SOME INTERESTING APPLICATIONS OF RADIOCARBON DATING TO ART AND ARCHAEOLOGY

A RADIOKARBON KORMEGHATÁROZÁS NÉHÁNY ÉRDEKES ALKALMAZÁSA

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Abstract

Radiocarbon dating is an important tool for the determination of the age of many samples and covers the time period of approximately the last 50,000 years. We can use radiocarbon dating to estimate the age of a wide variety of carbon-containing materials. Both organic or inorganic materials at the Earth's surface and in the oceans form in equilibrium with atmospheric carbon-14. This makes it an important tool for the understanding of processes during the time-scale of modern humans, from the last glacial-interglacial transition, to recent archaeological studies of art works. We present an overview of the technique, its advantages, assumptions and limitations. We also emphasize dating interesting objects. Radiocarbon has been applied to dating many historical artifacts and archaeological applications. Some specific examples including dating of famous artifacts of artistic, religious and scientific interest are discussed.

Kivonat

A radiokarbon kormeghatározás az abszolút kronológiai adatok meghatározásának fontos eszköze, sokféle mintán alkalmazható és alkalmas az utolsó 50 000 év leleteinek datálására. A módszert sokféle, szenet tartalmazó anyag korának meghatározására használhatjuk. Szerves és szervetlen mintákat vizsgálhatunk a Föld felszínéről vagy az óceánok mélyéről, bárhonnan, ahol a C-14 izotóp mennyisége egyensúlyban volt a légköri szén izotóp összetétellel. Ennek következtében fontos eszköze a fejlődési folyamatok megismerésének a modern ember létezésének idején, az utolsó jégkorszakot megelőző interglaciális időszak végétől egészen napjainkig. A tanulmányban áttekintést adunk a kormeghatározási eljárásról, előnyeiről, feltételeiről és korlátairól. Esettanulmányokat mutatunk be érdekes műtárgyakon. A radiokarbon kormeghatározást számos történeti és régészeti tárgyon próbálták ki, amelyek között néhány különleges művészeti, vallási vagy tudományos értékkel is bír.

KEYWORDS: RADIOCARBON DATING, ART, ARCHAEOLOGY

KULCSSZAVAK: RADIOKARBON KORMEGHATÁROZÁS, KÉPZŐMŰVÉSZET, RÉGÉSZET

Introduction

Radiocarbon (^{14}C) is produced in the upper atmosphere by the action of secondary cosmic-ray particles, which are thermal neutrons on nitrogen. It has a half-life of 5,700 years and the amounts of ^{14}C produced naturally cover the time scale of approximately 50,000 years (Jull 2013a; Kutschera 2013; Fifield 1999; Tuniz et al. 1998). Of course, this is also the period of interest to archaeology and many other fields. There are a large and diverse number of applications of ^{14}C (Jull 2013b). Originally, ^{14}C was counted by decay counting of the nuclide, however this has now been largely replaced by direct measurement of ^{14}C atoms using accelerator mass spectrometry (AMS). Indeed, accelerator mass spectrometry has become the method of choice for most measurements of longer-lived radionuclides, of which the most well-known is carbon-14 (Jull and Burr 2013a). This method allows for much smaller samples of carbon to be measured than were previously possible using decay counting, since counting atoms directly is inherently more efficient than by counting radioactive decay particles. In practice, the measurement of samples of carbon of 0.05 to 0.5 mg is easily performed (Jull and Burr 2013b).

Radiocarbon dating relies on a basic assumption that organic or inorganic materials are in equilibrium with ^{14}C, which is produced in the atmosphere and its removal into other reservoirs, and which establishes a constant level of ^{14}C at any given time. This relies on the radioactive decay equation (Rutherford and Soddy 1902), where the decay rate is determined by the number of atoms:

$$\frac{dN}{dt} = -\lambda N \quad [1]$$

Where N is the number of atoms, t is time and λ is the decay constant of the nuclide. When an animal or plant dies, it is removed from the atmospheric equilibrium and so the level of ^{14}C decays according to equally recognizable equation:

$$\frac{N}{N_0} = e^{-\lambda t} \quad [2]$$

Where N0 is the number of atoms present at the time of formation of the material. One can therefore easily solve for the apparent "radiocarbon age" of the sample, by rearranging this equation:

$$t = -\lambda \ln(\frac{N}{N_0}) \quad [3]$$

Where t is the "radiocarbon age" of the material. This "radiocarbon age" is an approximate age of the material, since there are other effects on the ^{14}C production in the atmosphere (Burr 2013). Usually, radiocarbon ages are quoted in "years before present" (yr. BP), where "present" is defined as 1950AD. In practice, the production rate of ^{14}C in the atmosphere varies with time, so that it is important to calibrate raw radiocarbon ages derived from eqn. 3 to a true "calendar age". This is achieved by using a calibration of radiocarbon ages against true age from tree-ring records up to 12,700 before present. Beyond that time, calibration is achieved by cross-referencing ^{14}C ages of other records, such as annually-layered lake sediments or by cross-correlation of other dating methods such as U-Th in corals and speleothems (Reimer et al. 2013). An example of a calibration is shown in **Fig. 1**.

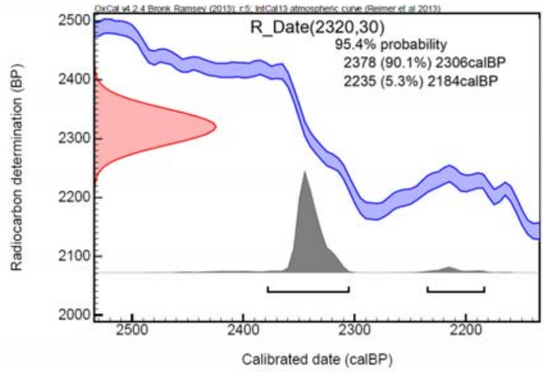

Fig. 1.: An example of a calibration of a radiocarbon age. The measured value is plotted on the vertical axis, with the 1σ error. The horizontal axis shows the combination of the analytical measurement with the calibration curve.

1. ábra: A radiokarbon koradat kalibrálása. A mért adatot 1σ hibával felvesszük a függőleges (y) tengelyre. A vízszintes (x) tengelyen leolvasható a kalibrációs görbe és a mért érték hibahatárral korrigált kombinációja.

The example shown is very precise, since the curve is very steep at this point. However, an intersect with a flatter or varying part of the calibration curve can obviously give much wider error ranges for the resulting calibrated age.

There are other effects that have changed the inventory of ^{14}C in the atmosphere. Fossil-fuel burning has raised the level of CO2 in the atmosphere from 280 ppm in the 18th century to almost 400 ppm today. This ^{14}C-free carbon added to the atmosphere dilutes the original signal, so that the value today is considerably depressed. After 1950AD, we have a different effect on the radiocarbon curve. There is a large increase due to the atmospheric testing of nuclear weapons, which raised the atmospheric value in the northern hemisphere to 1.8 times the pre-bomb value. Since this testing mainly ceased after 1963, the level in the atmosphere has now decreased to about 1.04 times the pre-bomb value, due to both exchange with the ocean and the addition of more "dead" carbon from fossil-fuel burning. Indeed, the radiocarbon in the surface ocean is now exchanging bomb carbon-14 back into the atmosphere, as shown in **Fig. 2** (Hua et al. 2013). This radiocarbon "spike" allows us to identify recent material, post-1950AD, by its characteristic excess in carbon-14.

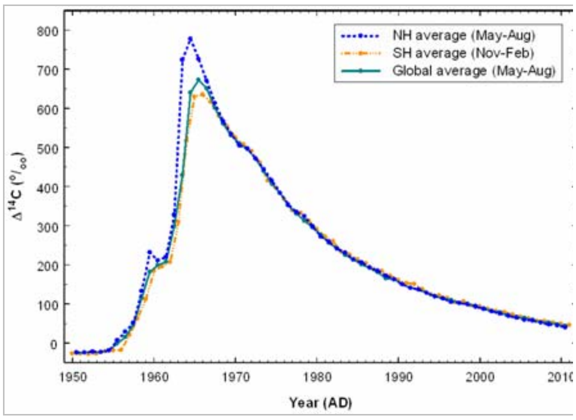

Fig. 2.: Average ^{14}C bomb pulse effect for the northern and southern hemispheres, from Hua et al. (2013)

2. ábra: Átlagos bomba impulzus hatás a ^{14}C értékekre az északi és a déli féltekén, Hua et al. (2013) alapján

In this paper, we will review the method, and then give some examples of applications that highlight the usefulness of these measurements to a wide variety of topics.

Basics of the AMS Method for adiocarbon

AMS covers a wide range of different types of instruments, from very large accelerator systems, to the latest compact AMS systems, such as the one at ATOMKI in Debrecen, shown in **Fig. 3** (Molnár et al. 2013). The trend in AMS design over the last 20 years has led to smaller machines. Although they can vary a lot in detail (Jull and Burr 2013b), AMS systems all have the following basic components:

a.) an ion source which generates negative carbon ions (20 to 100 µA C-) by Cs sputtering from a graphite target. In some newer systems, ions can be generated from gas samples.

b.) an injection magnet, which performs the initial separation of the negative ions by mass. At this point, molecular ions such as hydrides of carbon (CH-) are also present. N- is unstable so an important possible interference is removed. Usually, the different isotopes are pulsed through the magnet rapidly.

c.) the accelerator, which may have a voltage of as little as 200 kV or perhaps as much as 3 MV, which accelerates the C- ions towards the “terminal”, which is located in the central part of the machine and is at high voltage.

d.) the terminal, that includes a gas canal, usually known as the "stripper". Negative carbon ions enter the canal and interact with a gas. Because they are

Fig. 3.: The MICADAS AMS system installed at Debrecen (courtesy Dr. M. Molnár).

3. ábra: A Debrecenben telepített MICADAS AMS rendszer.

moving so fast, they lose several electrons from their electron cloud, and as a result become positively charged. Depending on the design of the AMS, this can vary from the C+ to C3+ charge state.

e.) an electrostatic analyzer that allows us to select ions of one energy/charge ratio.

f.) a magnet to separate ions of the correct mass/charge ratio for the given design.

g.) a solid-state or gas ionization detector, which can discriminate between ions of ^{14}C and other isotopes.

h.) a data analysis and storage system. At Arizona, we use the procedures detailed by Donahue et al. (1990) to calculate the radiocarbon ages from the isotopic measurements.

Chemical Pretreatment

It is important to use the appropriate chemical pretreatment scheme to clean the sample prior to extraction of the carbon in the form of graphite or CO2. In general, we use an acid-base-acid method for charcoal, wood, cellulose, plant material, animal tissue: After physical inspection, samples are cleaned with 1N HCl acid, 0.1% NaOH and 1N HCl (acid-base-acid (ABA) pretreatment), washed with distilled water until neutral, dried, and combusted to CO2 at 900oC with CuO. In contrast, carbonate samples are etched with H3PO4 to remove 50-85% of the carbonate, dried and hydrolysed with H3PO4 as discussed by Burr et al. (1992). More complicated cleaning is done for many kinds of art works, since the samples are also subjected to a Soxhlet extraction usually using hexane, then ethanol and finally methanol. Some laboratories also use acetone as an additional step in the Soxhlet protocol. All these steps are designed

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to remove various kinds of organic contaminants (Bruhn et al. 2001; Hatté and Jull 2013). For further details on sample pretreatment techniques for AMS analysis, the reader is referred to Hajdas (2009).

We have had some success dating iron archaeological artifacts by either melting the sample in an oxygen atmosphere and extracting CO2, or by using wet-chemical techniques (Park et al. 2010). In our laboratory, we use a radio-frequency induction furnace to melt the iron in a flow of oxygen. We have also had some limited success dating archaeological bronze items in this way (Jull, unpublished results).

An important material to consider for dating is bone. Bone degradation is a complex process and a lot of attention needs to be given to bone pretreatment because of the important role they play in archaeological studies. The basic method has remained unchanged for many years, which involves collagen extraction (Longin 1971). Collagen is a fibrous protein and the principal constituent of bone. This material degrades with time as a result of post-depositional alteration and eventually breaks down into individual amino acids. Because bone proteins break down over time, there is usually some type of pre-screening of bone samples, for example by measuring the elemental C/N ratio. Modern bone consists of ~22 wt% collagen and this value decreases with the age of the material. Samples with as low as 0.5% collagen may be datable, but often require specialized chemical pretreatment, such as the isolation and analysis of individual amino acids (Van Klinken 1999). Samples with <0.5% collagen are generally considered unsuitable for obtaining a useful date. Another refinement of the collagen extraction technique has become standard in the past decade, using ultrafiltration to isolate the >30 kD molecular weight fraction (Brown et al. 1988).

Groundwater and surface waters can be dated by extracting dissolved inorganic carbon by acid hydrolysis, in the same way as carbonates (Burr et al. 1992). We can extract dissolved organic carbon using either KMnO4 or K2Cr2O7 as an oxidizing agent, as well as in other ways (Leonard et al. 2013).

After cleaning and combustion, the carbon dioxide produced is converted to graphite using an iron catalyst. Finally, the graphite powder is pressed into a target holder and can be put into the accelerator ion source for analysis, along with other samples, known standards and blanks.

Some Aplications

Shroud of Turin

Perhaps the most famous example of radiocarbon dating involves the Shroud of Turin. This is a linen cloth which has the image of a crucified man. It is widely believed to be the burial cloth of Christ, although radiocarbon dating shows it to be of medieval age. Damon et al. (1989) reported a result of 691±31 radiocarbon years BP, which using the current calibration curve (Intcal13) gives a calibrated age of 1264-1388AD (95% confidence interval), the same range as originally reported. Freer-Waters and Jull (2010) discussed further characterization of the material. Intriguingly, there has been much discussion about these results and various challenges have been made to the original measurements. However, there has not been any reason to doubt the original studies. Many proposals have been made to do new dating measurements, however, so far none have been allowed by ecclesiastical authorities. Presumably, further work to confirm these results may be done in future.

Fig. 4.: The Book of Isaiah. This parchment is on display in the Shrine of the Book in Jerusalem. It dates to 209-59BC according to radiocarbon dating, or 150-125BC from studies of the writing style (Bonani et al. 1991; Jull et al. 1995). Image courtesy Israel Museum.

4. ábra: Ézsaiás Könyve. Ez a pergamen Jeruzsálemben látható a „Shrine of the Book” múzeumban. Kora a radiokarbon adatok szerint 209-59 BC, vagy 150-125 BC az írásmód vizsgálata alapján (Bonani et al. 1991; Jull et al. 1995).

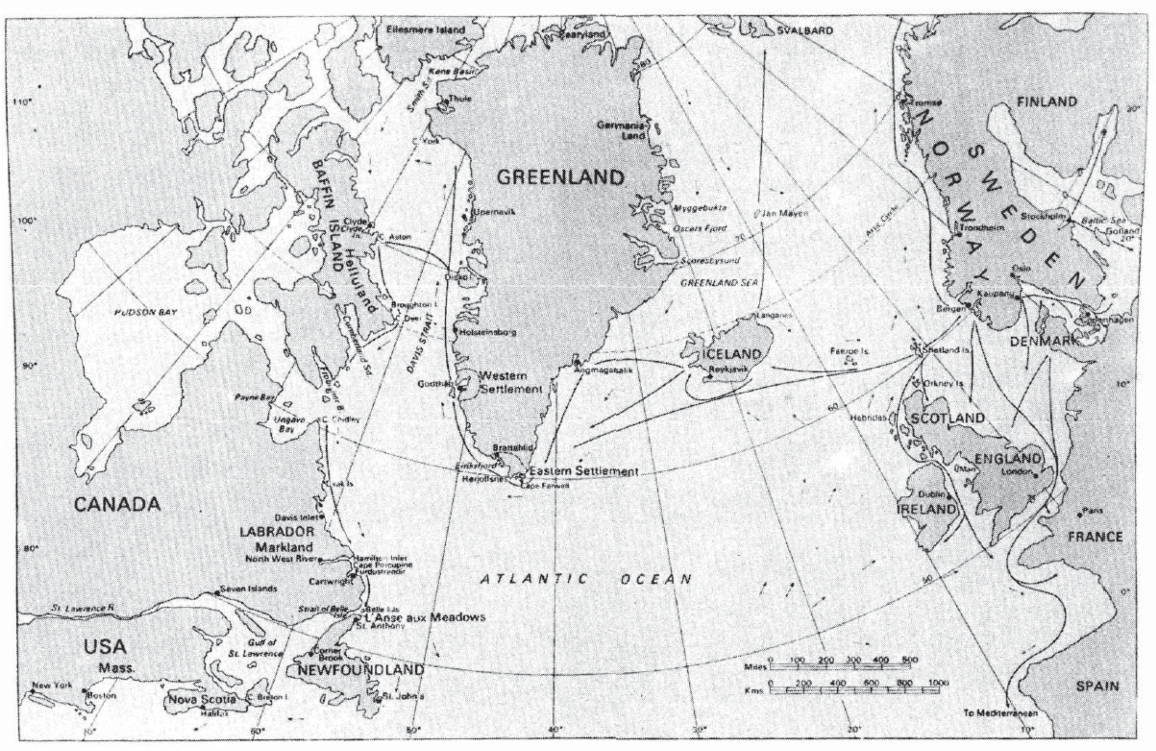

Fig. 5.: A map of Viking exploration routes, adapted from a drawing of G. Furuholmen (Canada Department of Mines and Technical Surveys) and reproduced by Nydal (1989) in Radiocarbon with permission.

5. ábra: A viking felfedezők utvonalainak térképe, G. Furuholmen (Canada Department of Mines and Technical Surveys) rajza alapján. (Nydal 1989 nyomán).

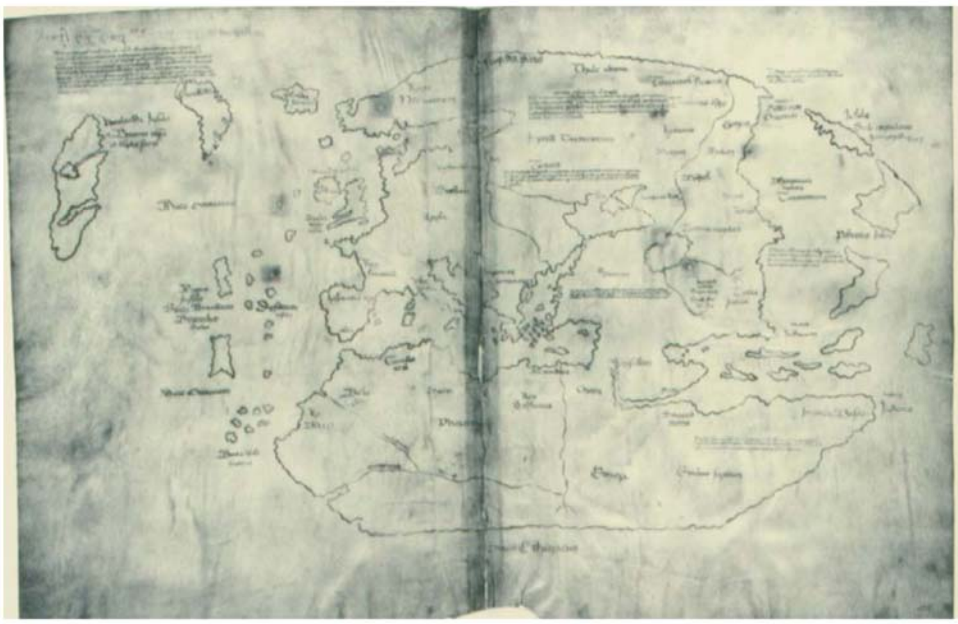

Fig. 6.: Vinland Map, dated by Donahue et al. (2002). Courtesy Beineke Rare Book Library, Yale University and Radiocarbon.

6. ábra: Vinland térképe, Donahue et al. (2002).kormeghatározása

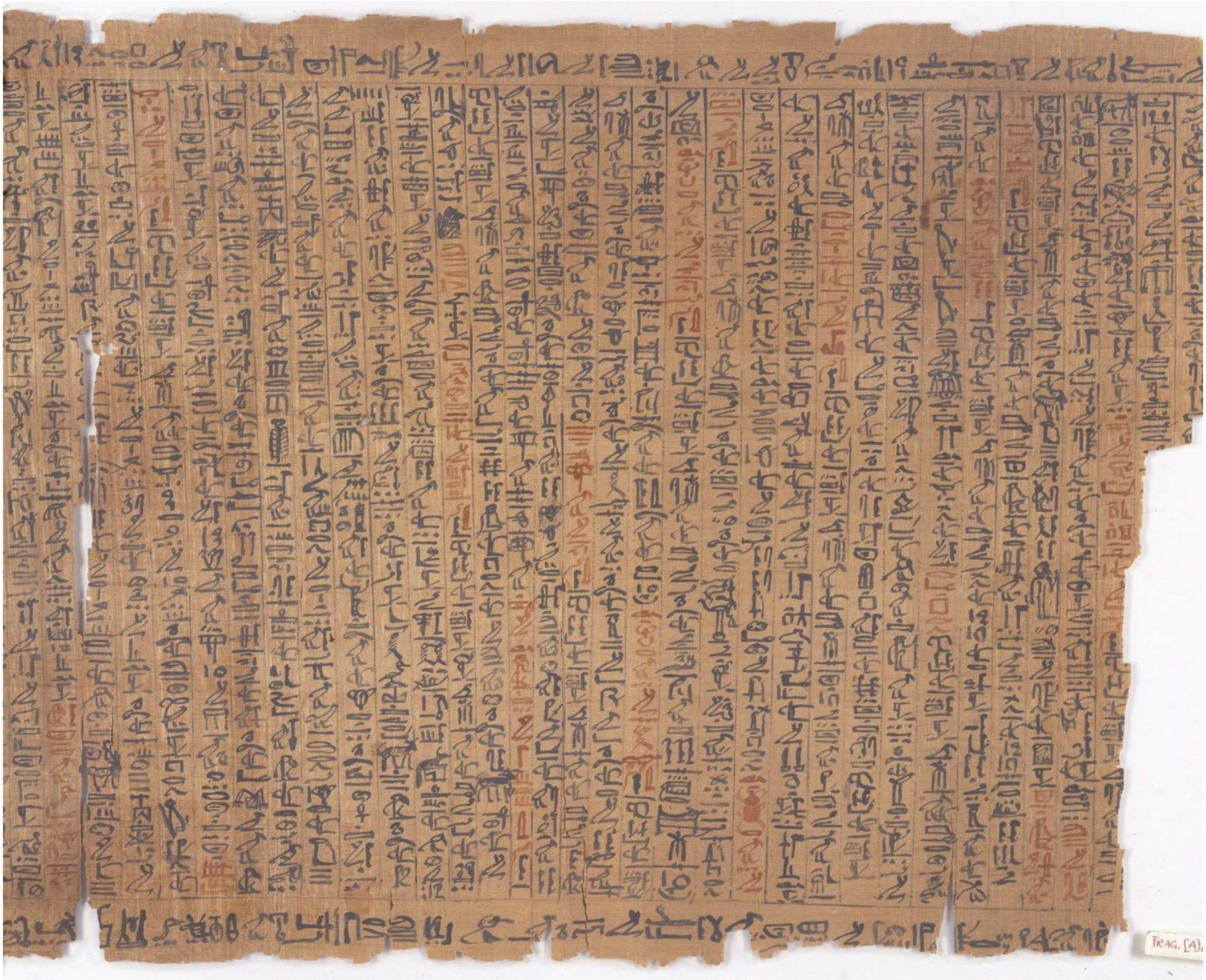

Fig. 7.: A papyrus page from the Egyptian Book of the Dead. Courtesy Brooklyn Museum.

7. ábra: Egy papirusz oldal a Holtak Könyvéből (Brooklyn Museum)

Dead Sea Scrolls

A remarkably uncontroversial example is the study of the Dead Sea Scrolls (Shanks 1993). These interesting documents, written on parchment or papyrus, contain detailed copies of books of the Old Testament, other religious commentaries on books of the Bible of an esoteric nature, as well as more mundane business documents, such as financial transactions. These documents date from the mid-2nd century BC, for example the Book of Isaiah shown in **Fig. 4**, to the first century AD. They are a fascinating depiction of some religious views from the Maccabean revolt to after the time of Christ, and are the subject of many discussions about their significance. The ages of these documents fit well with the expected results (Bonani et al. 1992; Jull et al. 1995).

The Gospel of Judas

The Gospel of Judas is a Gnostic manuscript written on papyrus which has been compiled into a codex, or book. The original text was the subject of extensive criticism by the Christian scholar Irenaeus, who wrote a document called "Against all heresies" in about 180AD. The document gives a radiocarbon age of 1767±16 yr BP, which is consistent with a calibrated age range of 220-340AD.

Voynich Manuscript

A most intriguing document, the Voynich manuscript is currently in the possession of the Beineke Rare Books Library at Yale University in the USA. The document was known since about the 16th century, since it was at one time in the possession of the Holy Roman Emperor Rudolph II. The document is enigmatic in that it is written in an indecipherable language, which is assumed to also be encrypted. Many have tried to decipher this code, but none have succeeded. The document consists of strange drawings of astronomical features, botanical drawings of unknown plants and ritual bathing. The purpose of the document remains unclear. The manuscript was dated to the 15th century, to 1404-1438AD (Hodgins 2011).

Vinland Map

Intriguingly, there is another document which is almost exactly the same age as the Voynich manuscript. This is called the Vinland Map, stored in the same library, which shows the New World on a map dated to the mid-15th century (1411-1468AD) (Donahue et al. 2002). This map (**Fig. 5**) used to be controversial, since it shows Newfoundland and Greenland on a map before the time of Columbus. However, we now know that there were earlier explorations to North America by the Vikings, well-dated by radiocarbon (Nydal 1989). There is a famous site at L'Anse aux Meadows in Newfoundland ("Vinland") giving ages of 975-1000AD, so the fact that Vinland appears on this map is not surprising. **Fig. 6** shows a map of Viking explorations shown by Nydal (1989).

Egyptian Book of the Dead

One interesting object that we have dated was made on papyrus and features several pages of the Egyptian Book of the Dead. This is shown in **Fig. 7**. This object gave a very nice radiocarbon result of 1620-1430BC, and the calibration result is useful to show, as it gives some idea of the fit of this result to the calibration curve. In **Fig. 8**, one can observe that the smooth Gaussian curve of the "radiocarbon age" becomes more complex when passed through the complex function of the calibration curve, giving the result shown on the horizontal axis. The chronology of Egypt used to be based solely on dynastic chronologies and "king lists". However, Ramsey et al. (2010) have been able to cross-correlate the dating of Egyptian sites from historical records with radiocarbon dates on materials found in tombs with the radiocarbon calibration curve (Reimer et al. 2013).

Conclusions

To conclude, dating art works is a fascinating topic that brings laboratory scientists into contact with a wide range of persons in different fields.

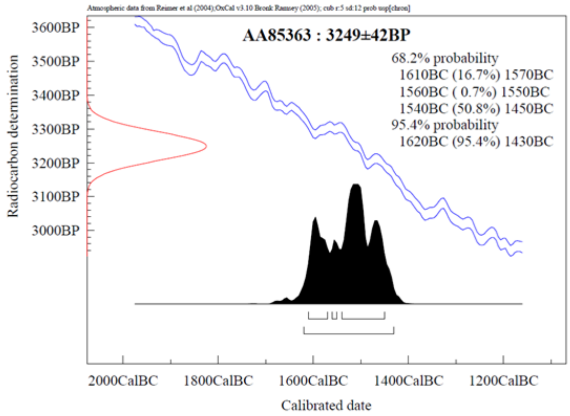

Fig. 8.: Calibrated age distribution for the papyrus page shown in **Fig. 7.**

8. ábra: A **7. ábrán** bemutatott papirusz kalibrált kora

Some are done for private individuals, so we cannot show those results here, although we do note that we also frequently receive art works for dating that turn out to be younger than expected. One example can be given which was dated both at Arizona and also in Debrecen. The result in both laboratories was the same and confirmed that the painting in question did not date from the early 16th century, as expected, but from the period of 1700-1950AD, when radiocarbon ages are subject to a number of fluctuations due to changes in solar activity and also the addition of "old" carbon due to the industrial revolution. Unfortunately, this is quite a common result. We attribute this to the copying of great works by later art students. Any visitor to an art gallery can observe enthusiastic students works on quite excellent copies of some great master's work. Hence, many copies of great art works circulate and the unsuspecting collector can be surprised when the expected Old Master turns out to be not quite so old. In any case, there are many excellent examples of radiocarbon dating applied to works of art and artifacts. We can only present a few of them here and we hope this brief introduction is helpful. Radiocarbon dating has an important place in the toolbox for the archaeologist and the art historian.

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