AMS AND RADIOMETRIC DATING OF AN ETRUSCAN LINEN BOOK AND ASSOCIATED MUMMY

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ABSTRACT. An important Etruscan linen "book," the *Liber linteus Zagrabiensis*, was preserved in wrappings of an Egyptian mummy. Stylistic estimates for the date of composition of the text vary. Three possible centuries were suggested, the 3rd and the 1st centuries BC and the 1st century AD. Radiometric and AMS dating of the linen book and the mummy has demonstrated multiple uses for differing aged materials. There seem to be at least two sets of linen wrappings of markedly contrasting ages, while separate fractions of the embalming unguent seemed to contain carbon of differing dates. ¹⁴C results suggest the most probable age range for the linen book is ca 360–210 cal BC, making the 3rd century BC stylistic date the most likely time of inscription.

INTRODUCTION

The Archaeological Museum in Zagreb has in its collections a linen "book" known as the *Liber linteus Zagrabiensis*. This liturgical calendar, with over 1200 words, represents the longest known Etruscan inscription. Although its origin is uncertain, the book eventually was used to wrap the body of a deceased Egyptian female (Mirnik & Rendić-Miočević, 1986). Estimates as to when the book was cut into long pieces to wrap the mummy vary considerably; most researchers favor a date between the 1st century BC and the 1st century AD. Estimates of the age of the book are based on the style of writing, but there is no agreement, since at least three possible dates have been suggested: the 3rd century BC, the 1st century BC, and the 1st century AD (Roncalli, 1980a, b). Thus, we decided to radiocarbon date the book and the mummy in an attempt to resolve this problem.

Since it was undesirable to directly date this valuable Etruscan manuscript by conventional methods because of the large sample needed, accelerator mass spectrometry (AMS) was applied to a much smaller sample (~10mg, ~1cm² of textile). Other associated datable materials from the mummy included the human tissue from the corpse, the embalming unguent (thought to consist of resins and balms extracted from a number of plant genera) and fragments of leaves. In some cases there was sufficient material for radiometric ¹⁴C dates to be obtained; these were made at the Ruder Bošković Institute in Zagreb and at the Center for Isotopic Research, University of Groningen. A series of AMS dates were also undertaken at the Radiocarbon Accelerator Unit in Oxford to date the body and participate in inter-laboratory comparison.

CHEMICAL ANALYSIS OF THE DATED MATERIAL

Before the different materials were pretreated for dating, a series of analyses were undertaken by Z Sliepčević of INA Petroleum Co to investigate the chemical composition of the materials and to check for potential contaminants (Srdoč & Horvatinčić, 1986, p 84–88). These analyses revealed red and black paint on the linen, which included such components as mercury sulphide and "ivory black" (charred ivory). We avoided these complications by sampling only from untreated areas.

We observed that some of the leaves, which had been sewn to form a wreath, appeared stained or painted. X-ray fluorescence (XRF) analysis showed that no paint containing lead, mercury or iron oxides was present and so we presumed an organic dye had been applied.

The embalming unguent was interesting in that, at one point, some of it had been softened by high temperature which caused a molten fraction to exude out of the matrix to form an asphalt-like substance. The original, non-exuded, unguent was dull grayish-black and very brittle. Upon extraction using CHCl₃, the insoluble unguent fraction disintegrated into small fragments, <1–2mm in diameter, which seemed to be composed of charred and crushed twigs, leaves and seeds of various plants. The percentage carbon content was 43%. Infra-red (IR) spectroscopy of the CHCl₃ insoluble fraction revealed mostly inorganic substances. The soluble fraction contained a mixture of organic compounds, characterized by –COOH and –COOR (aliphatic and esther) functional groups, and had a higher carbon content, ranging from 66–74%. The exuded unguent compound gave an almost identical spectrum to the soluble fraction.

PREPARATION OF SAMPLES FOR RADIOMETRIC DATING

The unguent and leaves were prepared for ¹⁴C dating by the Zagreb radiocarbon laboratory using their standard pretreatment method, namely 4% HCl, 4% NaOH at 80°C overnight, boiling in H₂O until neutral and dry. The linen was given a milder treatment, with the alkali wash being confined to 2 hr rather than overnight. All the samples were then combusted to CO₂, catalytically converted to CH₄, purified and stored for 14 days before being counted for ca 24 hr twice in a proportional counter.

The Groningen dates were obtained using the pretreatment procedures of Mook and Streurman (1983), except that the linen, because of its fragility, was given a milder pretreatment with only hot distilled water.

PREPARATION OF SAMPLES FOR AMS DATING

We knew that the mummy tissue had been soaked in kerosene in the last century to preserve the body. Thus, this sample was washed with CHCl₃ to remove the residues and then treated in accordance with the standard Oxford bone and skin methods detailed in Gillespie, Hedges and Humm (1986).

The linen wrappings were treated by two different methods: OxA-1387 was washed with CHCl₃ before pretreatment using the standard Oxford cellulose method, namely 1M HCl at 80°C/1hr, 0.1M NaOH at 80°C/1hr, 0.1M

NaOH cold overnight, 1M HCl at 80°C/1hr, 5% chlorite bleach (pH 3) at 80°C, wash H₂O until neutral and dry. OxA-1388 was prepared using the Zagreb Laboratory procedure: 4% HCl, 4% NaOH at 80°C/2hrs, boiling in H₂O until neutral and dry. The dicotyledon leaves were prepared using the Oxford cellulose method.

The embalming unguent was separated in Zagreb, using CHCl₃, into an insoluble and a soluble fraction. We thought that the insoluble fraction mainly consisted of cellulose-based plant remains, and so should have been treated as cellulose using the above method. However, we found that it was soluble in hot 1M HCl, and so pretreatment was discontinued, except for water washing. The CHCl₃ soluble fraction, considered to be the residues of organic resins and tars, was treated with hot HCl, but was unstable in alkali (1M NaOH), and so after acid treatment, was rinsed with water, redissolved in CHCl₃ and dried for combustion. The AMS dates were obtained using the methods described in Hedges *et al* (1989).

RADIOCARBON MEASUREMENTS: RESULTS AND DISCUSSION

The ¹⁴C determinations reveal several sharp age discrepancies between the different materials (Table 1). The AMS date on the body tissue (OxA-1386), and the two dates on the CHCl₃ insoluble unguent fraction (OxA-1390, Z-1682), contrast with the older, if different, dates for the soluble fraction and the exuded unguent. This tells us that one or several components used in the preparation of the unguent contained older carbon, and that it was used in a chemical form which was soluble in organic solvents. Organic materials, known to have been used in embalming, which could be responsible include mineral oils such as asphalt (possibly from Sinai shales) and fossil plant-derived resins. The soluble fraction made up only a small proportion of the original unguent, and thus it had little noticeable influence on the overall ¹⁴C age of the unguent. This is demonstrated by GrN-13860, the date on the original, non-exuded, brittle unguent (a mixture of both the CHCl₃ soluble and insoluble components) which gave a result indistinguishable from the two dates on the CHCl₃ insoluble fraction (Z-1682, OxA-1390). The difference between the AMS date for the soluble fraction (OxA-1391) and the radiometric date for the exuded unguent (Z-1675) is probably explained in terms of minor differences in composition.

One major age discrepancy is between the 2 AMS dates for the linen wrappings (OxA-1387, -1388) and the 3 radiometric dates (Z-1953, GrN-13875, -14442). Two points are worth noting here: 1) the wrappings dated at the three laboratories did not all come from the same piece of bandage, although the two AMS dates were from one piece of linen; 2) there is a small but significant discrepancy between the two high-precision dates (GrN-13875, -14442). This implies that not all the mummy bandages are of the same age. The fact that a linen book was cut into strips and "reused" by an embalmer suggests that re-used material was customarily used in mummy wrapping, which is important in assessing the implications of these dates.

The discordant ages of various parts of the mummy wrappings compared to the associated leaves, which supposedly accompanied the mummy as a wreath, clearly show that the mummy might have undergone a certain

 $\label{table 1} TABLE~1$ Radiometric and AMS ^{14}C dates on the linen "book", the mummy and associated material

Sample description	Sample no.*	¹⁴ C age (BP)	Calibrated range** (ca)
Liber linteus Zagrabiensis	OxA-1680	2110 ± 60	360 BC-AD 5
Human tissue from mummy	OxA-1386	2160 ± 80	395-40 BC
Linen wrappings of mummy	Z-1653	2290 ± 40	405-210 BC
	GrN-13875	2210 ± 13	365-205 BC
	GrN-14442	2335 ± 15	405-395 BC
	OxA-1387	$2760 \pm 90^{\circ}$	1015–800 BC
	OxA-1388	$2700 \pm 90^{*}$	
Leaves	Z-1654	2580 ± 60	900-520 BC
	GrN-14467	2825 ± 15	1015-925 BC
	OxA-1389	2920 ± 100	1400–905 BC
Embalming unguent	Gr-N-13860	2240 ± 13	385-255 BC
Embalming unguent fraction insoluble in CHCl ₃	Z-1682 OxA-1390	2230 ± 50 2160 ± 100	395–190 BC 400 BC–AD 40
Embalming unguent fraction soluble in CHCl ₃	OxA-1391	3010 ± 80	1430–1020 BC
Embalming unguent exuded			
fraction	Z-1675	4540 ± 120	3610-2920 BC

^{*} Z – Ruđer Bošković Institute

GrN - Center for Isotope Research, Groningen

OxA - Radiocarbon Accelerator Unit, Research Laboratory for Archaeology and the History of Art, Oxford

amount of 19th century repair work before being sold to the European collector who transported it to Zagreb. According to Helen Whitehouse (pers commun, 1988) of the Ashmolean Museum in Oxford, the process of mummification was not done as carefully in the last few centuries BC as in earlier periods and re-use of old materials is known. Also, local tomb robbers could have taken textiles from other tombs to partially rewrap the mummy, especially if it was in poor condition. This may explain the discrepancy between the ages of the mummy wrappings obtained by the three laboratories which obviously did not belong to the same piece of linen cloth.

The conspicuously older age of the leaves found with the mummy supports this explanation. At first, the older age for the leaves was explained at the Zagreb laboratory as a consequence of poor counting statistics due to large fluctuations in the count rate (Srdoč & Horvatinčić, 1986). Also, the sample of selected clean leaves was lost in a laboratory accident, so that the reported age was obtained on a less reliable sample containing rootlets, twigs, seeds and stained leaves. The independent dating of leaves at

^{**} Dendrochronological calibration based on high-precision curves (Stuiver & Pearson, 1986; Pearson & Stuiver, 1986; Pearson *et al*, 1986, using the computer calibration program of van der Plicht and Mook, 1989). Dates in calendar years BC unless otherwise indicated. Age range based on 95.4% (2σ) confidence level.

[†] Pretreated using the Oxford cellulose pretreatment method

^{*} Pretreated using the Zagreb pretreatment method

Groningen and Oxford confirmend the older age of the leaves compared to the mummy tissue and some of the wrappings.

CONCLUSIONS

This study emphasizes the caution needed in interpreting archaeological finds and associated ¹⁴C dates. Although the dates for the mummified female corpse, the linen wrappings with Etruscan inscription, and the charred plant material used in the embalming unguent appear contemporary in 14C terms, it is important not to use these results to draw conclusions for samples other than those measured. The AMS dates on the mummy wrappings and on the Liber linteus Zagrabiensis demonstrate that linen of at least two different ages was used. It is therefore possible that the radiometric wrapping samples may have included linen of two ages, possibly more, although none of the bandages used for these samples had writing on them. It is important not to use the high-precision dates on the wrappings to date the Liber linteus Zagrabiensis. Only the AMS date (OxA-1680) should be used, despite the result that the relatively large error on this date produces a calibrated age range of ca 360 cal BC - cal AD 5 (20 confidence level), meaning that, on the basis of this one date, the "book" could have been written in the 3rd or the 1st century BC, or perhaps even at the very beginning of the 1st century AD.

It is possible to refine the age estimate for the "book" if the dating evidence for the mummy is taken into account. The AMS date on the mummy tissue, and the two dates on the CHCl₃ insoluble fraction of the unguent (which represent various plant genera), are arguably direct measurements of the date of mummification. For this to be correct, first, the removal of non-contemporary, kerosene carbon from the mummy tissue by pretreatment would have to be successful; second, the plant material in the insoluble fraction would have to be fresh at the time of mummification. If these two assumptions are correct, then the pooled mean (Ward & Wilson, 1978) of these three dates is ca 2200 ± 40 BP, which gives a calibrated age range (2σ) for mummification of ca 390–170 cal BC.

This argument can be taken further. The influence of "old" carbon on the ^{14}C age of a sample is a function of the percentage content of the contaminant and the age difference between the contaminant and the true age of the sample (Olsson, 1968). The two dates Z-1682 (2230 \pm 50) and OxA-1390 (2160 \pm 100) were from the CHCl3 insoluble fraction (arguably the "true" age of the sample), whereas OxA-1391 (3010 \pm 80) dated the CHCl3 soluble fraction (the "old" carbon component). The difference between the pooled mean of Z-1682 and OxA-1390 (2216 \pm 45) and OxA-1391 is nearly 800 ^{14}C yr. The percentage of "old" carbon in the unguent was measured as <3% of total carbon content. From these figures, GrN-13860 is ca 20 ^{14}C yr too old due to the presence of 3% older carbon. Taking this into account and combining all four dates for the tissue, the insoluble fraction of the unguent, and the revised age estimate for the total unguent (GrN-13860 – 20 yr), produces a mean date for mummification of 2218 \pm 12 BP, which gives a 2 σ calibrated age range of ca 385–210 cal BC.

Using the two age ranges for the mummy (ca 385-210 cal BC,) and the book (ca 360 cal BC - cal AD 5), it follows that since the "book" was re-used to wrap the corpse, and mummification happened before ca 210 cal BC, the latest date for the Etruscan "book" must be ca 210 cal BC. Similarly, mummification could not have taken place before the "book" was written, ie, before ca 360 cal BC, thus, a revised range for the Liber linteus Zagrabiensis would be ca 360-210 cal BC, making the 3rd century BC stylistic date as the most likely period of composition.

In conclusion, by dating the various materials associated with the Etruscan Liber linteus Zagrabiensis and the Egyptian mummy, 14C determinations have helped resolve the details of the mummification process responsible for preserving this linen "book," and have also narrowed down the time range in which this liturgical calendar was written.

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