

RADIOCARBON DATING OF PLANT REMNANTS IN QUATERNARY SEDIMENTS AT STARUNIA PALAEOLOGICAL SITE AND VICINITY (CARPATHIAN REGION, UKRAINE)

Tadeusz KUC¹, Kazimierz RÓŻAŃSKI¹, Tomasz GOSLAR^{2,3} & Renata STACHOWICZ-RYBKA⁴

¹ Faculty of Physics and Applied Computer Sciences, AGH University of Science and Technology, Al. Mickiewicza 30, 30-059 Kraków, Poland, e-mail: kuc@novell.ftj.agh.edu.pl

² Poznań Radiocarbon Laboratory, Foundation of the Adam Mickiewicz University, ul. Rubież 46, 61-612 Poznań, Poland, e-mail: goslar@radiocarbon.pl

³ Faculty of Physics, Adam Mickiewicz University, ul. Umultowska 85, 61-614 Poznań, Poland

⁴ Polish Academy of Sciences, W. Szafer Institute of Botany, ul. Lubicz 46, 31-512 Kraków, Poland, e-mail: r.stachowicz@botany.pl

Kuc, T., Różański, K., Goslar, T. & Stachowicz-Rybka, R., 2009. Radiocarbon dating of plant remnants in Quaternary sediments at Starunia palaeontological site and vicinity (Carpathian region, Ukraine). *Annales Societatis Geologorum Poloniae*, 79: 289–296.

Abstract: Radiocarbon dating applied on several occasions in the past to date animal bones and animal soft tissue originating from the area of the Starunia palaeontological site turned out to be very controversial. Although investigations of Pleistocene flora in the Starunia area go back to the beginning of the 20th century, no published ¹⁴C dates of such material are available to date. Sixteen boreholes drilled in the area of the Pleistocene mammals discoveries, in the framework of a multidisciplinary research project (2006–2009), have been selected for radiocarbon dating of plant macrofossils. Moreover, five samples of plant remnants extracted from two outcrops in the area were ¹⁴C-dated. The nature of the dated plant material, in some cases soaked with oil, posed specific methodological problems. Although applied chemical treatment of macrofossil samples led to complete removal of contaminating hydrocarbons in the case of small pieces, some ageing effects in terms of radiocarbon dating cannot be completely ruled out. Radiocarbon ages of macrofossil samples originating from the close neighbourhood of the location, where the “second” woolly rhinoceros was found at a depth of 12.5 m, suggest that the minimum age of sediments in which the Pleistocene mammals were found is in the range of ca. 35–40 ka BP. A broad consistency between palynological reconstruction of Younger Dryas/Preboreal boundary and the corresponding radiocarbon ages of macrofossils is observed for locations, where the natural sedimentary sequence for late Glacial and early Holocene was not disturbed by extensive mining activity in the area.

Key words: radiocarbon dating, AMS dating, woolly rhinoceros, Starunia, Carpathian region, Ukraine.

Manuscript received 14 May 2009, accepted 18 September 2009

INTRODUCTION

Radiocarbon dating of plant remnants in Quaternary sediments in the Velyky Lukavets River valley (Starunia, SW Ukraine) was a part of a multidisciplinary research project carried out between 2006 and 2009 and focused on thorough geological, geophysical, and palaeontological investigation of an abandoned earth wax (ozokerite) mine (Kotarba, 2009). Starunia, located about 130 kilometres southeast of Lviv, Ukraine (Fig. 1), is the place where remnants of a mammoth and woolly rhinoceros were discovered in 1907. Furthermore, one nearly completely preserved rhinoceros carcass (called “second woolly rhinoceros”) and remnants of two other woolly rhinoceroses were found in

1929. This discovery in 1929 of a large Pleistocene mammal in the Starunia ozokerite mine was a spectacular scientific event on a world scale. A unique combination of oil and brine within the Pleistocene clayey mud, into which the animal had sunk, resulted in near perfect preservation of this specimen (Kotarba *et al.*, 2009).

Fossil flora from the Starunia site (SW Ukraine) was the subject of numerous scientific investigations aimed at identification and reconstruction of ecological conditions in the area, mainly in connection with findings of Pleistocene mammals (Bayger *et al.*, 1914; Hoyer, 1914; Szafer, 1930; Szafran, 1934; Granoszewski, 2002; Pawłowski, 2003; Ko-

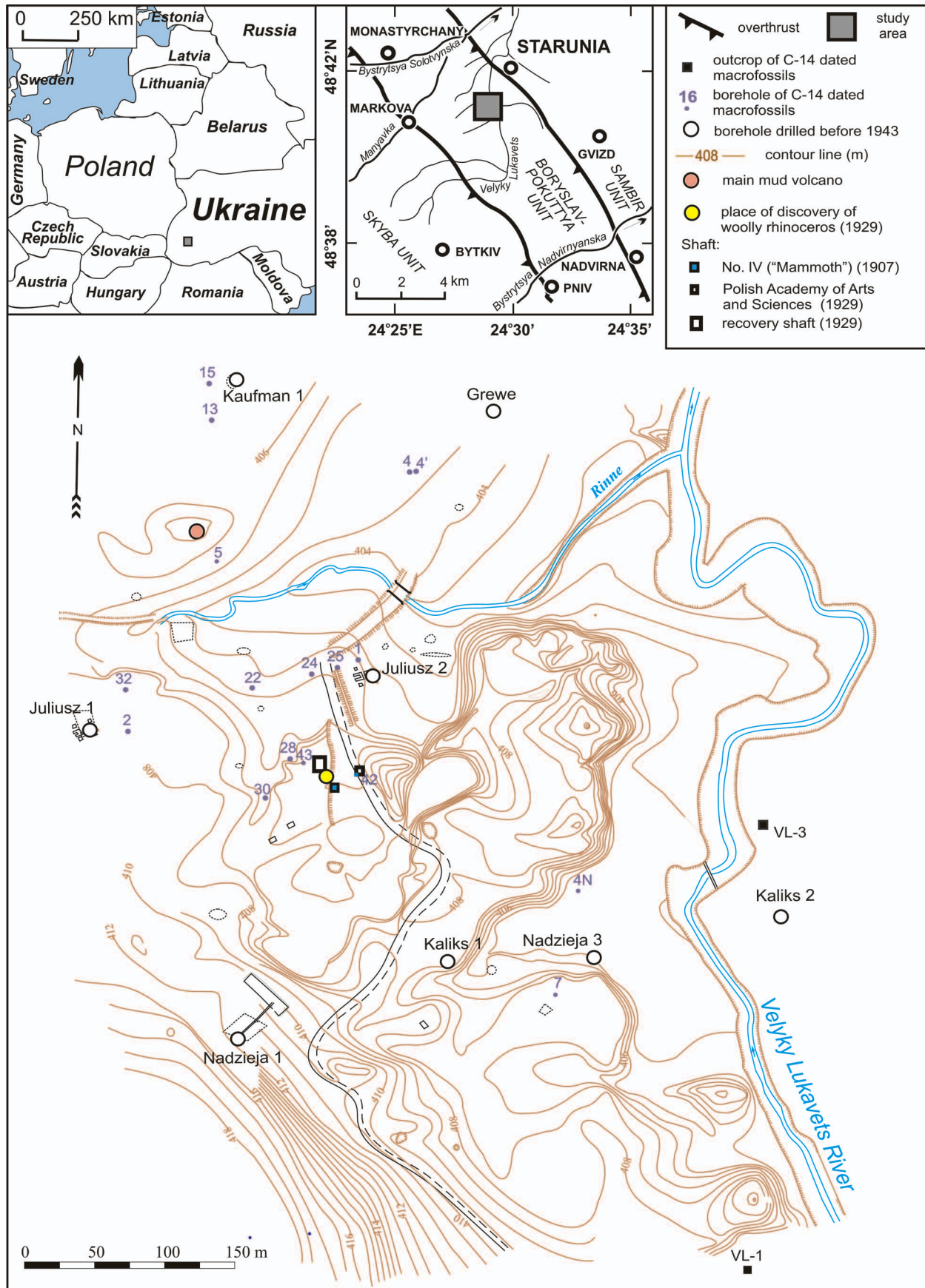


Fig. 1. Sketch map of the Starunia palaeontological site and surrounding area (Carpathian region, Ukraine) showing location of boreholes, from which extracted microfossils were radiocarbon dated

tarba, 2005; Kubiak & Drygant, 2005). Development of industrial activity in the Eastern Carpathians (saltworks and saline brines) goes back to the Middle Ages. In the Starunia area, saltworks operated between the 15th and 18th centuries (Alexandrowicz, 2004). Rich deposits of ozokerite discovered in this area stimulated fast growth of a mining industry in the 19th century and during the first decades of the 20th century. Afterwards, the exploitation was abandoned and industrial infrastructure disappeared.

Numerous radiocarbon dates of bones and animal soft tissue of Pleistocene mammals obtained in the past turned to be controversial what makes the true age of the "second" rhinoceros still unknown. Investigations undertaken, including radiocarbon dating, aimed at delivery of new information on the age of Pleistocene deposits, in which mammals carcasses were found. Correlation between ^{14}C data and a parallel palynological study enabled building depth distributions of most probable ages of sediments in the area. These studies around the Starunia palaeontological site are part of comprehensive, interdisciplinary investigations (Kotarba, 2009). The main goal of the undertaken works is to define and recognise the area, where the highest probability of finding undiscovered remnants of large Pleistocene mammals exists, and to provide future explorers with necessary knowledge.

GEOLOGICAL SETTING

Interdisciplinary studies performed in Starunia between 2006 and 2009 (Kotarba, 2009) were confined to the area of an abandoned mine of earth wax, extending over a surface of *ca.* 10.5 ha in the valley of Velyky Lukavets River. The study area belongs to the foreland of the Eastern Carpathians, within Gorgany Fore-Carpathians, in the Boryslav-Pokuttya Unit (Carpathian Foredeep Basin). The Pleistocene and Holocene deposits in the Velyky Lukavets River valley are resting on the Lower Miocene Vorotyshcha salt-bearing beds, underlain by the Upper Cretaceous–Lower Miocene flysch succession, in the upper part of which black shales, siltstones, sandstones and hornstones of the Menilite beds can be distinguished.

The Vorotyshcha beds (thickness up to 2000 m) are made up of salt-cemented sandy-clayey breccia with intercalations of sandstones and shales, among which layers, lenses or veins of marls, gypsum, anhydrite, rock and potash salts, and ozokerite are observed (Mitura, 1944; Korin, 2005; Koltun *et al.*, 2005). A completely preserved carcass of woolly rhinoceros found in 1929 in fine-grained Pleistocene sediments was impregnated with brine and oil coming from the Vorotyshcha beds. Surface appearances of saturated salt brine and oil, together with mud volcanoes, are observed also nowadays in the area (Kotarba *et al.*, 2005).

MATERIALS AND METHODS

Three drilling field campaigns were performed: in May and October 2007 and in May 2008. From 16 boreholes drilled at distances no exceeding 210 m from shaft No. IV

(called "Mammoth") and the location of the rhinoceroses finding, core samples for radiocarbon dating of plant macrofossils have been selected (Fig. 1). Apart from the material from boreholes, 5 samples of plant remnants extracted from two outcrops were ^{14}C -dated. The investigated outcrops are located close to the Velyky Lukavets River, while the analysed core samples were obtained from boreholes in the area between the Velyky Lukavets River and the Rinne Stream, and north of it. The majority of them are located at less than 100 m distance from the place of palaeontological finds, and three of them (boreholes Nos 28, 42 and 34) at a distance of *ca.* 25 m. Exact positions of the sampled boreholes and outcrops are shown in Fig. 1. Samples for radiocarbon dating were extracted from different depth intervals of the selected boreholes, generally between 1 and 10 m depth (Stachowicz-Rybka *et al.*, 2009a, b).

Plant macrofossils were chosen as a basic material for ^{14}C age determination, mostly because they were relatively easy to separate and usually occurred in sufficient quantities. Individual macrofossils after visual identification were picked out by hand from the bulk core sample. Standard separation procedure was as follows: sample of core material (*ca.* 20 cm³) was washed with distilled water (*ca.* 100 cm³) in a glass pan and identified plant fragments, mostly vegetative, were picked out by tweezers. A mixture of organic fragments obtained in that way differed from sample to sample. Some of the separated macrofossils were typical for remnants of small green plants, *e.g.* grass, sedge, while others consisted mainly of broken pieces of sprigs or small branches, resembling material deposited by water (see Fig. 2A). Also chips of wood originating from bigger fragments, small pieces of bark or even tiny parts of scales and cuticle could be identified (Fig. 2B). More details on botanical characteristics of the extracted plant material are presented elsewhere (Stachowicz-Rybka *et al.*, 2009a).

Chemical treatment of the separated macrofossils was aimed at removal of contaminants, first of all of hydrocarbon origin. Investigations carried up to date and available historical data document surface appearances of hydrocarbons in the area (Kotarba, 2002; Kotarba *et al.*, 2005; Kuc *et al.*, 2005). As hydrocarbons are of infinite radiocarbon age, their possible interactions with organic material in the sediments pose serious problems for radiocarbon dating. The adopted cleaning procedure is schematically presented in Table 1. After initial rinsing with distilled water, the separated macrofossils were oven-dried and left overnight in CH_2Cl_2 . Then they were rinsed 3–4 times in C_6H_6 until colour disappeared, followed by rinsing in acetone for *ca.* 5 min, washed in distilled water and finally oven-dried at 120°C. Individual masses of the macrofossil samples delivered to the Accelerator Mass Spectrometry (AMS) laboratory (Goslar *et al.*, 2004) did not exceed 150 mg.

In the AMS laboratory, the samples were checked and mechanically cleaned under binocular microscope. It is worth to mention that the largest piece of wood analysed (sample 2/4.20), revealed the presence of hydrocarbons in its inner part (Fig. 3) even after the cleaning steps described above, so for the further treatment, only much smaller pieces of the plant remnants were taken. The selected samples were then treated with 4% HCl (room temperature,

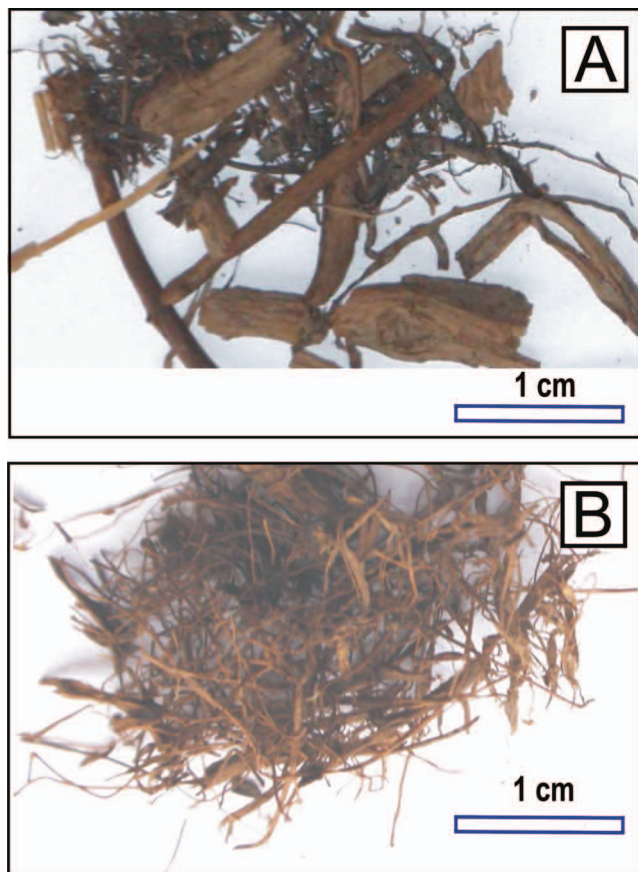


Fig. 2. Examples of macrofossils (A) sample No. 2/4.20 consisting of different plant remnants and (B) sample No. 22/4.80 composed mostly of thin fragments of green plant tissue remnants. Photo by T. Kuc

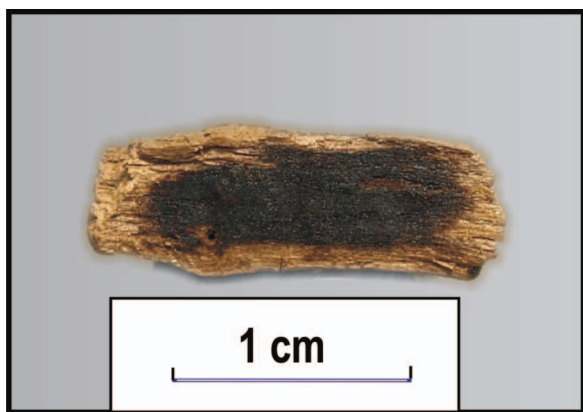


Fig. 3. Cross-section of large macrofossil from sample No. 2/4.20 with black inner part still containing hydrocarbons. Photo by T. Goslar

12h), 0.5M NaOH (60°C, up to 2h), and again 4% HCl (room temperature, 2h). Combustion to CO₂ was performed in a closed quartz tube together with CuO, and Ag wool in 900°C. Obtained CO₂ sample was then reduced with H₂ over Fe powder as a catalyst (*ca.* 2 mg) and the resulting carbon/iron mixture was pressed into a pellet in the target holder (Czernik & Goslar, 2001).

Table 1

Preparation procedure of macrofossils for the AMS dating

No.	Step	Parameters
1	Hand separation of plant remnants from bulk sample	Bulk material (<i>ca.</i> 20 cm ³) digested in 100 cm ³ distilled water, picking out visible fragments
2	Initial washing	Distilled water, room temperature
3	Drying	100°C, several hours
4	CH ₂ Cl ₂ treatment	100 ml, room temperature, overnight
5	Rinsing with C ₆ H ₆	3-4 times, 5 min., room temperature, till colourless aliquot
6	Rinsing with acetone	100ml, 5 min., room temperature
7	Washing in distilled water	2 times, 100 ml
8	Oven drying	120°C, overnight
9	Visual checking, selection best material	Binocular (magnifying lens)
10	AAA treatment	HCl (4%, 20°C, 12h), NaOH (0.5M, 60°C, up to 2h), HCl (4%, 20°C, 2h)
11	Combustion to CO ₂	CuO + Ag wool, 900°C
12	Reduction to graphite	H ₂ + Fe (catalyst)
13	Formation of pellet	Graphite/Fe mixture pressed into target holder

Abbreviations: AMS – Accelerator Mass Spectrometry; AAA – acid, alkali, acid applied consecutively

Radiocarbon concentration in the samples was measured in the mass spectrometer “Compact Carbon AMS” (Goslar *et al.*, 2004) by comparing the simultaneously collected ¹⁴C, ¹³C and ¹²C beams with those of Oxalic Acid standard CO₂ and coal background material. Conventional ¹⁴C ages were calculated with δ¹³C correction for isotopic fractionation (according to Stuiver & Polach, 1997), based on the ¹³C/¹²C ratio measured by the AMS-system simultaneously with the ¹⁴C/¹²C ratio. For determination of the measuring uncertainty (standard deviation) both the counting statistics of the ¹⁴C measurement and the variability of the intermediate results were observed, and the larger of the two values was adopted as the measurement uncertainty. To this we added the uncertainty connected with the subtraction of the background. The quoted 1-sigma uncertainty (*cf.* Table 1) is thus our best estimate for the overall uncertainty of the measurement, not based just on counting statistics alone.

The results of ¹⁴C measurements are presented in Table 2 in the form of conventional radiocarbon ages (Stuiver & Polach, 1997).

RESULTS AND DISCUSSION

Radiocarbon dating was applied on several occasions in the past to date animal bones and animal soft tissue originating from palaeontological finds in the Starunia area. The obtained radiocarbon ages turned out to be highly controversial (Kuc *et al.*, 2005). Although investigations of Pleistocene flora in the Starunia area go back to the beginning of the 20th century (Szafer, 1930; Szafran, 1934; Kucowa,

Table 2

Radiocarbon dates of plant macrofossils

No.	Sample Code	Description		¹⁴ C yr BP (1 σ)	Remarks
		Borehole/outcrop	Depth (m)		
1	1/1.80	1	1.80	9740 ± 50	***
2	2/4.20	2	4.20	21100 ± 100	***
3	4/3.50	4	3.50	12230 ± 70	*
4	4'/0.95	4'	0.95	3655 ± 35	
5	4'/1.30	4'	1.30	625 ± 30	
6	4'/2.35	4'	2.35	10190 ± 50	
7	4'/4.65	4'	4.65	8460 ± 50	*
8	4N/2.20	4N	2.20	1505 ± 30	
9	5/5.60	5	5.60	3915 ± 35	
10	7/5.70	7	5.70	210 ± 30	*
11	7/10.8	7	10.8	390 ± 30	*
12	13/2.90	13	2.90	13010 ± 60	***
13	15/3.60	15	3.60	240 ± 30	
14	15/4.30	15	4.30 - 4.40	6160 ± 40	
15	22/1.70	22	1.70	12240 ± 60	
16	22/4.80	22	4.80	43100 ± 1100	***
17	22/5.80	22	5.80 - 5.90	40000 ± 700	
18	24/2.10	24	2.10	13690 ± 70	**
19	25/1.70	25	1.70	9550 ± 50	*
20	28/1.00	28	1.00	325 ± 30	*
21	28/2.50	28	2.50	26850 ± 200	*
22	28/4.00	28	4.00	34000 ± 500	*
23	28/6.0	28	6.0	33250 ± 300	*
24	30/2.50	30	2.50	5490 ± 40	***
25	30/6.80	30	6.80	48200 ± 1800	
26	32/4.70	32	4.70	2915 ± 30	
27	42/3.90	42	3.90	230 ± 30	
28	42/9.50	42	9.50 - 9.60	16260 ± 80	
29	42/9.70	42	9.70 - 9.80	22900 ± 150	
30	43/5.35	43	5.35	40 ± 30	
31	43/8.90	43	8.90 - 8.95	modern	
32	VL-1/1.00	VL-1	1.00 - 1.05	4505 ± 35	
33	VL-1/2.20	VL-1	~ 2.20	11110 ± 60	
34	VL-1/2.30	VL-3	~ 2.30	11430 ± 60	
35	VL-3/0.05	VL-3	0.05 - 0.10	1275 ± 30	
36	VL-3/1.20	VL-3	1.20 - 1.25	125 ± 30	

BP: before present (i.e., before 1950)

Content of hydrocarbons in CH₂Cl₂ washed out from macrofossils: (***) – very much; (**) – medium-level; (*) – very small; without asterisk – not noticeable

1954; Granoszewski, 2002), no published ¹⁴C dates of such material are available to date.

The history of the area, with extensive mining activities in the past and resulting possible modifications of the sedimentary sequence, make the current attempts to establish undisturbed chronostratigraphy of the Quaternary cover in the Starunia area rather difficult. For instance, samples retrieved from borehole No. 43, which is located very close to the shaft of 1929, represents typical remnants of mining activities. The ¹⁴C age of macrofossils found at a depth of ca. 9 m in this borehole is modern, strongly suggesting relatively recent earth works of re-cultivation after old mining exploitation. Stratigraphy of Quaternary deposits in the Starunia area is presented in detail elsewhere (Sokołowski *et al.*, 2009; Sokołowski & Stachowicz-Rybka, 2009). Here, only selected boreholes are discussed from the perspective of radiocarbon ages obtained in the course of this study.

Young radiocarbon ages of some hundred years for macrofossils extracted from depths less than ca. 4 metres from boreholes Nos 4' (1.3 m), 15 (3.6 m), and 42 (3.9 m) are the result either of reworking due to mining activities or of incomplete removal of deep penetrating roots of young herbaceous plants growing there. Consequently, these dates should be discarded from further interpretation. Similarly, the age of 125 years BP from the outcrop VL-3 (depth: 1.20–1.25 m) is considered too young, most probably due to contamination with recent root remnants.

Macrofossil sample 7/5.70, originating from borehole No. 7 and collected ca. 50 cm below a 5.2-m-thick layer of mixed material typical for mine dump, reveals the age of 210 years BP corresponding to the calibrated age of 1760–1800 AD. Apparently, it represents mining activity in the past. The age of macrofossils equal 390 years BP (calibrated age: 1440 – 1520 AD) at a depth of 10.8 m in sample No. 7/10.8 should be also considered as a result of human activity in the area, although in this particular case there was no clear evidence of anthropogenic influences at the depth of the dated sample (Sokołowski *et al.*, 2009; Sokołowski & Stachowicz-Rybka, 2009).

Boreholes Nos 28, 30, and 42 surround the location, where the “second” rhinoceros was found at a depth of ca. 12.5 m (Alexandrowicz, 2004). The macrofossil extracted from borehole No. 30 at a depth of 6.8 m revealed the oldest radiocarbon age (ca. 48 ka BP), while in logs of two other boreholes Nos 28 and 42 the maximum ages found were on the order of 33 ka BP, and 23 ka BP, respectively (Table 2). The Miocene strata occur in these boreholes below the dated horizons (*cf.* Fig. 4). The spot where the “second” rhinoceros was found is located between boreholes Nos 42 and 30 (Fig. 1). Linear interpolation between the highest ages found in these two boreholes yields the age range of ca. 35–40 ka BP as approximate minimum age of the sediments in the exact location, where the second rhinoceros was found.

The group of boreholes Nos 1, 25, 24 and 22, located to the north of the 1929 recovery shaft, situated very close to each other, reveal consistent radiocarbon ages of microfossils (9.55 ka BP, 13.69 ka BP, 9.24 ka BP, ca. 40 ka BP) retrieved from the sediment horizon (1.5, 2.0, 1.9, and 0.5, re-

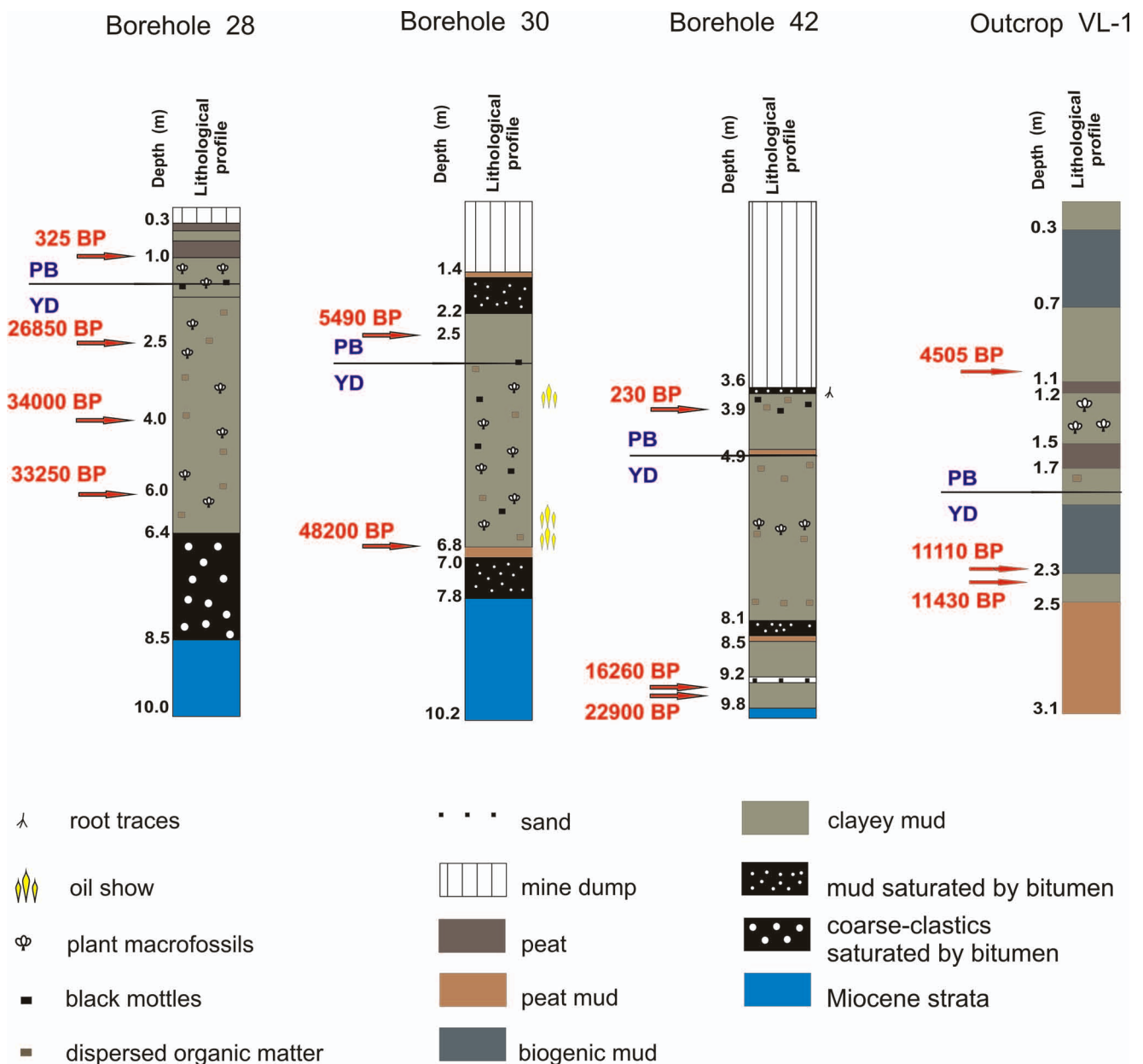


Fig. 4. Selected lithological profiles of boreholes Nos 28, 30 and 42, and outcrop VL-1 located in the Starunia palaeontological site. Results of radiocarbon dating of plant remnants are expressed as conventional age. BP – before present (*i.e.*, before 1950), YD – Younger Dryas, PB – Preboreal

spectively) above the Miocene level. Borehole No. 2, located *ca.* 100 metres in the SW direction at a different morphological position, shows at a depth of *ca.* 0.1 m above the Miocene strata a radiocarbon age equal 21.1 ka BP. It is worth pointing out that macrofossils from boreholes Nos 1 and 2 revealed substantially higher content of hydrocarbons than the remaining samples in this group.

Age distribution of macrofossils extracted from boreholes Nos 4, and 4' located *ca.* 150 metres NE of the 1929 recovery shaft reveals two age inversions: at depths of *ca.* 1.3 and 4.65 m, respectively. The only plausible explanation of these inversions seems to be an admixture of younger material during sampling. A similar situation of too young a macrofossil age occurs in borehole No. 15, where the sam-

ple originating from a depth of 3.6 m has an age of 240 years BP, while only *ca.* 75 cm deeper (sample 15/4.30) the radiocarbon age rapidly increases to 6,160 years BP.

Palynological profiles constructed for some boreholes (Stachowicz-Rybka *et al.*, 2009a) with a well-defined Younger Dryas horizon allow us to place the YD/PB transition on the time scale. The youngest ages of samples belonging to the YD horizon are: 10,190 years BP (sample 4'/2.35), 12,240 years BP (sample 22/1.70) and 11,110 years BP (sample VL-1/2.20). The above mentioned samples were situated just below the palynologically defined YD/PB transition: 5, 40 and 60 cm, respectively. This distribution demonstrates broad consistency of palynological reconstruction with the obtained radiocarbon ages of macro-

fossils for locations, where the natural sedimentary sequence at the transition from the Late Glacial to the Holocene was preserved.

CONCLUSIONS

Thirty-six radiocarbon dates of plant macrofossils retrieved from logs of 16 boreholes and two outcrops in the area of Starunia (SW Ukraine) allowed a unique insight into the age structure of Quaternary sediment cover directly at the site of famous palaeontological findings of Pleistocene mammals at the beginning of the 20th century. The obtained radiocarbon ages generally confirm the rich mining history of the region, which resulted in heavy disturbances of the natural sedimentary sequence in many places of the study area.

The peculiar nature of the dated plant material, in some cases soaked with oil, posed specific methodological problems for radiocarbon dating. Although chemical treatment of macrofossil samples aiming at complete removal of contaminating hydrocarbons from the plant tissue leads to satisfactory results in the case of small pieces of macrofossils, some ageing effects in terms of radiocarbon dating cannot be completely ruled out.

Radiocarbon ages of microfossil samples originating from the close neighbourhood of the location where the "second" woolly rhinoceros was found at a depth of 12.5 m suggest that the minimum age of sediments, in which the mammals were found, is in the range of ca. 35–40 ka BP.

Broad consistency between palynological reconstructions of the Younger Dryas/Preboreal boundary and the corresponding radiocarbon ages of macrofossils were found for locations, where the natural sedimentary sequence for the Late Glacial and early Holocene was not disturbed.

Acknowledgements

Financial support from the Ministry of Science and Higher Education (project No. 139/UKR/2006/01) is kindly acknowledged. Review comments and suggestions by Teresa Madeyska of the Polish Academy of Sciences in Warsaw and Adam Walanus of the AGH University of Science and Technology in Kraków were very helpful. Authors would like to thank Mrs. Joanna Gawęda and Mr. Sebastian Ptak for their perfect technical assistance at drawing profiles.

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