

IAEA virtual training on measurement techniques for natural and artificial radionuclides and radiochemical methods for their determination in marine environment

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Session 52. **Basic principles of C-14 dating**

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MYANMAR

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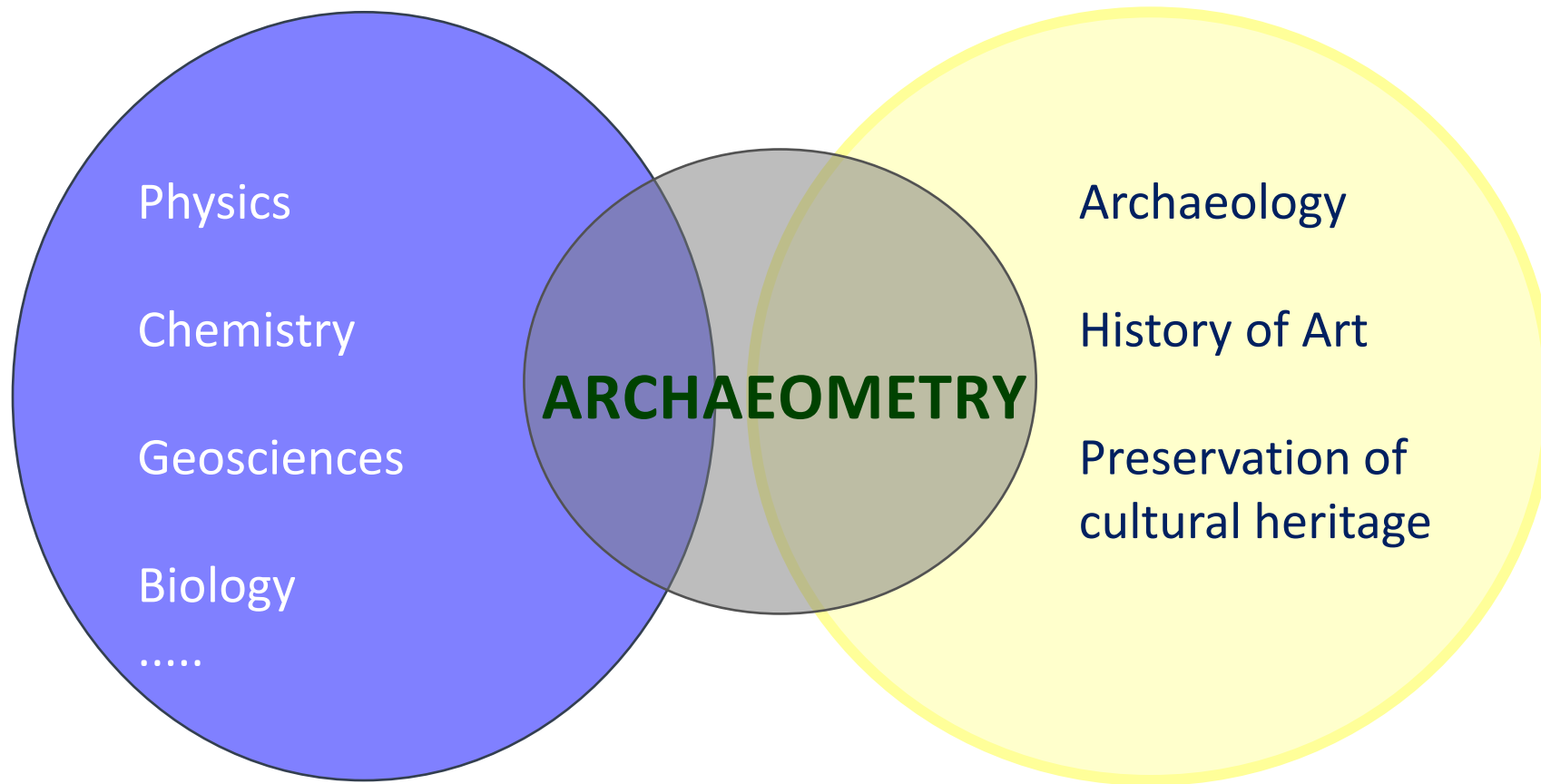
ARCHAEOMETRY

Various (slightly different) definitions:

- application of analytical techniques from the field of engineering and related natural sciences
- the analysis of archaeological materials using analytical techniques borrowed from the physical sciences and engineering
- application of the physical, chemical and biological sciences to archaeology and the history of art

Archaeometry - application of methods and concepts of natural and technical sciences in order to contribute to the solution of cultural-historical questions and problems.

→ **multidisciplinary field**



Archaeometry is the interface between
the natural and the cultural-historical sciences.

The topics covered by archaeometry include:

dating methods, artefact studies, material structure and composition studies, **nuclear techniques**, spectroscopy techniques, mathematical methods, remote sensing techniques, conservation science, environmental reconstruction, biological anthropology, archaeological theory...

Modern archaeometry began with the discovery of radiocarbon dating in the 1950s. Introduction of radiocarbon dating caused “radiocarbon revolution” in archaeology, and changed the way of looking at various cultures, their appearance, development and fall, and interdependencies, influences etc.

Dating

An important objective in cultural heritage studies is **to order past events chronologically** by analyzing materials associated with past human activities.

Radiocarbon dating, or ^{14}C dating, is probably one of the most widely used and best known absolute dating methods.

Another important dating technique for archaeologists is **luminescence dating**, either as the thermostimulated (TL) or optically stimulated (OSL) version, depending on the radiation used to release trapped electrons in quartz and feldspar grains.

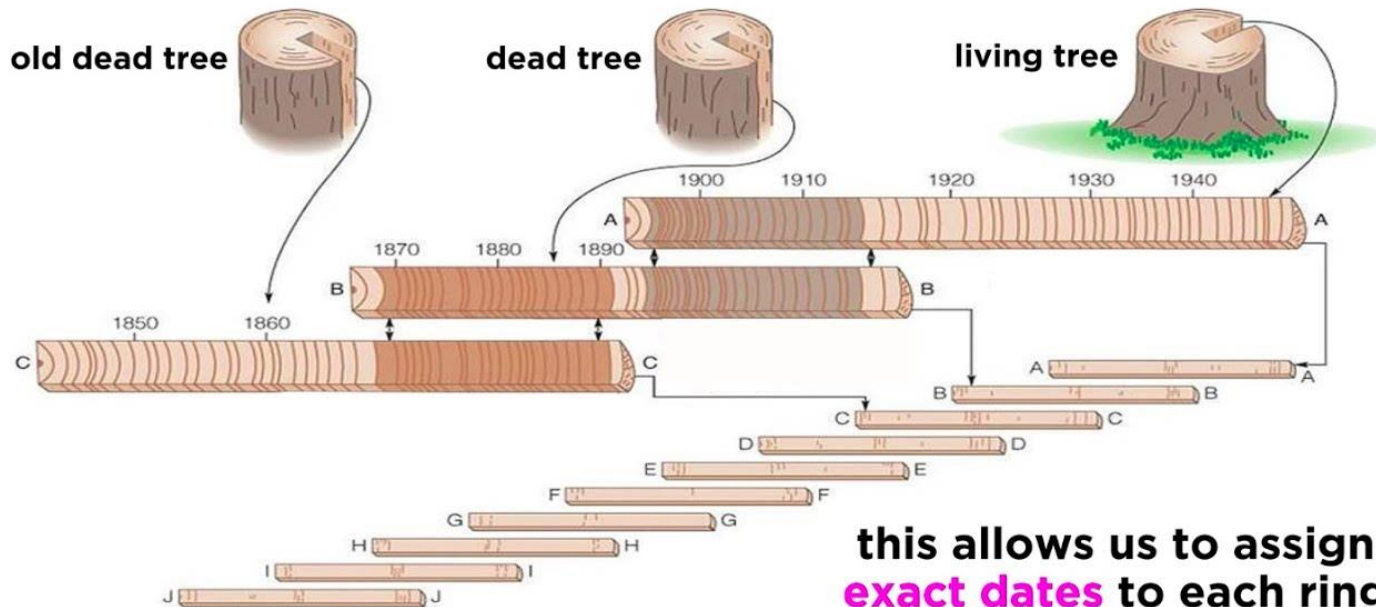
For wood and wooden samples, **dendrochronology** can be applied.

Dendrochronology

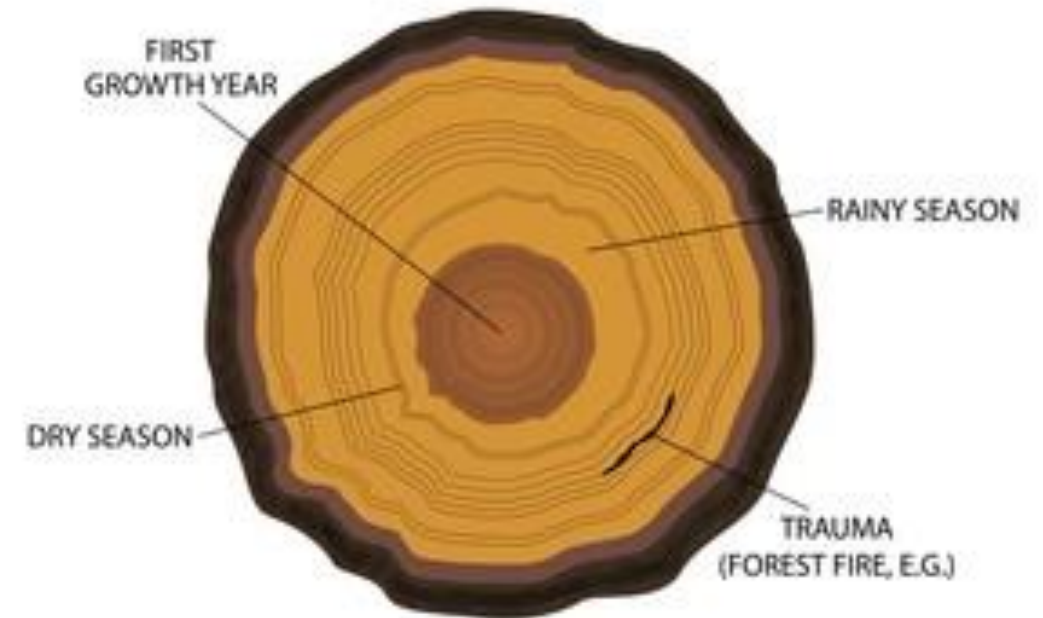
- dating of wood, i.e., wooden objects – of art, furniture, beams in buildings...)
- used also for calibration of conventional ^{14}C ages
- used for paleoenvironmental reconstruction

Principle of Crossdating

comparing **different trees** to find the ring patterns



Dendrochronology

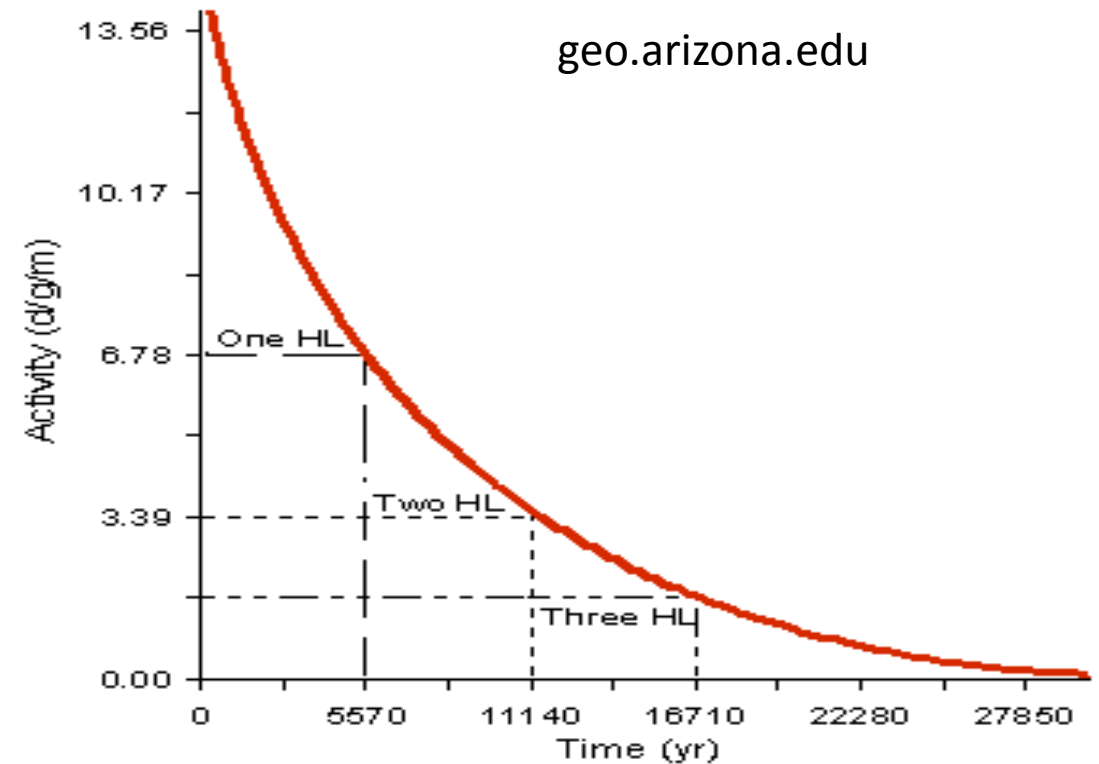


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Radiometric methods

- The decay rate expressed as half-life of an isotope, $T_{1/2}$, which is not dependent on environmental conditions or material
- Radioactive decay is a base for an accurate „clock” for measurement of time and establishment of an absolute time scale
- Time period that can be dated depends on $T_{1/2}$ of the isotope, it is about $10 \times T_{1/2}$

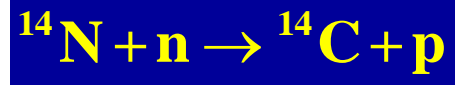
Isotope	$T_{1/2}$ (y)	Decay product
^{87}Rb	4.8×10^{10}	^{87}Sr
^{238}U	4.46×10^{10}	^{206}Pb
^{232}Th	1.4×10^{10}	^{208}Pb
^{40}K	1.3×10^9	^{40}Ar
^{235}U	7.04×10^8	^{207}Pb
^{26}Al	7.16×10^5	^{26}Mg
^{36}Cl	3.0×10^5	^{36}Ar
^{14}C	5730*	^{14}N



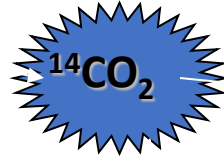
5568 y – Libby half-life

Radiocarbon dating

- Radiocarbon dating, or carbon dating, is a radiometric dating method that uses the naturally occurring radioisotope ^{14}C to estimate the age of carbonaceous materials up to about 57,000 years.
- Generation of radioactive ^{14}C occurs primarily in the upper troposphere, but C (mostly as CO_2) mixes thoroughly in the atmosphere, and is incorporated into living organisms
- When plants fix atmospheric carbon dioxide (CO_2) into organic material during photosynthesis, they incorporate a quantity of ^{14}C that approximately matches the level of this isotope in the atmosphere (a small difference occurs because of isotope fractionation, but this is corrected after laboratory analysis).
- After plants the ^{14}C fraction of this organic material declines at a fixed exponential rate due to the radioactive decay of ^{14}C .
- Comparing the remaining ^{14}C fraction of a sample to that expected from atmospheric ^{14}C allows the age of the sample to be estimated.
- After about ten half-lives (~ 57 ka) there is almost no more ^{14}C left in the tissue.



O_2

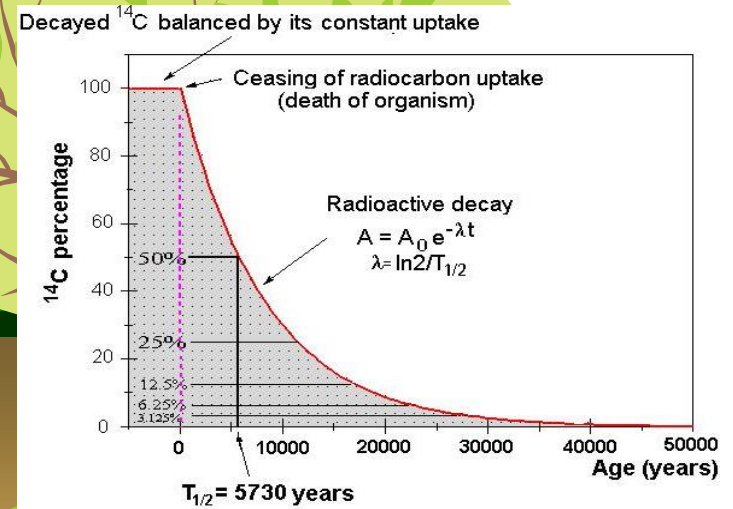
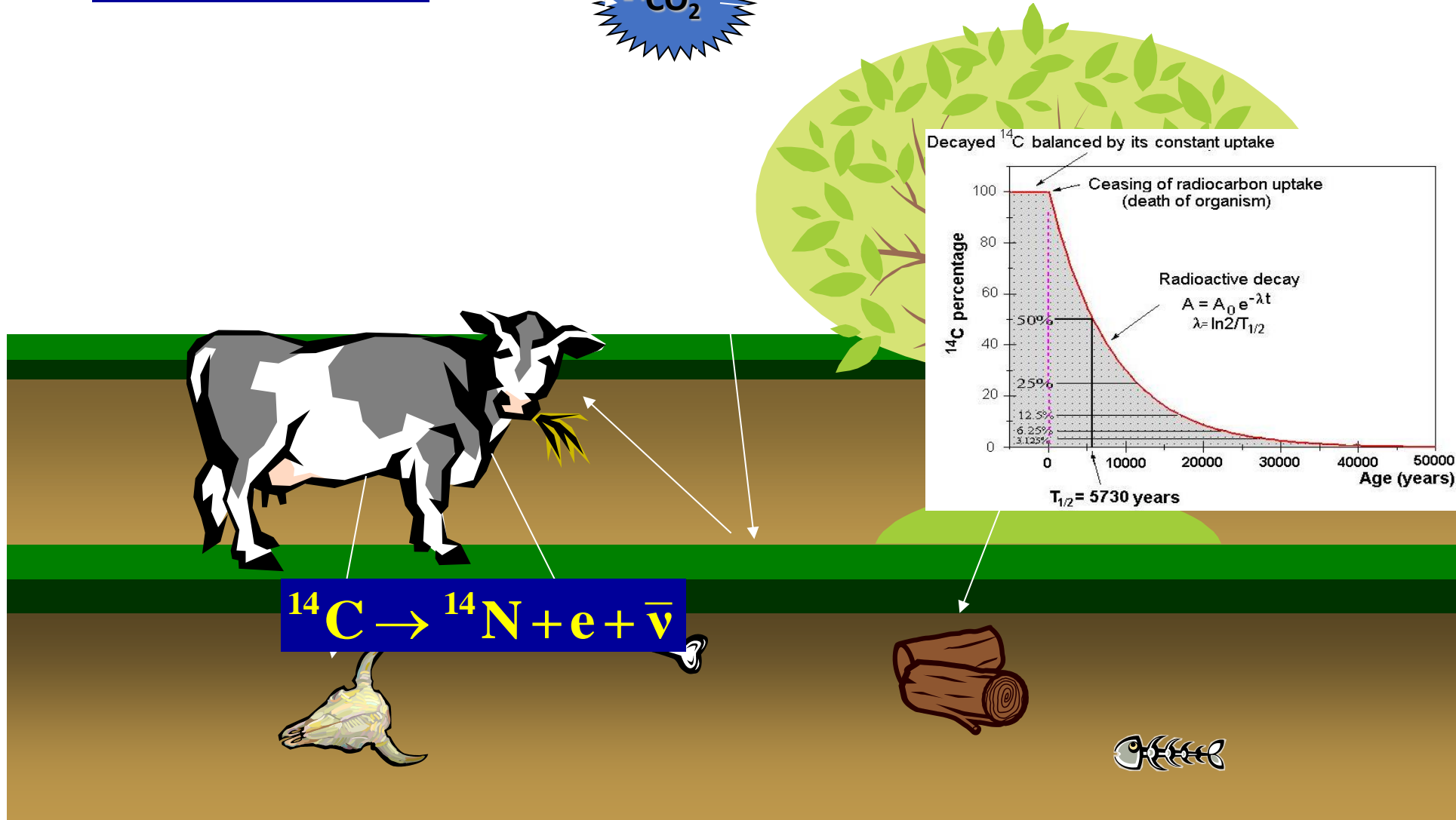


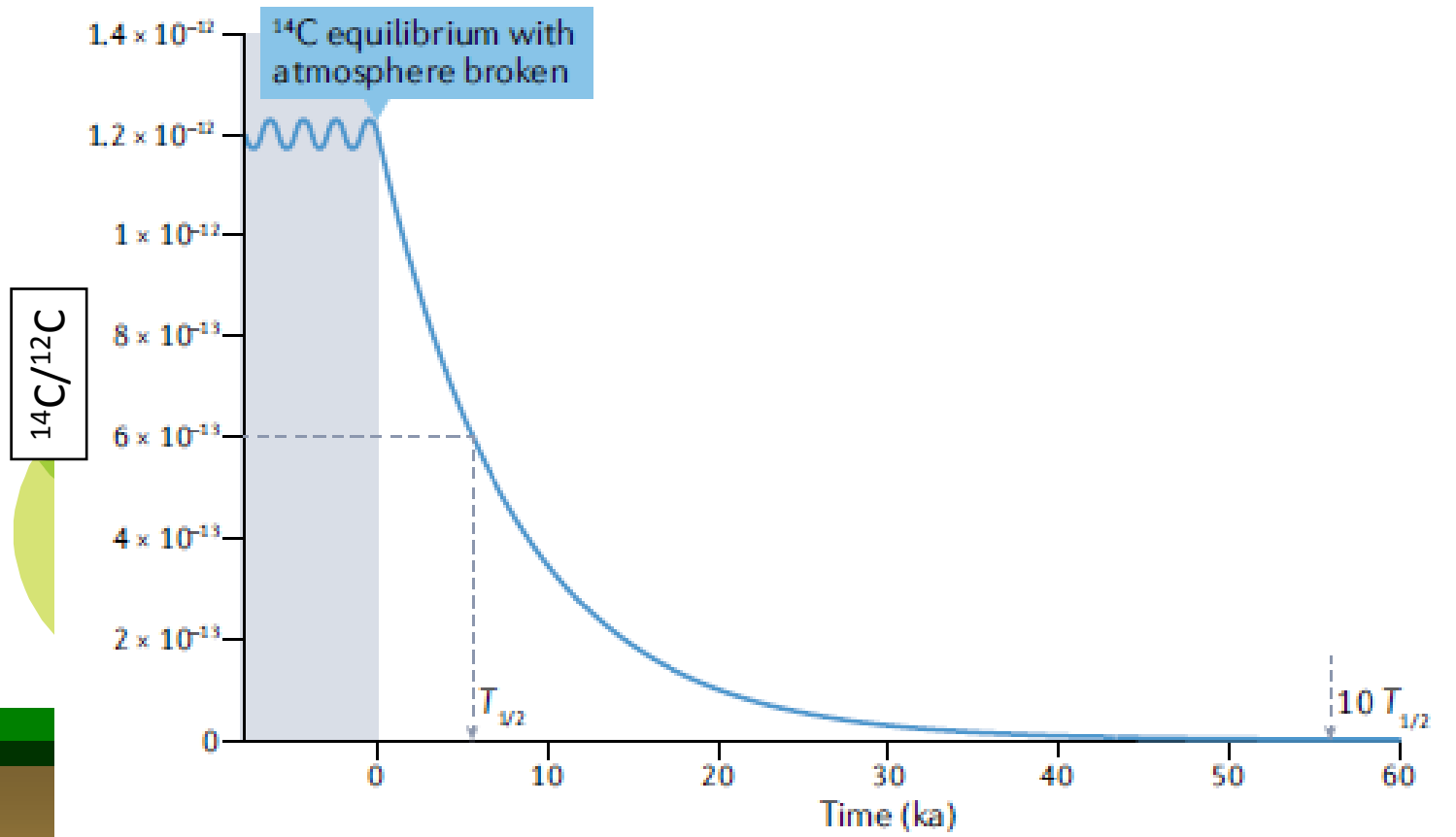
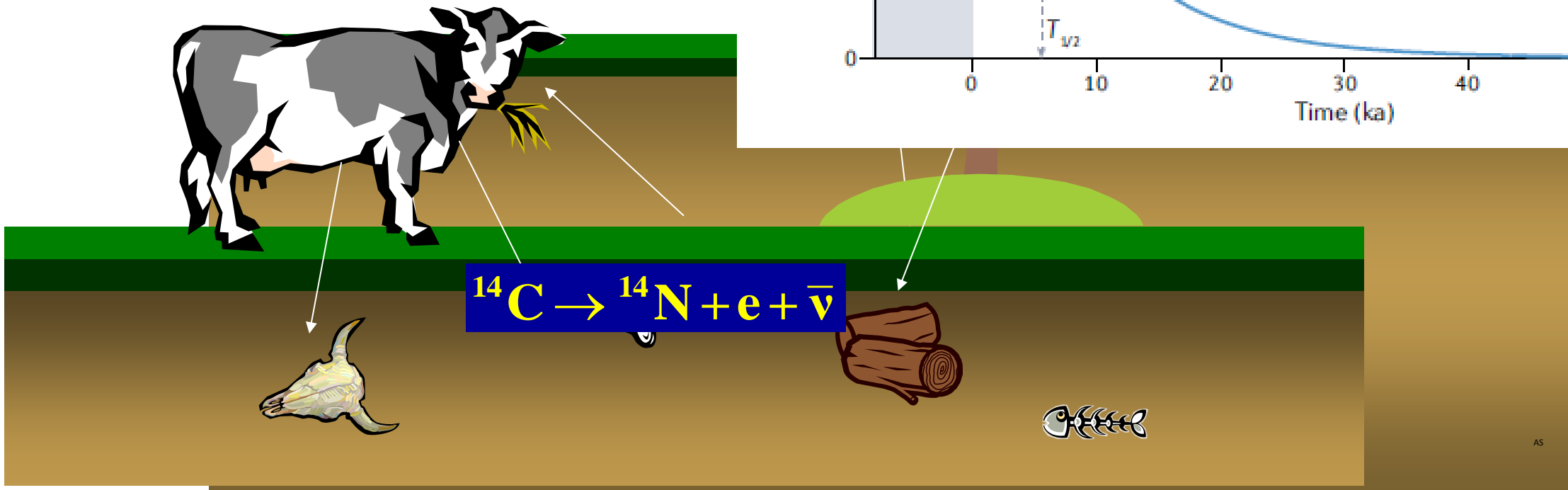
Carbon on Earth

^{12}C : 98.89 %

^{13}C : 1.1 %

^{14}C : 1.18×10^{-12} %





Discovery of ^{14}C

Obituary

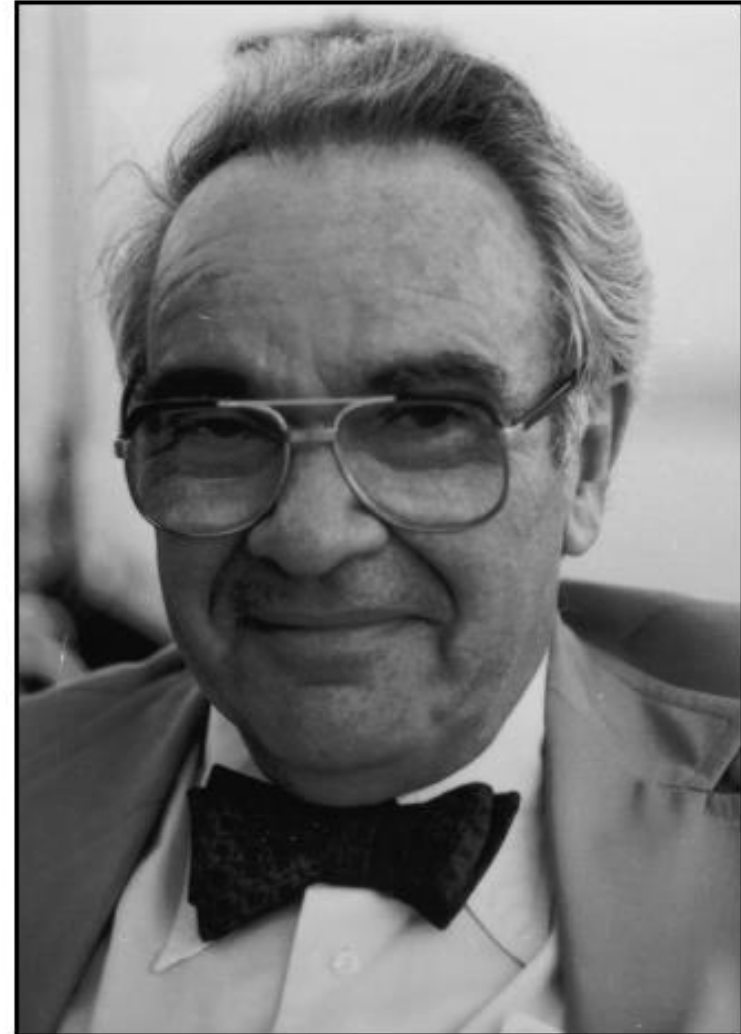
Martin Kamen

Scientist, co-discoverer of the isotope that gave archaeology carbon-dating, and innocent victim of America's Communist witchhunts

(Pearce Wright , [The Guardian](#),
Monday 9 September 2002)

The American scientist Professor Martin Kamen was the co-discoverer of the radioactive isotope carbon-14. The finding transformed biochemistry as a tracer following chemical processes in plants, while its use in the carbon dating of fossils and ancient artifacts between 500 and 50,000 years old revolutionized archaeology.

MARTIN DAVID KAMEN



27 AUGUST 1913 • 31 AUGUST 2002

Science 10 May 1963:
Vol. 140 no. 3567 pp. 584-590
DOI: 10.1126/science.140.3567.584

Sam Ruben and Martin Kamen co-discovered the isotope carbon-14 on February 27, 1940, at the University of California Radiation Laboratory, Berkeley, when they bombarded graphite in the cyclotron in hopes of producing a radioactive isotope of carbon that could be used as a tracer in investigating chemical reactions in photosynthesis. Their experiment resulted in production of carbon-14.

Ruben S, Kamen MD (1940)
Radioactive carbon of long-half life.
Phys. Rev. 57: 549

Martin Kamen, has been named one of two winners of this year's (1995) Enrico Fermi Award. The 82-year old Kamen is joined by 83-year-old physicist Ugo Fano, who won for his pioneering contributions to the theory of atomic and radiation physics.

Early History of Carbon-14

Discovery of this supremely important tracer was expected in the physical sense but not in the chemical sense.

Martin D. Kamen

When, how, and why was carbon-14 discovered? As T. S. Kuhn has remarked (1), discovery is seldom a single event that can be attributed wholly to a particular individual, time, or place. He notes that some discoveries, such as those of the neutrino, radio waves, and missing isotopes or elements, are predictable and present few problems, as far as establishment of priority is concerned. Others, such as the discoveries of oxygen, x-rays, and the electron, are unpredictable. These put the historian in a "bind" when he tries to decide when, how, who, and where the discovery was made. Much more rarely does he have a basis for an answer to the question "Why?"

I propose in this account of the "prenatal" history of carbon-14 to provide the answers to my leading questions (2). These make a story which is a fragment of the whole record. That record must be constructed by future historians who seek to probe the events of a period in which there has been an unparalleled impact of intellectual curiosity and scientific creativity on the structure of society.

The tremendous outburst of technology in the past half century, the result of the rise of nuclear science, has crowned man's quest for the philosopher's stone so successfully as to be hardly credible even to the most optimistic alchemist. Tracer methodology, an offspring of nuclear science, has provided essential support for the ever-widening and deepening knowledge of structure and function in biological systems, expressed as the dynamic science of molecular biology.

These developments have profound, but unknown, implications for the future of our social structures. They obviously bring with them an unexampled load of grist for the mills of cultural historians, social scientists,

and philosophers. Perhaps the novelists will dig into the record of these exciting times for fresh insights into the age-old drives of mankind.

Carbon-14, the long-lived carbon isotope, is the most important single tool made available by tracer methodology, because carbon occupies the central position in the chemistry of biological systems. Thus it plays, and will continue to play, an essential role in the elucidation of biochemical mechanisms, knowledge of which is essential in the further development of molecular biology. Obviously, the circumstances surrounding its discovery are valid objects of interest for the historian (3).

Initial Phases, 1934-36

In the early 1930's, nuclear physics, immersed in the great traditions of the Cambridge school led by Ernest Rutherford, was concerned primarily with observations of processes associated with the scattering of elementary nuclear particles by various atomic nuclei. Reports in those times show painstaking determinations of range-energy relations for the fundamental projectiles (protons, deuterons, alpha particles). The energies used did not exceed approximately 10 Mev, because of the limitations set by the relatively primitive accelerators and by the radiation characteristics of the naturally radioactive materials that were available. The rationale for such work, which often involved tedious attention to detail and much labor, was that if enough precise facts were put together, accurate binding energies for nuclei could be deduced. From these energies, it was reasoned, there could be derived a solid basis for further attack on the problem of the nature of nuclear forces.

By 1933, such data—binding energies, angular distributions in scattering experiments, and so on—had demonstrated that nuclear forces could be described as analogous to saturation exchange forces like those postulated previously for chemical bonding. The so-called "alpha-particle" model of the nucleus already contained the seeds of what was to be the full-fledged modern "shell" theory of nuclei, to be developed later by Maria Mayer, Eugene Feenberg, and others.

As to my part in this, I was a young, eager student and had just begun doctoral research, using the Wilson cloud chamber to study the angular distribution of neutrons scattered in collisions with protons and other nuclei. These researches were part of a general program initiated in the laboratory of W. D. Harkins in the chemistry department at the University of Chicago (4). My decision to work in this field was largely a result of the influence of D. M. Gans, Harkins' associate and an assistant professor in the department (5).

Most significantly for this history, similar work was also under way at Yale, where F. N. D. Kurie, investigating neutron-induced disintegration of light elements, had obtained certain anomalous results for the angular distributions of protons in collisions with neutrons. In 1934 he proposed a radical interpretation (6) of certain events he noted in the cloud chamber. When nitrogen was exposed to fast neutrons, for instance, he noted that in some cases the ejected nucleus produced a very long, thin track. This he ascribed to a proton, rather than to an alpha particle. Thus, he supposed that the usual reaction, $N^{14}(n,He^4)B^{11}$, was accompanied by a less frequent but readily observable reaction, $N^{14}(n,H^1)C^{14}$. (As far as I am aware, this is the first suggestion in the literature that C^{14} might exist.) Kurie also suggested, however, that the tracks he was observing might arise from H^2 , or even H^3 , and thus that the reactions $N^{14}(n,H^2)C^{13}$ and $N^{14}(n,H^3)C^{12}$ were also possibilities. In fact, he felt the reactions with emission of H^1 and H^2 were the more likely because they resulted in nuclei of known stability.

The author is professor of chemistry at the School of Science and Engineering, University of California, San Diego. This article is adapted from a paper which he presented at a meeting of the American Chemical Society in Los Angeles in April 1963, when he received the Society's 1963 Award for Nuclear Applications in Chemistry. This paper is also being printed in the May issue of the *Journal of Chemical Education*.

After discovery of radioactive carbon-14, Ruben and Kamen found that it had a half-life of about **5,700** years and that some of the nitrogen in the atmosphere was turned into carbon-14 when hit with cosmic rays. Thus, an equilibrium was reached, the newly formed carbon-14 replacing the carbon-14 that decayed, so that there was always a small amount in the atmosphere.

Ruben S, Kamen MD (1940) Radioactive carbon of long-half life. Phys. Rev. 57: 549

Ruben and Kamen had to abandon attempts to experiment with ^{14}C in 1942.

Willard Libby, at the University of Chicago, experimented with carbon-14 further. Through a series of tests, he calculated the atom's half-life to be **5,568** years. He figured that plants would absorb some of this trace carbon-14 while they absorbed ordinary carbon in photosynthesis. Once the plant died, it couldn't absorb any more carbon of any kind, and the carbon-14 it contained would decay at its usual rate without being replaced. **By finding the concentration of carbon-14 left in the remains of a plant, you could calculate the amount of time since the plant had died.** He continued to refine the concept for the next decade, calculating the age of an ancient Egyptian barge using wood samples. The science of archaeology was revolutionized and, for his efforts, **Libby received the Nobel Prize in Chemistry for 1960.**

Inventor of radiocarbon dating



Willard Frank Libby
(1908-1980)

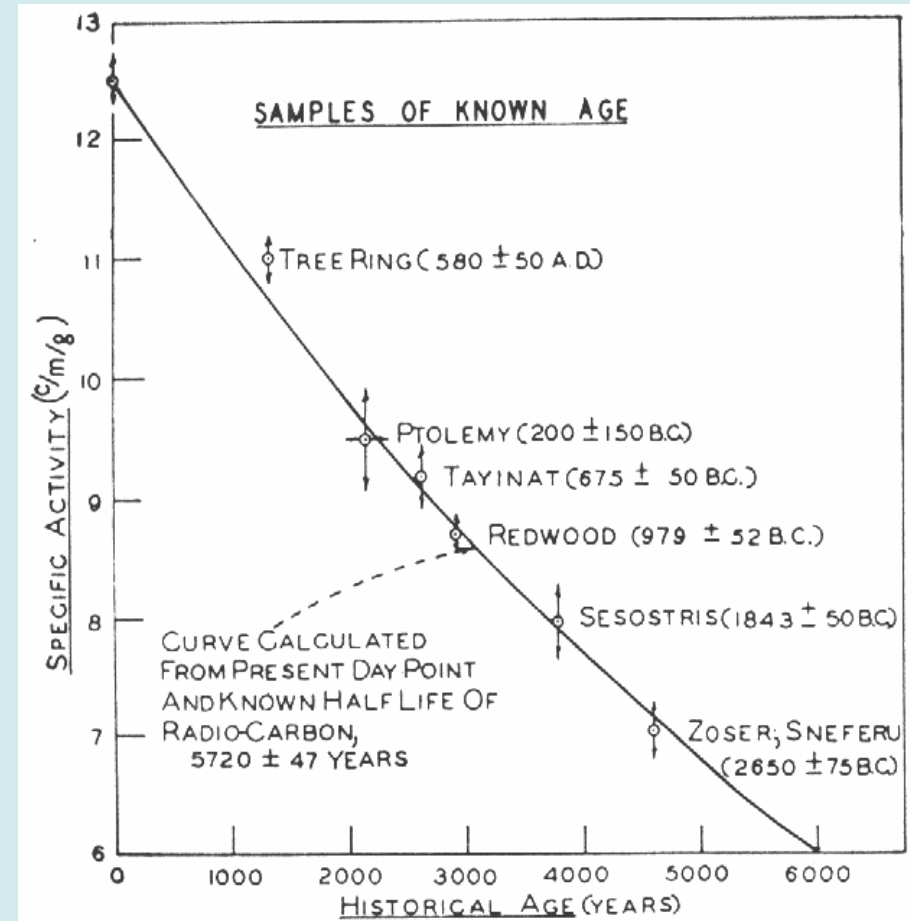
Work within the **Manhattan Project** (1942 to 1945), directed by Gen. Leslie Groves, to create the atomic bomb.

„**Radiocarbon revolution**”: Great impact upon the human sciences

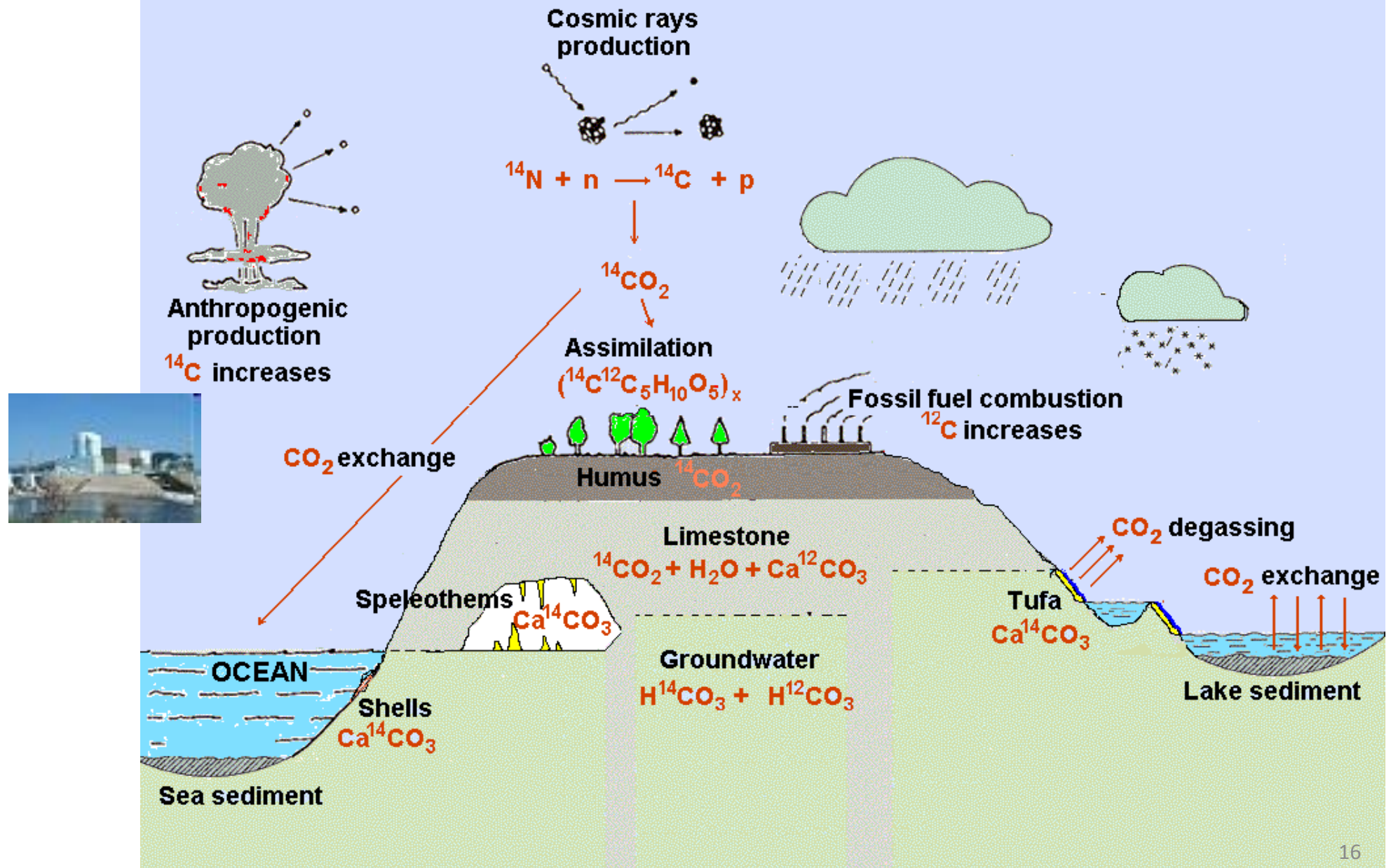
Renfrew, A.C., 1973, *Before Civilisation, the Radiocarbon Revolution and Prehistoric Europe*, London

Arnold, J.R., Libby, W.F: *Age determinations by radiocarbon content: checks with samples of known ages*. *Science* 110 (1949) 678-680

Curve of Knowns



Distribution of ^{14}C



Applications of ^{14}C

- **Dating** (determination of age) of **organic samples** (wood, grains, leaves/plants, seed, charcoal, leather, textile, linen, bones, teeth, ivory, antler and horn, peat, soil, organic sediment, dissolved organic carbon...)
- **Dating** of secondary carbonates (inorganic lake sediment, speleothemes, tufa, corals, mollusks, algal rims, dissolved inorganic carbon in water)
- Carbon cycle in nature (including atmospheric CO_2)
- Environmental monitoring (nuclear facilities)
- Oceanology, climatology
- **Forensic** science
- Medical biochemical, pharmacological applications
- Determination of biofuel fraction
- ... and many others

Sample Preparation

- Extract all carbon from a sample
(fractionation)
- All carbon only from the sample
(contamination)

“Each sample is an individual”
and should be treated so,
→ one should collect all possible information about
the sample and its environment



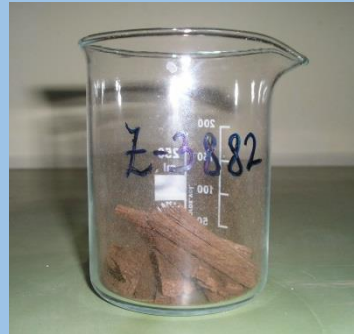
Bone



Bone collagen



Wood



Pretreated wood



Carbonized wood



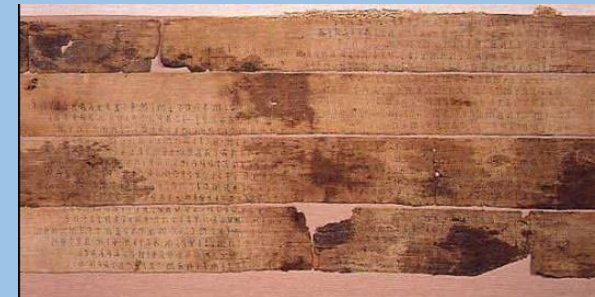
Speleothem



Lake mud - carbonate



Shell



Linen – *Liber Linteus Zagradiensis*

Materials that have been radiocarbon dated

- charcoal, wood, twigs, seeds, peat, pollen, resins
- bones
- hair, leather, parchment, fabrics, papers
- lake mud, soil, water, shells, corals, speleothem

Virtually all samples containing carbon from atmospheric origin



Unfortunately, not everything is easy to date. This woolly mammoth leg bone found on Gabriola does not contain enough collagen for ^{14}C analysis. (limit – efficiency of collagen extraction 1 %)

Bone is mostly *hydroxy-apatite*, a form of calcium phosphate: $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$
The bone probably dates back to the Port Moody Interstade *ca.* 18,000 BP, but that's a guess.

“Each sample is an individual”
and should be treated so,
→ one should collect all possible information
about the sample and its environment

Measurement - radiometric

- low relative abundance of ^{14}C atoms ($<10^{-10}\%$)
- low electron energy ($<156\text{ keV}$)
- low activity of ^{14}C in natural materials, $< 13\text{ decays/min/g}$ of carbon



Radiometric – number of decays per time (i.e., decay rate) of ^{14}C per mass of carbon

required mass of C: 1 - 5 g

Required mass of samples: 10 - 50 g

Gas proportional counters (GPC)

Liquid scintillation counters (LSC)

All sample pretreatment and preparation techniques, as well as measurement techniques of large efficiency

Measurement - AMS

Accelerator Mass Spectrometry (AMS) – number of ^{14}C atoms is counted, together with the number of ^{12}C and ^{13}C

Required mass: <2 mg C, <1 g sample

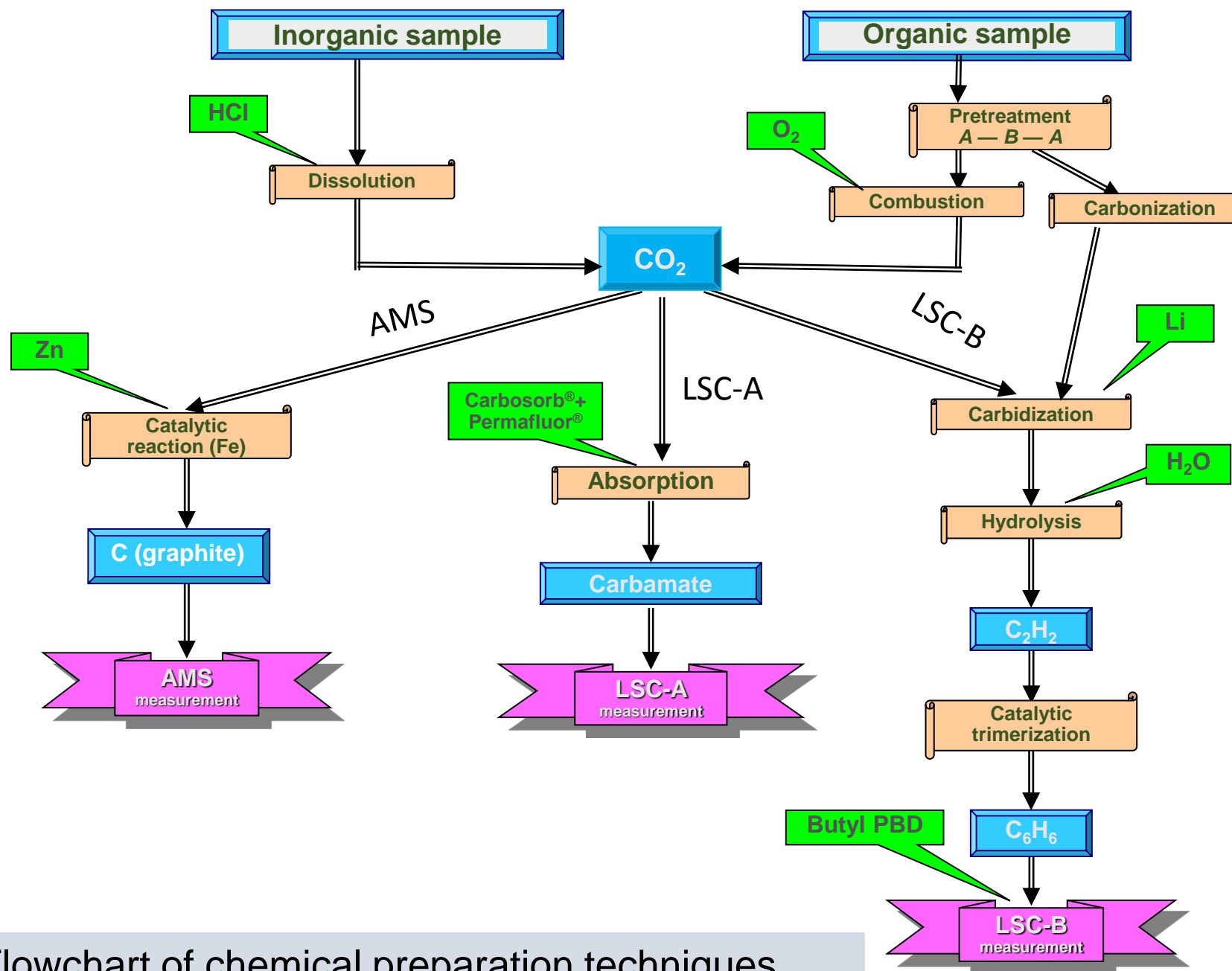
AMS dating is relatively more expensive, requires only about a gram of a sample, short measurement, high throughput

All sample pretreatment and preparation techniques, as well as measurement techniques of large efficiency

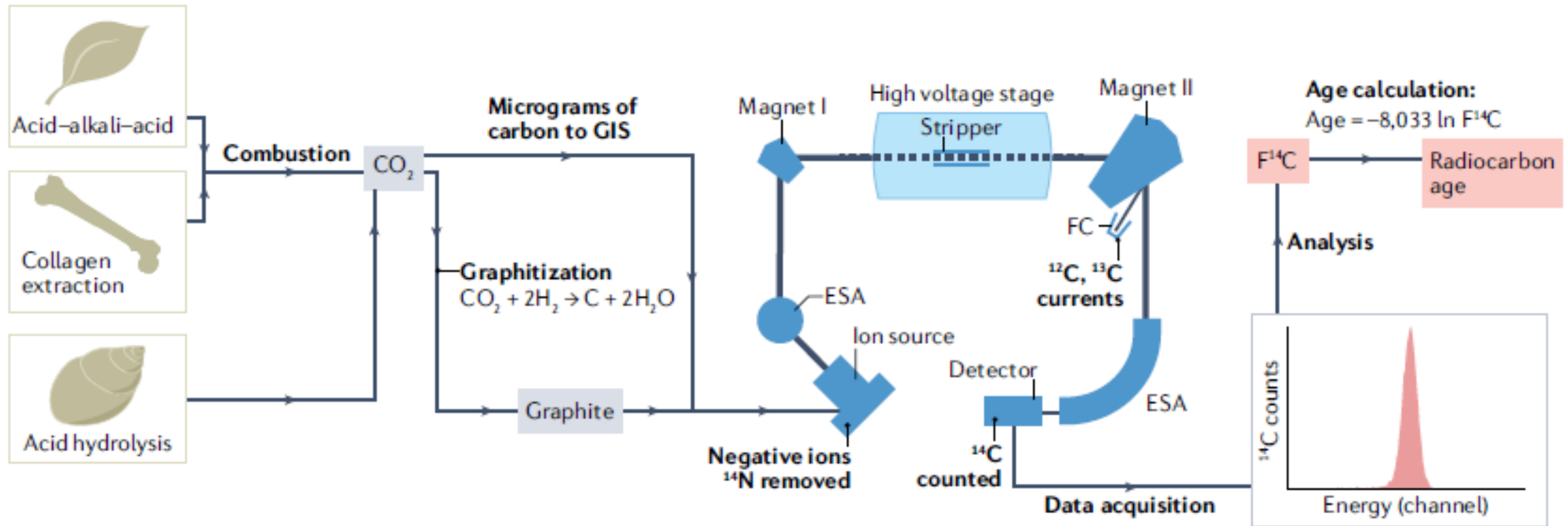
Comparison of characteristics (precision, complexity, and price) of various techniques of the ^{14}C method.

Measurement technique	Sample types	Required mass of carbon	Complexity *	Precision *	Price *	Main drawback	Good for dating
AMS	all	~1 mg	3	4	4	representativeness of the sample	
LSC-benzene	all	~4 g	4	3	3	time-consuming	
LSC- CO_2	all	~0.6 g	2	2	2	high uncertainty low sensitivity	
LSC-direct	liquid fuels	10 ml of liquid	1	1	1	quenching	

* The higher the number, the more complex the method
/ the lower the uncertainty / the higher the price



Flowchart of chemical preparation techniques for **AMS**, **LSC-A**, **LSC-B** methods



Hajdas I. et al, Radiocarbon dating, Nature Reviews, Methods Primers, article ID (2021)1:62

Radiocarbon dating – basic assumptions

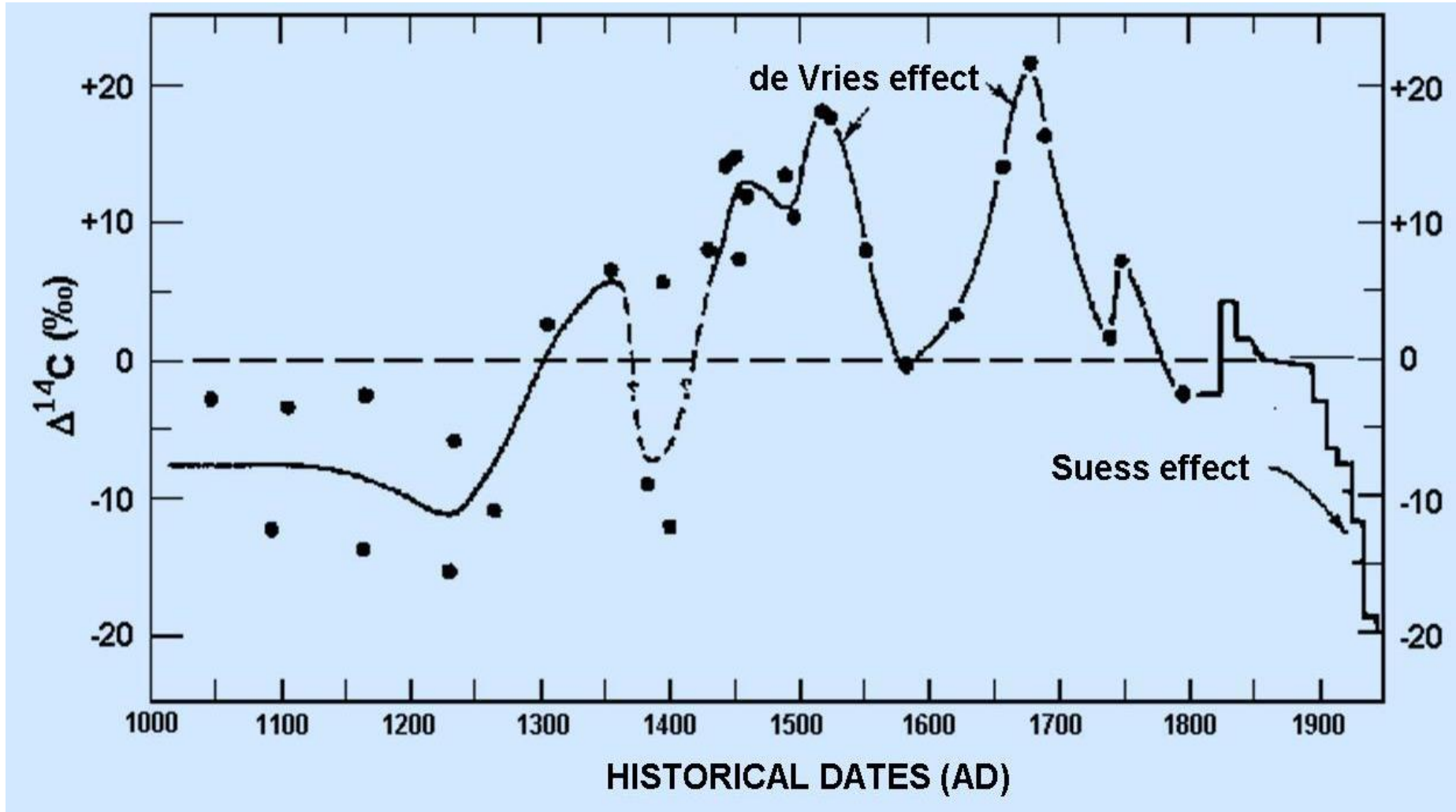
1. **Constant production of ^{14}C** during last 60 000 years
→ calibration!
2. **Uniform distribution of ^{14}C** in the biosphere
(stationary, well-mixed reservoirs)
→ $\delta^{13}\text{C}$ normalization
3. **Carbon originates only from the sample**
→ avoiding contamination...
4. **Origin of carbon in a sample known** („closed system”)
→ reservoir effect

Constant production? - Calibration

The measurement of ^{14}C decay provides the raw radiocarbon age of any carbon-based material under the **supposition that the specific ^{14}C activity over millenia has been constant** (100 pMC). However, over time there have been small fluctuations in this activity in the atmosphere caused by variations in cosmic-ray intensities due to:

- **Fluctuations of Earth's magnetic field** - strong field shields Earth from cosmic rays causing modulation of the production of radiocarbon in the upper atmosphere;
- **Solar variability:** The sun produces a powerful solar wind that deflects cosmic rays;
- **Carbon cycle:** Fluctuations in Earth's carbon reservoirs, especially circulation of deep ocean waters.

Variations in ^{14}C production produce differences between obtained radiocarbon ages and calendar ages.



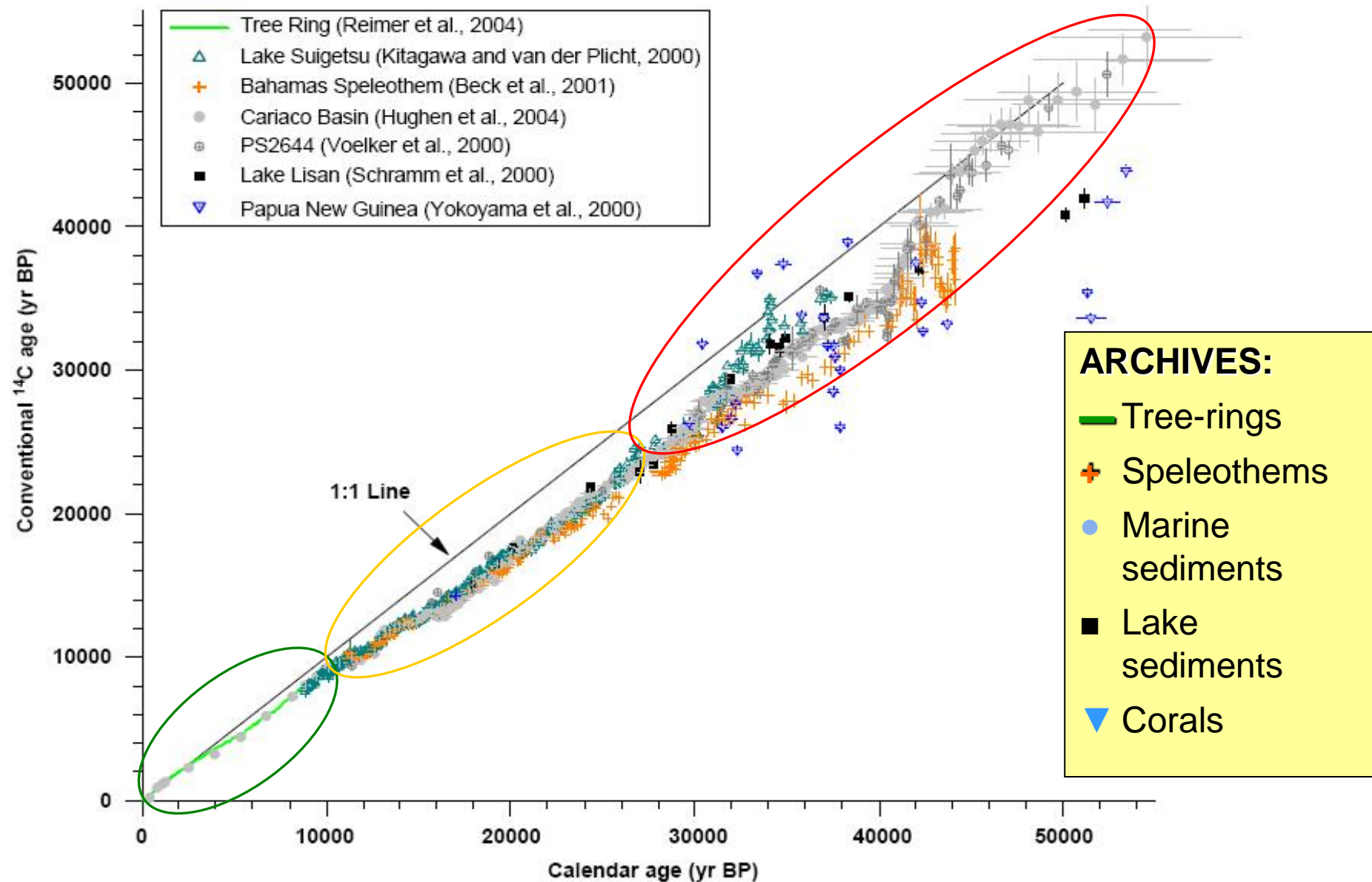
Fluctuations in radiocarbon activity over last 1000 years

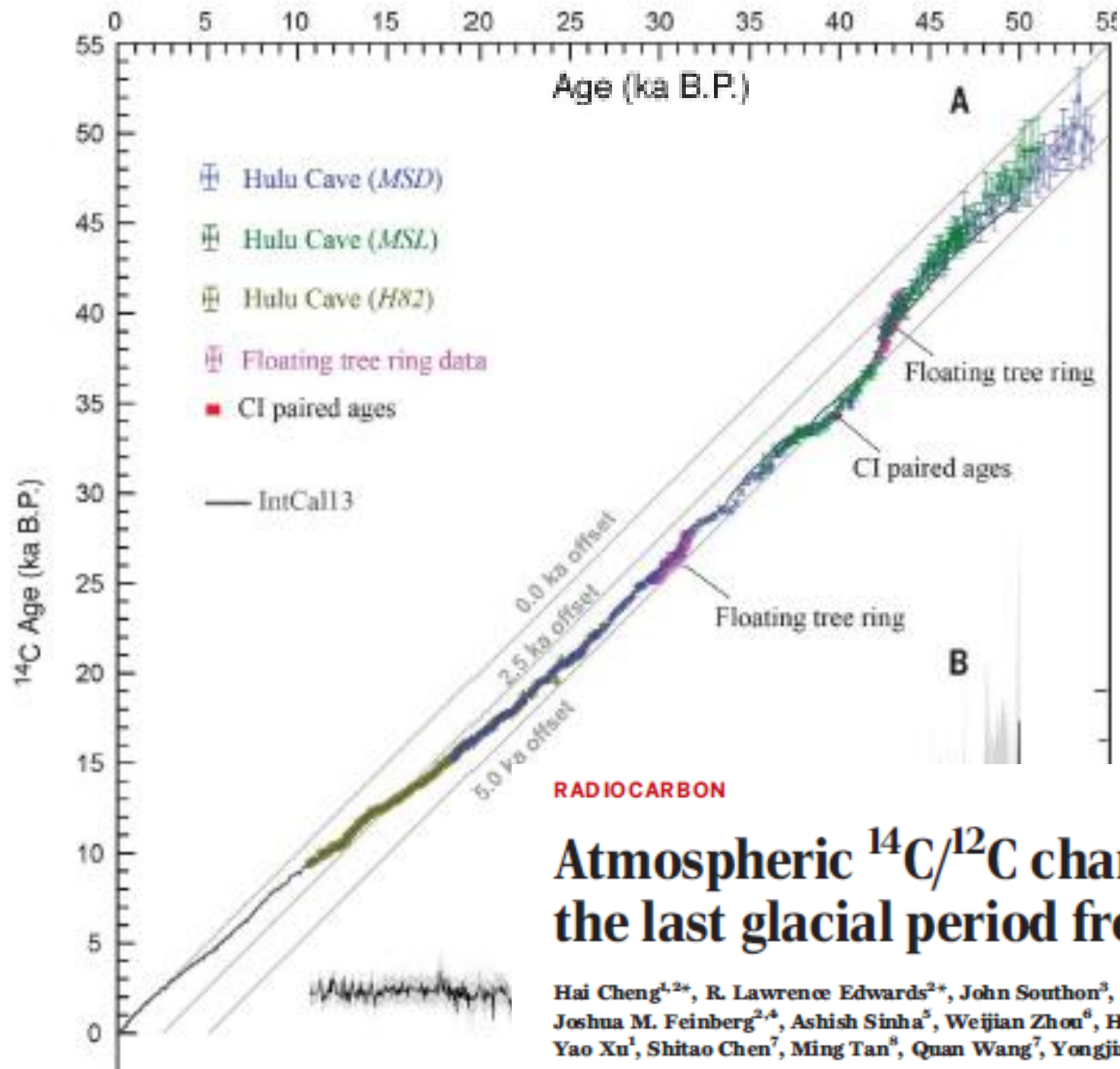
- conventional ^{14}C years do not directly equate to calendar years because atmospheric ^{14}C concentration varies through time due to changes in the production rate
- a **calibration is required** to convert the conventional radiocarbon age to the calendar age
- **calibration curves** should be based on absolutely dated record that has carbon incorporated directly from the atmosphere at the time of formation

Four decades of joint research by the **dendrochronology** and radiocarbon communities have produced a radiocarbon calibration data set of remarkable precision and accuracy extending from the present to approximately 12 000 calendar years before present. The extension to more than 50 000 years has been achieved by high precision paired $^{230}\text{Th}/^{234}\text{U}/^{238}\text{U}$ and ^{14}C age determinations on pristine **coral samples** that created a calibration curve from 12 000 to 50 000 years before present.

Radiocarbon calibration curves

R.G. Fairbanks et al. / Quaternary Science Reviews 24 (2005) 1781–1796

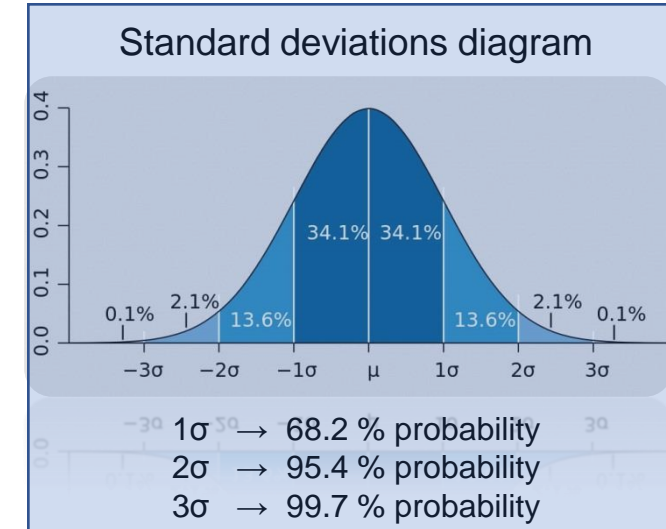
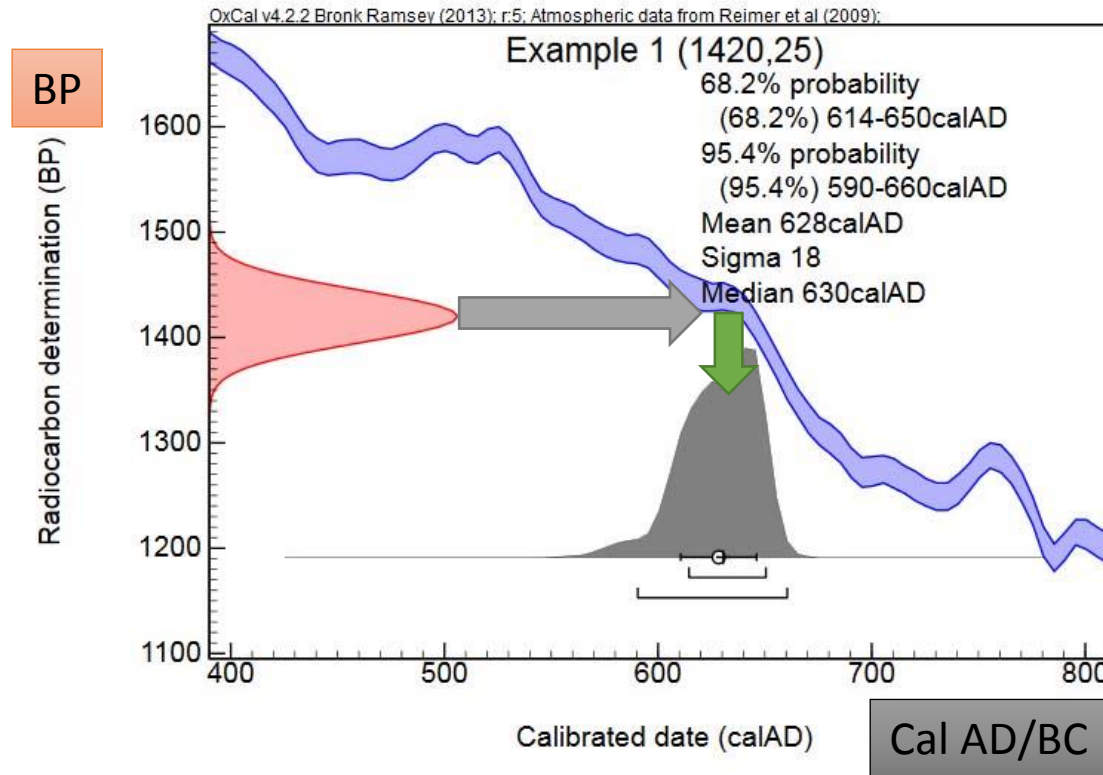




Cheng et al., Science 362, 1293, 2018

How to perform calibration

<https://c14.arch.ox.ac.uk/oxcal.html>

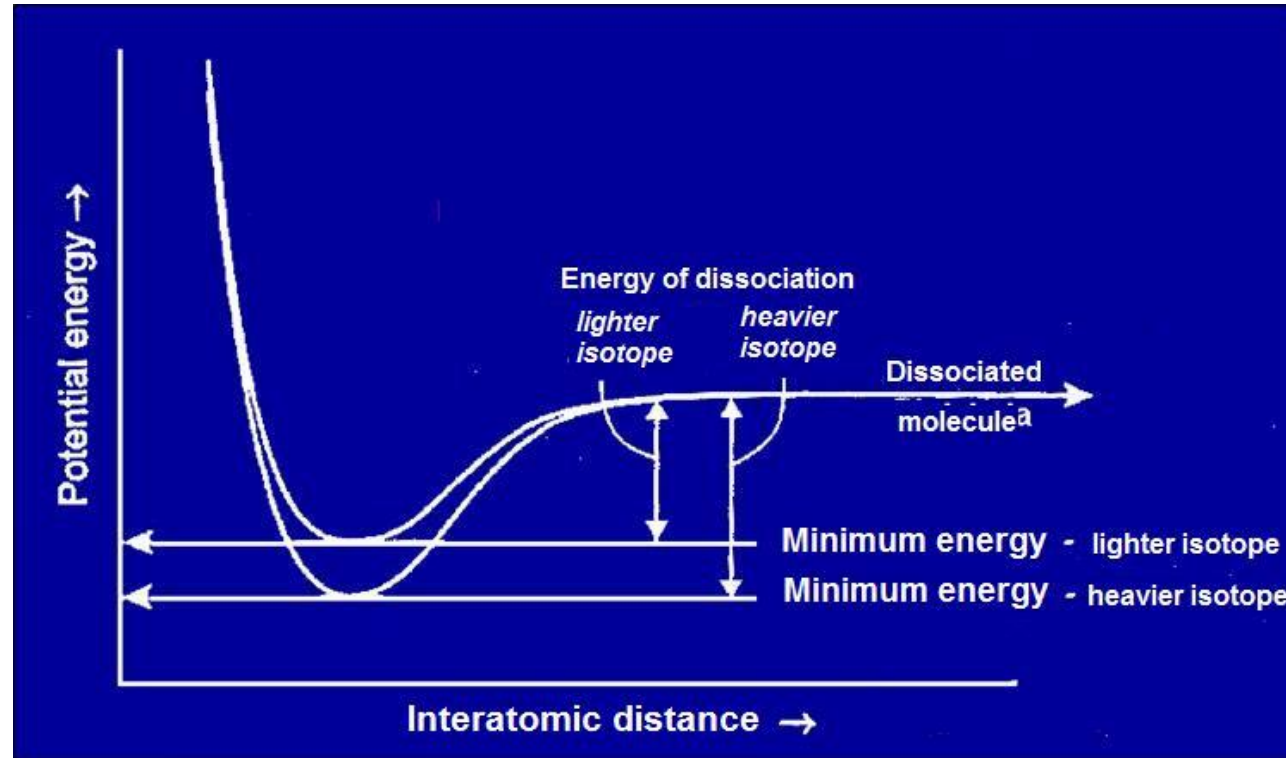


Example of a single calibrated date

Conventional radiocarbon ages (ordinate) in years BP are represented as the Gaussian curve with mean and standard deviation (uncertainty) being 1420 ± 25 BP. Calibrated values, in calendar years, are obtained by transferring the values on ordinate over calibration curve to the abscissa. Results can be presented by 1σ , 2σ or 3σ probabilities and by mean or median values.

Isotopic fractionation

Isotopes behave differently in physical, chemical or biochemical processes. Molecules composed of heavier isotopes are more stable (have a higher dissociation energy) than those composed of lighter isotopes.



Distribution of carbon isotopes (^{12}C , ^{13}C and ^{14}C) in various substances and organisms is **not equal**. Enrichment of one isotope relative to another in a chemical, physical or biochemical processes is called **isotopic fractionation**.

Isotopic fractionation

- Isotopic fractionation is defined as the relative partitioning of the heavier and lighter isotopes between two coexisting phases in a natural system - various physical and chemical processes are mass dependent and thus they cause isotopic fractionation
- Usually, the ratio of the heavier, less abundant, isotope to the lighter, most abundant, isotope ($^{13}\text{C}/^{12}\text{C}$) is compared in various compounds

$$\delta^{13}\text{C} = \frac{R_{\text{sample}} - R_{\text{standard}}}{R_{\text{standard}}} \quad R = ^{13}\text{C}/^{12}\text{C}$$

the quantity δ as the relative difference, expressed in ‰, of the ratio R in a sample and R in the standard

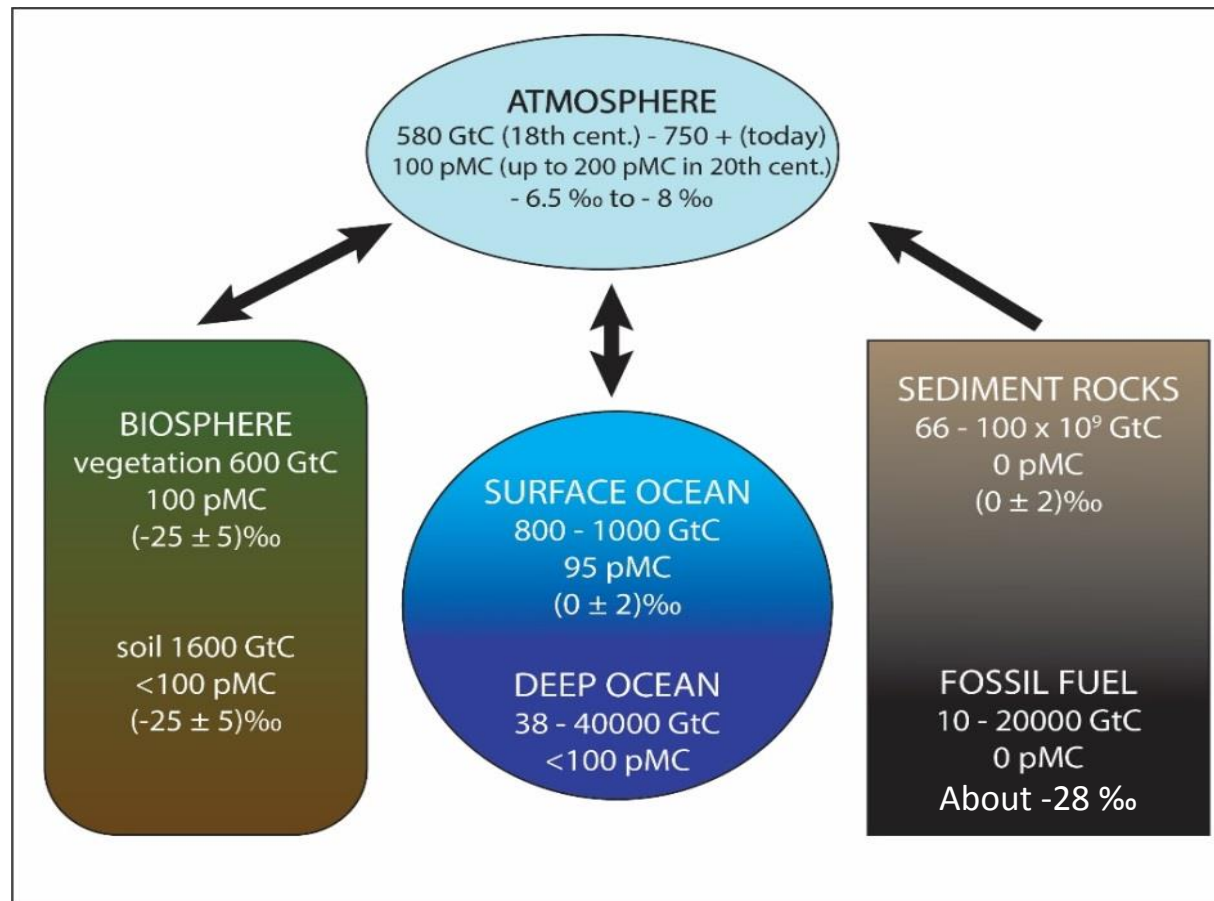
The international standard for $\delta^{13}\text{C}$ is **VPDB** (Vienna PDB, for Pee Dee Belemnite)

$$\delta^{13}\text{C} = 0 \text{ ‰}$$

The δ value is usually small, and therefore it is expressed in ‰.

Isotopic fractionation occurs to both ^{13}C and ^{14}C , and the fractionation for ^{14}C is known to be about twice that for ^{13}C , the $\delta^{13}\text{C}$ value of the sample material can be used for fractionation correction of the measured ^{14}C activities.

It has become common practice to normalize ^{14}C results to the value $\delta^{13}\text{C} = -25 \text{ ‰}$



Schematic presentation of carbon reservoirs that take part in carbon cycle. For each reservoir the following values are given: carbon inventory in GtC (Gigatone carbon), relative specific activity $a^{14}\text{C}$ in pMC, and $\delta^{13}\text{C}$ in ‰.

Isotopic fractionation

If a sample shows a lower ratio of ^{13}C to ^{12}C than exists in the atmosphere, it is reasonable to expect that the amount of ^{14}C to ^{12}C has also been reduced, making the sample appear older than it actually is.

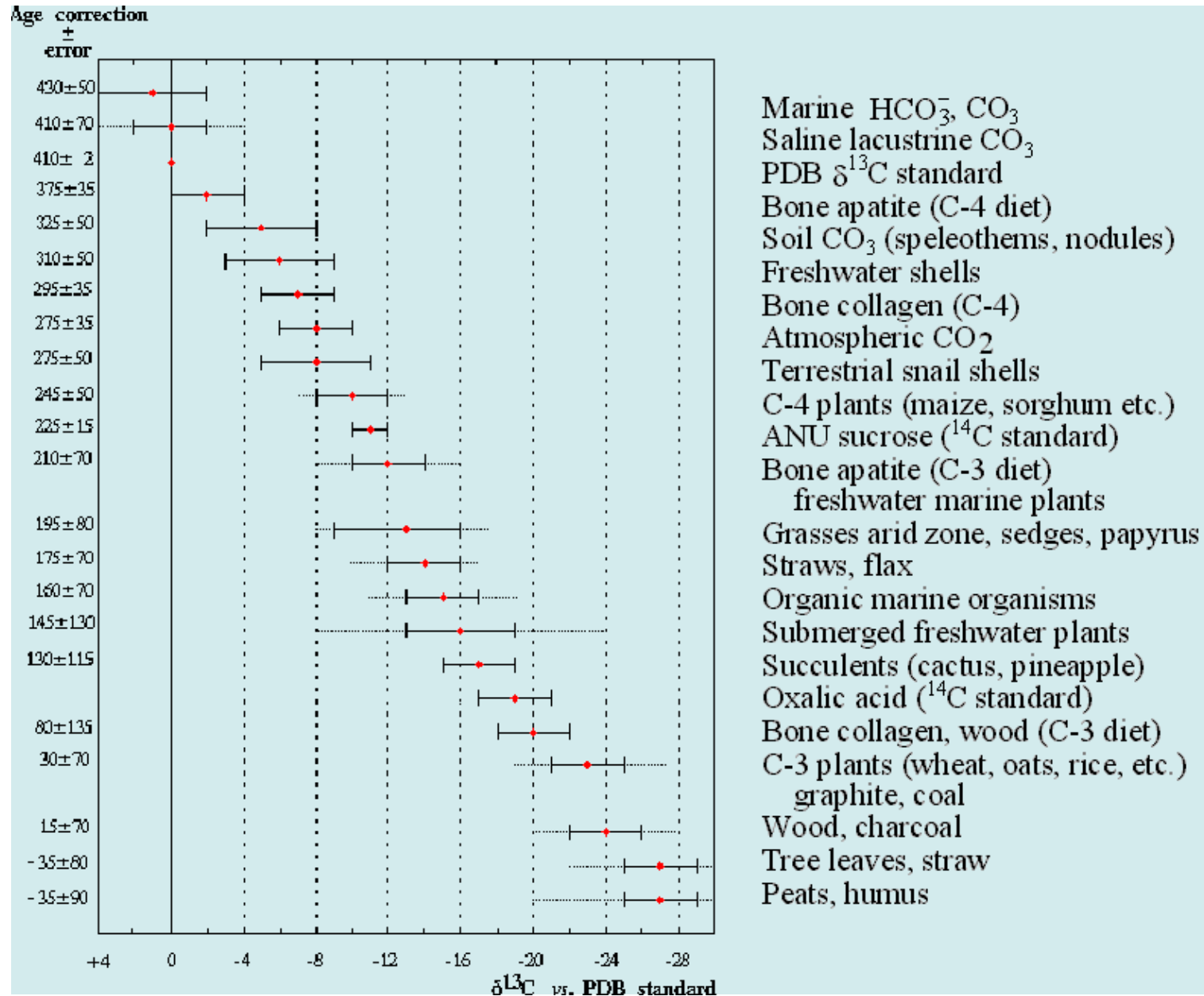
It is standard practice to correct for deviations of ^{13}C to ^{12}C from the norm.

These will be reported as a "delta 13 C" normalization (also sometimes called correction for ^{13}C). The $\delta^{14}\text{C}$ fractionation is commonly taken in calculations to be 2 times greater. **The norm for $\delta^{13}\text{C}$ is -25 ‰.**

$$\epsilon^{14}\text{C} = 2 \cdot \epsilon^{13}\text{C} (\text{‰})$$

It is sometimes important to note that measurements published in articles before *ca.* 1990 either did NOT make this adjustment, or used a different standard such as 0.0 ‰. These have to be re-evaluated before being compared with more recent measurements.

The introduction of isotopic fractionation corrects the conventional age of various sample materials according to the following table:



Contamination of samples

Sample to be measured should not be subjected by any process other than natural radioactive decay. Anything that affects the carbon content of a sample is considered as contaminant.

Natural Contaminants

Limestone, humic or fulvic acids, and plant roots are examples of natural contaminants because they introduce additional ^{14}C on the sample. They are introduced into the artifacts from the surrounding material, e.g. soil.

Artificial Contaminants

Artificial contamination happens during post excavation particularly during collection, conservation, and packaging of the artifacts.

The sample age t can be contaminated by fraction F_c of carbon of age t_c .

The effect of contamination depends on:

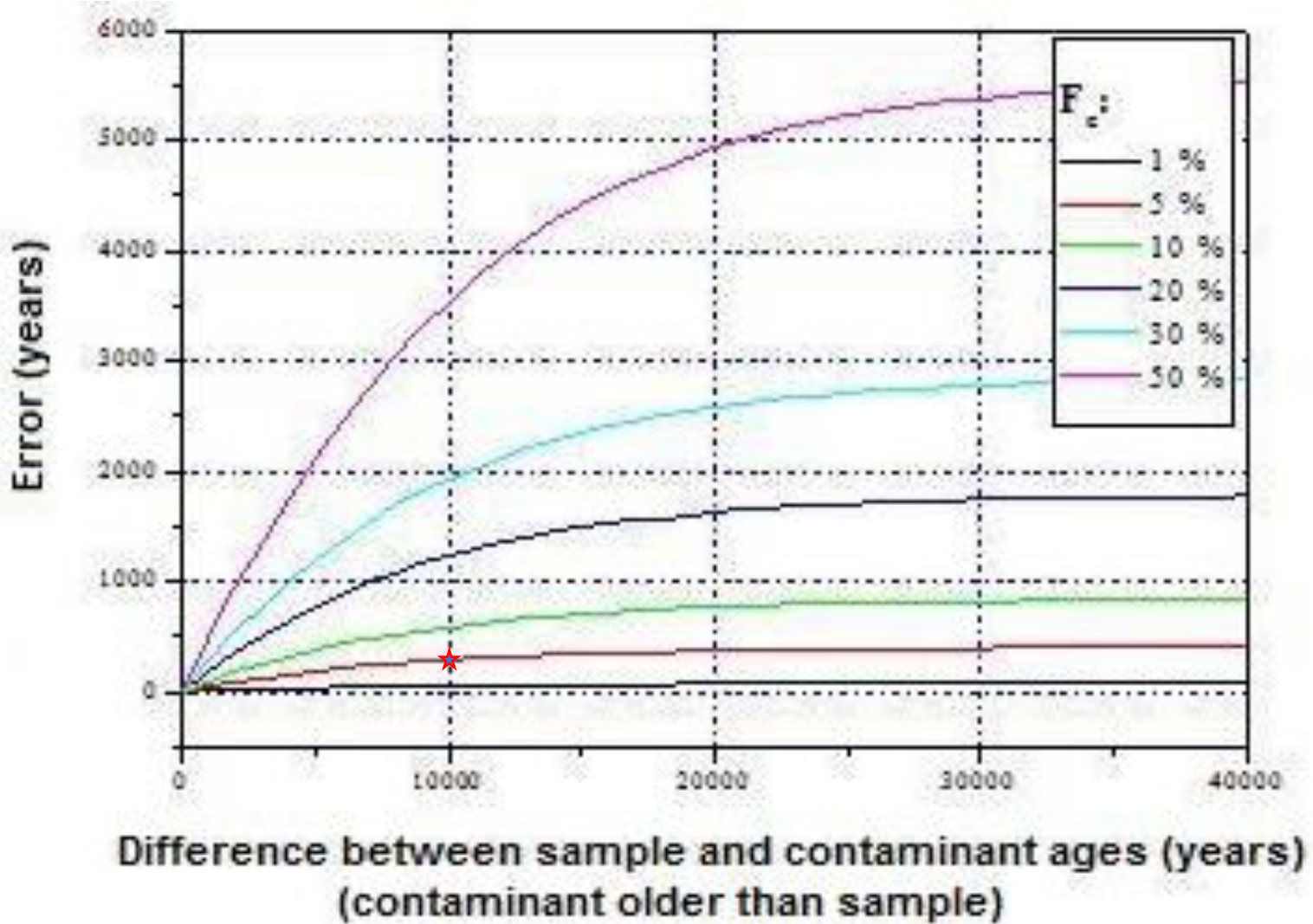
- A) The difference in age of the sample and the contaminant ($t - t_c$)
- B) "Course" of contamination (contaminant may be older or younger than the sample)
- C) Portion (fraction) of the contamination F_c .

Effects of sample contamination by „¹⁴C-free” carbon

Contamination by „old” carbon

$a_c^{14}\text{C}=0\%$ → $t_c>50\,000\text{ y}$

„True“ sample age t (y)	Apparent age t _{meas} (y) after contamination with „old“ carbon			
	F _c =5%	F _c =10%	F _c =20%	F _c =50%
500	900	1 300	2 200	6 000
5 000	5 400	5 800	6 700	10 500
10 000	10 400	10 800	11 700	15 500
20 000	20 400	20 800	21 700	25 500



Portion (fraction) of the contamination F_c

Weak influence on „old” samples

Effects of sample contamination by modern carbon

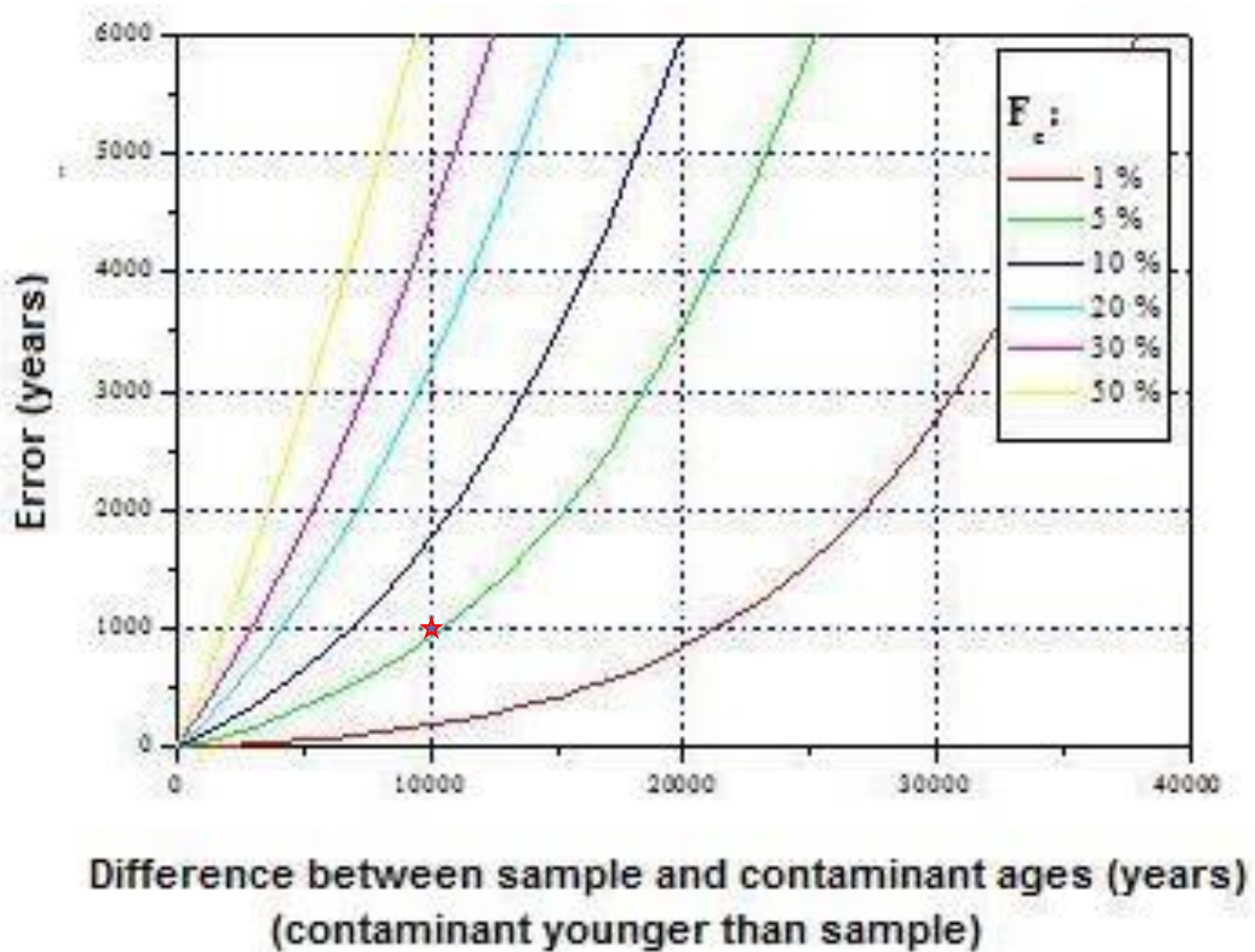
Contamination by „modern” carbon

$$a_c^{14}\text{C}=100\% \rightarrow t_c=0 \text{ y}$$

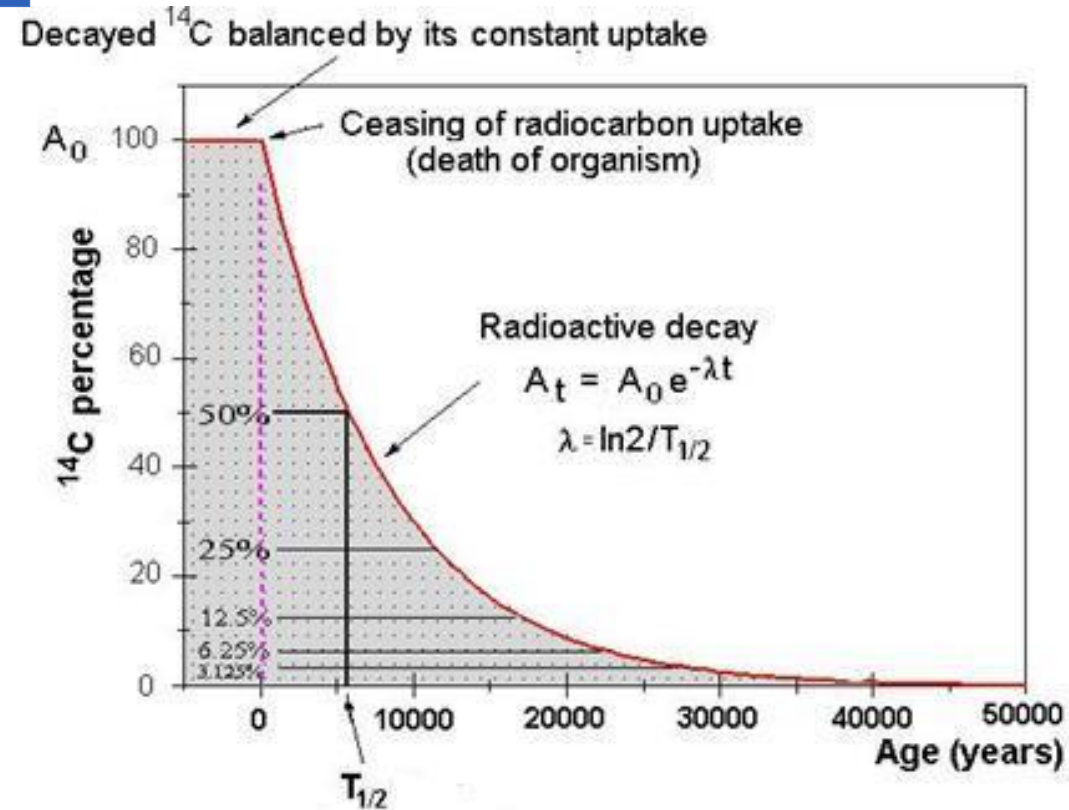
„True“ sample age t (y)	Apparent age t_{meas} (y) after contamination with „modern“ carbon			
	$F_c=1\%$	$F_c=5\%$	$F_c=20\%$	$F_c=50\%$
5 000	4 950	4 650	3 700	2 100
10 000	9 800	9 000	6 800	3 600
20 000	1 910	16 500	10 600	5 000
30 000	27 200	21 000	12 200	5 400

Portion (fraction) of the contamination F_c

Large influence on „old” samples



AGE CALCULATION



A_0 initial specific activity of sample in the moment of death (activity of **modern standard**)

A_t final specific activity of sample when measured (time t elapsed after the death)

$$T_{1/2} = \ln 2 / \lambda = \tau \cdot \ln 2$$

$T_{1/2}$ half-life (years) λ decay constant (years^{-1}) τ mean lifetime (years)

Maximal age to be measured is limited by half-life ($\sim 10 \cdot T_{1/2}$): $\sim 50\,000$ to $60\,000$ years

Conventional radiocarbon age

Radioactive decay law gives:

$$A_{SN*} = A_{ON*} \cdot e^{-t/8030} \quad \rightarrow \quad t = -8030 \cdot \ln \frac{A_{SN*}}{A_{ON*}}$$

where

8030 represents the ^{14}C mean lifetime according to conventional ^{14}C half-life 5568 y

A_{ON*} is the isotopically normalized net activity of the **modern standard**

A_{SN*} is the isotopically normalized net activity of the **sample**

Radiocarbon age is obtained under internationally accepted conventions:

- a **half-life of 5568 years** (consequently, mean lifetime is 8030);
- use **appropriate modern radiocarbon standard**;
- Normalization for sample **isotopic fractionation** ($\delta^{13}\text{C}$);
- use of **1950 AD as 0 BP**, i.e. all ^{14}C ages head back in time from 1950;
- assumption that all **^{14}C reservoirs** have remained **constant** through time.

Radiocarbon half-life

**Conventional or Libby
half-life**

5568 years

the Libby 'half-life' is *uncertainty-free*.

True or Cambridge half-life

5730 \pm 40 years

Godwin, H, 1962. *Half-life of radiocarbon*. Nature
(1962), p.195:984

Modern and absolute ^{14}C standards

Modern standard depends on year of measurement (y) and has to be corrected to decay between 1950 and year (y) of actual measurement date in order to obtain absolute international standard activity:

$$A_{\text{ABS}} = A_{\text{ON}*} \cdot e^{\frac{(y-1950)}{8267}}$$

where 8267 is decay constant corresponding to true ^{14}C half-life of 5730 years

Absolute radiocarbon standard (A_{ABS}) is the activity of 1890 wood corrected for radioactive decay to 1950. Thus 1950, is year **0 BP** (*Before Present*) by convention in radiocarbon dating and is deemed to be the 'present'. It corresponds to the hypothetical specific activity of atmospheric carbon in year 1950, measured in 1950, under assumption that this atmosphere is free from human perturbations and normalized to $\delta^{13}\text{C} = -25\text{‰}$. Specific activity is:

$$A_{\text{ABS}} = 226 \text{ Bq/kg carbon}$$

This specific activity, equivalent to natural equilibrium atmospheric ^{14}C activity, is in radiocarbon articles often expressed in units **pMC (percent of modern carbon)** as:

$$A_{\text{ABS}} = 226 \text{ Bq/kg C is equivalent to } a^{14}\text{C} = 100 \text{ pMC}$$

QUANTITIES AND UNITS SPECIFIC FOR ^{14}C

There are several conventions for expression of radiocarbon activities.

Basic quantity is specific activity $A^{14}\text{C}$, i.e., activity per unit mass of carbon, Bq/kgC.

However, more common unit is relative specific activity $a^{14}\text{C}$,

$$a^{14}\text{C} = \frac{A^{14}\text{C}}{A_{\text{ref}}^{14}\text{C}}$$

$A_{\text{ref}}^{14}\text{C}$ is the specific activity of a referent material from AD 1950
(AD = Anno Domini) equal to 226 Bq/kgC

The value of $a^{14}\text{C}$ for natural samples (before AD 1950, i.e., before anthropogenic disturbances) lies between 0 and 1. Recently, a new name of the quantity has been introduced: $F^{14}\text{C}$ (fraction modern carbon), $a^{14}\text{C} = F^{14}\text{C}$, and their values are <1 for old samples (before AD 1950) and usually >1 for recent samples from “bomb period” or post-bomb

$a^{14}\text{C}$ – it is expressed as percentage of modern carbon, and such “unit” is called pMC – percent of modern carbon

$$a^{14}\text{C} = 100 \text{ pMC}$$

$$F^{14}\text{C} = 1$$

$$A^{14}\text{C} = 226 \text{ Bq/kgC}$$

All these values are equivalent

The principal radiocarbon standard:

Oxalic Acid I (OXI), $C_2H_2O_4$, was made from a crop of 1955 sugar beet. Modern radiocarbon standard is 95 % activity of OXI corrected for $\delta^{13}C$. When the stocks of OXI were almost fully consumed, a secondary standard, **Oxalic Acid II (OXII)**, was made from a crop of 1977 French beet molasses with ^{14}C activity 1.2933 times of OXI.

List of radiocarbon standards

Standard	Material	$\delta^{13}C$	$a^{14}C$ (pMC)
OxI (NIST* SRM 4990 B)	Oxalic acid	-19.3	105.26
OxII (NIST* SRM 4990 C)	Oxalic acid	-17.8	134.08
IAEA-C6 (ANU*)	Sucrose	-10.8	150.61
IAEA-C7	Oxalic acid	-14.48	49.53
IAEA-C8	Oxalic acid	-18.3	15.03

* NIST – National Institute of Standards and Technology; Gaithersburg, Maryland, USA
* ANU – Australian National University, Canberra, Australia

Expressions for normalized modern standard using OXI and OXII are:

$$A_{ON*} = 0.95 \cdot A_{OXI} \left[1 - \frac{2(19 + \delta^{13}C)}{1000} \right]$$

$$A_{ON*} = 0.7459 \cdot A_{OXII} \left[1 - \frac{2(25 + \delta^{13}C)}{1000} \right]$$

Basic definitions

Depletion or enrichment of archaeological samples are defined in regard to the **modern standard** as:

$$d^{14}\text{C} = \frac{A_{\text{SN}} - A_{\text{ON}^*}}{A_{\text{ON}^*}} \cdot 1000 (\text{‰})$$

When isotopic fractionation normalization to $\delta^{13}\text{C} = -25\text{‰}$ is applied:

$$D^{14}\text{C} = \frac{A_{\text{SN}^*} - A_{\text{ON}^*}}{A_{\text{ON}^*}} \cdot 1000 (\text{‰}) \approx d^{14}\text{C} - 2(\delta^{13}\text{C} + 25) (\text{‰})$$

Conventional age (t) which includes isotopic fractionation correction:

$$t = -8030 \ln \frac{A_{\text{SN}^*}}{A_{\text{ON}^*}} = -8030 \ln \left(1 + \frac{D^{14}\text{C}}{1000} \right)$$

For environmental samples and post-bomb applications frequently is used **relative specific activity** expressed in **percent of modern carbon (pMC)**, and defined as:

$$a^{14}\text{C} = \frac{A_{\text{SN}^*}}{A_{\text{AN}^*}} \cdot 100 (pMC) = \left(\frac{D^{14}\text{C}}{10} + 100 \right) (pMC)$$

Fraction of modern carbon ($F^{14}\text{C}$) is defined as:

$$F^{14}\text{C} = \frac{A_{\text{SN}^*}}{A_{\text{AN}^*}} = \frac{a^{14}\text{C}}{100}$$

$D^{14}\text{C}$, $a^{14}\text{C}$, $F^{14}\text{C}$ and ^{14}C age all report the ratio in the year of measurement, which will not vary as time goes on because radioactive decay in standard and sample occurs at the same rate ($T_{1/2}$).

Example: Comparative table of basic values used in ^{14}C calculations

Depletion/enrichment	$D^{14}\text{C} = 10 \cdot a^{14}\text{C} - 1000$	-200‰
Percent modern	$a^{14}\text{C} = D^{14}\text{C}/10 + 100$	80%
Fraction modern	$F^{14}\text{C} = a^{14}\text{C}/100$	0.80
Conventional age	$-8030 \cdot \ln(F^{14}\text{C})$	1792 y

For geochemical samples depletion or enrichment are defined in regard to the **absolute international standard** as:

$$\delta^{14}\text{C} = \frac{A_{\text{SN}} - A_{\text{ABS}}}{A_{\text{ABS}}} \cdot 1000 (\text{‰})$$

normalization to $\delta^{13}\text{C} = -25\text{‰}$ gives:

$$\Delta^{14}\text{C} = \frac{A_{\text{SN}^*} - A_{\text{ABS}}}{A_{\text{ABS}}} \cdot 1000 (\text{‰}) \approx \delta^{14}\text{C} - 2(\delta^{13}\text{C} + 25) (\text{‰})$$

What is the results of the measurement?

- **conventional radiocarbon age of the sample**, expressed in years Before Present (BP), where **0 BP = 1950 AD**

What is needed to have

- Background (sample with no ^{14}C , prepared the same way as your samples)
- Reference Standard material (Oxalic Acid II) *NIST* SRM 4990C
- Quenching and/or other parameters needed for correction
- Measured or assumed $\delta^{13}\text{C}$ of the material dates

conventional radiocarbon age

- Libby half-life 5568 yr
- Years Before present (BP) – or $F^{14}\text{C}$ for modern samples
- Correction/normalization to $\delta^{13}\text{C} = -25 \text{ ‰}$

Calibrated age span

- ✚ **Calendar age ranges in years cal BC or cal AD***, obtained by calibration of conventional age results using recommended calibration curves;
- ✚ **Probabilities** of finding age ranges (in %) within the **ranges**: 68.3% (1σ), 95.4% (2σ) and/or 99.7% (3σ). The **68.3% (1σ)** range is used mostly;

Example for conventional age

1375 ± 25 BP

1σ range – 68.3 % probability:

Cal AD 645 - 666 (68.3 %)

2σ range: - 95.4 % probability

Cal AD 605 - 629 (8.4 %)

Cal AD 635 - 679 (88.9 %)

*** Abbreviations:**

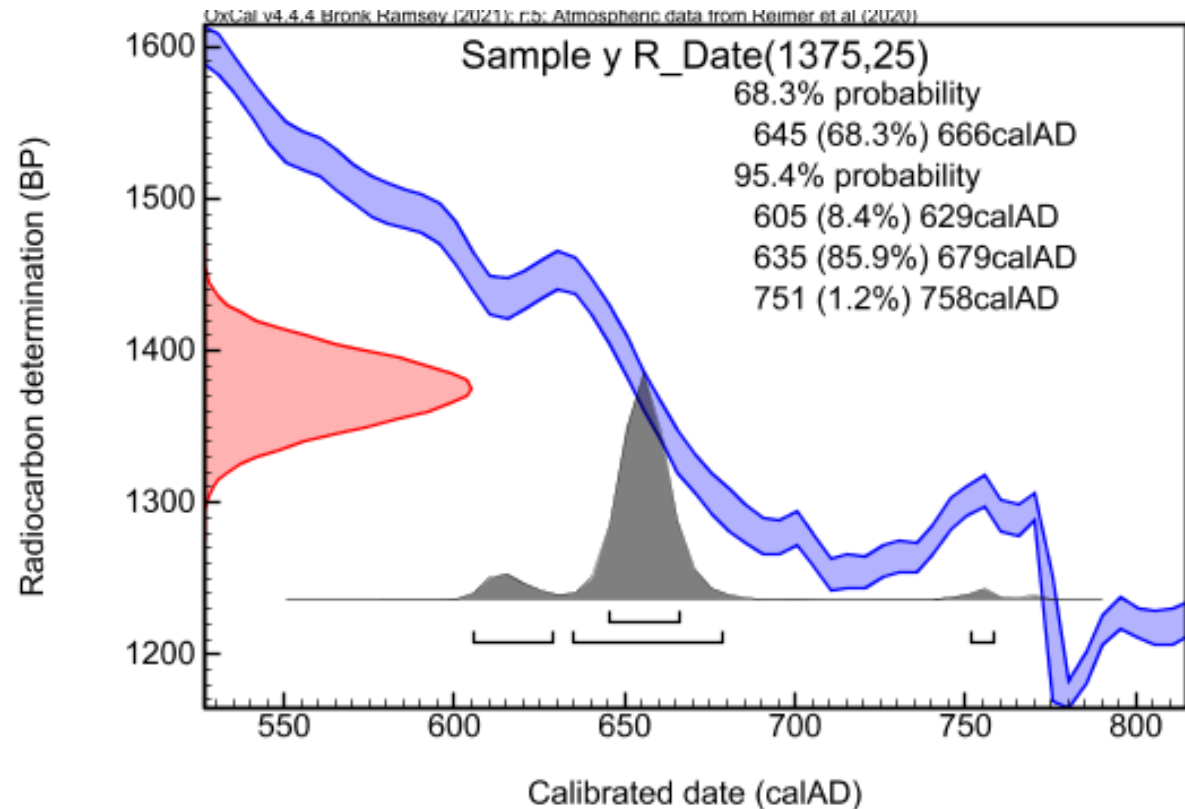
AD – Anno Domini (Latin: year of our Lord)

BC – Before Christ

Sometimes in use also:

CE – Common Era

BCE – Before Common Era

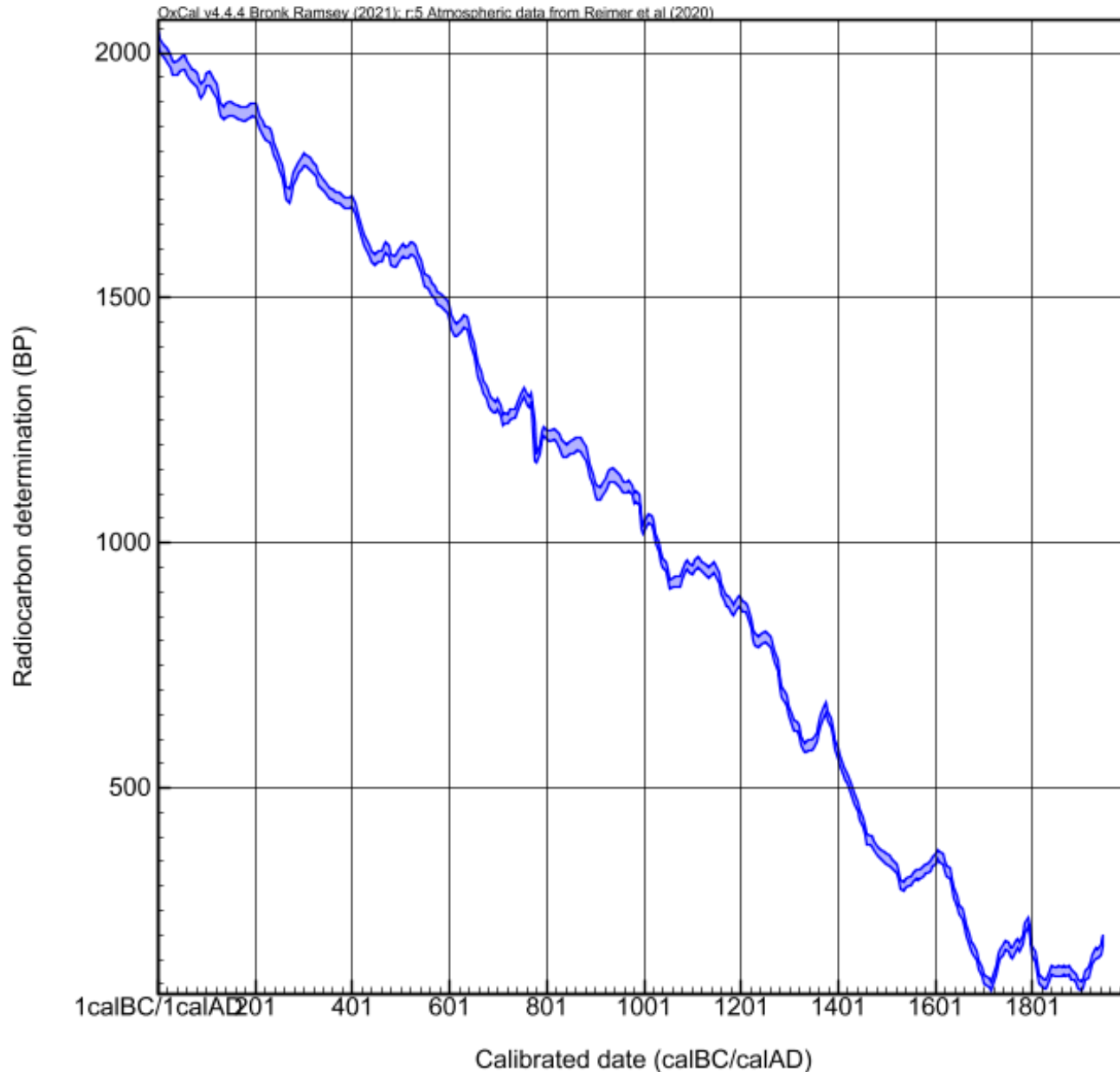


OxCal v.4.4.4, Bronk Ramsey 2021

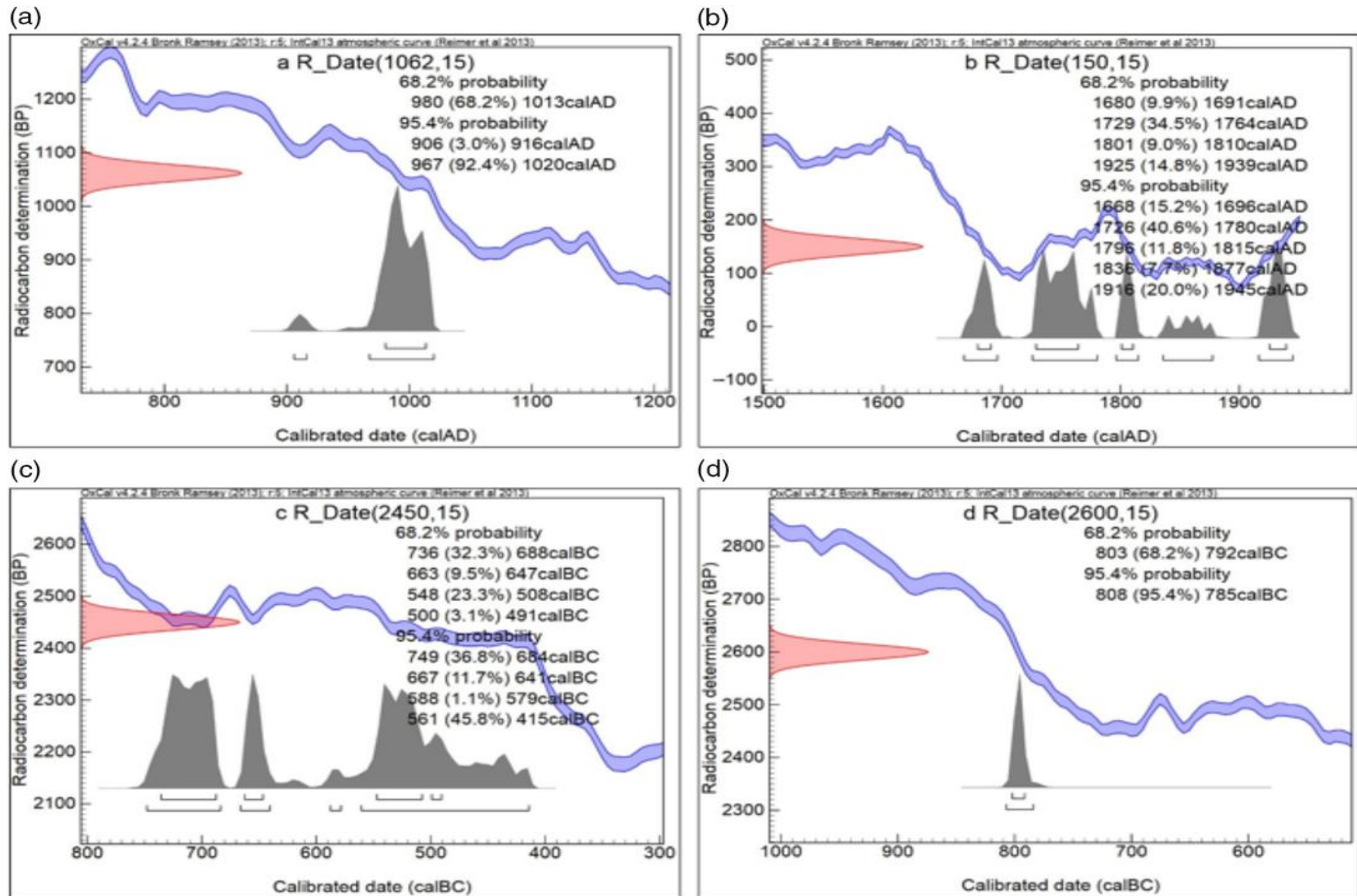
Atmospheric data from Reimer et al (2020) **IntCal20**

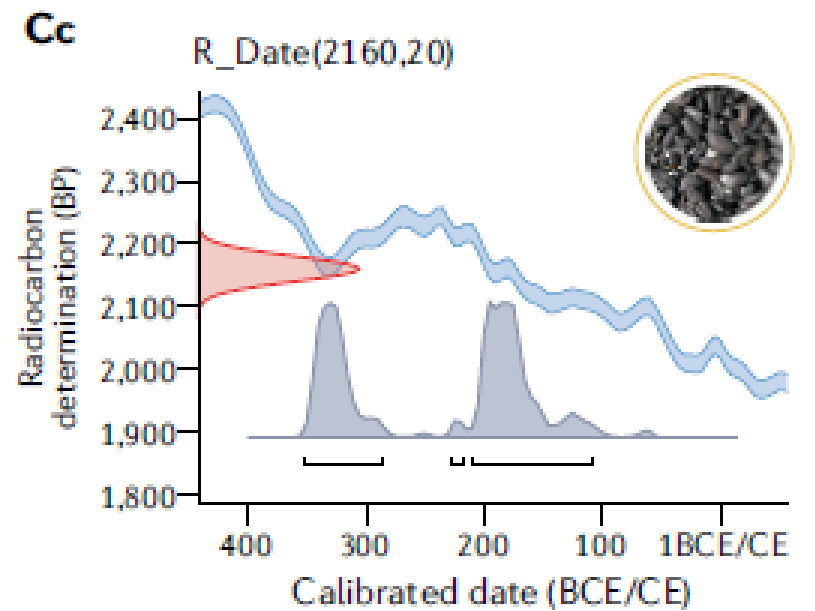
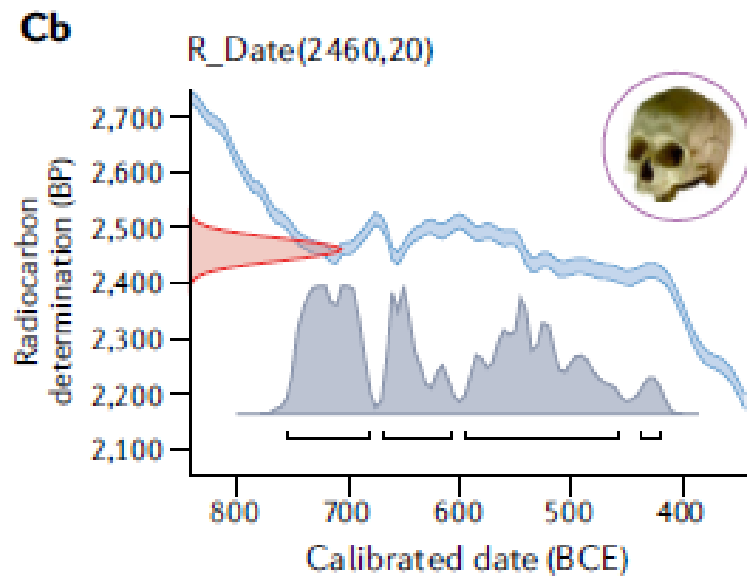
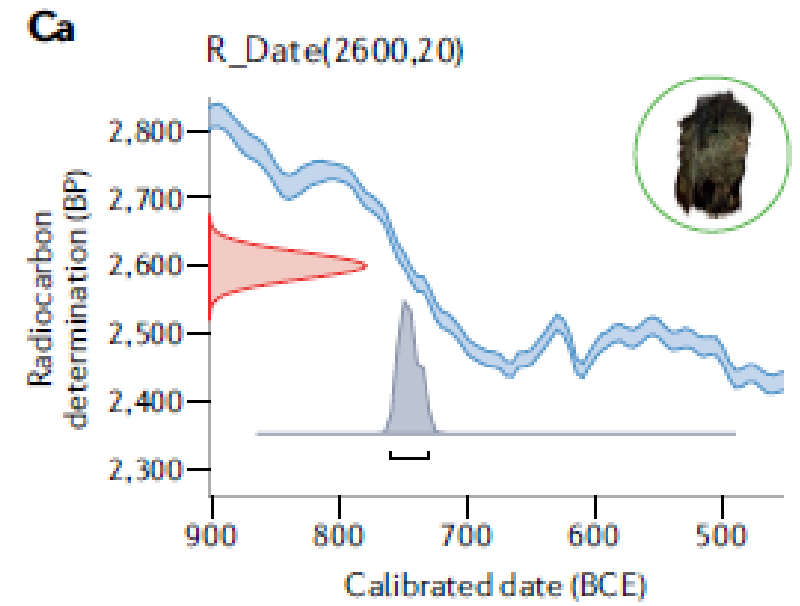
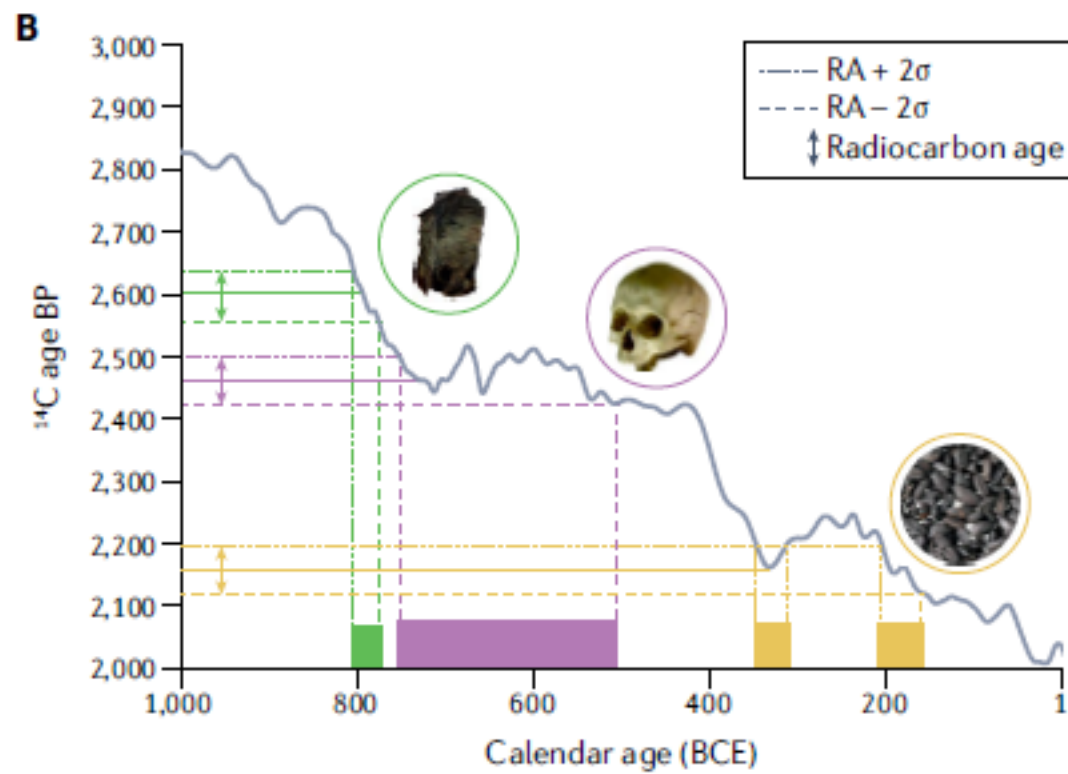
Example of calibration curves

Range from 2000 BP to now



The calibration curve is not smooth and monotonic. The differential production of ^{14}C makes “**wiggles**” in the calibration curves, and these wiggles can result in a single radiocarbon age corresponding to more than one calendar age.





Hajdas I. et al, Radiocarbon dating, Nature Reviews, Methods Primers, article ID (2021)1:62

CONVENTIONS FOR REPORTING RADIOCARBON DETERMINATIONS

Andrew R. Millard

1. Conventional radiocarbon age (BP) or $F^{14}\text{C}$ corrected for ^{13}C fractionation (normalized to $\delta^{13}\text{C} = -25\text{‰}$)
2. Laboratory code
3. Sample material, pretreatment method [measurement method], control measurements
4. The calibration curve used (INTCAL20), any reservoir offset
5. Calibrating software with its version, any option or model used, citation
6. Calibrated data given as a range (ranges) with associated probability (probabilities), clearly identified time scale (cal BP, or cal AD/cal BC, or cal BCE/CE)

Reservoir effect

Radiocarbon dating is an extremely useful technique for determining the ages of **geological materials** (*that have some organic-derived carbon in them*), and it is highly applicable to the study of Quaternary materials (that are younger than 50 000 years).

But, interpretation of radiocarbon data can be quite complex, and several factors need to be taken into account to understand what the results actually mean.

For samples containing carbon from reservoirs other than the well-mixed atmosphere or terrestrial biosphere, the measured radiocarbon ages usually require corrections before they can be placed on a common calendar timescale.

The correction is performed after all other calculations have been already performed (except calibration)

Marine radiocarbon reservoir effect

- A consequence of the spatio-temporal variability of marine radiocarbon records
- The MRE is the product of global scale mechanisms in marine environments (extended residence of ^{14}C in the marine reservoirs once water is circulated away from the air-water interface at the ocean surface, the rate of gas transfer of CO_2 across the interface)
- Correction for the MRE applies a value for the global average of about 400 radiocarbon years
- this is quite constant in places like the eastern Atlantic Ocean
- Prior to 2004, it used to be standard practice to subtract 400 years from the conventional radiocarbon age of marine samples. This is called $R(t)$, the pre-industrial "global reservoir correction"
- Since 2004, marine samples have been calibrated using a separate database from that used for terrestrial samples. This database includes the global reservoir correction, which does vary slightly from year-to-year from the previous standard of 400 radiocarbon years.

Radiocarbon years – more variations in reservoirs (local)

Research has shown that this "constant" was far from constant during the late-Pleistocene/early-Holocene transition. This is undoubtedly because the age of the carbon in the ocean depends on deep-water circulation patterns in the Pacific, and these were different during the ice age.

- Comparisons between the radiocarbon ages of wood and shell found at the same location in ice-age deposits have shown differences in excess of 1000 years. One paper records a total reservoir correction ($R(t) + \Delta R$) that for younger samples would be 790 radiocarbon years, as 1250 years at the time of deglaciation.
- This requires further research. Apart from improving the accuracy of the dates for late-Pleistocene/early-Holocene samples, it might provide interesting data on changes in ocean circulation in the north Pacific at that time.
- Recent research also indicates different MRE for different species

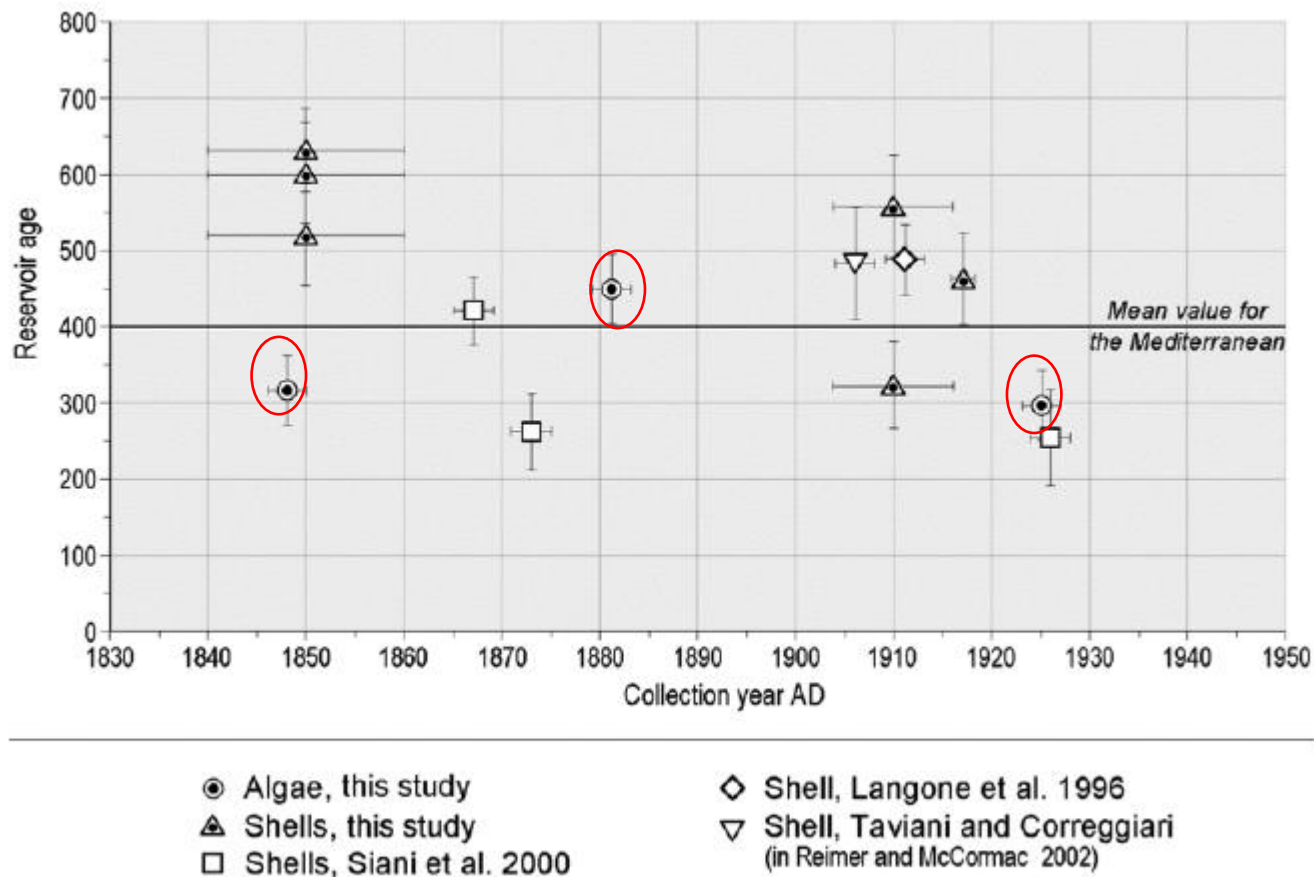


Figure 2 Reservoir age R as a function of the collection year for samples from the Adriatic Sea. The solid line represents the mean value of the Mediterranean (from Reimer and McCormac 2002).

Radiocarbon, Vol 57, Nr 4, 2015, p 527–538

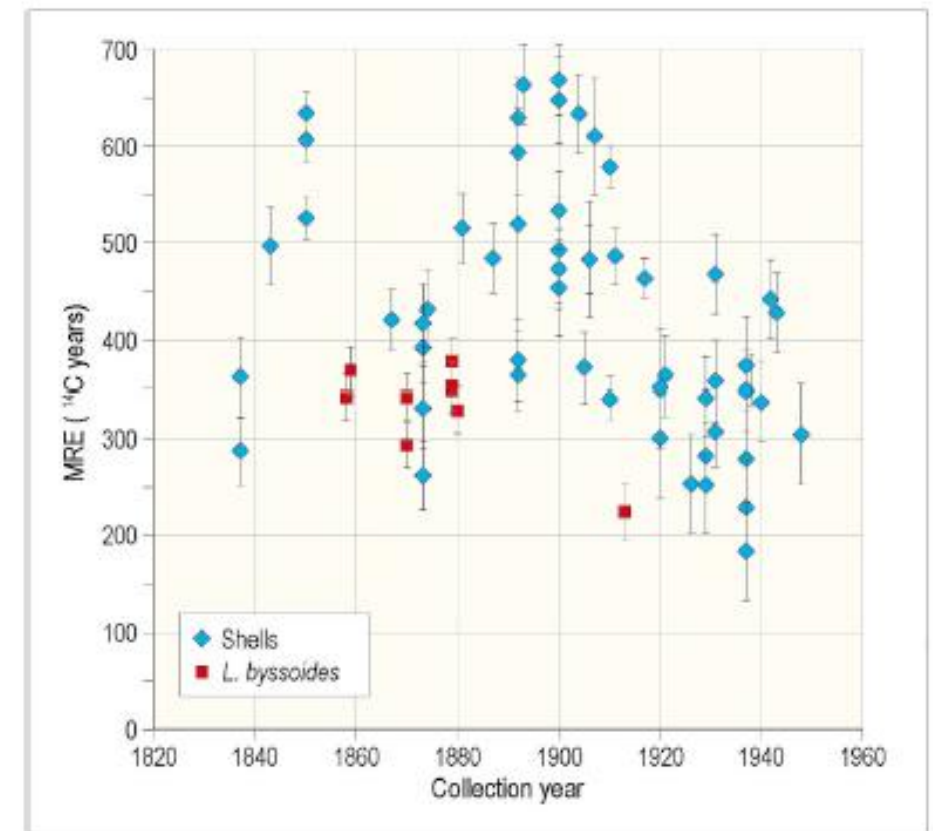
DOI: 10.2458/azu_rc.57.18452

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NEW DATA ON MARINE RADIOCARBON RESERVOIR EFFECT IN THE EASTERN ADRIATIC BASED ON PRE-BOMB MARINE ORGANISMS FROM THE INTERTIDAL ZONE AND SHALLOW SEA

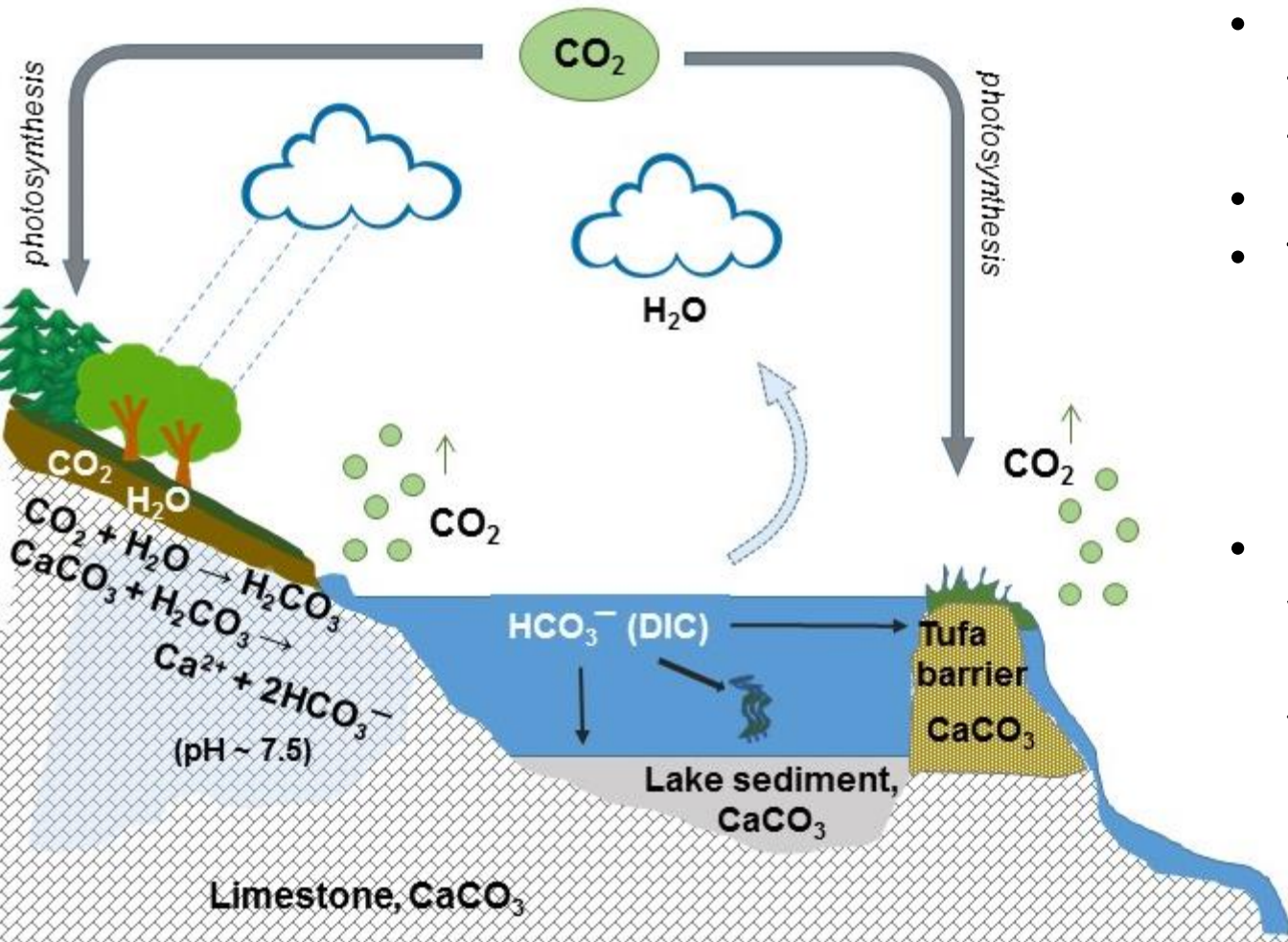
Sanja Faivre^{1,2} • Tatjana Bakran-Petricioli³ • Jadranka Barešić⁴ • Nada Horvatinčić⁴

Coralline algae from the Adriatic had a considerably lower reservoir age (355 ± 34 ^{14}C yr) and ΔR value (-9 ± 34 ^{14}C yr) than molluscs ($R(t)$ 513 ± 53 ^{14}C yr; ΔR 154 ± 52 ^{14}C yr)



Faivre S. et al., Quaternary Geochronology 51 (2019) 15-23

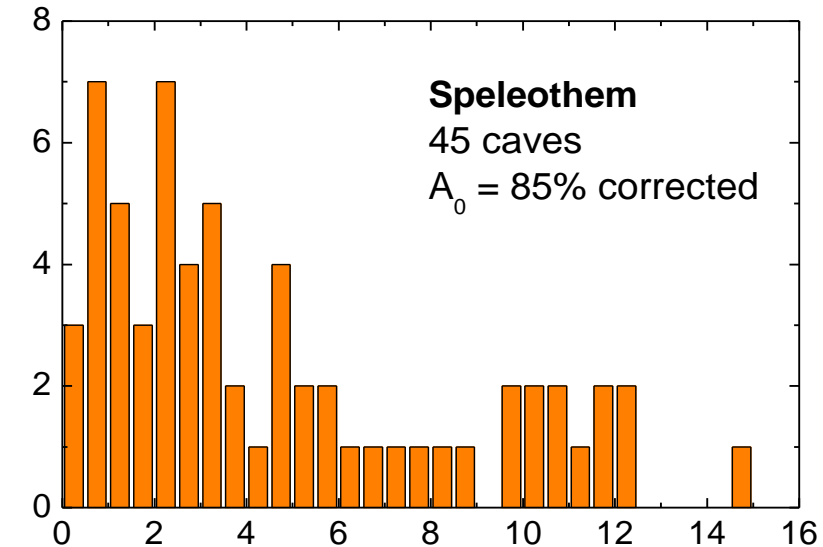
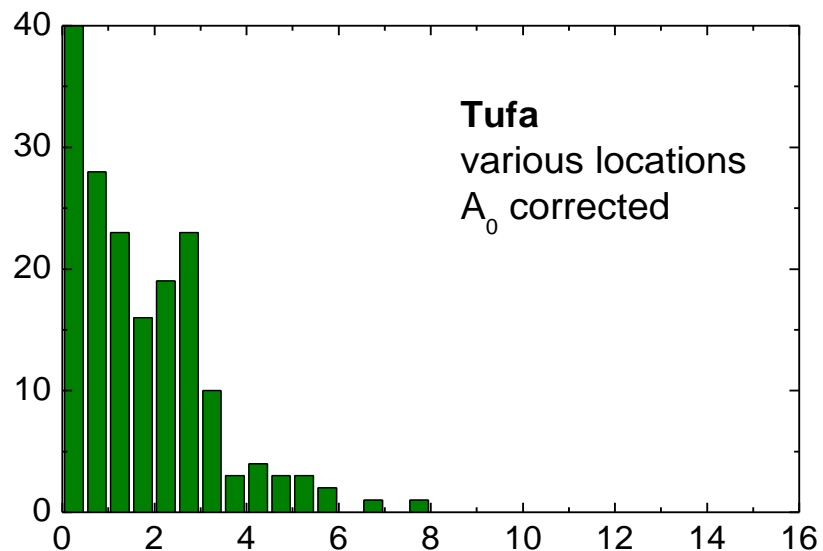
Carbon cycle in karst region – fresh-water effect



- The ^{14}C reservoir effect in karst waters refers to the hard-water or **fresh-water effect** which is a consequence of geochemical formation of secondary carbonates.
- A certain amount of “dead” carbon is incorporated, making its initial radiocarbon activity (a_0) lower.
- Measured radiocarbon dates appear older; therefore, they should be corrected by the reservoir age to obtain the real age
- It should be determined locally
- The hard-water effect can be solved - a_0 can be obtained from radiocarbon activity of freshly sampled: (1) dissolved inorganic carbon (DIC), (2) carbonate precipitated on (an inert material) pad, or (3) actively precipitating tufa
- It is important to emphasize that it is not known if the value of the fresh-water effect established in this manner is constant or changes through time, similarly to the reservoir effect in oceans (marine reservoir effect, MRE)

HOLOCENE

Number of samples



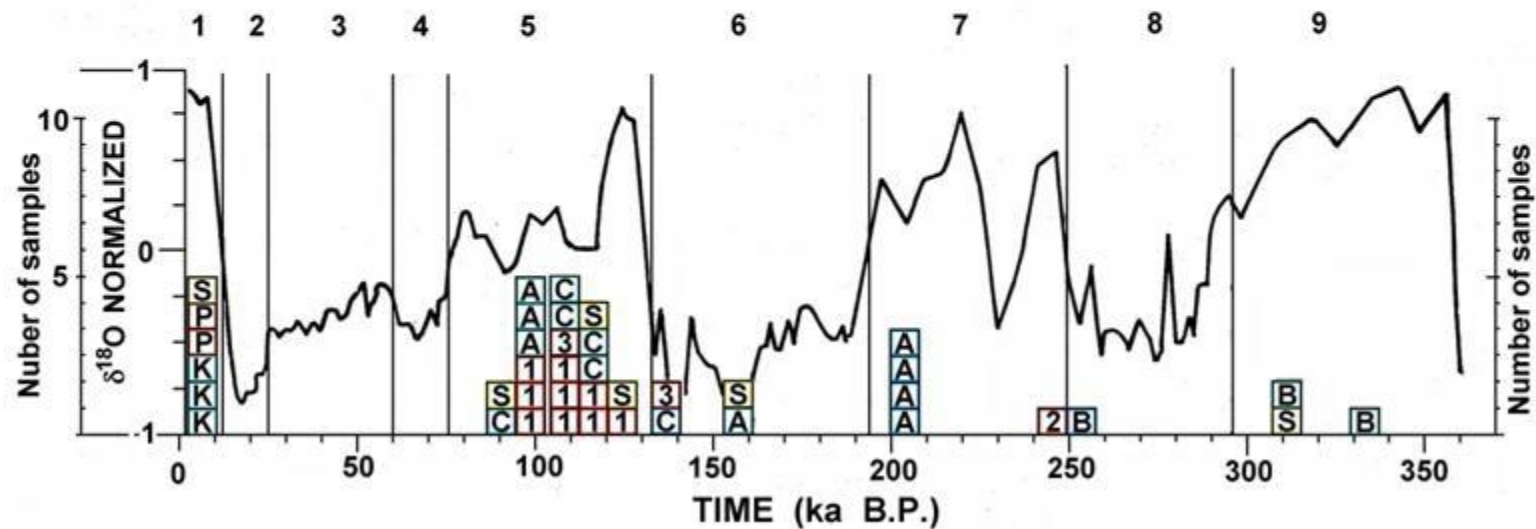
^{14}C age (kyr BP)

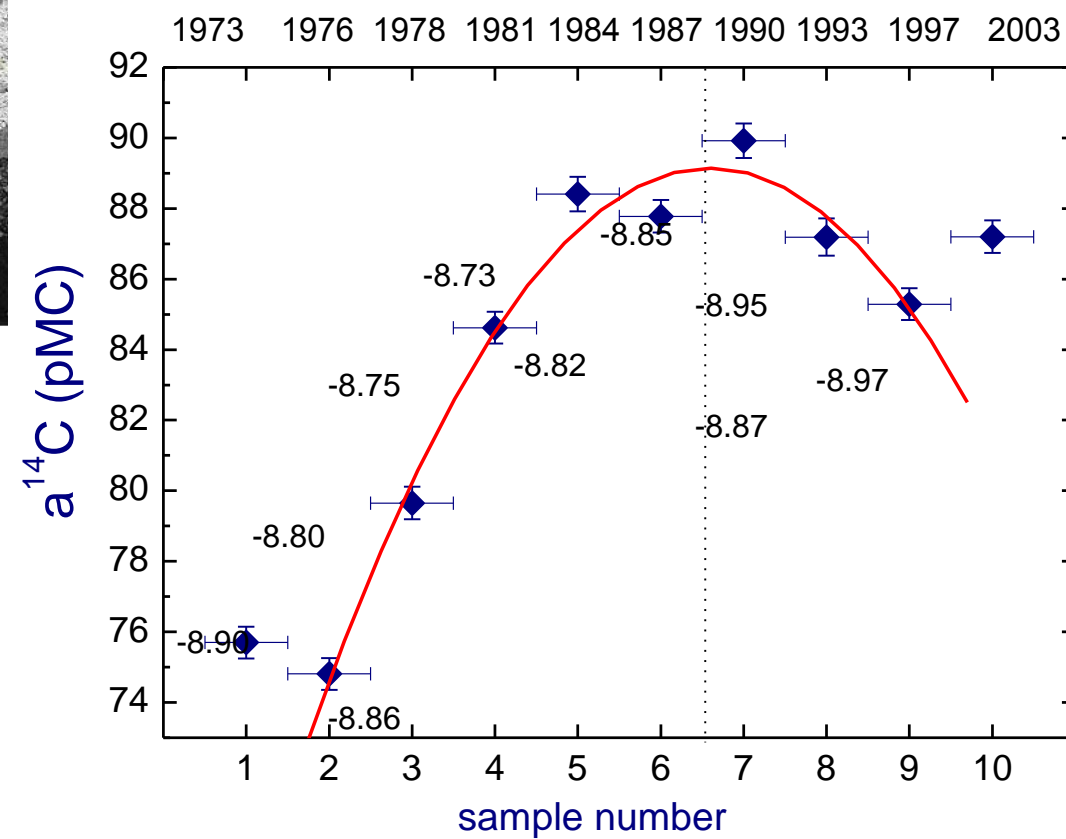
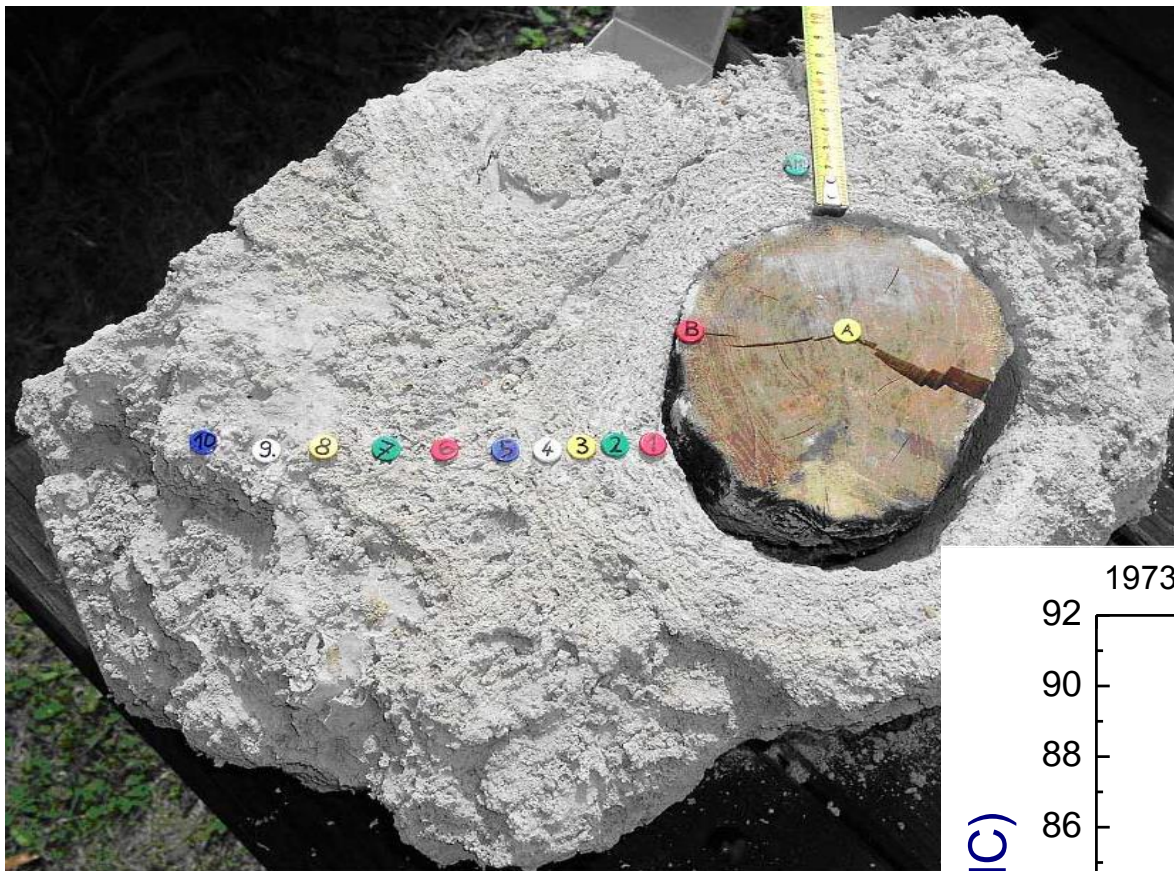
KRKA RIVER

- A** Krčić
- B** Topolje quarry
- C** Kaliča Kuk
- K** Holocene tufa from Krka r.

PLITVICE LAKES

- 1** Plitvički Ljeskovac
- 2** Smolčića Cave
- 3** Bijela Rijeka r.
- P** Holocene tufa from Plitvice
- S** Speleothem





Dead carbon proportion, DCP

- it is often used term in discussion of speleothem ages
- Carbonates precipitating in cave environments have a different ^{14}C signature to the atmosphere
- DCP reflects a difference between the atmospheric ^{14}C content and the signal recorded in carbonates at the time of their formation
- Speleothems are complex carbon records, however, they can be precisely dated by the U-Th dating method
- DCP usually ranges between 5 and 20 %, resulting in age correction from 400 to 1800 years

Age determination of human remains.

- Radiocarbon dating of human remains can be used to estimate the age of a person, and can significantly assist authorities when attempting to solve unidentified homicides as well as facilitate identification in mass disasters.
- Dependent on the stage of preservation, analysis of bone lipids, hair and skin can provide information on the year of death
- As bone undergoes remodeling across the lifespan, the age of bone does not always reflect person age, and cannot be used to reliably estimate the date of birth
- Radiocarbon dating of tooth enamel has been used to estimate the date of birth of an individual. Tooth enamel is not remodeled or exchanged during the lifespan and this analysis revealed that when calibrated for the enamel formation time, dental enamel provides a remarkably accurate estimate of a person's year of birth.
- In one example, where radiocarbon dating (and other forensic methodology) was applied to a cold case, the legal identification of a young boy who had died approximately 40 years earlier was made, resulting in the repatriation of the boy's remains and closure for the family

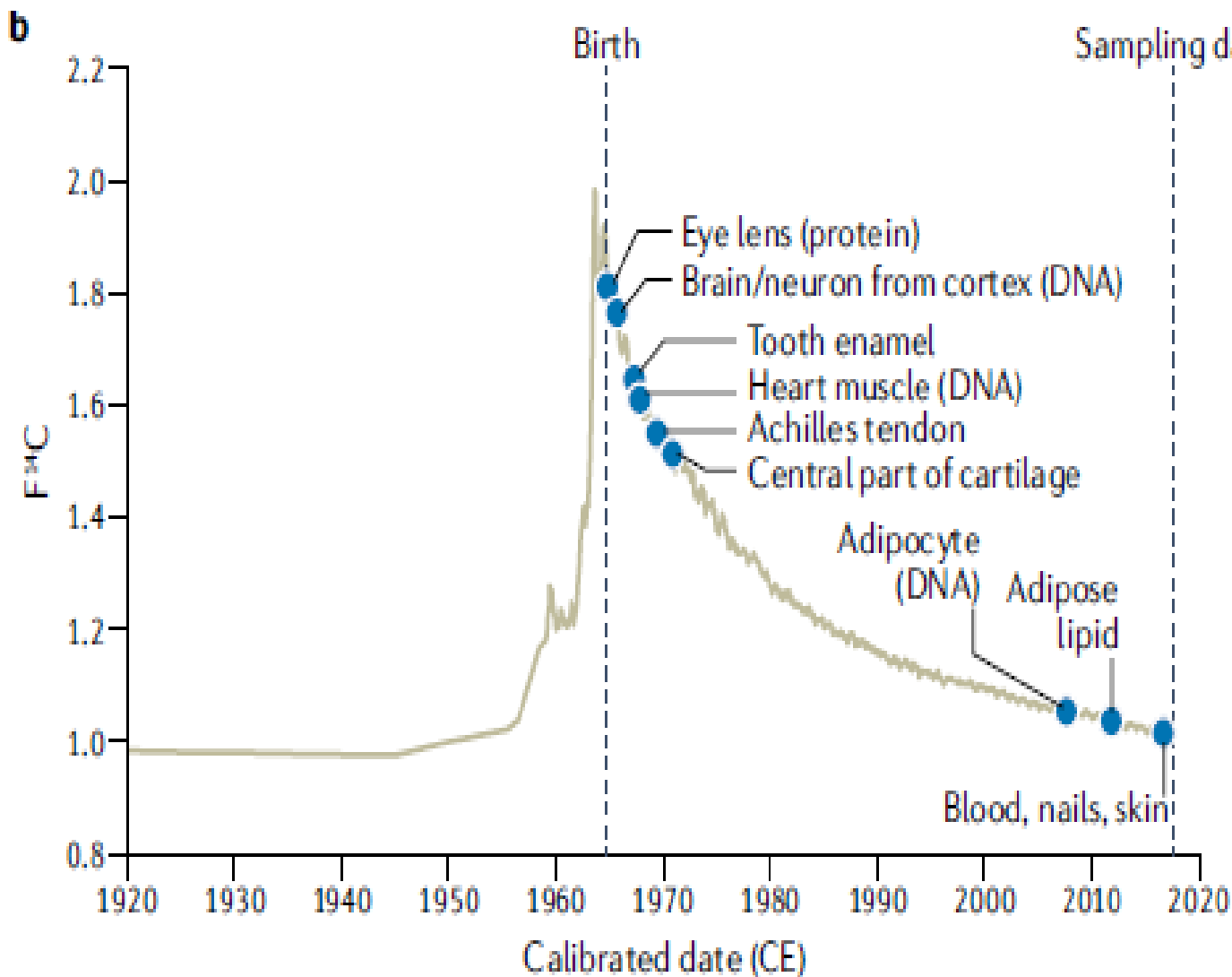
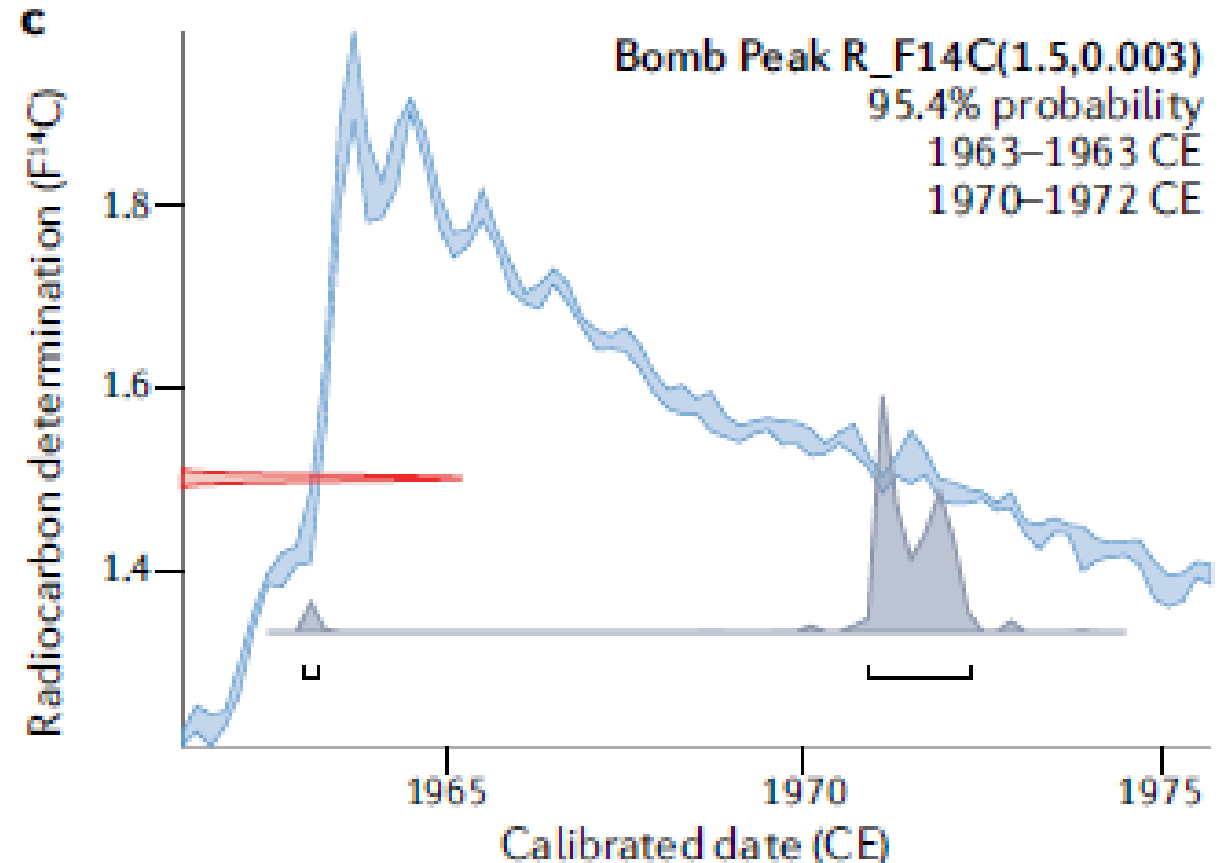
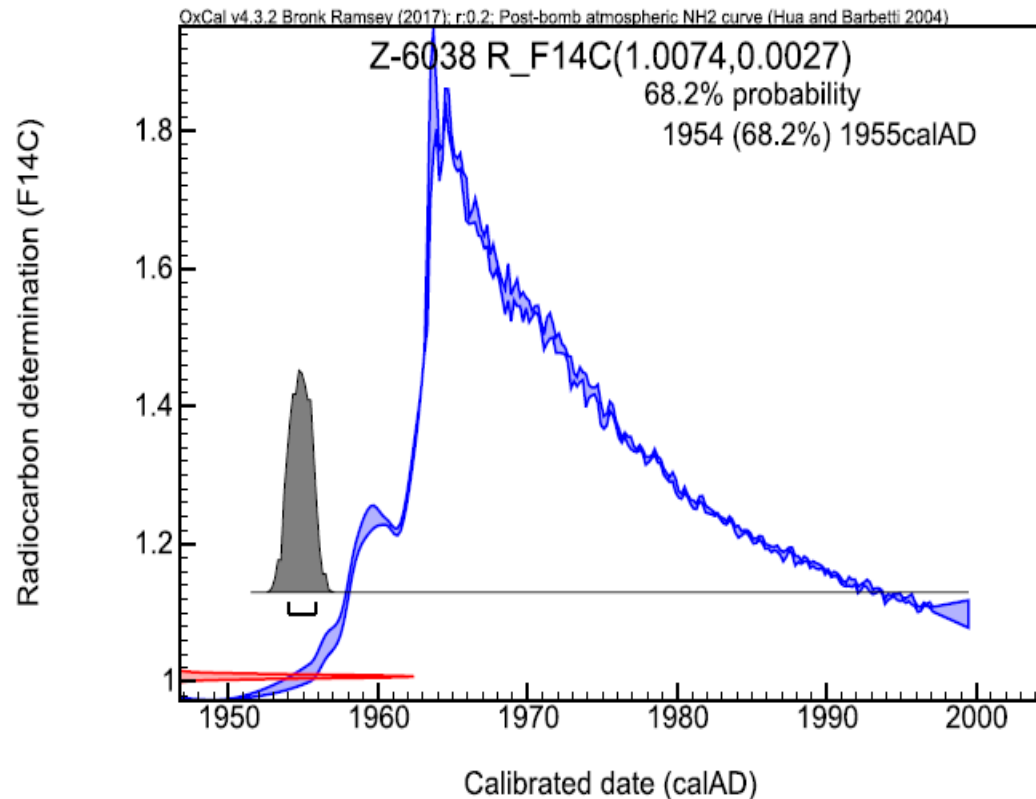


Fig. 7 | Distribution of ^{14}C in the human body. Bomb peak provides a unique opportunity

- eye lens proteins and neurons from the cerebral cortex retain carbon corresponding to the year of birth.
- tooth enamel does not turn over carbon after completion of enamel formation, allowing it to be used forensically to determine the date of birth of an individual.
- $F^{14}C$ analyses in other organs provide insight into the dynamics of cell and tissue formation and turnover rates, such as renewal of cardiomyocytes in the heart muscle decreasing with age
- formation of cartilage and Achilles tendon is completed before adulthood.
- $F^{14}C$ in fat cells, blood, nails, hair, skin collagen and bone lipids, which are constantly renewing, is in equilibrium with the atmospheric $F^{14}C$

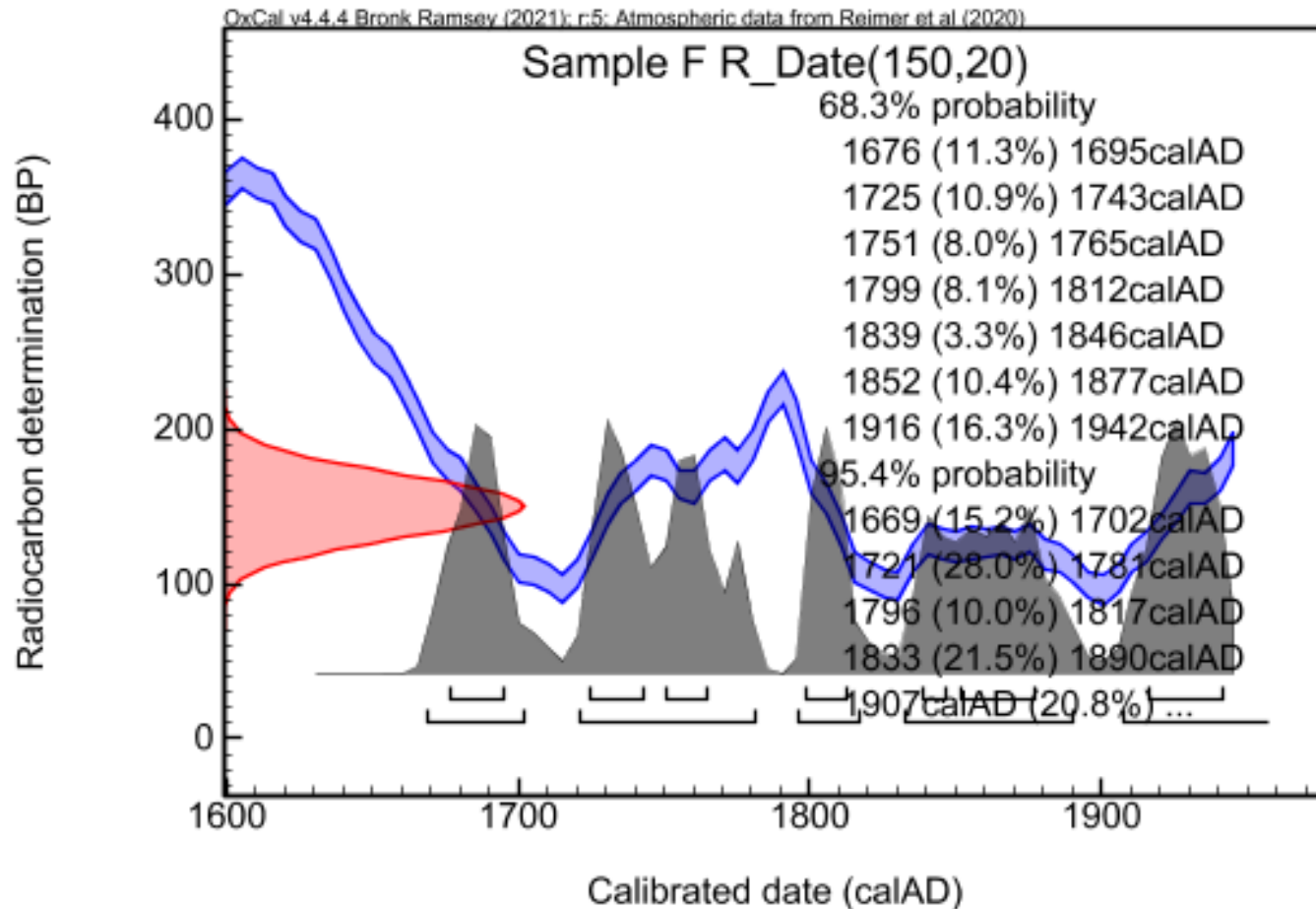
Forgeries – „art-history forensic”

- Potential of the method to detect anachronism of materials of cultural heritage that can indicate forgeries
- The bomb-peak can be successfully used to distinguish origin of the material “before” of „after” the peak



Forgeries

- It can be used as additional proof of forgery detected or suggested by other analyses
- However, it cannot be used if the old canvas is reused
- Or if the material originates from between the mid-17th to mid-20th century



Some examples of radiocarbon dating

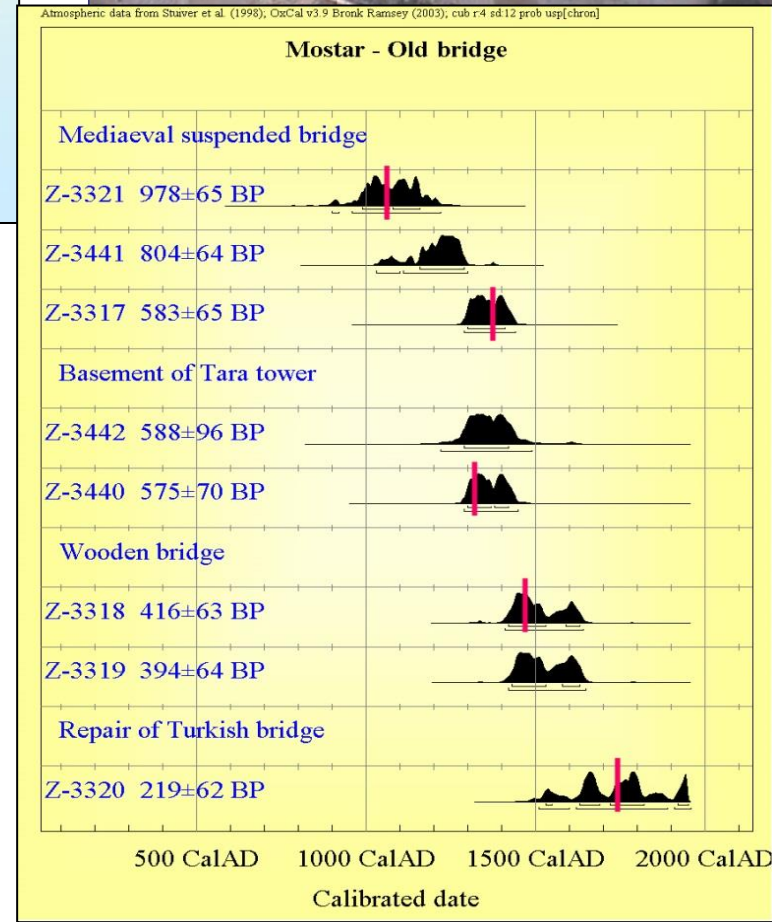
Old Bridge in Mostar



Stone bridge (span 28.60m) by order of the Sultan **Süleyman the Magnificent** in 1557 (architect Kodja Mimar Sinan) Completed **AD 1566**

¹⁴C dating (Obelić & al., 2007, Radiocarbon 49:617): Six wood and two charcoal samples from different periods of construction of the bridge.

Dendrochronological analyses (Kuniholm et al. 2004): 16 wood samples with visible tree rings sent to the Cornell University, Ithaca (NY, USA).



The Fojnica Armorial, Expected – most likely 17th century

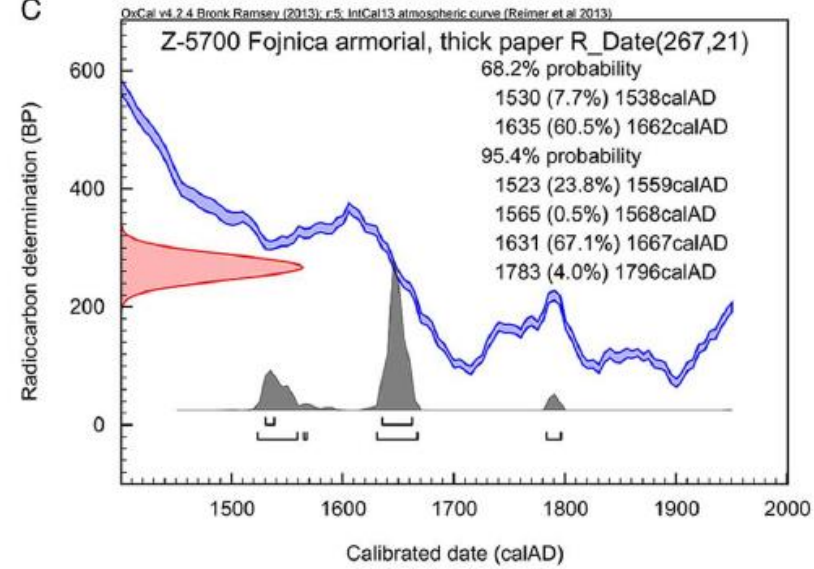
A



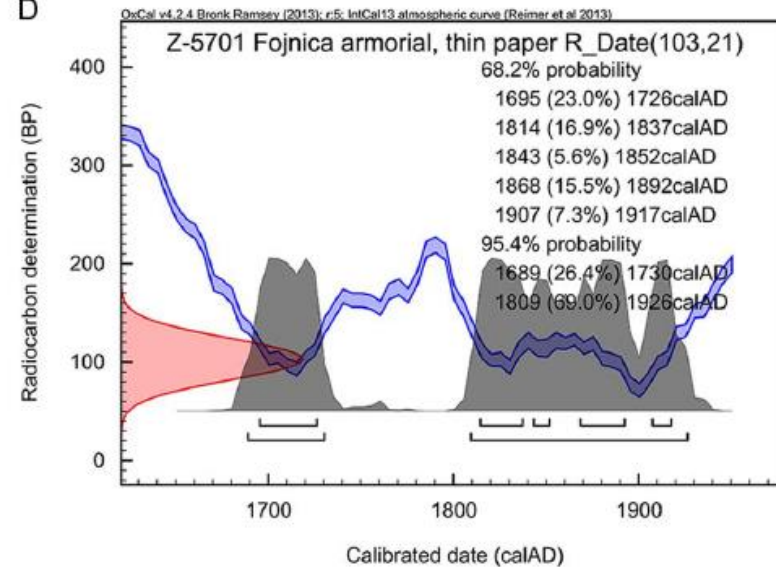
B



C



D



The Cathedral of St. Domnius in Split, Croatia

received a massive gilded wooden door on the Feast of St. George in 1214.

The doors were made by the local craftsman Master Andrija Buvina.

530 cm high and the two wings 360 cm wide



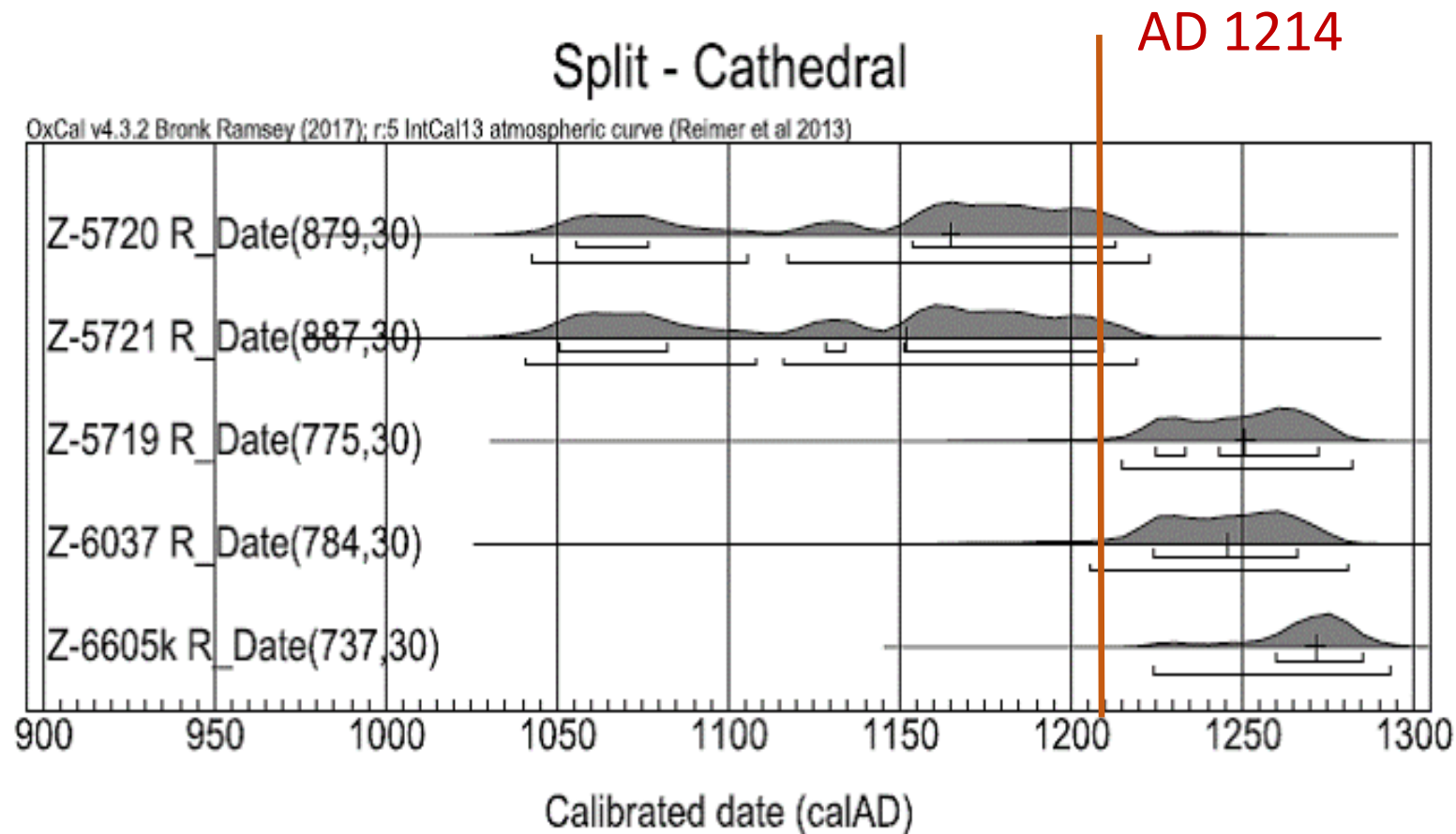
Split-Cathedral – conventional ^{14}C ages

Z-	Sample desc.	$\delta^{13}\text{C} \text{ ‰}$	AMS Age	σ	comm
5469	Wood, Buvina's door, Split - Cathedral, br.inv. M6S 6671-1	-25.0	313	80	LSC
5719	Wood, Buvina's door, Split - Cathedral, cassette 24, (<i>Uzašće</i>) - walnut, #1	-23.6	775	22	
5720	Wood, Buvina's door, Split - Cathedral, between cassetes 21-22, walnut, #2	-24.3	879	21	
5721	Wood, Buvina's door, Split - Cathedral, middle part of the door - oak, #3	-27.1	887	21	
6037	Wood (<i>Juglans</i> L.), Buvina's door, Split - Cathedral, cassette 1, #1	-23.2	784	22	
6038	Wood, Split - Cathedral, Choir bench, #2	-27.1	100.74	0.27	pMC
6605	Wood, Split - Cathedral, Choir bench, south- west verticale 13th century, #3	-26.9	737	21	

Summary

Samples Z-5720 and Z-5721 from the door give the calibrated age spans cal AD 1155 – 1210 and cal AD 1054 – 1191, respectively. It may be concluded that these two wooden pieces belong to the original door from AD 1214.

Samples Z-5719 and Z-6037 are somewhat younger (cal AD 1245 – 1272 and cal AD 1225 – 1266, respectively) indicating that these pieces could not have been built into the door in AD 1214.





A CONTRIBUTION TO THE
HISTORY OF DUBROVNIK,
CROATIA - RADIOCARBON DATING
OF BURIAL AND CHARCOAL
SAMPLES FROM CHURCH OF ST.
STEPHEN in Pustijerna,
DUBROVNIK

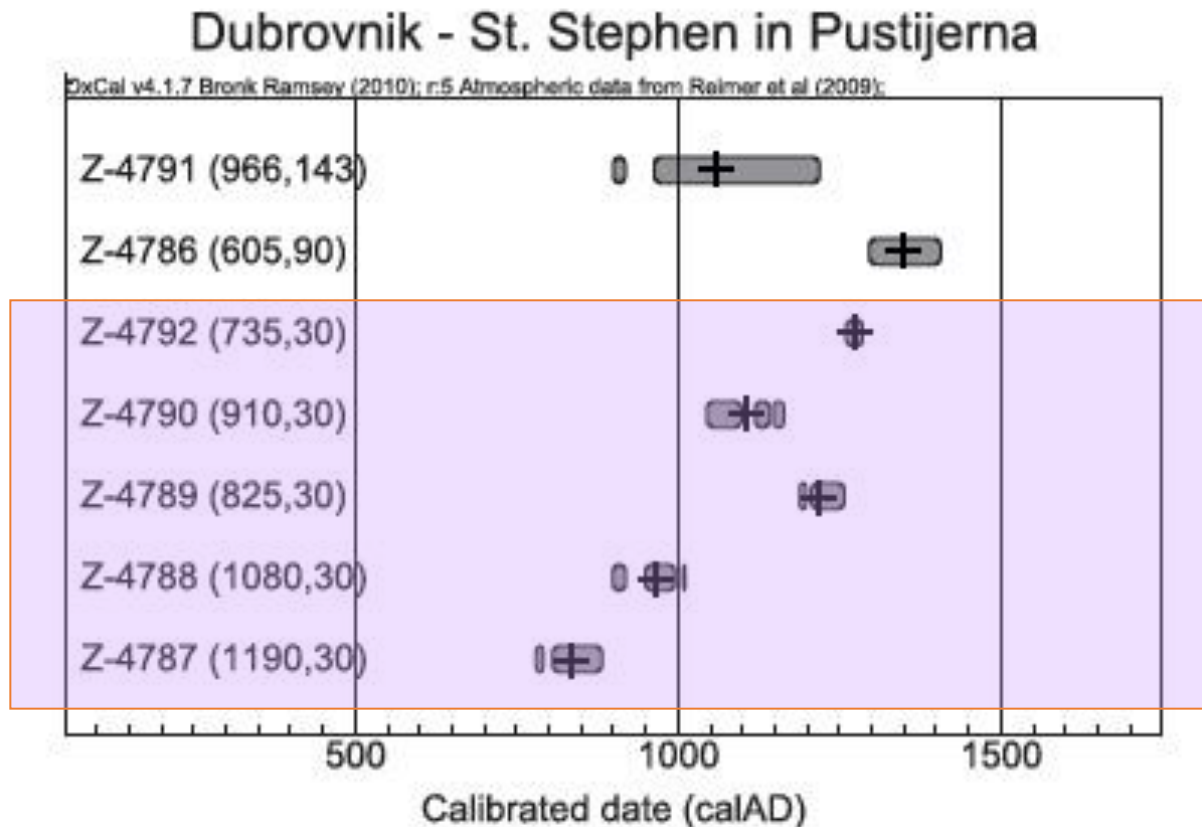
The Byzantine emperor **Constantine VII Porphyrogennetos** in **949 AD** in his book *De administrando imperio* explicitly mentioned the church of St. Stephen in Pustijerna.

However, the construction period of St. Stephen's must be earlier according to archaeological excavations, i.e. archaeological finds and architectural remains which indicate to late 8th / beginning of the 9th century as initial period of this church.

St. Stephen in Pustijerna - one of the 24 oldest churches in the historical core of Dbk.

B o n e s

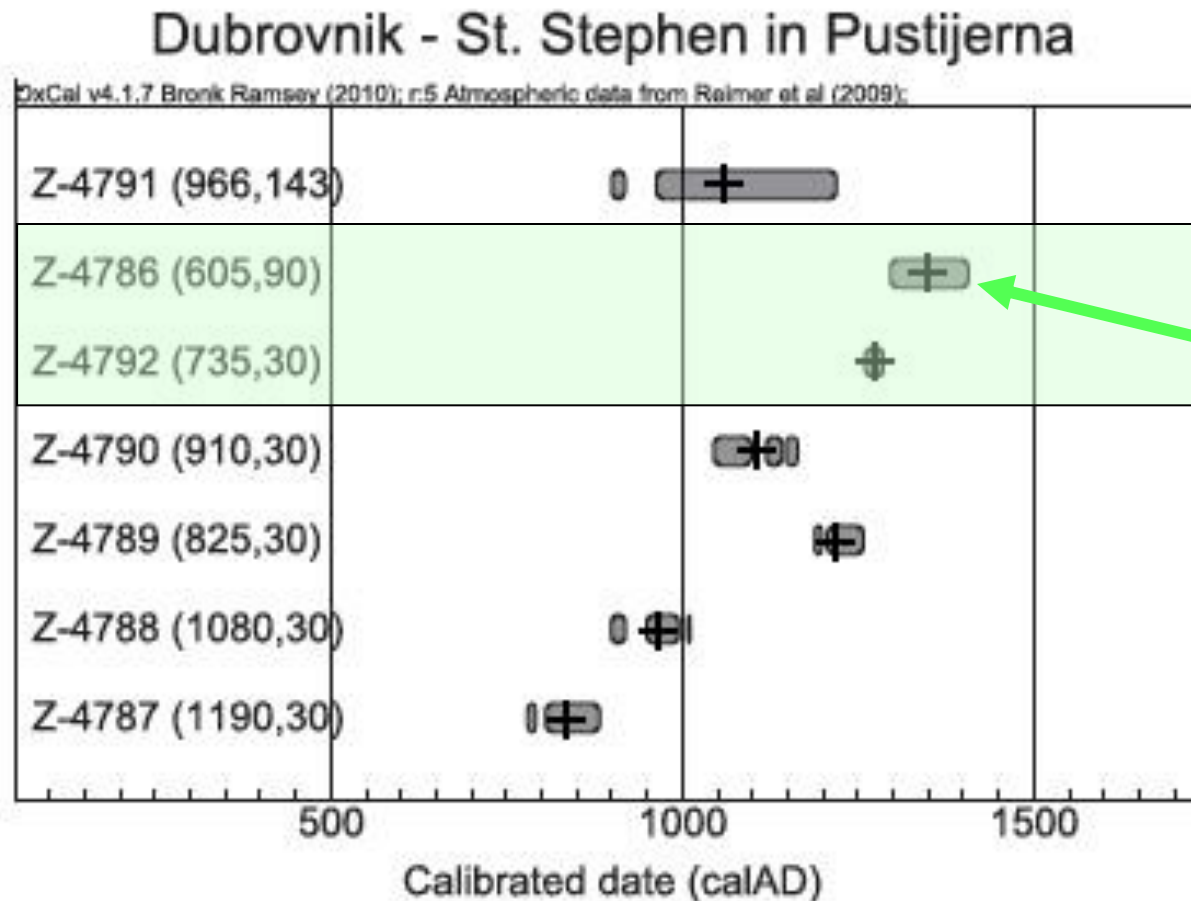
The earliest 806-882 cal AD, and the latest to 1260-1286 cal AD. These results indicate the existence of pre-Romanesque church before 806 AD, because there must be earlier built church beside the cemetery. The graveyard continuously existed for several centuries, analyses prove period 806-1286 cal AD, but burials belonging to early modern period (Renaissance, Baroque) were also found.



Charcoal

The charcoal samples (963-1219 cal AD and 1296-1407 cal AD) but the context of these layers is still not completely clear.

They can be related to burning during transition of pre-Romanesque to Romanesque period and to Gothic period of the church.



The median of Z-4786, 1351 cal AD, corresponds to the restoration of the church after the earthquake in 1348 AD, documented in the archives of the Republic of Dubrovnik.

Old Olive tree, Brijuni Islands

Olive trees do not form clear circular annual rings, and the most central wood is frequently rotted. Thus the direct dating of the tree based on ring counting in cores or radiocarbon dating of the pith cannot be carried out.



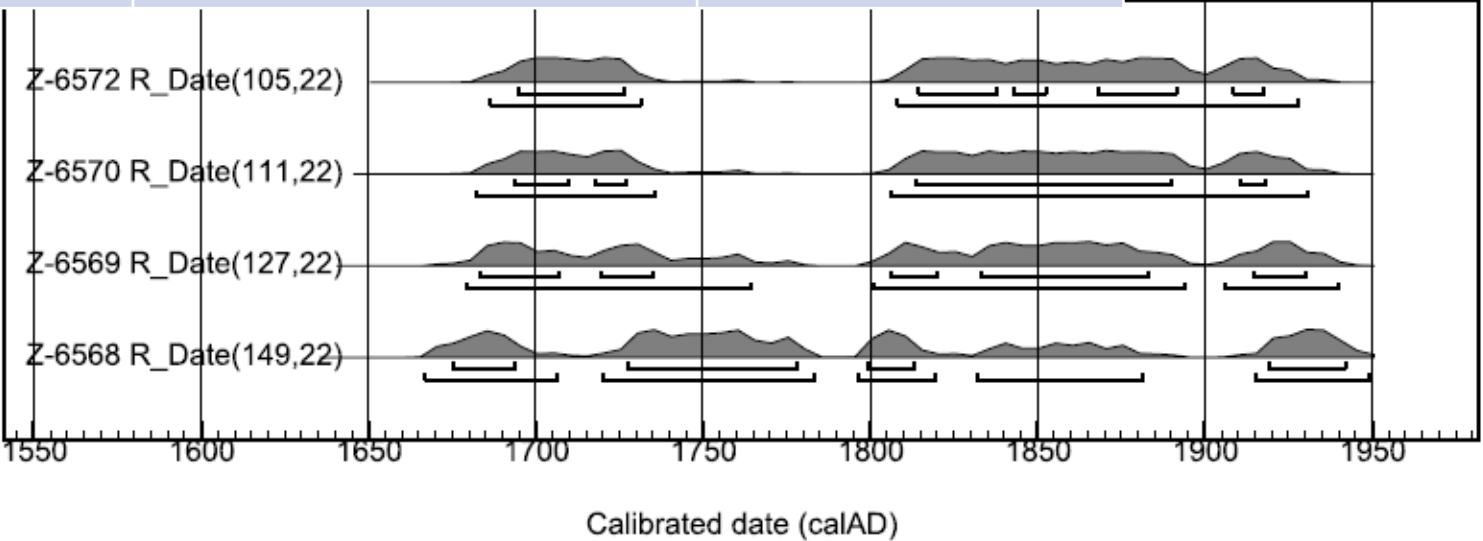


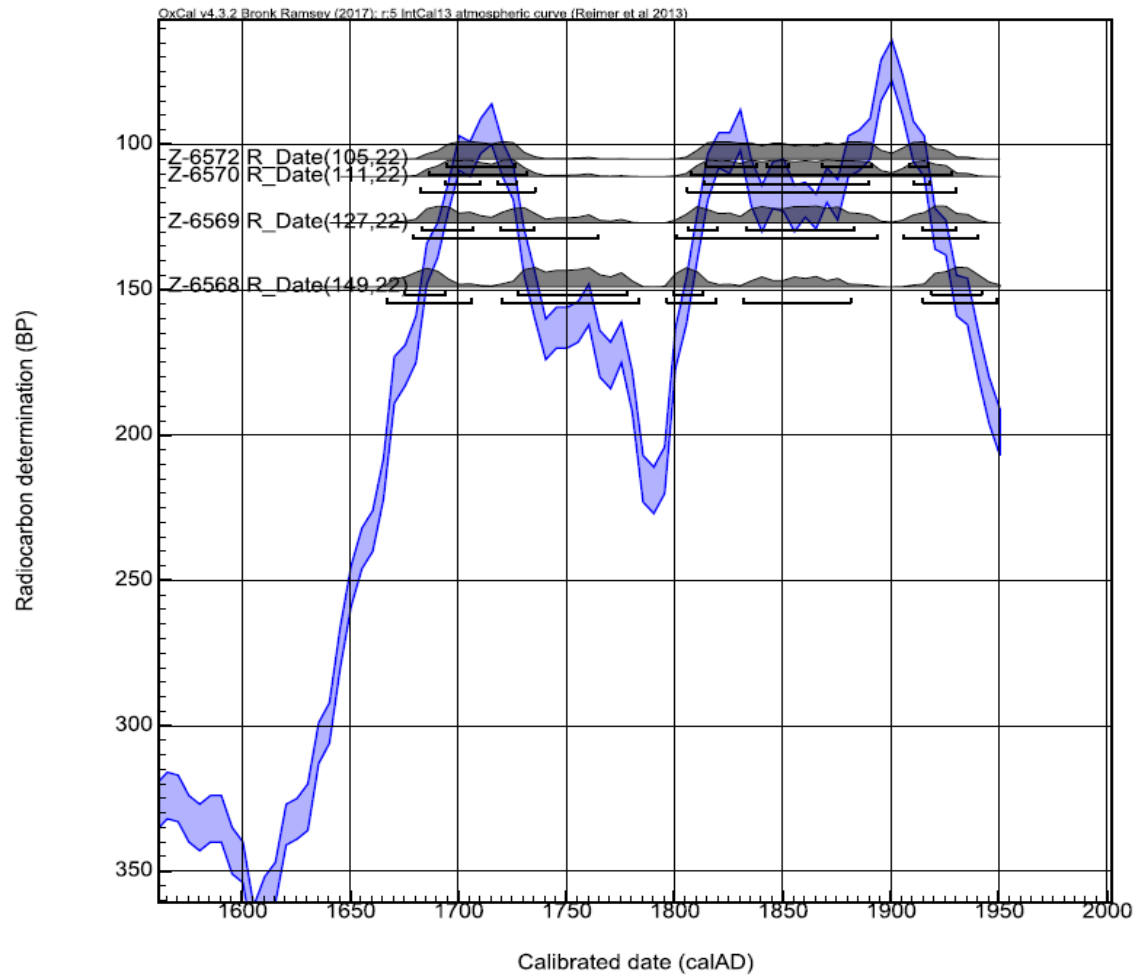
Sample 2



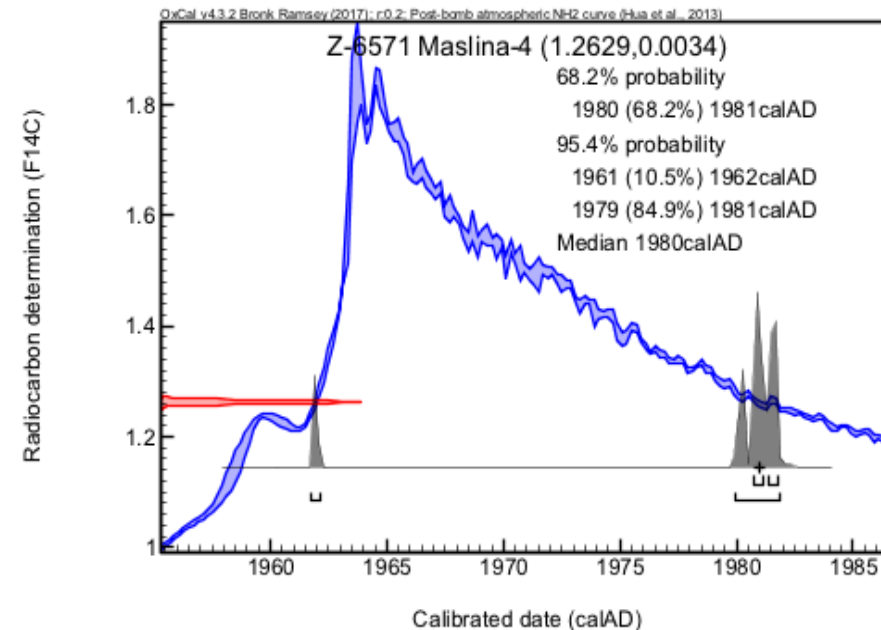
Sample 4

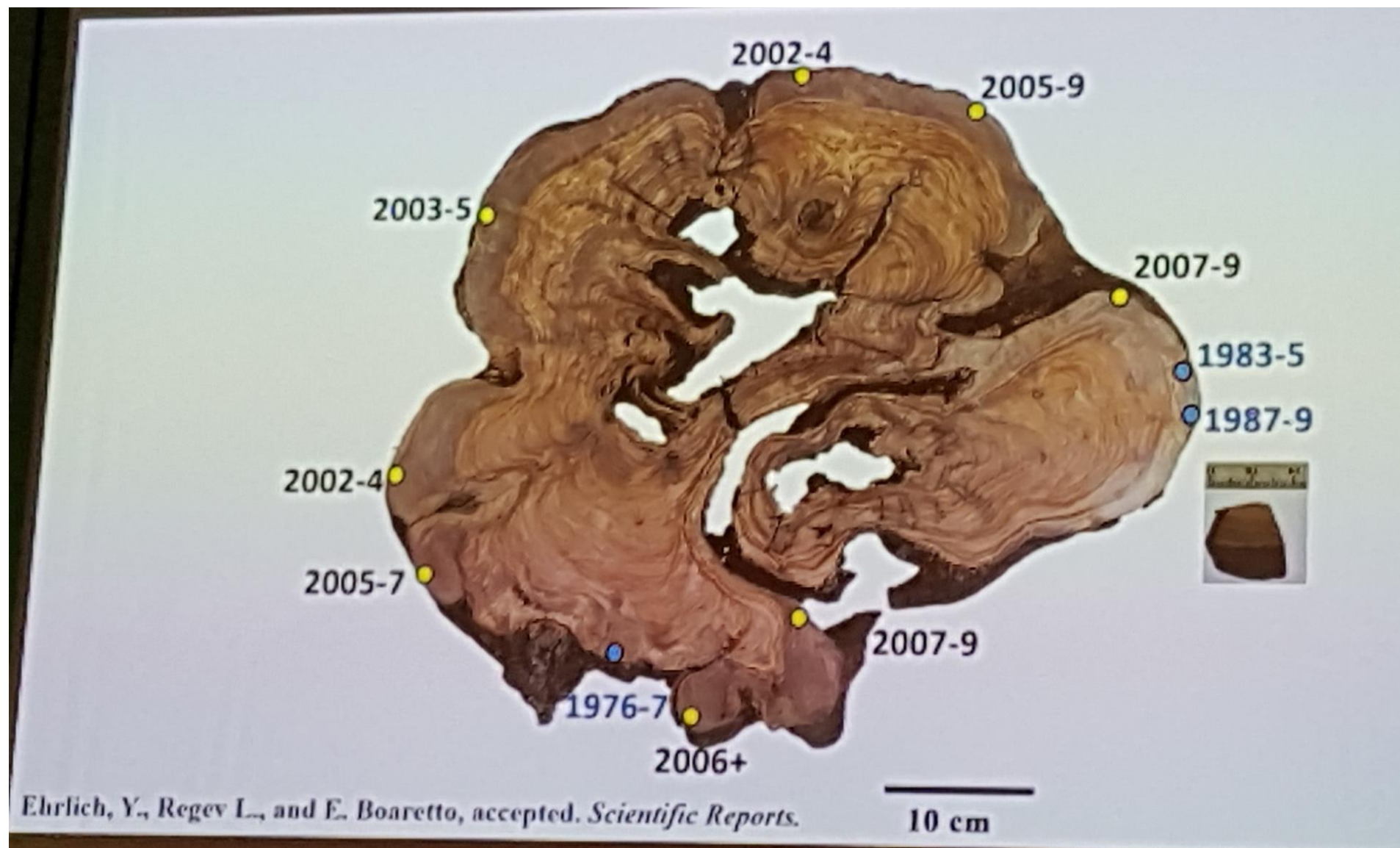
Sample code	Sample name	$\delta^{13}\text{C}$ (‰)	Conventional ^{14}C age (BP)	Calibrated age span (cal AD)
Z-6568 A1575	Brijuni, <i>Olea europaea</i> , sample 1		150±20	1675 – 1942
Z-6569 A1576	Sample 2	-23.6	130 ± 20	1683 – 1930
Z-6570 A1577	Sample 3	-25.5	110 ± 20	1694 – 1890
Z-6571 A1578	Sample 4	-24.2	126.3 ± 0.3 pMC Modern sample	1980 – 1981
Z-6572 A1579	Sample 5	-23.7	105 ± 20	1695 – 1917

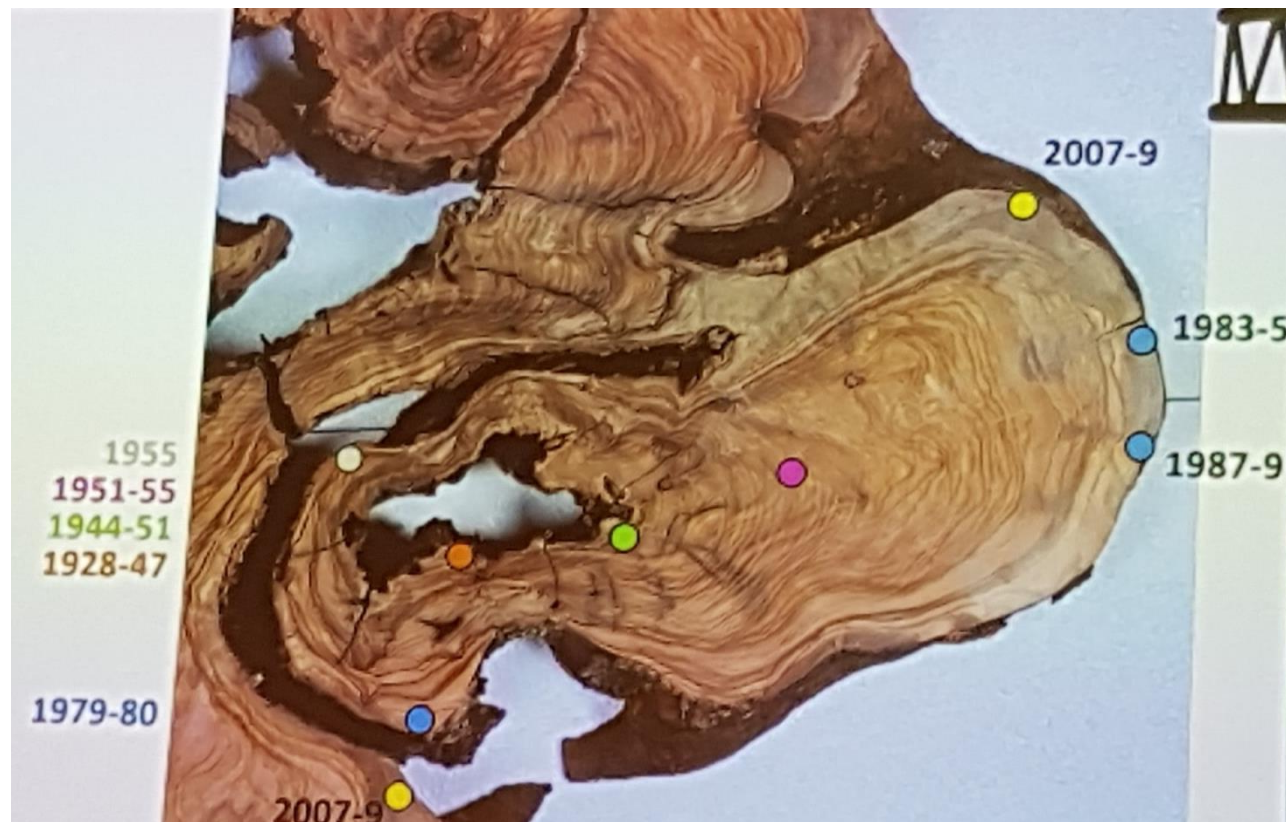




This is in agreement with most other existing inner wood within live olive trees which has not been dated to older than 200–300 years.







Radiocarbon Dating of an Olive Tree Cross-Section: New Insights on Growth Patterns and Implications for Age Estimation of Olive Trees

Yael Ehrlich¹, Lior Regev¹, Zohar Kerem² and Elisabetta Boaretto^{1*}

To remember:

- ❖ Radiocarbon (^{14}C) dating is a convenient and accurate method of absolute dating of organic materials - such as wood, charcoal, bones, grains, paper, parchment, textile, etc
- ❖ Radiocarbon dating gives **the age of material**
- ❖ Radiocarbon dating cannot give a single year – **a range of years** is obtained with a certain probability
- ❖ There are defined rules how to report radiocarbon results
- ❖ Interpretation of results should be performed in **close collaboration** of art historian and radiocarbon specialists

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