

Andrew Lawler BA (Hons) Cantab  
Archaeologist, London, UK

S3 – BT9

## AMS RADIOCARBON DATING OF A WOOD SAMPLE FROM TUNNEL RAVNE: METHODS, RESULTS AND IMPLICATIONS FOR FURTHER RESEARCH

### SUMMARY

This paper discusses the discovery of a piece of wood discovered embedded in the sediments of the tunnel Ravne complex, Visoko, Bosnia and Herzegovina. The wood was sampled, and sent for Radiocarbon analysis to two independent establishments. Below the procedures involved in sampling, and a brief outline of the methodologies and principles applied in the AMS  $^{14}\text{C}$  dating are detailed, as well as an appraisal of implications for further work and directions of research regarding the tunnel system.

#### Key words:

### 1. INTRODUCTION

During work on Tunel Ravne November 2007, workers discovered a dark, soft compressible material preserved embedded within the sides of the tunnel, at a height of 1.4 m, during the clearance of debris and installation of wooden support beams to facilitate visitor access. Initial examination on 12<sup>th</sup> November by employees of the Foundation led to the assumption that the material was organic in nature, and potentially capable of yielding dating evidence. The Permanent Archaeologist was alerted to this, and work in the immediate vicinity was temporarily halted.

Originally, the material measured approximately 35 cm in length and 8 cm in width, with an unknown depth (now estimated to have been approximately 12 cm), but a small collapse prior to 10<sup>th</sup> December reduced its width by approximately 8 cm.

On 15<sup>th</sup> January 2008, the Permanent Archaeologist took a small sub-sample of the material, and confirmed it to be wood. After discussion with directors of the Foundation, it was decided that this wood should be sampled, and C-14 analysis of the samples should be undertaken by independent institutions, in order to determine its age.



Figure 1 - The wood in situ with label

## 2. SAMPLING

On 29<sup>th</sup> January 2008, the Permanent Archaeologist, along with an assistant, photographed the wood in situ, and removed 4 samples (<5a>, <5b>, <5c>, <5d>), each weighing approximately 5 g. These were placed in labelled polythene bags, in accordance with guidance given in a telephone conversation with a member of staff at the Research Laboratory for Archaeology and the History of Art, University of Oxford.

After removal, a label was embedded into the sediments, denoting the location and date of sample removal, alongside the index number. The samples were then taken and stored in a refrigerator at 4°C, whilst the institutions to carry out the analyses were selected.

*Figure 2 - The four samples*

Eventually, it was decided that two separate AMS laboratories were to perform the analysis. Those chosen were the Research Laboratory for Archaeology and the History of Art, University of Oxford, UK, and the Leibniz-Laboratory for Radiometric Dating and Stable Isotope Research, of Christian-Albrechts University, Kiel, Germany. After completing all necessary paperwork and data sheets, these samples were packed in protective envelopes, and sent by recorded delivery on 26<sup>th</sup> February, 2008.



*Figure 3 - Oxford sample and data sheets ready to be sent*

Notification that the samples were delivered to the relevant institutions was received by the Foundation on the 5<sup>th</sup> and 7<sup>th</sup> of March from Oxford and Kiel respectively.

## 3. RESULTS FROM KIEL

On 6<sup>th</sup> May 2008 the Foundation received the results of the analysis undertaken by the Leibniz-Laboratory for Radiometric Dating and Stable Isotope Research, at Christian-Albrechts University, Kiel, via e-mail. Below is a summary of them:

*Table 1 - Summary of <sup>14</sup>C AMS Results*

Fraction	Corrected pMC†	Conventional Age	δ <sup>13</sup> C(‰)‡
Wood, alkali residue, 1.2 mg C	2.22 ± 0.14	30 600 + 540 / - 510 BP	- 26.47 ± 0.08

## 4. PREPARATION, METHODOLOGY AND CALCULATION

After receipt, the wood sample was checked under a microscope and all material suitable for dating was selected. The demineralisation process consisted of processing through solutions of 1 % HCl, 1 % NaOH at 60°C and again 1 % HCl (to eradicate any alkaline residue). The combustion of the demineralised material into CO<sub>2</sub> was performed in a closed quartz tube together with CuO and silver wool at temperature of 900 C. The sample CO<sub>2</sub> was reduced with H<sub>2</sub> over about 2 mg of Fe powder as catalyst, and the resulting carbon/iron mixture was pressed into a pellet in the target holder, ready for AMS analysis. Alongside sample 5C, samples of the international isotopic standards used in AMS <sup>14</sup>C – dating, being reduced CO<sub>2</sub> derived from oxalic acid (for ‘present day’ levels) and from background coal (considered to be radiocarbon ‘dead’) were also included in the particular AMS ‘run’, for comparison.

#### **4.1. Equipment**

The AMS system employed by the Leibniz-Laboratory for Radiometric Dating and Stable Isotope Research is a HVE 3 Million Volt Tandemron 4130 AMS system, equipped with a single caesium sputter ion source.

#### **4.2. Methodology**

The AMS methodology achieves accurate and reliable <sup>14</sup>C dates by measuring the ratios of different carbon isotopes through the use of a high-powered electromagnet to separate single ionised atoms in a gaseous state according to atomic mass. These are deflected by the electromagnets into separate ‘chambers’, where their high-velocity impacts are counted individually. The fact that atom impacts are individually counted allows the accurate measurement of carbon isotope ratios from relatively low amounts of organic material (around 20 mg is the generally accepted minimal amount). In the case of <sup>14</sup>C AMS dating, the isotopes studied are those of all three carbon isotopes (<sup>12</sup>C, <sup>13</sup>C, <sup>14</sup>C).

In nature, Nitrogen with an atomic mass of 14 also occurs naturally (as well as several <sup>12</sup>C and <sup>13</sup>C - bearing molecular ions). To eradicate the risk of inaccuracies through the accidental measurement of <sup>14</sup>N (which would give a much younger date, as particles with an atomic mass of 14 would be much more abundant in results), the gaseous samples are initially run in a negative ion beam, as N - ions are not stable. The AMS machine’s counters are then set to measure atomic masses of 12, 13, and 14, by adjusting the strengths of the electromagnets, and the individual ions are propelled into their relevant counters.

After the ions have been propelled into the counters and counted, the ratio of the three carbon isotopes is then calculated in order to calculate the age of the material studied.