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BALTIC-PONTIC STUDIES

VOLUME 7 • 1999

BALTIC-PONTIC STUDIES
61-809 Poznań (Poland)
Św. Marcin 78
Tel. (061) 8536709 ext. 147, Fax (061) 8533373

ADAM MICKIEWICZ UNIVERSITY
INSTITUTE OF EASTERN STUDIES
INSTITUTE OF PREHISTORY
Poznań 1999
ISBN 83-86094-06-0
ISSN 1231-0344

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Cover Design: Eugeniusz Skorwider

Lingvistic consultation: John Comber

Printed in Poland

Computer typeset by PSO Sp. z o.o. w Poznaniu

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Editor's Foreword

This volume of the *Baltic Pontic Studies* focuses on the results of the research carried out so far into the absolute (radiocarbon) chronology of the area lying between the Vistula and Dnieper or the bio-cultural borderland between the West and East of Europe. Absolute chronology is treated here both as a research goal and fundamental premise in the broader studies of the chronometric and development synchronization of "borderland" cultural systems. In a series of articles devoted to individual taxa a considerable number of new ^{14}C dates have been compared. The dates concern source materials that have been chosen from the point of view of their representativeness and chronometric value ("short-lived" materials were preferred to minimize a potential error). The vast majority of analyses were purposefully made in the same ^{14}C laboratory of the *State Scientific Center of Environmental Radiogeochemistry of Ukrainian Academy of Sciences* in Kiev taking advantage of funds generously provided by the *Polish Committee for Scientific Research*.

The volume devoted to the "dark" section of the "borderland" history (3150--1850 BC) is the first but not the last publication on the broader issues mentioned above that we intend to present in the near future.

Editorial comment

1. All dates in the B-PS are calibrated [see: Radiocarbon vol.28, 1986, and the next volumes]. Deviations from this rule will be point out in notes.
2. The names of the archaelogical cultures and sites are standarized to the English literature on the subject (e.g. M. Gimbutas, J. P. Mallory). In the case of a new term, the author's original name has been retained.
3. The spelling of names of localities having the rank of administrative centres follows official, state, English language cartographic publications (e.g. *Ukraine, scale 1 : 2 000 000*, Kiev: Mapa LTD, edition of 1996).

Baltic-Pontic Studies
vol. 7: 1999, 27-33
PL ISSN 1231-0344

Vadim V. Skripkin, Nikolay N. Kovalyukh

RADIOCARBON LS DATING OF BONE MICRO-SAMPLES

Samples of bones from archeological excavations are reliable and often exclusive material for radiocarbon dating. At the same time radiocarbon dating of fossil bones is associated with some difficulties. Porous structure of bones when being in ground absorbs water soluble organic substances, which have age different from collagen of bones. Besides, bone collagen when being in ground is subjected to bacteria and micro-fungus destroying influence. Micro-biota influence leads to breaking of carbon isotopes primary correlation, so called "isotopic fractionating".

The factors mentioned above have an influence particularly on the bone micro-samples dating, in which carbon total contents does not exceed one gram. Micro-samples form not less than 50% in a whole archeological material available for radiocarbon dating. Micro-samples of bone material are the only possible objects in determination of absolute age by isotopic methods for many archeological monuments. It should be given the special attention for primary chemical processing of samples under investigation. An important and inconsistent problem is decided on this stage: as much as possible full removing of introduced organic substances and bad admixtures with keeping simultaneously as much as possible amount of bone collagen, and of bone coal - for burnt bones. It is important for the samples with significant biological destroying of collagen (more than 50%), that stable carbon isotopes correlation with subsequent correcting of radiocarbon age should be defined by masses-spectrometric method.

Methods and strategies. A new complex technology is designed in our laboratory of bone micro-samples primary chemical processing and subsequent carbon dating fractions deposition in a kind of benzene.

Primary processing. Traditional strategy of bone samples primary processing comprises a stage of collagen deposition in a pure substance type [Arslanov 1987]. For this aim the sample reduced to fragments is processed by 0.5% - 2% solution of hydrochloric acid at room temperature. Mineral part of bone consisting of phosphates and calcium and magnesium carbonates is dissolved in hydrochloric acid,

but collagen stays as a jelly-like material. It is important to note that a certain portion of the bone organic material is also dissolved and, moreover, it disappears forever. Subsequent processing of collagen is impeded and requires much time. Hydrated form of collagen is extremely uncomfortable for washing, centrifugation and drying. A great amount of phosphorus and sulphur compounds is abundant in the end product - dry collagen. Phosphorus and sulphur are the most harmful admixtures in lithium carbide producing, and that is why it is necessary to oxidize a collagen beforehand till carbon dioxide with subsequent gas purifying. Multi-stage of traditional technology leads to inevitable losses of carbonaceous substances, that is undesirable particularly for micro-samples. It is impossible at all to select a dating carbon fraction from some types of bone material by traditional technology. So for instance burnt bones contain semi-destroyed collagen and fine-dispersed bone coal. Both components are completely available for the purpose of undistorted radiocarbon age determination, but semi-destroyed collagen is practically completely dissolved in acids, and fine-dispersed bone coal can be deposited only with the help of super-speed and lowefficient centrifugal machines.

Technology developed makes possible lithium carbide production from collagen or bone coal without preliminary deposition of them in a pure kind [Skripkin, Kovalyukh 1998]. The bones for this purpose are reduced to fragments, and after washing with trisodium phosphate solution they are processed by 1-3% hydrofluoric acid. This acid transforms carbonate and partly calcium phosphate into fluoride. Calcium fluoride does not dissolved practically in weak acids, but change of CO_3^{-2} and PO_4^{-3} volumetric anions for compact F leads to genesis of mineral matrix which is porous and cleaned from organic-silicate complex. Collagen in this case exists in semi-bound nonhydrated state. Essential advantage of hydrofluoric acid is its ability to dissolve silicates and humic acids as well as products of bacteria vital activity absorbed on them. It make it possible to remove introduced organic substances and carbonic carbon, to wash and dry the processed sample easy and qualitatively. The losses of bone organic substances or bone coal are minimum under such processing [Kovalyukh, Skripkin, Sobotovich 1996].

The sample subsequently is reduced to fragments and mixed with manganese dioxide for lithium carbide production by technology of "vacuum pyrolysis" *(the direct chemisorption into a lithium alloy of carbonaceous gases produced by the controlled thermal degradation of organic materials under vacuum)*.

This technology is based on combination of two processes: thermal breakdown of organic sample and chemical absorption of gaseous products by lithium. Reactor construction and elements of technology are shown on the figure 1. Synthesis is carried out within the reactor made of stainless steel with metallic lithium placed on the bottom, but the sample is seated inside the titanium glass. The glass with the sample is held at the optimum height in the tubular holder, which directs a gas flow for the melted lithium. Such location of lithium and carbonized sample permits

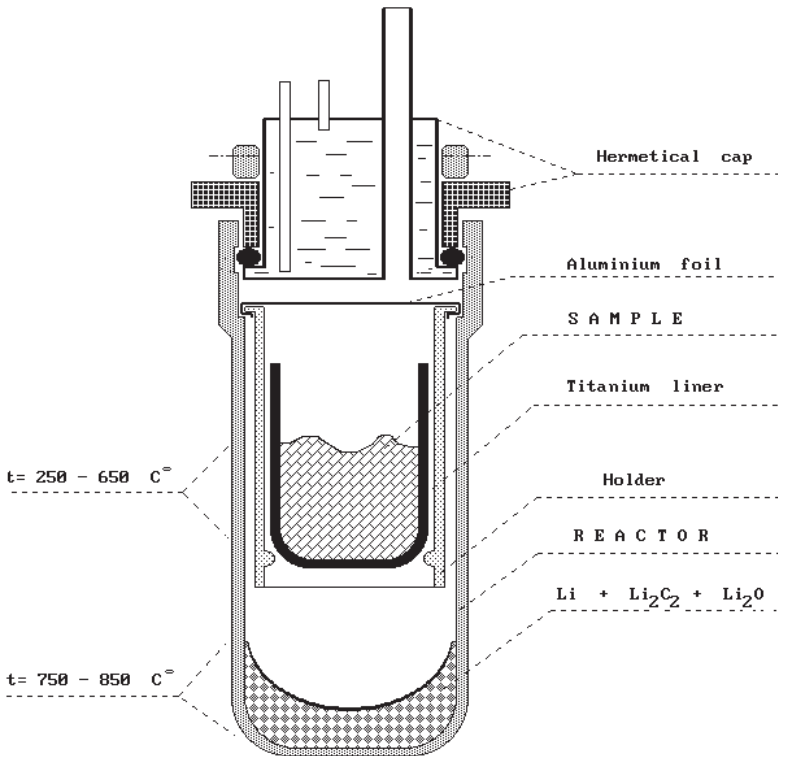

Fig. 1. Reactor construction and elements of technology

temperature regulation to be made within the area of thermal breakdown (and consequently gassing velocity) without temperature changing within the melted lithium area. Reactor is inserted into the stove at optimum depth, and is fixed in such a position. When temperature conditions are properly chosen a velocity of chemical absorption of thermal breakdown gaseous products by metallic lithium slightly exceeds a sample thermal breakdown velocity. The reactor pressure is stabilized at 0.1-0.2 atm. This promotes raising of thermal breakdown light - volatile products gassing and prevents condensation of these materials for reactor walls.

As a result of running processes the collagen is converted into a volatile organic combinations and into a bone coal. Addition of manganese dioxide plays an important role. When the temperature is above 550°C the manganese dioxide disintegrates with active oxygen liberation all over the volume of mixture. Oxygen liberation runs quietly, under the broad range of temperatures (550-940°C). Fine-dispersed bone coal therewith is oxidized till carbon oxide and dioxide, and in such a kind it is absorbed by melted metallic lithium. An essential feature of manganese oxides is

their ability to link phosphorus and sulphur in thermal stable combinations. This allows getting lithium carbide of high quality, and what is more - practically from the whole carbon content of bone organic substances. Formation of lithium carbide runs without complications with high output. Lithium oxide, hydride and nitride are also formed in parallels with carbide. Lithium hydride and nitride are completely destroyed under short reaction volume vacuuming at the end of process. The reactor of reduced volume (400 ml^3) was designed for fossil bones micro-samples that has made it possible to reduce greatly the losses on this stage.

Lithium carbide is subjected to hydrolysis, and gassing acetylene is converted into benzene on vanadium catalyst. Vacuum system for benzene syntheses is made from materials, which adsorb not at all acetylene and benzene. Internal volume of vacuum line has its minimum possible meaning. Constructive particularities mentioned above allow to reach 95-97% benzene output to the total exclusion of the memory effect. As a result of summation of new complex technology advantages the possibility appears for bone samples dating carrying-out with collagen total contents up to 250-300 mg.

The complex chemical technology of lithium carbide production from fossil bones samples had been tested comprehensively. The samples of different type bones as well as of their different preservation after their reducing to fragments and quartering were processed in parallels by both traditional and new methods. The results obtained were compared and analyzed. The samples age in parallel series coincided within the limits of instrumental error under measurement practically in overwhelming majority of cases. Moreover there was a success in dating some tests by only new method of chemical bones processing application. Samples with obviously denominated rejuvenating influence of humic acids were equally well cleaned from the last ones both by washingout with alkali or trisodium phosphate and by the action of hydrofluoric acid. Laborious process of the new complex approach and a temporal period spent for it was found 5-10 times less than for the traditional method of bone material radiocarbon analysis. The most typical results of two methods comparisons are shown in the Table 1.

Traditional technology of collagen deposition stipulates humic substances removing by 0.1% NaOH solution processing. As a rule, humic substances are of younger age with respect to collagen, and they are present within the bone samples in a kind of silicon oxide and soil organic material re-precipitated conglomerate. If used new technology the humic substances are removed from the bone samples together with silicates at the processing stage by hydrofluoric acid. Silicon oxide is completely dissolved in hydrofluoric acid solution, and humic substances precipitated on silicates form pseudo-solution.

One can see from comparative experiments carried out that new technology completely satisfies requirements for the samples purification from humic substances. Comparison was carried out for the samples of different preservation and ar-

Table 1

Comparative dating of fossil bone samples by the traditional technology and by "vacuum pyrolysis" method

No.	Sample		Lab. numb	Age ^{14}C, BP
1	Ordzhonikidze (excavations 1997), b.11, g.8	Traditional method	Ki-6827a	3890±50
		Vacuum pyrolysis	Ki-6827b	3910±45
2	Golovkovka, b.3, g.1	Traditional method	Ki-6718a	3905±55
		Vacuum pyrolysis	Ki-6718b	3920±60
3	Ordzhonikidze (excavations 1980), gr. Chorna Mogila, b.3, g.17	Traditional method	Ki-6553a	3710±60
		Vacuum pyrolysis	Ki-6553b	3745±50
4	Semionovka (excavations 1990-91), b.1	Traditional method	Ki-6688a	6800±60
		Vacuum pyrolysis	Ki-6688b	6980±65
5	O. Surskoy b.II (excavations 1946), sq.7	Traditional method	Ki-6691a	7230±55
		Vacuum pyrolysis	Ki-6691b	7245±60
6	Solonoe Ozero IV (excavations 1990)	Traditional method	Ki-6202a	12805±95
		Vacuum pyrolysis	Ki-6202b	12890±100
7	Novovladimirovka II	Traditional method	Ki-6203a	19290±85
		Vacuum pyrolysis	Ki-6203b	19340±95
8	Dmitrievka, Upper late Paleolithic layer of seat	Traditional method	Ki-5826a	16495±100
		Vacuum pyrolysis	Ki-5826b	16520±95

cheological age. If the samples come under the age period of 12-19 th. BP, «vacuum pyrolysis» method gives even more ancient results, that points to the fact of more complete and selective removing of introduced organic substances. The collagen losses are likely be reduced considerably when humic substances removing technology by hydrofluoric method of organo-silicate component dissolving is in use.

Measurement of benzene micro-samples is carried out into the specially developed micro-vials with the help of "Quantulus" - low-background spectrometer [Buzinny, Skripkin 1995]. Micro-vials are made of high pressure non-porous teflon. Holders for micro-vials are made of high purity titanium, and they are provided with by the screen to prevent "cross-talk" effect. Constructive particularities as well as the chosen materials enable high counting features to be get (Table 2).

As a result of biological processes, which have been going on within the system bone - soil microorganisms, the natural relation between three main carbon isotopes (^{12}C, ^{13}C and ^{14}C) experiences certain changes. These changes can be increased additionally during the process of carbon collagen form chemical transformations into benzene one.

Table 2

Counting features

Vial volume (ml)	Benzene loss per 24 hour (mg)	Bg (cmp)	^{14}C efficiency (%)	FM	FM (E2/BG)	t_{max} (year)	t_{max} (year)
0.85	less than 0.10	0.11	82	23.2	61127	48050	80

Table 3

Comparative dating of fossil bones samples macro and micro increments (Yamnaya culture)

No.	Sample		Lab. numb	Age ^{14}C, BP
9	Protopopovka, b.1, g.4	Macro	Ki-6733a	3945±50
		Micro	Ki-7130	3920±70
10	Protopopovka, b.2, g.3	Macro	Ki-6734a	3925±55
		Micro	Ki-7131	3910±60
11	Ordzhonikidze (excavations 1997), Shakhta 22, b.2 g.6	Macro	Ki-6833	3900±55
		Micro	Ki-7132	3930±70
12	Golovkovka, b.6, g.8	Macro	Ki-6719	3970±55
		Micro	Ki-7133	3960±60
13	Golovkovka, b.11, g.5.	Macro	Ki-6723a	4030±60
		Micro	Ki-7134	4035±60
14	Golovkovka, b.5, g.5	Macro	Ki-6731	4005±55
		Micro	Ki-7135	4020±70
15	Golovkovka, b.7, g.4	Macro	Ki-6722	3980±60
		Micro	Ki-7136	3940±70

In evaluating radiocarbon age the correction is being taken in account for biological isotopic fractionation. Undertaking such an operation is possible due to welldefined relationship between the deflection of ^{13}C isotope concentration and the degree of ^{14} isotope fractionation. Practically $\Delta^{14}C = (\delta^{13}C)^2$ equality is persisting. For this aim the determination is made on variation in concentrations of ^{13}C isotope in the ready benzene by mass-spectrometric method. This factor usually falls within the limits $-18.5 < \delta^{13}C < d^{13}C < -20.7$ promille. For such kind of samples, which had been under considerable effect of soil microorganisms, this factor can reach - 16 promiles, whereas together with chemical fractionation it may

even come to 14 promile. It means in practice that samples misrepresentation by age in something like BP=5000 years can reach 250 years. Correction entering for the isotopic fractionation is currently central for micro-samples, since such samples have as a rule a fine bone layer, being easy permeable for natural destroying agents.

The work carried out in our laboratory on Yamnaya culture archeological monuments dating is an inherent example of radiocarbon method applying for fossil bones micro-samples.

As one can see from the data mentioned above in the Table 3 the ages of the bones micro-samples derived are coincided within the limits of error in measurement with those ages derived from the same samples, taken in macro amounts. Small rejuvenation for the micro-tests is caused by comparatively most background effect stipulated by the cosmic radiation. These deviations can be taken into account subsequent to the complex comparative checking, including mathematical statistics are carried out.

Conclusions. Complex technology has been designed with the purpose of fossil bones micro-samples dating, which has shown reliability of high degree for resulting dating. All the stage of new technology had passed all-round inspection for the convergence of results with respect to the traditional methods. Represented technology allows getting reliable results for those kinds of samples, which could be not dated earlier by traditional method.

Translated by authors

ABBREVIATIONS

AO	– Arkheologicheskiye otkrytya, Moskva.
AJA	– American Journal of Archaeology, New York.
BPS	– Baltic-Pontic Studies, Poznań.
EA	– Eurasia Antiqua, Berlin.
FPP	– Folia Praehistorica Posnaniensia, Poznań.
KSIA	– Kratkiye soobshcheniya Instituta Arkheologii, Moskva.
KSIA AN USSR	– Kratkiye soobshcheniya Instituta Arkheologii AN USSR, Kiev.
KSIIMK	– Kratkiye soobshcheniya Instituta Istorii Materialnoy kultury, Moskva.
KSOGAM	– Kratkie Soobshcheniya Odesskogo Gosudarstvennogo Arkheologicheskogo Muzeya, Odessa.
MIA	– Materialy i issledovaniya po arkheologii, Moskva.
NA IA NANU	– Naukovy Arkhiv Instituta Arkheologii Nacionalnoi Akademii Nauk Ukrainu, Kiev.
SA	– Sovetskaya Arkheologia, Moskva.
SpA	– Sprawozdania Archeologiczne, Kraków.
ZFA	– Zeitschrift für Archäologie, Berlin.

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Vol.1: *Weapons of the Tribes of the Northern Pontic Zone in the 16th–10th Centuries B.C.*, by Viktor I. Klochko.
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Orders regarding B-PS should be adressed directly to the Editorial Office (Baltic-Pontic Studies, Institute of Prehistory, Św. Marcin 78, 61-809 Poznań, Poland).

The publications is carried out within the research project No. 1H01G05912 (in part No. 1H01G01810) financed in the years 1997–1999 by the Committee for Scientific Research and supplied with funds of the Ministry of National Education and Adam Mickiewicz University Foundation.

ISBN 83-86094-06-0
ISSN 1231-0344