organization can leave an online application on the site for the conduct of joint research with BINP using AMS. Заявки в УНУ УМС

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http://www.inp.nsk.su/nauka/issledovatelskaya-infrastruktura/nauchnyeustanovki/uskoritelnyj-mass-spektrometr/podat-zayavku-ams The ratio ${}^{14}C/{}^{12}C$ in modern carbon (living organics) is about $1.2*10^{-12}$ The radiocarbon concentration in "dead" (very old organics) sample is almost zero.

The half-life of a radiocarbon is 5730 years.

The main advantages of AMS method compared to conventional radiometric method are the use of three orders of magnitude smaller samples and the measurement time is an order of magnitude shorter. Only about 1 mg of carbon samples is needed for AMS analysis. This is very important because usually samples either represent a great historical value or large sizes sample are not available.

Atomic and molecular isobars of radiocarbon

- ¹⁴N (The negative nitrogen ions are unstable.)
- ¹³CH, ¹²CH₂ and other molecular ions of mass 14 (The ratio between molecular isobars ions and radiocarbon ions more than 10⁸ in the carbon sample)

Such molecular ions are stable from 1- to 2+ charge state.

Background ions problem: The scattering and charge exchange processes allow backgrounds ion to pass through electrostatic and magnetic filters. (The ions can interact with molecules of residual gas and parts of vacuum chamber.)





BINPAMS The BINPAMS is based on a folded type electrostatic tandem accelerator type of the source is used for AMS analysis. The negative ions are produced by bombarding



BINP AMS layout: 1 - ion source, 2 - low energy beam line magnet, 3 - first accelerating tube, 4 - magnesium vapors stripper, 5 - 180⁰ electrostatic bend, 6 - second accelerating tube, 7 - high energy beam line magnet, 8 -TOF telescope

The multi-cathode (for 23 samples) sputter ion source is used for AMS analysis. The the graphite target with positive cesium ions and are horizontally extracted from the ion source. Then the beam is analysed at low energy with 90° magnet and vertically injected into the first accelerating tube through injection channel. The negative ions are accelerated to the 1 MV high voltage terminal and stripped to 3+ charge state in magnesium vapors stripper. Then they pass through the 180° electrostatic bend working as separator and then again vertically accelerated into the second accelerating tube to the ground potential. The ions after tandem accelerator are separated in 90° magnet and rare ions move horizontally to the final detector through high energy channel.

Most important distinguishing features of BINP AMS

- The ion energy selection just after molecular destruction (by 180° electrostatic bend into the high voltage terminal) \rightarrow effective filtration of the molecular fragments, because energy of fragments always less then ion energy (at this moment).
- The magnesium vapor target as a molecule destroyer → localized molecular destruction
- 2D time of flight detector → accurate recognition of each ion





TOF telescope calibration by different atoms detection. AMS settings were changed to pass each atom type.

The TOF channel width is about 70 ps. The atoms are well detected by the TOF telescope. But, when an electrostatic breakdown occurs in an ion source (or in other electrostatic elements), a large number of background ions can pass through the full system of selection and reach the TOF telescope. Tails from intense ion peaks can reach to the TOF detection area of other atoms. The moment of time for ion detection is registered with 16 us channel width in BINP AMS. This data is used for calculation of number of detected ions per unit time, that allows to numerically discard the background ions from electrical breakdowns.

Sample preparation

For AMS analysis, all natural samples must be converted to a universal form by sample preparation procedure. For these purposes, a sample is combusted in vacuum. Then the carbon from formed CO2 gas catalytically deposited on iron powder. The Fe-C mixture is pressed in aluminum sample holder (cathode for ion source). Sample preparation for the BINP AMS is carried out by chemists from LRMA NSU



Carbon on the iron powder.

Graphitized samples - cathodes for ion source



Ion source



Sample wheel for 23 samples



The sputtering by Cs beam region of the sample is only about 0.5 mm in diameter.

Now at BINP AMS used two types of sample holders: with inner diameter of 2 mm (for about 3 mg of carbon sample) and with inner diameter of 1 mm (for 1 mg or less of carbon sample).

Atmospheric carbon is permeated into the samples during sample preparation. Samples used for radiocarbon dating must be handled carefully to avoid contamination. The contamination level in the samples during the sample preparation procedure is estimated by the radiocarbon content in graphite MPG ("dead" sample) with and without sample preparation.



The content of radiocarbon atoms in graphite MPG without sample preparation is 15 orders of magnitude smaller than the content of carbon atoms. But the content of radiocarbon is increased by an order of magnitude after sample preparation. Typically, the quantity of pollution (radiocarbon concentration) composes approximately 1.5% of the concentration level in the modern samples.

The mass spectrums at the exit of BINP AMS for modern organic (1) and for "dead" samples with (2) and without (3) sample preparation procedure.

To switch between the isotopes:

When measuring the concentration of radiocarbon in the samples, the switching algorithm is used. The isotope ¹⁴C is detected by TOF telescope and ¹³C currents are measured at the exit of AMS by shifted Faraday cup. For switching algorithm the high voltage of ion source is changed. The energy of the cesium ions remains constant. The electrostatic lens and correctors at the exit of the ion source are changed for each isotope. Thus, the passage of isotopes is carried out through a dipole magnet in low energy channel without changing the magnetic field. The magnetic field in high energy beam line magnet is not changed to, because the radial aperture is wide enough for passing radiocarbon ions to TOF detector and ¹³C ions to shifted FC.



Algorithm for measuring of the radiocarbon concentration on BINP AMS



 $^{13}C^{3+}$ currents (a), radiocarbon counts $^{14}C^{3+}$ (b), and radiocorbon conctntration (c) depending from the time

For each sample, the ¹⁴C ions are counted four times (10 seconds each) and twice the ¹³C currents are measured for each 10 seconds counting. After that, the samples wheel is turned to the next sample for process repetition. Measuring of whole sample wheel (20 samples) takes about 15 minutes. For a set of statistics the wheel are moving to the second turn, third, etc. Typically, the measurement will take approximately 5 hours, with a statistical error of measurement for modern samples less than 1%.

Statistics Set



Example of accumulation of counted radiocarbon ions and a change in the measured value of the radiocarbon concentration in the standards, depending on the wheel turn number.

BINP AMS analysis reproducibility.



Individual values of the radiocarbon concentration in the OXII standards.

 Radiocarbon concentration, pMC

The distribution of radiocarbon concentrations for OXII standards and the approximation by the Gaussian function.

The rate of radiocarbon concentration measurement is 20 samples in about 5 hours. It is seen that the average error value is slightly less than 1%.

An example of a radiocarbon analysis of samples

In order to increase the reliability of the samples dating, the samples are prepared and measured in pairs. The duplicating samples are circled. It is seen, that the measured values of the radiocarbon concentration in identical samples are statistically identical. The international standard reference materials OXI (SRM 4990 B) and OXII (SRM 4990 C) are used for calculation radiocarbon age of dated samples by normalization to samples with a known radiocarbon concentration.



In 2017, about 1000 samples was analyzed at BINP AMS for 25 user organizations:

- Федеральное государственное бюджетное учреждение науки Институт криосферы Земли Сибирского отделения Российской академии наук
- Федеральное государственное бюджетное учреждение науки Институт геологии и минералогии им.В.С. Соболева Сибирского отделения Российской
- Федеральное государственное бюджетное учреждение науки Лимнологический институт Сибирского отделения Российской академии наук
- Федеральное государственное бюджетное учреждение науки Институт земной коры Сибирского отделения Российской академии наук
- Федерального государственного бюджетного учреждения науки Институт археологии и этнографии Сибирского отделения Российской академии наук
- 6. Томский Государственный Университет
- Научно-производственный центр по сохранению историко-культурного наследия Новосибирской области
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- 17. Меморнальный музей А.С. Попова СПбГЭТУ «ЛЭТИ»
- 18. Ульяновский государственный педагогический университет
- 19. ООО "КРАСНОЯРСКАЯ ГЕОАРХЕОЛОГИЯ"
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- 22. Хакасский Государственный Университет
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- 25. Амурский государственный университет

Example of BINP AMS using in user research. Radiocarbon age of lake Ebeity sediments, depending on the depth from surface level



The deposits accumulation rate at small depths is an about order of magnitude higher than at deep depths.

Example of BINP AMS using in user research.

Intermediate lakes of the Chulym and Kargat river valleys and their role in the evolution of the Lake Chany basin.

S.K. Krivonogov (IGM SB RAS) , et al.



Radiocarbon dates of sediments of intermediate lakes of the Kargat and Chulym river

Location	Depth, cm	Material	Laboratory number	Measured ¹⁴ C age	Corrected ¹⁴ C age	Calendar age (median value)
Lake Sargul	10-20	Pisidium shells	LRMA K-219	3519 ± 98	3658 ± 99	3991
	50-60	Pisidium shells	LRMA K-220	4262 ± 96	4401 ± 97	5027
	80-90	Pisidium shells	LRMA K-221	4503 ± 94	4642 ± 95	5371
	120-130	Pisidium shells	LRMA K-216	5193 ± 89	5332 ± 90	6112
	160-170	Bithynia shells	LRMA K-223	5911 ± 93	6050 ± 94	6913
	180-190	Bithynia shells	LRMA K-224	6638 ± 98	6777 <u>±</u> 99	7633
Starogornostalevo basin near-shore zone	25	Peat	LRMA K-264	1867 ± 41	1867 ± 44	1804
	25	Lymnaea shell fragments	LRMA K-233	1560 ± 84	1699 ± 86	1613
	53-55	Lymnaea shells	LRMA K-234	3537 ± 94	3676 ± 95	4017
	55-57	Peat	LRMA K-265	4288 ± 45	4288 ± 48	4858
Starogornostalevo basin central part	45–50	Peat	LRMA K-266	1247 ± 36	1247 ± 39	1197
Suma basin near-shore zone	16-25	Lymnaea shell fragments	LRMA K-235	2756 ± 83	2895 ± 85	3037
	90–93	Lymnaea shell fragments	LRMA K-236	4976 ± 106	5115 ± 107	5855
Suma basin central part	20-25	Peat	LRMA K-268	1884 ± 44	1884 ± 47	1826
	25-30	Anadonta shell fragments	LRMA K-238	2211 ± 83	2350 ± 85	2413
	25-30	Lymnaea shell fragments	LRMA K-248	2514 ± 56	2653 ± 58	2777
	30-35	Anadonta shell fragments	LRMA K-249	2691 ± 61	2830 ± 63	2944

Example of BINP AMS using in user research.

BINP and **NSU** determined the age of the **Meshoko** rock shelter. The **dating confirmed the assumption** that the third layer of the camp belonged to **Maikop culture**.



Radiocarbon age: 4778 ± 35 years

Calendar age : 3645 – 3516 BC (92.5%)



et al 2013

Example of BINP AMS using in user research. The **DEATH** of **MAMMOTHS** was **NATURAL** and it not related to the human activity.

Leshchinskiy S.V. (TSU), et al. "WOLF RIDGE IS THE LOCATION WITH THE LARGEST CONCENTRATION OF THE REMNANTS OF MAMMOTHS IN ASIA"



More than half of the new datings of mammon bones are in the range 19-17 thousand years BP.

SUMMARY

- The BINP AMS with additional ion selection properties is demonstrated the sustained performance on 1MV running and the good radiocarbon ions identification.
- The BINB AMS is used for radiocarbon analysis of graphitized natural samples for users. About 1000 samples were measured last year for 25 user organizations.
- The statistical error of radiocarbon analysis for modern samples less than 1% when measuring 20 samples per 5 hours.
- The concentration of radiocarbon in the very old samples is approximately 10^{-14} ($^{14}C/^{12}C$) due to contamination by atmospheric carbon during sample preparation of natural samples.