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STATUS AND PROSPECTS OF THE ACCELERATOR MASS SPECTROMETRY CENTER OF THE INSTITUTE OF APPLIED PHYSICS OF NASU

The paper deals with the issues related to development of the Accelerator Mass Spectrometry CF of IAP of NASU. The first archaeometrical results have been presented. The main tasks and directions of further CF development have been discussed.

Key words: accelerator mass spectrometry, center for collective use, ^{14}C carbon isotope, archeology, radiochemistry

Within the framework of the Core Facility of Institute of Applied Physics of NASU (hereinafter referred to as «the IAP CF») the key research and studies have been conducted in the following fields:

- Nuclear energy and nuclear safety (environmental monitoring of ^{14}C, ^{129}I, ^{236}U, ^{239}Pu, ^{240}Pu, ^{241}Pu, ^{242}Pu);
- Archeology, geology, cultural heritage artifacts (carbon dating: ^{14}C);
- Radiation ecology (identification of delayed radiation effects in living organisms, water circulation in the context of Chernobyl accident: ^{14}C, ^{10}Be, ^{129}I);
- Biomedicine and pharmacology (monitoring of body state based on the distribution and kinetics of introduced radioisotope; metabolism of pharmaceutical products: ^{26}Al, ^{14}C);
- Registration of long-living isotopes in nature, in a wide range of applications (technosphere, biosphere, atmosphere, hydrosphere, and cosmosphere) [1—4].

During the study, there have been used a lot of methods and techniques, including accelerator mass spectrometry (AMS), a cutting-edge, ultra-sensitive method for isotopic analysis of substances.

The mass spectrometric analysis of substance is based on classifying and registering the ionized atoms or molecules according to their relative mass-to-charge ratio (m/q). Despite the use of this method for solving a wide range of analytical tasks, some challenges remain inaccessible for traditional mass spectrometry, even for that with record-breaking sensitivity (10^{-10}). In addition to the problem of sensitivity there is limited resolution as well. The separate quantitative registration of isobars and molecular ions, which have the same mass as measured isotope, is principally unattainable with the use of conventional schemes of mass spectrometric separation of ions.

The specific tasks requiring high-resolution sensitivity from mass spectrometry should include the measurement of extremely rare isotopes constituting 10^{-12}—10^{-15} of major radionuclide (e.g., long-living cosmogenic radionuclides) concentration [5—7]. The cosmogenic nuclides are the products of nuclear reactions of cosmic rays with the Earth's substance. The cosmic rays coming to the Earth possess a sufficient energy to induce nuclear reactions. The flux of cosmic rays entering the Earth's surface is relatively small and is about a half of background radiation. However, near the troposphere-stratosphere interface their intensity reaches a maximum [3]. Among the cosmogenic radionuclides ^{3}H and ^{14}C make the largest contribution to the natural radioactivity. The most interesting objects to be studied are long-living cosmogenic nuclides ^{3}H, ^{10}Be, ^{14}C, ^{26}Al, ^{36}Cl, ^{129}I (Table 1) formed in the atmosphere.

A remarkable feature of cosmogenic radionuclides is independence of their concentration from the initial content in the Earth's matter. They are used for carbon dating, particularly, in archeology, geology, and earth sciences. Of course, the scope of application of the methods for accurate measurement of main-to-«rare» isotope ratio can be extended.

Such measurements become possible as a result of combining an ion accelerator and a mass spectrometer in one analytical device. Nowadays, throughout the world, there are about one hundred operating AMS centers. Most of them deal with analyzing the carbon isotopic composition (^{14}C) for a variety of tasks, including archaeometry.

IAP NASU ACCELERATOR MASS SPECTROMETER

The IAP AMS core facility (the only one within the territory of Ukraine) was launched in 2010. During three years, at the IAP, a Tandetron 1.0 MV accelerator mass spectrometer (model 4110Bo-AMS, manufactured by HVEE Europa B.V., Netherlands) was being installed and commissioned.

The Tandetron 1.0 MV accelerator mass spectrometer is a model of new generation (Figs. 1, 2) characterized by compactness, versatility, and ability to add options. In this device, there have been implemented new original engineering solutions, which give it an advantage over the previous models. For example, the removal of molecular interference (in case of ^{14}C, ^{13}CH and $^{12}CH_2$) is achieved due to the design of ion recharge chamber and increased density of "stripping" gas rather than due to the high-charged state of ions and accelerating potential. The separation of isobars and molecular ions occurs in the ion source, accelerator recharge chambe, and in detector chamber of recording system. The hybrid ion source (Model SO-110) allows the researchers to analyze the sample in both the solid and the gaseous states.

Fig. 1. AMS Tandetron 1.0 MV, model 4110Bo-AMS

The structure of Tandetron 1.0 MV model 4110Bo-AMS can be conditionally divided into three parts: the *low energy mass spectrometer*, the *ion accelerator*, and the *high-energy mass spectrometer* (Fig. 2). The first group of devices includes the ion source and the initial electromagnetic analyzer. They provide a highly effective entry of

Table 1

Cosmogenic radionuclides [5, 7]

Isotope	Half-life period	Product of decay
^{3}H	12.33 years	β-particles
^{10}Be	1.51×10^{6} years	β-particles
^{26}Al	7.2×10^{5} years	β-particles
^{14}C	5730 years	β-particles
^{36}Cl	3.01×10^{5} years	β-particles
^{129}I	1.57×10^{7} years	β-particles

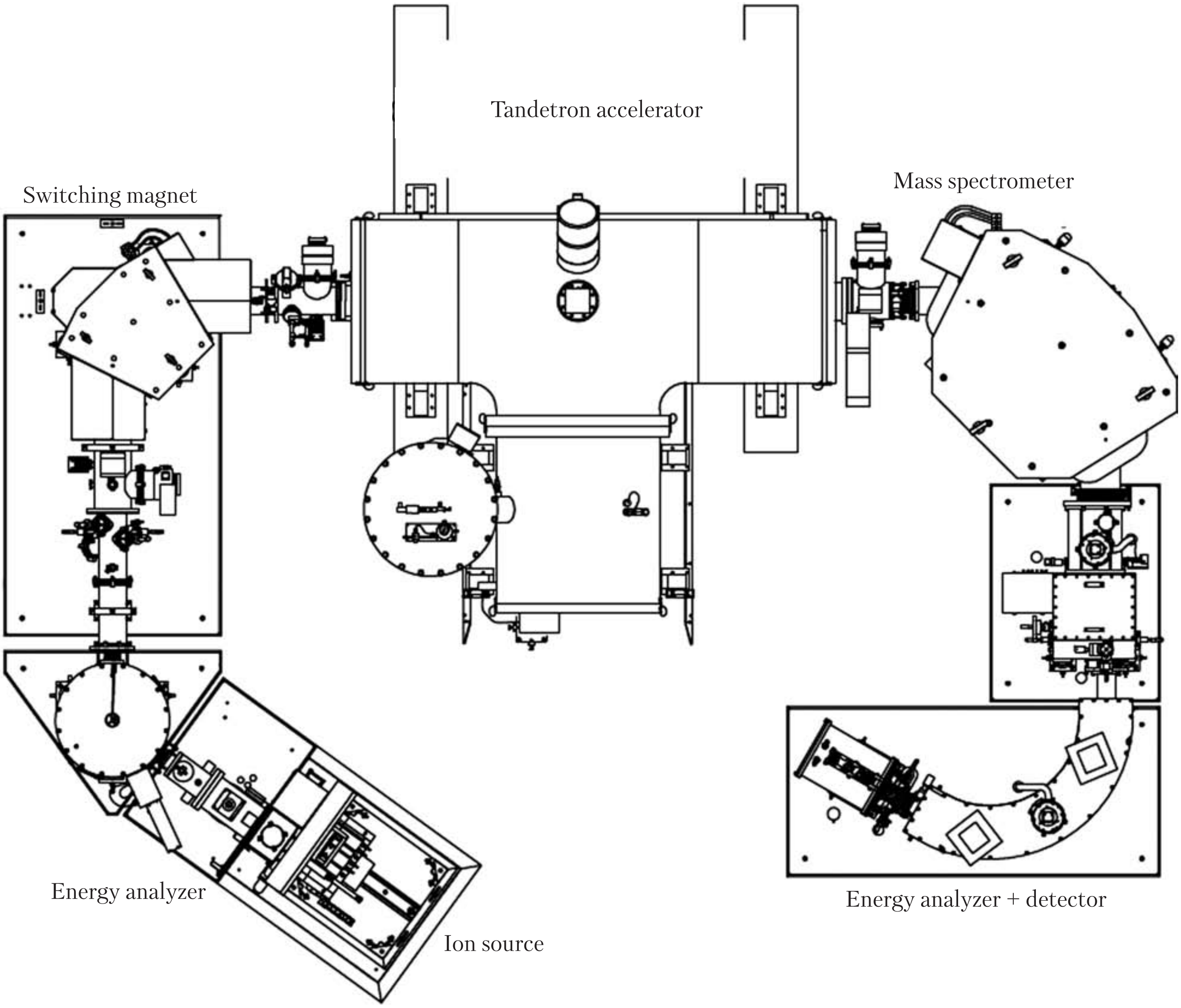

Fig. 2. Diagram of AMS Tandetron 1.0 MV, model 4110Bo-AMS

target nuclide into acceleration mode. In the ion source, under the influence of Cs^+ ions flux, the substance is ionized and evaporates from the surface of test sample. The atoms knocked out of the solid sample are partially ionized, with the isotope negative ions being among them.

The positively charged cesium ions are relatively easily desorbed from metallic surface while it is heated. They generate a sufficiently dense ionizing beam. Furthermore, they are focused onto the target by an electric field of the same polarity, which induces the emission of negative ions from the test sample. Since the interfering impurities often do not form stable negative ions (e.g., ^{14}N in case of ^{14}C or ^{26}Mg for ^{26}Al), the input source plays a role of the first selection device. Then the second step, selection based on *m*/*q* criterion, takes place. Having passed through the filter the ions are focusing and directed to the entrance of accelerator.

The tandem accelerator belongs to the class of electrostatic accelerators where the charged particles gain energy from a constant electric field and are accelerated by potential difference of several million volts. To raise the dielectric strength the accelerator body is placed in a vessel filled with insulating gas (SF_6 at a pressure of 4—6 bars). The negative ions from the low energy mass spectrometer pass through the first tandem section and are accelerated in positive potential. Then, having acquired energy of several MeV, they pass through the recharge cell where they lose some electrons. The resulting positive multi-charged ions are accelerated again in the second tandem section, from high potential to zero. Typically, the resulting energy is equal to several million electron volts. Having been accelerated, the particles are selected again by m/q factor. The radius of curve along which the ions are moving in magnetic field depends on the ion mass, charge, and kinetic energy. Consequently, the ions passing through the slit behind the magnet are distributed by mass, charge, and kinetic energy.

The high energy acquired by the ions that have passed through the whole acceleration cycle affords a possibility to make the last stage of selection using a $\Delta E - E$ detector providing for independent determination of specific energy loss and particle energy. Having passed through the special window the accelerated ions enter the gas discharge chamber where they generate the traces of secondary ions. Insofar as the ions entering the detector have the same velocity and charge, the interaction between the ions and the gas molecules depends mainly on ion nuclear charge. Under the influence of electric field the traces of secondary ions move to the collector plates where their average length is measured. Upon the results of these measurements the number of analyzed ions (e.g., ^{14}C) is calculated. After minor improvements and adjustments the accelerator mass spectrometer Tandetron 1.0 MV, model 4110Bo-AMS, originally designed to measure isotopes ^{14}C, ^{10}Be, and ^{26}Al can make the analysis of heavy isotopes, such as ^{129}I and even ^{239}Pu, ^{240}Pu, and ^{242}Pu [8, 9]. Table 2 contains the main tool characteristics.

The decisive factor for the operation of accelerator mass spectrometer in a given mode is to maintain the relevant parameters of microclimate in the laboratory room. The infrastructure (gas, water cooling system device, etc.) has to meet the special requirements. Therefore, the IAP specialists have designed and manufactured a device for drying and reduction of sulfur hexafluoride (SF_6) used as an insulating agent (Fig. 3), as well as a water cooling system.

PREPARATION OF SAMPLES FOR ANALYSIS

The sample preparation for the purposes of accelerator mass spectrometry is a rather complicated and expensive procedure. Firstly, there are neither commercial facilities nor complex laboratories for such preparation in the world market. Each laboratory designs and manufactures the required facilities on its own. The total cost of facility ranges from 0.2 to 1 million euros, which exceeds the cost of device itself. Secondly, to place the equipment for sample preparation it is necessary to have a basic chemical analytical laboratory meeting the strict requirements of GLP (Good Laboratory Practice) [10]. Currently, the laboratory must meet a whole range of requirements, namely: the Labor Protection Regulations (LPR) 0.00-1.27-09, the Government Sanitary Regulations 3.3.6.042-99, GOST 12.1.005-88, GOST 12.1.005-88, the Fire Safety Regulations A.01.001-2004, LPR 0.00-1.27-09, LPR 40.1-1.21-98, GOST 12.1.004-91, GOST 12.1.005-88, GOST 12.1.007-76, GOST 12.1.010-76, GSTU GOST 12.2.061:2009, GOST 12.3.002-75, the CMU resolutions of 13.04.2011, no. 469, and of 26.05.2004, no. 687, and the order of 11.09.2012, no. 1192 [11].

Table 2

Characteristics of AMS Tandetron 1.0 MV, model 4110Bo-AMS

Isotopic ratio	Sensitivity	Error
$^{14}C/^{12}C$	3×10^{-15}	≤0.5 %
$^{13}C/^{12}C$	1×10^{-14}	≤0.3 %
$^{10}Be/^{9}Be$	3×10^{-14}	≤3.0 %
$^{26}Al/^{27}Al$	5×10^{-14}	≤3.0 %
$^{129}I/^{127}I$	1×10^{-12}	≤5.0 %

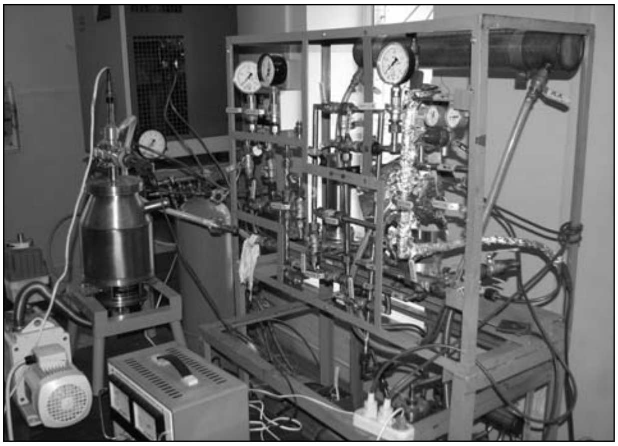

Fig. 3. Device for drying and reduction of electronegative gas (SF_6)

For the AMS purposes the samples are prepared by extracting from the sample the total carbon fraction as gaseous carbon dioxide CO_2. Firstly, it is necessary to conduct the preliminary sample purification due to separating sequentially the macro and micro contaminants by mechanical and chemical pretreatment with the use of acids and alkalis. Further, depending on the sample type (organic or carbonate (mineral)), the sample is either combusted (the organic samples) or decomposed with the use of acids (the carbonate samples). The organic materials are combusted in evacuated quartz ampoules, with CuO as a source of oxygen, or in the respective blocks of standard elemental analyzers for burning of organic substances, which are widely used in organic chemistry. The resulting CO_2 is purified from impurity gases (in particular, from nitrogen oxides, halogens, and oxygen) with the help of filter system, absorbents (silver wire, $PbCrO_4$, etc.) and cryogenic technique for freezing CO_2 by liquid nitrogen and removing trace gases by «dry» turbo-molecular pumps and oil-free backing vacuum pumps. Having been purified, CO_2 is either graphitized by the Bosch reaction with formation of solid phase «targets» for mass spectrometric analysis, or is directly fed to the ionization source of mass spectrometer. The reaction is based on reduction of carbon dioxide with formation of solid elemental carbon (graphite) on the catalyst surface of highly purified fine iron powder (*Alfa Aesar* iron powder, –325 mesh, reduced, 98 %) at a temperature of 400—650 °C during 0.5—6 hours:

$$CO_2 + 2H_2 \rightarrow C + 2H_2O$$

(550—650 °C; catalyst is Fe).

In the majority of laboratories there has been used graphitization which is rather complicated to implement, inasmuch as the installation is made of hard glass, contains precision vacuum valves of quartz (*J. Young*) or metal (*Swagelok*), digital vacuum pressure gauges and vacuum manometers (*Pfeiffer Vacuum*), temperature sensors, tube furnaces (*Watlow*) for heating up to 400 and 600 °C, and Peltier elements. In addition, the installation must withstand the residual pressure of up to 5×10^{-2} Pa provided by oil-free backing pump in combination with «dry» turbo pump (for example, *Adixen Alcatel Drytel*). The mandatory component of graphitization is an element analyzer for high-temperature combustion of organic samples in oxygen atmosphere (*Vario Micro Cube*).

Unlike the graphitization, the gas injection technique does not provide for obtaining solid carbon. The purified CO_2 is fed directly into the ionization source, which significantly reduces the cost and complexity of process, although it affects, to some extent, the accuracy of analysis. Nevertheless, currently, many AMS laboratories are improving this approach and have achieved impressive results. It should be noted that for the purpose of carbon dating the graphitization technique is mainly used, while for the biological applications of AMS the gas injection is getting more widely used. In the recent decade, a relatively low current of negative ions is considered to be the principal disadvantage of ^{14}C measurements in gaseous phase (which entails a longer duration and a lower accuracy in comparison with the solid phase graphite samples). In the last 4—5 years, the gas injection technique has showed a significant progress providing a decrease in the sample mass required for obtaining reproducible results.

Although for any type of AMS installation the analysis by gas injection technique lasts slightly longer as compared with the analysis of graphite samples, the entire preparation of these samples by classic graphitization method takes hours versus dozens of minutes required for the high-temperature combustion of organic matter to obtain gaseous carbon dioxide. For example, in Swiss Federal Institute of Technology, Zurich (Eidgenössische Technische Hochschule, Zürich), on MICADAS (MIniCArbon System) installation, there have been achieved the parameters allowing the researchers to eliminate virtually the gap between graphitization and gas injection with respect to the analysis of gaseous samples weighing 1—50 micrograms (in this case the ion current reaches 12—15 mA and the accuracy is 5 per cent). This makes it possible to confidently determine the amount of ^{14}C in such samples at the level of dozens of nanograms [12, 13]. To analyze the micro-samples they are burnt in quartz tubes. Copper oxide is used as oxygen source [14]. The further development of gas injection technique has led to designing integrated plants for obtaining CO_2 from the samples with the use of laser ablation of carbonate samples ($CaCO_3$) of stalagmites and corals [15]. A simpler method for obtaining CO_2 from carbonates is the treatment of samples by phosphoric acid with consequent purification and supply of gas to the source [16].

A group of British scientists has found that when using the gas injection technique the stable and reproducible ion currents of up to 12 μA can be reached at the CO_2 supply rate ranging within 1.5 ± 0.5 μl × × min^{-1} [17]. However, the general rule for gas injection techniques irrespective of sample combustion and purification method (laser ablation, gas-liquid chromatography, hydrolysis, high-temperature combustion) is as follows: the rate of carbon dioxide inflow to the gas source should not exceed 3 μl × s^{-1}. The larger or smaller flows (net of carrier gas contribution) can cause vacuum failure or disruption of ionizer operation. The constant gas flow at a rate of 3 μl × s^{-1} (at a pressure of 1 atmosphere) is ensured by selecting the inlet capillary length [18].

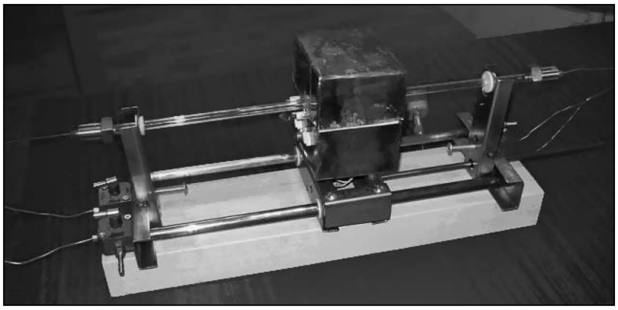

Fig. 4. Furnace for burning of organic samples in complex installation for sample preparation for AMS

An impressive example of using the gas injection technique for AMS in the context of proteomics problems related to the development of ultra-sensitive methods for tracking proteins and their metabolites or *in vivo* post-translational modification is accomplishments of researchers from the Center for Accelerator Mass Spectrometry at the Livermore National Laboratory (USA). They have showed that the LS-AMS installation allows the users to measure zeptomolar (10^{-21}) amount of ^{14}C in submicrogram quantities of specific protein extracted by high-performance liquid chromatography (HPLC) from complex protein mixture. It should be noted that in this installation, having been separated, the protein is immediately combusted, with carbon dioxide being fed directly into the ionization source. The comparison of the classical graphitization and the gas injection techniques has showed an almost complete agreement of analytical results (Table 3). However, the performance of gas injection technique is higher by an order of magnitude than that of the classical graphitization [19]. Taking into account the above mentioned considerations the IAP of NASU has designed an integrated installation of sample preparation for AMS.

The objective of this project is to create a system for combining two technologies for the preparation of samples in single installation unit by combining the unit for high-temperature combustion of organic matter (Fig. 4) with the unit for fine purification of CO_2. The basic component of the latter is a gas chromatograph (*Selmi-*

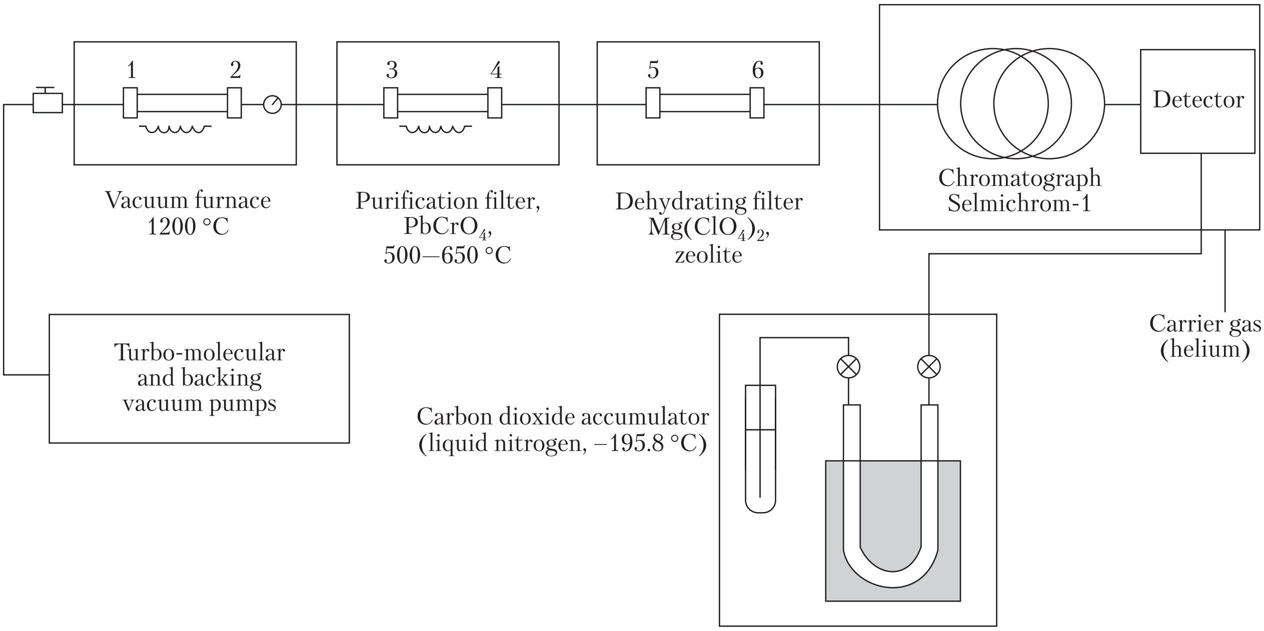

Fig. 5. Principal diagram of AMS gas injection system (IAP of NASU)

chrome-1, *SELMI*, JSC, Sumy) (Fig. 5). The next stage of sample preparation is to feed gaseous carbon dioxide into the AMS ionizer by means of CO_2 off-line mobile accumulation system (Fig. 6) (in future, by means of on-line capillary system and/or original CO_2 graphitization system developed by IAP of NASU for obtaining solid-phase «target samples» (Fig. 7)).

The next step in the development of analytical chemistry of carbon isotopes in the AMS context is the equipment for biochemical analysis laboratory: before the transformation of carbon contained in the sample into graphite it must be cleaned from «impurities» and «modern» carbon. The sample is treated with chemicals that selectively interact with certain types of carbon compounds. The «true» carbon component can be identified, on the basis of which the sample age is established. For example, the bones could absorb organic compounds or undergo chemical aggression of fungi and bacteria. These processes are a source of «foreign» carbon in bones, which affects the accurate determination of initial amount of ^{14}C, especially if the sample is very old. The main organic material of bones is collagen. One of the amino acids constituting collagen, oxyproline, can be found mainly in bones. Therefore, one can state certainly that in the sample containing oxyproline the carbon was incorporated in its structure during the formation of bone, not afterwards. For this kind of analysis it is necessary to use the state-of-the art methods of biochemical analysis, including, high performance liquid chromatography (HPLC).

Thus, when launching the AMS there was implemented a complex of commissioning operations, including testing and check of functional characteristics of all the AMS units, a series of

Table 3

Content of ^{14}C isotope in bovine serum albumin (BSA) samples modified by ^{14}C-iodine acetamide [19]

Sample	Atto-mole, ^{14}C / mg of BSA	
	Graphitization	LS-AMS
BSA (control)	57.2 ± 0.8	57.2 ± 1.1
BSA, native, modified by ^{14}C-iodine acetamide	3074 ± 35	2900 ± 130
BSA, reduced, modified by ^{14}C-iodine acetamide	2342 ± 21	2313 ± 59

test measurements of carbon isotope ratios on standard samples, and optimization of AMS ion optical systems. The basic infrastructure elements of installation, namely: the sulfur hexafluoride reduction system, the closed-loop water cooling system, and the facilities ensuring the operation of pneumatic and ventilation systems. The techniques for carbon isotope analysis have been improved and optimized using sets of standard samples. The equipment for analytical combustion of material and sample gas injection into the ion source of mass spectrometer has been designed and tested, which is of particular interest for biomedical industry (e.g., molecular biology, oncology, biochemistry, and pharmacology).

THE FIRST EXPERIMENTAL RESULTS

In collaboration with specialists from the Institute of Archaeology and Ethnography of the Siberian Branch of RAS the IAP of NASU started to develop methods for preparation of solid samples (analytical combustion and graphitization) for AMS-analysis and to create a database for building the radiocarbon age calibration curves. A series of archaeological samples (50 pcs.) have been made analyzed.

The IAP of NASU fruitfully cooperates with the IAEA within the framework of IAEA regional technical assistance project RER/0/034 *Improvement of Characterization, Preservation, and Protection of Cultural Heritage*.

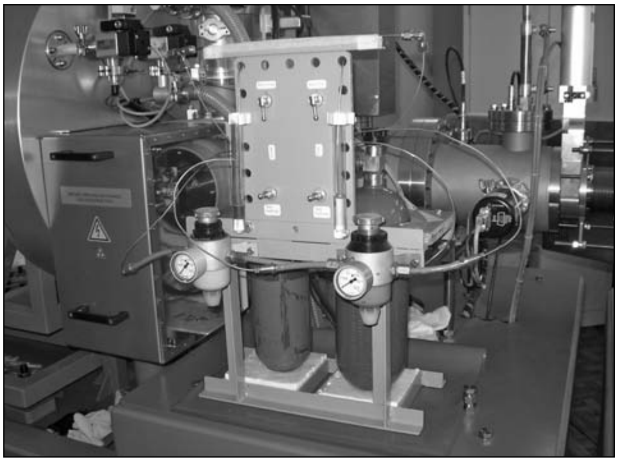

Fig. 6. CO_2 accumulation system

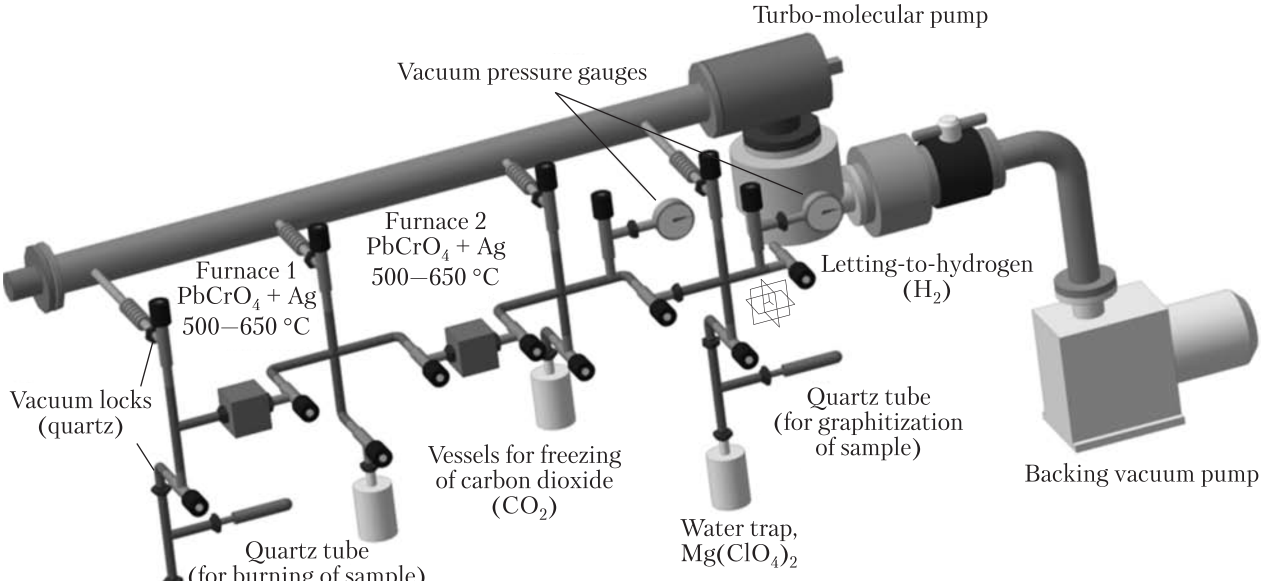

Fig. 7. Diagram of organic samples combustion-graphitization installation for AMS

The IAP staff has analyzed the samples of charcoal found by archeologists of Institute for Archaeology of NASU near the village of Hodosiivka (Dibrova area, 1988) and preliminary dated by experts as having an age of about 3.4—3.5 × × 10^3 years (the Bronze Age). Our studies have almost absolutely confirmed the archeometrical results, which is especially important considering a very small mass of the sample (0.34 g). The AMS advantage in comparison with other archeometrical methods (a possibility to study the extremely small mass organic samples) is vividly illustrated by the results of analysis of 0.07 g charcoal sample from Bukovaya mound (2010). It is dated 3.4–3.6 × 10^3 years old (the Bronze Age).

While setting the device configuration we used direct AMS analysis of solid pyrolytically and chemically oxidized samples of ultrapure sucrose and charcoal.

The main issues to be addressed are as follows:

- To provide the CF with additional state-of-the-art analytical equipment for addressing numerous cross-disciplinary problems;
- To take a set of measures for metrological certification and accreditation of CF equipment, including the creation of new research and engineering specialty occupations required for CF operation;
- To determine the CF administrative and financial status which would optimize effective cooperation between the CF and the users within the framework of budget, tax, and civil codes of Ukraine.

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СТАН ТА ПЕРСПЕКТИВИ РОЗВИТКУ ЦЕНТРУ ПРИСКОРЮВАЛЬНОЇ МАС-СПЕКТРОМЕТРІЇ ІНСТИТУТУ ПРИКЛАДНОЇ ФІЗИКИ НАН УКРАЇНИ

Розглядаються питання розвитку Центру колективного користування «Прискорювальна мас-спектрометрія» при Інституті прикладної фізики НАН України. Наведені результати перших експериментів з датування археологічних зразків. Сформульовані базові завдання для подальшого розвитку Центру колективного користування.

Ключові слова: прискорювальна мас-спектрометрія, Центр колективного користування, ізотоп вуглецю ^{14}C, археологія, радіохімія.

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СОСТОЯНИЕ И ПЕРСПЕКТИВЫ РАЗВИТИЯ ЦЕНТРА УСКОРИТЕЛЬНОЙ МАСС-СПЕКТРОМЕТРИИ ИНСТИТУТА ПРИКЛАДНОЙ ФИЗИКИ НАН УКРАИНЫ

Рассмотрены вопросы развития Центра коллективного пользования «Ускорительная масс-спектрометрия» Института прикладной физики НАН Украины. Приведены результаты первых экспериментов по датированию археологических образцов. Сформулированы основные задачи, направленные на дальнейшее развитие Центра коллективного пользования.

Ключевые слова: ускорительная масс-спектрометрия, Центр коллективного пользования, изотоп углерода ^{14}C, археология, радиохимия.

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