The dating game

What links the evolution of horses in North America to the history of human settlement in the Fertile Crescent, the extinction of the gigantic Irish elk and the volcanic eruption that destroyed the Minoan civilisation? **Tom Marshall** visited the Oxford Radiocarbon Accelerator Unit to find out.

Unlike most of Britain's radiocarbon-dating facilities, the Oxford Radiocarbon Accelerator, or ORAU, specialises in archaeological work, with a particular focus on bones.

Most of its peers, including its NERC-funded sister-facility in East Kilbride, Scotland, focus on environmental areas like dating sediment sequences with pollen to research ancient climates.

Since the lab started up in the early 1980s, this archaeological emphasis has drawn it into areas ranging from the spread of early humans to the provenance of antique spirits.

'We're unique – the only radiocarbon facility in the world that specialises in archaeological samples,' says Dr Tom Higham, deputy director at ORAU.

The lab uses an advanced technique for radiocarbon analysis known as accelerator mass spectrometer (AMS) dating, which lets it work with tiny samples of material, often as little as 20-40 milligrams. This means researchers don't need to destroy as much of a precious archaeological find to date it.

Radiocarbon dating works by measuring levels of a radioactive isotope of carbon, known as carbon 14, or ^{14}C for short, in samples of once-living matter like wood, leather or bone. Living things absorb radiocarbon while they are alive in quantities dependent on how much is in the atmosphere at the time.

There has been a major leap forward in archaeological science and its acceptance by archaeologists.

After they die, the ^{14}C decays at a steady rate, letting researchers calculate how many years ago the animal was alive – or rather, how many years before 1950. This is used as the baseline for radiocarbon dating because around this point nuclear weapons testing caused a marked shift in the radiocarbon signature in living organisms, forming a neat threshold.

ORAU's income comes from three sources. The Natural Environment Research Council (NERC) and the Arts and Humanities Research Council (AHRC) award grants to support work that is an integral part of major research projects, and the lab provides dates to academics and institutions successful in the grant process. Any researcher in Britain can apply to have samples dated, as long as they can get funding.

Work for the wider academic community accounts for around a quarter of the unit's revenue, with a similar proportion coming from independent commercial jobs done for clients like English Heritage, road builders or the police.

Some of this is straightforward work on archaeological digs, such as dating samples from sites that are being excavated before they are built over. Other work, especially for English Heritage, leads to ground breaking academic research. Doing for-profit work has also revealed that people want a surprising range of things dated.

The fraud squad

The thriving trade in old whiskies has attracted fraudsters who try to pass off more modern liquids as antique; auction houses and collectors have responded by asking for ORAU's help in confirming that some whisky is as old as it's claimed to be.

Sometimes the answer has been a resounding no – analysis of some supposedly antique bottles of whisky has revealed the unmistakeable signature of elevated levels of ^{14}C from the atomic era, implying that no matter how old the packaging, the contents can't date from before around 1950.

Efforts to preserve elephant populations and deter poaching by cracking down on the ivory trade have been complicated by the fact it's still legal to trade in old ivory. Criminals have taken to disguising ivory from freshly-killed elephants as antique in order to get round the ban. Dating work in Oxford has helped conservationists tell the difference.

The art market is another occasional source of work, with galleries and collectors looking to check claims that a famous artist produced a painting of uncertain provenance.

ORAU scientists are even occasionally asked to work on human remains. When someone finds part of a body and the police don't know if it's recent or centuries old, radiocarbon can help.

Most remains turn out to be medieval or even older, but there have been occasional surprises – a skull found by a dog-walker in Newcastle, which the police assumed was probably centuries old, turned out to date from within the last decade.

Sample being removed from bone for carbon dating using accelerator mass spectrometry (AMS).

James King-Holmes/Science Photo Library

Hard graft

Once a sample arrives, it's cleaned, taking care to avoid contamination with carbon of a different age. Each kind of sample demands its own style of pre-treatment – preparing charcoal for analysis is a very different procedure from preparing bone. The lab uses around 40 different procedures in different situations, most of them involving numerous and complex chemical steps.

The next stage is to burn the sample and turn it into CO_2 gas. The carbon in that gas is reduced onto iron and converted into graphite.

This is bombarded with caesium ions in a particle accelerator and the resulting charged particles focused into a tight beam. Radiocarbon ions are separated with electromagnets before finally entering a detector to be counted.

It's a complex procedure, relying on sophisticated instrumentation first developed for nuclear physics research. But the lab's AMS dating techniques let scientists work with much smaller samples than earlier methods – vital when dating irreplaceable archaeological finds or artwork.

Once the amount of radiocarbon in the sample has been measured, its age can be estimated. But the process doesn't end there; the estimated date needs to be calibrated. This is a crucial part of the lab's work, since the raw 'before present' dates the tests spit out aren't comparable to the calendar dates archaeologists need.

This is because levels of carbon in the atmosphere aren't constant, but fluctuate according to factors like the amount of solar radiation hitting the Earth, the planet's magnetic field and even its climate. More recently, human carbon emissions have muddied the waters still further.

Raw dates assume a constant level of ^{14}C at 1950 levels. To correct for this false premise radiocarbon dates need to be 'calibrated'. This involves comparison with samples whose dates are known independently by other methods, such as tree rings, cores of ice or lake-bed sediments, corals or stalagmites and stalactites in caves.

Measuring these known-age samples using high-precision radiocarbon dating provides a 'calibration curve' – a measure of how far off the mark a radiocarbon date is likely to be at different points through history. Even after calibration, the result can be a range of possible dates, with varying degrees of likelihood.

Professor Christopher Bronk Ramsey, ORAU's director, joined in the mid-1980s after training as a physicist. He has pioneered a statistical approach to calibrating radiocarbon dates more accurately.

The fate of the Irish elk

The Irish elk was long thought to have died out along with many other outsized versions of modern creatures – or 'megafauna' – around 10,000 years ago, but scientists were unsure what had caused this wave of extinctions.

Did humans hunt them into oblivion, or was a changing environment responsible?

ORAU analysis of surviving elk bones in collaboration with a NERC-funded project run by Dr Tony Stuart at UCL and Professor Adrian Lister at the Natural History Museum in London proved that some of the animals survived until around 7000 years ago in the Russian Ural mountains, bringing forward the date of the species' demise by more than three millennia.

The Irish elk's extinction now appears to stem from changes in its environment's climate and flora, not exclusively from human hunting.

The research will help scientists build up a picture of how the megafauna of northern Eurasia died out, and may be relevant to the study of other species such as the woolly mammoth, whose path to extinction may have been more complex than hitherto assumed.

Natural History Museum, London

Thera and the downfall of the Minoans

Increasing numbers of archaeologists and ancient historians have come round to the idea that natural disasters could have been responsible for the mysterious demise of several ancient cultures.

The sudden disappearance of the Bronze Age Minoan civilisation on Crete has often been blamed on the eruption of Mount Thera on the island of Santorini – one of the most violent volcanic eruptions on record.

The event spread ash over hundreds of miles and has been credited with phenomena ranging from the myth of Atlantis and the Biblical plagues of Egypt to the failure of harvests in China around the same time. But precisely dating the eruption has proved controversial; archaeologists have tended to settle on an approximate estimate of 1500BC.

Groundbreaking research by ORAU, in a NERC research project led by Dr Stuart Manning, then at Reading University, helped change that. Published in *Science* in 2006, the work involved the radiocarbon dating of 127 samples from digs in the eastern Mediterranean to refine the accepted chronology, proving with 95 per cent confidence that the eruption happened somewhere between 1613BC and 1600BC.

The results closely match those of another team, published in the same edition of the journal. The discovery of a small olive branch buried in ash on Santorini allowed the second team to produce another dating series, suggesting (again with 95 per cent confidence) a date between 1627BC and 1600BC.

The results are of wider historical significance, because they disprove the idea that Minoan culture was closely linked with the New Kingdom of Egypt. Instead, Minoan civilisation would have been reaching its zenith at around the same time as the early Hyskos dynasty.

The results are likely to mean the whole chronology of the period has to change; well-known shaft graves found by Schliemann, the nineteenth-century rediscoverer of Troy, must be far older than previously assumed.

Bob Turner/Alamy

Known as OxCal and available free online, Ramsey's software uses Bayesian statistical techniques to refine the chronology and give a much tighter range of possible dates by combining the results of radiocarbon dating with other information about the sample being dated.

'A single date may only be precise to a century or two,' explains Ramsey. 'But if we have other material that is associated with what we are trying to date, we can use statistical methods to refine our range of possible dates. Sometimes this reduces it to as little as 20 years.'

A better calibre of radiocarbon

ORAU staff are working with other scientists, led by Dr Takeshi Nakagawa of Newcastle University, to refine calibration curves still more by investigating certain promising ideas. At present, many dates can be calibrated only as far back as around 26,000 years.

But analysis of Lake Suigetsu in Japan promises to push this beyond 40,000 years. The lake's bed comprises thousands of layers or 'varves', each one laid down over the course of a year.

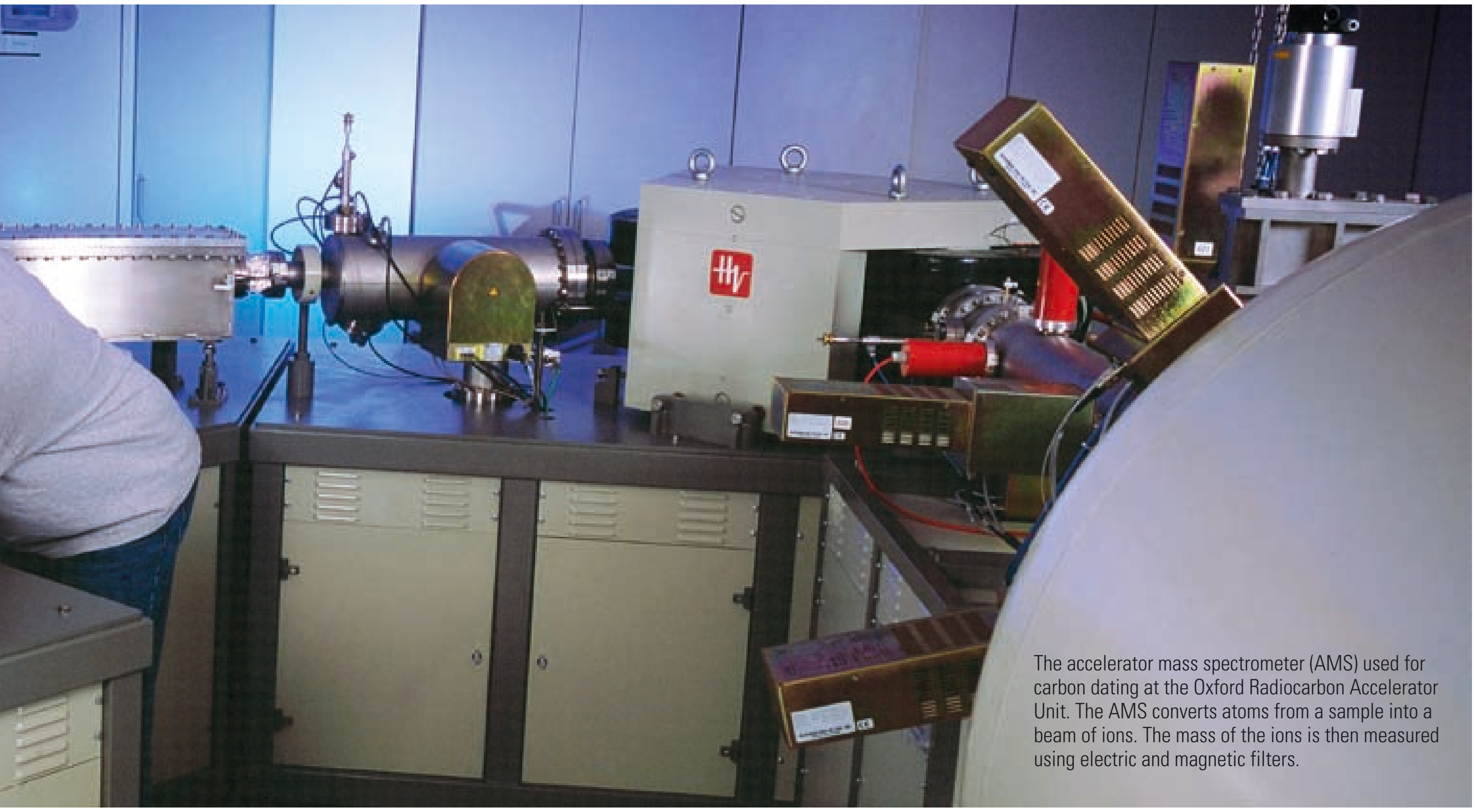
Each varve comprises a light layer formed from decaying algae, and a dark layer formed from clay. Each also contains organic matter

This will revolutionise our understanding of early prehistory.

that was in the lake at the time, such as ancient pollen or small leaves washed in during annual storms. The varves are therefore a perfect record of atmospheric carbon going back millennia.

Cores from the lake bed have been analysed before, but researchers are working on new triplicate core samples that should enable even more accurate dating of samples from between 10,000 and 50,000 years ago.

'Once this project is complete, the much more accurate calibration curve it will give us will revolutionise our understanding of early prehistory,' explains Bronk Ramsey.



James King-Holmes/Science Photo Library

The accelerator mass spectrometer (AMS) used for carbon dating at the Oxford Radiocarbon Accelerator Unit. The AMS converts atoms from a sample into a beam of ions. The mass of the ions is then measured using electric and magnetic filters.

A constant focus is how to make the lab's dates more accurate, and to reduce the size of sample it needs. Researchers also work to broaden the range of materials they can date – lately, it's turned out that chitin from insect carapaces can be dated with the right methods. Because insects are very dependent on specific conditions, establishing when certain species were alive can give important insights into ancient climates.

Another project deals with radiocarbon dating of ancient Egyptian samples. For the last three years, scientists from ORAU have been working with archaeologists and Egyptologists to revisit the chronology found in ancient Egyptian historical accounts, testing it against ^{14}C dating of contemporary material.

Much of the time, historical records and science agree. For example, at the site of Tell el-Amarna, the capital of Egypt under the pharaoh Akhenaten, all the dates of Egyptian historical records accord precisely with those produced by radiocarbon dating samples.

But some sites are more problematic, particularly those that seem to have been inhabited over long periods. Dating the organic material in the clay used to make ancient bricks has suggested some settlements could be a lot older than accepted chronologies suggest.

Where archaeology meets science

Another focus of research is refining our understanding of the Middle to Upper Palaeolithic period in Europe, between 30,000 and 40,000 years ago – a subject that should be illuminated by the improvements in calibration expected to come from the Lake Suigetsu research.

One of the biggest topics is the rise and rapid spread of modern humans, and the mysteriously swift disappearance of the Neanderthals.

^{14}C dating this far back is extremely tricky, as most of the carbon that was originally in samples from the period has already decayed, leaving only around 2.5 per cent of the original amount.

A technique called ultrafiltration, which lets researchers prepare bone collagen samples more effectively for dating, removing more impurities and hence improving the accuracy of findings, has made it easier to date hominid and animal remains from the period. Lab staff noticed it also seemed to make a big difference to the dates they arrived at.

They revisited numerous dates from Britain, Spain, France and Italy. Many of the previously dated Neanderthal remains were revealed to be a lot older than had been assumed once they were ultrafiltered. The Neanderthal remains found in the Croatian site of Vindija, for example, turned out to date from around 32,500 years ago, rather than 28,000 as had been thought.

The lab's development of ultrafiltration techniques has extended the limits of bone dating in European archaeology to around 50,000 years ago, compared with 40,000 years until recently. And it has greatly increased our knowledge of the period; the programme has already produced around 120 new dates, and NERC funding into 2009 should let it eventually find three times that number.

The result is already a more continuous picture of early humans' movements around Europe – including, for instance, a more nuanced understanding of patterns in the occupation of Britain, with periods of human habitation alternating with periods of abandonment, probably due to climate change.

All this shows how much closer archaeology and the hard physical sciences have moved over recent decades.

It used to be the case that many archaeologists had limited interest in the complexities of radioactive carbon decay rates and calibration curves.

That's now changed for good. 'In the last few decades there has been a major leap forward in archaeological science and its acceptance by archaeologists in general,' says Higham. 'Most now fully appreciate how much the information that radiocarbon dating gives can help them better understand their finds.' ❖

MORE INFORMATION

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