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A dataset of radiocarbon dates from Holarctic mammal collagen purified with high-quality chemistry

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Radiocarbon dates from megafaunal remains provide insights into climatic and anthropogenic factors shaping past ecosystems. Chronologies have advanced through rigorous chemical purification (pretreatment) of fossil vertebrate collagen for accelerator mass spectrometry (AMS) radiocarbon dating. We present MEGA14C, a comprehensive dataset of late Quaternary AMS radiocarbon dates for Holarctic large-bodied mammals, based on collagen purified by ultrafiltration (92% of records), XAD-2 purification (7%) and hydroxyproline isolation (1%). MEGA14C includes 11,715 dates spanning 8 orders, 23 families, 78 genera, 133 species and 18 subspecies, 27% from extinct taxa, and dominated by *Equus*, *Bos*, *Mammuthus*, *Rangifer*, *Bison*, *Ursus*, *Cervus*, *Canis*, *Coelodonta* and *Sus*. Where available, geolocation, genetic and isotopic data are provided. Pretreatment is critical for accurate and reproducible radiocarbon measurements, yet 44% of published dates lack this information. We addressed this gap through over 10,000 personal communications (out of >100,000 emails) with researchers and AMS laboratories among the parties involved in fossil dating. This unique dataset supports (pre)historical research and provides a foundation for future expansion and/or integration into a global radiocarbon repository.

Background & Summary

Radiocarbon (^{14}C) dating by accelerator mass spectrometry (AMS) is the most common chronometric method for tracking climate and ecological changes, human migrations, faunal extinctions and biodiversity shifts over the past 50,000 years[1–3] — spanning the late Quaternary, which includes the late Pleistocene and the Holocene[4]. A major research focus has been determining the timing of the global extinction of most mammal species with an adult body mass exceeding 44 kg[5,6], commonly referred to as 'megafauna'[7]. These extinctions began in Australia >40 thousand years ago[8] and have been documented thereafter across all continents[9–11] and major island groups[12,13], excepting Antarctica.

Competing hypotheses explaining late Quaternary megafaunal extinctions include climate change, disease, ecological and ecosystem changes, human exploitation or combinations of these drivers[9,14–18]. These hypotheses are tested by correlating species' ^{14}C ages with chronologies for human activity, climate and/or habitat descriptors[19–23]. Megafauna ^{14}C data are combined with ancient DNA1 to infer demographic histories[24–26], lineage replacements[27–29] and domestications[30–32]. With the biosphere facing the selective extirpation of megafauna from the late Quaternary to the present[33–35], this research improves our understanding of the ecological role of megafauna and can inspire actions to mitigate contemporary and future biodiversity loss[36–43].

Except for rare occurrences of permafrost mummies[44–48], and arid-cave coprolites and skin and other desiccated soft tissues[1,46,49], the majority of late Quaternary megafaunal remains are antlers, bones, horns and teeth — hereafter termed 'bone'. However, the accuracy of chronologies based on these fossils has been questioned

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since the development of ^{14}C dating[50–57]. Bone ^{14}C dates have been, and often continue to be considered, unreliable and are still ranked as yielding the lowest-quality ^{14}C dates[58,59]. This problem occurs because organic and inorganic compounds are physically and chemically incorporated secondarily into bones during burial and diagenesis with the passage of time[60–64]. Failure to remove exogenous carbon is the major factor causing ^{14}C dating inaccuracy[65]. Carbon-rich contaminants can be older, younger or coeval with the sample's original carbon content[66–68], resulting in differences between a fossil's measured ^{14}C age and its 'true' age of up to several millennia [see Methods]. Consequently, bones must be subjected to rigorous 'pretreatment' protocols designed to remove all foreign carbon prior to ^{14}C dating[68–72].

Databases compiling ^{14}C geochronological data (e.g., *CARD*[73], *IntChron*[74], *Neotoma*[75], the *Paleobiology Database*[76], *XRONOS*[77]) lack systematic recording of pretreatment protocols. However, researchers require information about pretreatment methods to determine the accuracy of the dates on which they base their archaeological and palaeoecological interpretations[55,66,78], or risk having those interpretations questioned or dismissed[79]. This lack of information is partly due to poor reporting standards as publications often report ^{14}C measurements, but do not describe ^{14}C chemistry[68]. Therefore, bone pretreatment remains the elephant in the room for ^{14}C dating[65], with specialists asserting that "…unless rigorous pretreatment protocols have been used, [bone] radiocarbon dates should be assumed to be inaccurate until proven otherwise"[80].

We present MEGA14C, a dataset of AMS ^{14}C dates of late Quaternary bone collagen purified by ultrafiltration, XAD-2 purification and isolation of hydroxyproline. We focus on remains of extant and extinct Holarctic Eurasian and North American mammalian megafauna. Data collection and curation involved a comprehensive review of the scientific literature, along with multifaceted efforts to obtain unpublished metadata from scientists, research sites, AMS ^{14}C facilities, government agencies, museums and universities. Our premise is that robust conclusions about the timing and cause of extinctions rely on accurate dating. MEGA14C answers calls "…to establish a large dataset of reliable radiocarbon dates … made directly on securely identified megafaunal remains. The need is for much more high quality data, not more debate based on imperfect evidence"[81]. In a companion study[82] (Fig. 1), we outline the broader challenges posed by the lack of standardized and transparent collagen pretreatment reporting, how inadequate reporting standards for ^{14}C results and collagen chemistry impede the effective curation of chronological datasets, and advocate for international solutions including open and replicable protocols, facility-led documentation and a global ^{14}C database. Together, these two studies are interdependent and should be read in tandem, as each provides essential context and insights necessary to fully understand the other.

Compiling a dataset that emphasizes rigorous sample-preparation chemistry to minimize the occurrence of inaccurate ^{14}C dates comes at a price. MEGA14C is exclusive not inclusive, and our approach required us to reject many dates of presumably high quality, particularly from high-latitude permafrost sites. Users should also bear in mind that cases will exist where even the most stringent chemical protocols cannot remove some contaminants[66,68,83]. Overall, MEGA14C is unique because previous efforts have not attempted to uncover the ephemeral, unpublished and vaguely understood chemical data necessary for ranking the accuracy of ^{14}C dates into reliable megafauna chronologies. Herein, we use the abbreviation ^{14}C (radiocarbon) to refer to the isotope carbon-14 that is used in the dating method; RC (RadioCarbon) to refer to estimates of bone age that have not been calibrated to calendar years [Usage Notes]; and square brackets […] to link text in a given section to other sections or subsections of the manuscript.

Methods

We developed MEGA14C over several postdoctoral appointments held by the first author (SHP): first at the University of Adelaide, Australia (April 2017 to December 2020), and later at the Spanish National Research Council, Spain (January 2021 to February 2023, and again from February 2024 to October 2025), with an intervening period of freelance work from March 2023 to January 2024. This effort amounts to over 30,000 person-hours, with more than 80% of the time dedicated to tracking unpublished information, particularly pretreatment. In the next four subsections, we describe the three chemical protocols considered to build MEGA14C [Target Pretreatments], our methodology for compiling ^{14}C dates from the literature [Literature Review], and the fields of information collated per record [Data Compilation].

Target pretreatments. What distinguishes MEGA14C from other datasets focused on ^{14}C dates is the systematic documentation of the specific pretreatment method applied to *each* collagen sample prior to dating. Historically, chemical protocols for bone ^{14}C dating have evolved from dating "whole bone" (meaning unprocessed bone material, whether from powder, fragments or entire bones) to demineralized bone, then to total collagen, and finally to gelatinized collagen[57]. A major advancement in this process was introduced by Robert Longin[84], who developed a protocol for collagen gelatinization. This method involves removing the bone's mineral component using an acid, typically hydrochloric acid (HCl)[85], followed by the extraction of soluble collagen (gelatin) in hot water[65,86]. Known as the 'Longin method,' it remains the standard pretreatment technique used by most AMS laboratories worldwide for ^{14}C dating of bone.

The senior author (SHP) engaged with 132 experts[65] who use and generate bone ^{14}C dates (from top research institutions and AMS facilities in 25 countries) and learned that 95% would trust the Longin method providing that additional purification steps were used, namely ultrafiltration[87], XAD-2 purification[88,89] or hydroxyproline isolation[90]. AMS ^{14}C dates from purified *versus* non-purified collagen gelatin can vary by decades to tens of millennia for the same bone[91–103] due to incomplete removal of contaminants. As a follow-up to the former research enquiry, MEGA14C focuses on ^{14}C dates of mammalian megafauna fossils that have undergone these three specific pretreatments, which we succinctly describe below (see[65,82]).

Ultrafiltration[87] removes contaminants by separating molecules based on their molecular weight. The method assumes that compounds larger than 30 kDa originate from bone collagen, while smaller molecules are

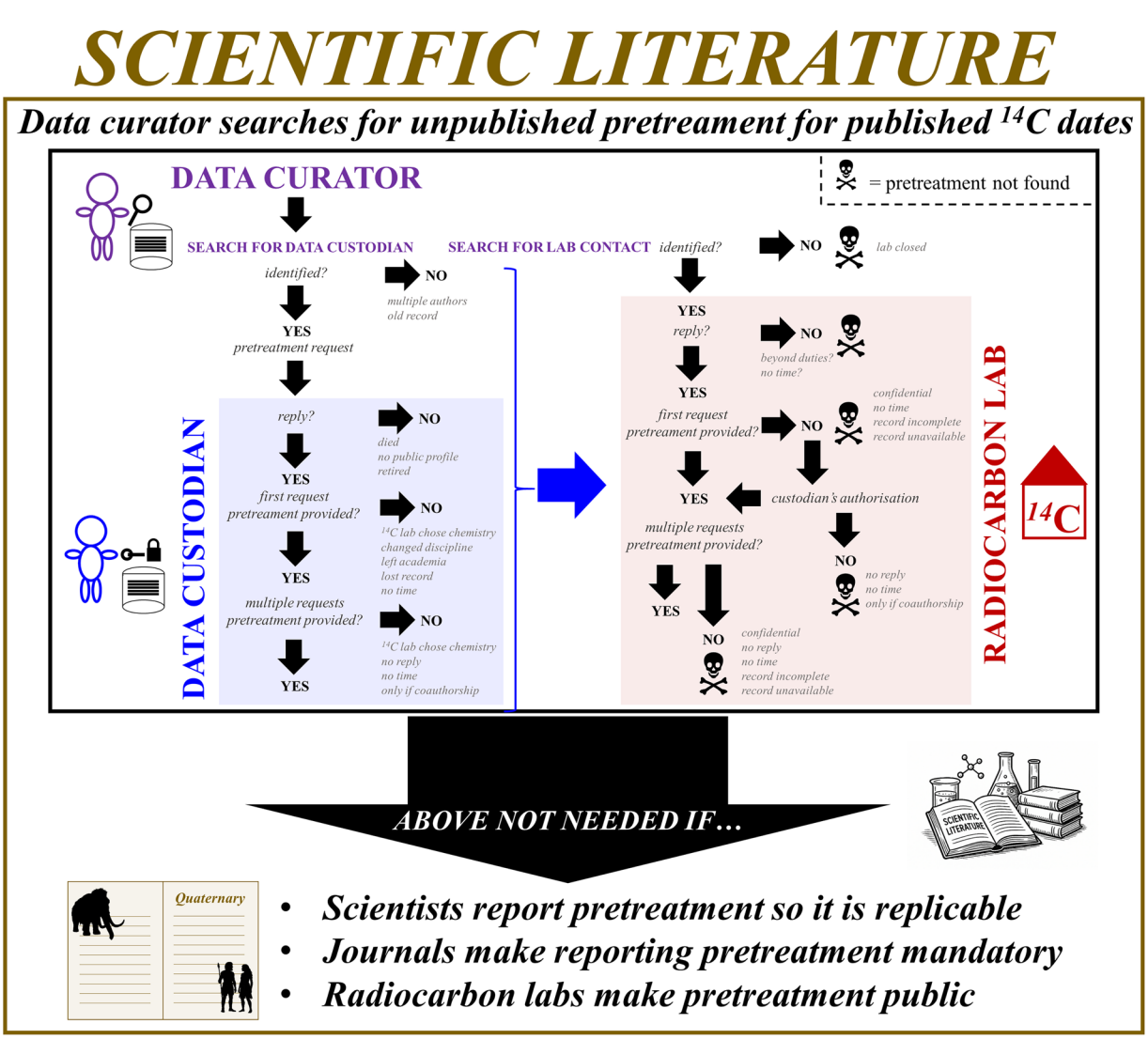


Fig. 1 Flow of enquiries used to uncover the radiocarbon (^{14}C) chemistry of records in MEGA14C for ^{14}C dates published in the scientific literature without pretreatment details: "The process of enquiry exhibits fractal behaviour, as communication with a particular party often opens multiple additional pathways of communication with other parties"[82]. These enquiries typically involve a data curator, a data custodian and an AMS ^{14}C facility. The data curator is the researcher who compiles and curates ^{14}C data from the literature, while the data custodian is the owner of the ^{14}C data, often the individual who funded the dating service, owned the sample(s) and/or submitted it(them) to the AMS facility. The enquiry process begins with the data curator contacting the data custodian; if the custodian cannot provide pretreatment information, the curator then contacts the AMS facility. The 'skull and crossbones' symbol indicates a "dead end," where a chain of enquiries is blocked and pretreatment cannot be located.

contaminants[104]. The process involves pipetting liquid gelatin into tubes with filtering membranes, centrifuging and recovering the retained high-molecular-weight fraction (>30 kDa) for ^{14}C dating[105]. XAD-2 purification[89] eliminates contaminants through hydrophobic resin chromatography. In this method, hydrolyzed collagen gelatin is passed through XAD-2, a non-polar resin, resulting in a purified hydrolyzate composed of 18 amino acids, including both primary and secondary amino acids[88]. Hydroxyproline isolation[90] also begins with gelatin hydrolysis, producing free amino-acid cations. These are then separated by liquid chromatography to isolate pure hydroxyproline for dating[106,107].

Each ^{14}C date reported in MEGA14C is categorized according to one of those three pretreatment methods. When available, we also recorded if bone samples underwent alkali and solvent treatments prior to gelatinization[65]. Typically, raw bone samples are rinsed with a dilute alkali solution, such as sodium hydroxide (NaOH) or potassium hydroxide (KOH), to remove some humic substances[108,109]. Additionally, museum bones are often treated with adhesives, coatings and consolidants, which can introduce exogenous carbon and compromise ^{14}C accuracy[110,111]; these substances can be removed, though with varying success, using only alkali or alkali and organic solvents combined[67,112].

Genus	Records	Genus	Records	Genus	Records	Genus	Records
Aenocyon (X)	**101**	***Crocuta*** (TX)	**138**	***Mammut*** (X)	**110**	*Poephagus* (T)	1
Alces (T)	**169**	*Cuon* (T)	2	***Mammuthus*** (X)	**857**	*Praemegaceros* (X)	3
Antilocapra (T)	3	*Dama* (T)	28	*Marmota* (T)	25	*Praeovibos* (X)	5
Arctodus (X)	34	*Dasypus* (TX)	11	*Martes* (T)	5	*Procapra* (T)	10
Bison (TX)	**784**	*Elasmotherium* (X)	23	*Mazama* (T)	1	*Procavia* (T)	12
Bootherium (X)	35	*Elephas* (X)	2	***Megaloceros*** (X)	**122**	*Procyon* (TX)	3
Bos (TX)	**894**	*Enhydra* (T)	1	*Megalonyx* (X)	16	*Puma* (T)	3
Bubalus (T)	1	***Equus*** (TX)	**1,211**	*Meles* (T)	5	***Rangifer*** (T)	**844**
Camelops (X)	93	*Erethizon* (T)	1	*Miracinonyx* (X)	2	*Rupicapra* (T)	12
Camelus (TX)	7	*Euceratherium* (X)	4	*Mustela* (T)	5	*Saiga* (T)	92
Canis (T)	**399**	*Felis* (T)	73	*Nothrotheriops* (X)	6	*Sinomegaceros* (X)	4
Capra (T)	**151**	*Gazella* (T)	2	*Odocoileus* (T)	50	*Smilodon* (X)	87
Capreolus (T)	83	*Gulo* (T)	5	*Oreamnos* (T)	3	*Spirocerus* (X)	4
Capromeryx (X)	1	*Haringtonhippus* (X)	25	***Ovibos*** (TX)	**184**	*Stephanorhinus* (X)	5
Castor (T)	26	*Hemiauchenia* (X)	2	***Ovis*** (T)	**245**	***Sus*** (T)	**332**
Castoroides (X)	12	*Homotherium* (X)	13	*Palaeoloxodon* (X)	3	*Taxidea* (T)	1
Cervalces (X)	7	*Hyaena* (T)	7	***Panthera*** (TX)	**205**	*Urocyon* (T)	3
Cervus (T)	**692**	*Lutra* (T)	3	*Paramylodon* (X)	17	***Ursus*** (TX)	**756**
Coelodonta (X)	**338**	*Lynx* (T)	8	*Platygonus* (X)	14	*Vulpes* (T)	62

Table 1. Number of records in the dataset MEGA14C (11,715 accelerator-mass-spectrometry radiocarbon dates of late Quaternary Eurasian and North American mammal records) that could be assigned to the Linnaean taxonomic level of *Genus*. A subset of 2,212 records (19% of the dataset) could not be identified and their counts are excluded herein. Genera contributing >100 records are boldened; of those only ten exceed 300 records: *Equus*, *Bos*, *Mammuthus*, *Rangifer*, *Bison*, *Ursus*, *Cervus*, *Canis*, *Coelodonta* and *Sus* (in that order). Capital letters indicate 'extinct' (X) or 'extant' (T) genera and, if extant, genera that include both extant and extinct species (TX).

Literature review. Here we undertook a comprehensive review of ^{14}C dates published in the scientific literature following six steps:

(1) We compiled the Linnaean names of late Quaternary megafaunal mammals from Eurasia and North America following Koch and Barnosky[6] and Faurby *et al.*[113]. The final tally ('target taxa') comprised 29 extinct genera along with 9 extant genera that include extinct species (Table 1). We also collected ^{14}C dates from 37 additional extant mammal genera reported along with ^{14}C dates of the target taxa (Table 1). Many of the latter were from cervids and domesticated species (cat, cattle, dog, goat, horse, pig, sheep), and some included mammal species with maximum body mass below 44 kg (body mass).
(2) On 01/06/2018, we retrieved the set of Scopus-indexed research papers matching a Boolean search with the string of key words [*RADIOCARBON AND* [genus of target taxa OR *MEGAFAUNA*]].
(3) We replicated step (2) in the online search menus of *ScienceDirect, Cambridge University Press, Elsevier, Oxford Academic, SAGE, Springer, Taylor & Francis and Wiley*.
(4) We accessed the full text of all the primary literature retrieved in steps (2) and (3), and compiled the references cited therein that could potentially report additional ^{14}C dates, which included other research papers and most of the secondary literature we reviewed (books, conference proceedings, government reports, Honours/Masters/PhD theses). Ultimately, we selected only those publications screened in steps (2) to (4) that did contain ^{14}C dates.
(5) We also reviewed published datelists as potential sources of megafauna ^{14}C dates. The first ^{14}C dates of megafauna and human bones published in the 1950s were seen as an extraordinary scientific breakthrough, and ^{14}C datelists appeared in *Science*[114–116] and *Nature*[115,117–119] until editors argued that these journals were "not an archive for any branch of science"[120]. Some laboratories continued to publish datelists in specialized journals such as *Archaeometry*, *Journal of Archaeological Science* or *Radiocarbon*, but as technological advances led to increased sample throughput, the effort involved in assembling such lists (plus concerns about intellectual property) led to some laboratories abandoning this process by the early 1970s[53]. With the development of AMS producing an additional 5-to-10-fold increase in throughput, publishing lists of dates has now become the exception rather than the rule. Currently, the Oxford Radiocarbon Accelerator Unit (ORAU) is the only AMS facility still publishing datelists, covering 1984[90] to 2018[121], with the next edition scheduled for publication within 12 months (Emma Henderson, pers. comm., 16/09/2025).
(6) From 01/08/2018 to 12/09/2025, we automated a fortnightly alert in *Scopus* and *ScienceDirect* that matched the same string of key words used in step (2), and resulted in additional publications containing ^{14}C data. Therefore, over the study period, we combined data curation with incorporation of new data to keep the content of MEGA14C up to date. All publications ($n > 5{,}000$) revised were stored in an EndNote™ library with a copy of the full text whenever available. The last ^{14}C date was added to MEGA14C on 13/09/2025.

Field	Column	Heading	Description
Record code	A, BB	*N_Record*	Record number in MEGA14C
	B, BA	*Ref_ID*	First author's surname and year of publication of ^{14}C date
Taxonomy	C	*Class*	Linnaean taxonomic category
	D	*Order*	Linnaean taxonomic category
	E	*Family*	Linnaean taxonomic category
	F	*Genus*	Linnaean taxonomic category
	G	*Species*	Linnaean taxonomic category
	H	*Genus_Species*	Linnaean taxonomic category
	I	*Body_Size*	megafauna (>44 kg body mass) *versus* mesofauna
	J	*Global_Status*	extant *versus* extinct taxa
	K	*Wild_Domesticated*	domesticated *versus* wild taxa
Chronology	L, AZ	*Code*	Dating code assigned by AMS facility to ^{14}C date
	M	*Date*	Non-calibrated ^{14}C date in years BP
	N	*Error*	Non-calibrated 1 SD error of the ^{14}C date in years
	O	*Replicates*	Dating codes for ^{14}C dates from the same fossil/individual
	P	*Caveat_Type*	Source of data inaccuracy and/or imprecision
	Q	*Caveat_Description*	Description of data inaccuracy and/or imprecision
	R	*Caveat_Reliability*	Category of data reliability
Chemistry	S	*Pretreatment*	Chemical purification of fossil bone prior to AMS ^{14}C dating
	T	*Psource*	Source of ^{14}C pretreatment
	U	*CNratio*	Carbon-to-nitrogen mass ratio (C:N)
	V	*δ13C*	^{13}C/^{12}C in ‰ δ^{13}C
	W	*δ15N*	^{15}N/^{14}N in ‰ δ^{15}N
Material & Genetics	X	*Find*	Whether fossil found on land or submerged in the sea
	Y	*Material*	Type of skeletal material dated (e.g., humerus, molar)
	Z	*Collection_ID*	Specimen catalogue code in fossil collection (e.g., museum)
	AA	*Sample_ID*	Sample code given by researcher, chemistry and/or genetics lab
	AB	*Genetics*	Availability of mitochondrial or nuclear sequences
	AC	*Genetics_ID*	Sequence accession number in online repository
Locality	AD	*Site*	Name of study site
	AE	*Region*	Geopolitical region of study site
	AF	*Country*	Country of study site
	AG	*Geolocation*	Broad geographical area
	AH	*Lat_D*	Raw latitudinal degrees as collected from literature
	AI	*Lat_M*	Raw latitudinal minutes as collected from literature
	AJ	*Lat_S*	Raw latitudinal seconds as collected from literature
	AK	*Lon_D*	Raw longitudinal degrees as collected from literature
	AL	*Lon_M*	Raw longitudinal minutes as collected from literature
	AM	*Lon_S*	Raw longitudinal seconds as collected from literature
	AN	*Lat*	Latitudinal value in decimal numbers
	AO	*N*	N = Northern coordinate
	AP	*Long*	Longitudinal value in decimal numbers
	AQ	*East/West*	E = Eastern coordinate, W = Western coordinate
	AR	*Latitude*	Final latitudinal estimate
	AS	*Longitude*	Final longitudinal estimate
	AT	*Lat_band*	5° band for latitude
	AU	*Lon_band*	5° band for longitude
Bibliography	AV	*Primary_Reference*	Literature source of ^{14}C date
	AW	*Primary_Access*	DOI or website giving access to primary reference
	AX	*Secondary_Reference*	Literature relevant to ^{14}C date and site

Table 2. Fields of information collected for each record in MEGA14C, a dataset of radiocarbon (^{14}C) dates of late Quaternary Eurasian and North American (Holarctic) mammals. Missing information for any of those fields is tagged with 'NA' in the dataset.

Data compilation. For each publication selected in our literature review, we identified those ^{14}C dates estimated by AMS on mammal bone gelatin pretreated by ultrafiltration, XAD purification or hydroxyproline isolation[82]. Every row in MEGA14C comprises a unique record representing a fossil with a unique ^{14}C date. For

each record (row), we filled cells with 'NA' for those fields' columns for which information could not be found [Technical Validation]. For fossils dated multiple times, one row is used for each individual date. For each record we collected and stored eight fields of information across a total of 53 columns (Table 2) as follows:

- *Record* = 4 columns A, B, BA and BB
- *Taxonomy* = 9 columns C to K
- *Chronology* = 8 columns L to R and AZ
- *Chemistry* = 5 columns S to W
- *Material & Genetics* = 6 columns X to AC
- *Locality* = 18 columns AD to AU
- *Bibliography* = 3 columns AV to AX

Record

We labelled each ^{14}C date with a consecutive numeric code at unitary steps from top to bottom of the dataset (columns A and BB with heading = *N_Record_v1*), and an alphanumeric code indicating the first author and year of the publication of the date (columns B and BA = *Ref_ID*). All records in MEGA14C were sorted alphabetically by taxonomic genus, then alphanumerically by the dating code. Specimens with non-diagnostic taxonomy at the order, family or genus levels are listed at the end of the dataset.

Taxonomy

We present the Linnaean taxonomic *Class* (column C), *Order* (column D), *Family* (column E), *Genus* (column F) and *Species* (column G, including the *Species* and the *Subspecies* if identified) assigned to each dated mammal fossil per ^{14}C date. *Genus* along with *Species* (and *Subspecies*) names together are provided in column H. When a taxonomic level below *Class* (Mammalia) was unknown, we used the abbreviations *ord*., *fam*., *gen*. and *sp*., respectively. Records with a likely taxonomy are flagged with the abbreviation *cf*. (*confer*). For instance, we used *Mammuthus cf. primigenius* for fossils that *compared to* woolly mammoths, *cf. Artiodactyla/Perissodactyla* for herbivores or ungulates, or *cf. Bison/Bos cf. bonasus/primigenius* for a bone that could belong to either European bison or aurochs.

We routinely asked the authors of publications if the taxonomy of material belonging to uncertain specimens had been revisited since the publication of the ^{14}C dates, and refined their taxonomic status in MEGA14C accordingly. For a number of records with published genetic information (particularly *Bison* and *Equus*) we used the species identity reported in USA National Center for Biotechnology's *GenBank* (www.ncbi.nlm.nih.gov) or the European Nucleotide Archive's *ENA* (www.ebi.ac.uk/ena). Finally, when the taxonomic identity of the samples allowed so, we assigned fossils to three categories: (1) 'megafauna' or 'mesofauna' depending on whether males or females at the species level exceed a maximum body mass greater or lower than 44 kilograms, respectively (column I = *Body_Size*), (2) 'extant' or 'extinct' (column J = *Global_Status*) depending on whether the taxon in question has survived or not to modern times, and (3) 'domesticated' or 'wild' (column K = *Wild_Domesticated*) depending on whether or not the taxon has been globally domesticated by humans. Fossils identified to *Species* could be allocated a status for each of the three categories, whereas fossils identified only to *Genus* (or a higher taxonomic rank) were only allocated a status when it was shared by all species to which that fossil could plausibly belong. For instance, European and North American fossils of the order Proboscidea, or the family Rhinocerotidae, unequivocally belong to *extinct* and *wild megafauna* species irrespective of their species-level taxonomy, whereas specimens of *Equus caballus* or *E. ferus* older than (conservatively) 5000 RC years unequivocally belong to *wild megafauna*. Among the samples with unknown taxonomy, we conservatively assigned to *mesofauna* those reported as 'medium' body-sized species, and to megafauna those described as large mammals; for example, equids, large ungulates, cf. *Bison/Bos* and the like.

Chronology

We reported the dating code given by an AMS facility to every ^{14}C date (columns L and AZ = *Code*), the non-calibrated ^{14}C date (column M = *Date*) and its one standard deviation (column N = *Error*) in RC years BP (where Present = 1950 AD[122]). For fossils or individuals that had been *directly* dated multiple times (with and without our target pretreatments), we provide the dating codes of all dates given by the relevant AMS facilities (column O = *Replicates*).

We flagged aspects that rendered a ^{14}C date *potentially* inaccurate in three columns: *Caveat_Type* (column P) classifies the type of caveat, with caveats mostly including confirmed or suggested contamination, faulty quality indicators such as low collagen yield, ultrafiltered fraction different from >30 kDa (only Rancho La Brea fossils), ^{14}C measurements retracted by authors or AMS laboratories, younger or older than expected dates given the depositional context of a bone, and outliers in chronological models. *Caveat_Description* (column Q) describes the caveat, and *Caveat_Reliability* (column R) assigns one of six labels of reliability to each record — all described below [Technical Validation].

Chemistry

For each record, pretreatment is presented in column S (*Pretreatment*). All ^{14}C dates in MEGA14C have been obtained from bone samples subjected to the Longin method including HCl demineralization and gelatinization [Methods]. We report if a bone sample was further treated with (1) alkali, organic solvents, or both, prior to gelatinization, and (2) ultrafiltration, XAD-2 purification or hydroxyproline isolation following gelatinization and preceding AMS ^{14}C dating. For example, 'Hydroxyproline-Gelatin (NaOH/Collagen)' indicates a

sample of demineralized bone collagen extracted with NaOH, collagen gelatin hydrolyzed to free amino acids and hydroxyproline isolated with high-performance liquid chromatography. Likewise, 'XAD-Gelatin (KOH/Collagen)' indicates that the demineralized bone collagen was rinsed with KOH, gelatinized, hydrolyzed and purified with XAD-2 resin. A question mark signifies uncertainty, e.g., 'Ultrafiltration (>30 kDa)-Gelatin(NaOH?/Collagen)' indicates the dating of ultrafiltered gelatin but the inclusion of a sample wash with alkali during pretreatment could not be confirmed. When solvents are not mentioned for a given date this might indicate that solvents *were* or *were not* used.

When the chemistry purification for a ^{14}C date was not reported where it was published, we noted whether we obtained it through personal communications with data custodians or laboratory personnel, or other sources (column T = *PSource*) — the nature, variety and challenges of those communications are described in the companion study[82] (Fig. 1) and briefly described below. Whenever available, we also collected the atomic Carbon/Nitrogen ratio (C/N) (column U = *CNratio*) and $\delta^{13}C$ (column V = $\delta^{13}C$) and/or $\delta^{15}N$ (column W = $\delta^{15}N$) values.

When a ^{14}C date selected from the literature for MEGA14C had been published without data for some of the fields described in Table 2, dating codes were essential to request missing information from AMS facilities and/or the custodian of the ^{14}C date (who might differ from the author submitting the sample to the AMS facility and/or publishing the date). Communications with AMS facilities and researchers were done via email. We first contacted the lead and/or corresponding author publishing the data; if they lacked the pieces of requested information or did not answer our emails, we then contacted the AMS facility[82] (Fig. 1). Some AMS facilities filled our gaps of information straight away. Some could not do so if the effort needed for extracting the requested data from their files was too great. Some requested that we send them written consent from data custodians to release the information directly to us. Others required data custodians to make the request themselves and then share the information with us. The implication is that AMS facilities interpret data confidentiality in their own specific terms when it comes to releasing their archived information to third parties[82].

The number of personal communications required to obtain missing data ranged from one to tens of emails per ^{14}C date as authors (in no particular order) forwarded our request to colleagues, government and private agencies that paid for the dating, museums and other institutions holding the dated specimens and/or AMS facilities. This was a time consuming, difficult and often frustrating process for all parties involved, taking several years for some records[82] (Fig. 1).

Material & Genetics

If documented in the literature, in column X (*Find*) we reported if a fossil was found on land (*terrestrial*) or in the sea (*marine*), and the type of dated skeletal material (column Y = *Material*) including: antler, bone name, horn, tusk, tooth name or, if the latter were unknown or not reported, *bone*, *tooth* or *unnamed skeletal material*. We also strived to collate the collection or museum catalogue number of the specimen (column Z = *Collection_ID*), the sample number given by authors and/or chemistry or genetics laboratories (column AA = *Sample_ID*), and the sequencing of mitochondrial (*mtDNA*) and/or nuclear (*nDNA*) genetic data (column AB = *Genetics*) and the accession numbers for those sequences in online genetic repositories (column AC = *Genetics_ID*). Accession numbers mostly represented nucleotide sequences (partial or complete) for single loci and (mito)genomes and SAM (Sequence Alignment Maps) files from *GenBank* and *ENA*. For many records, the institutions hosting specimens were not reported or were cited with acronyms that could not be unequivocally linked to institutions[82]. When we were unable to differentiate an alphanumeric code from institution-based catalogue identities *versus* author-based samples, we reported them in column AA (*Sample_ID*).

Matching genetic accession numbers with ^{14}C dates for a given bone/sample/specimen was a complex task for various reasons, the most important being that ^{14}C dates (code and/or date ± errors) are rarely part of the fields of information submitted to *GenBank* and *ENA*[82]. Additionally, labels for the same bones/samples/specimens were often inconsistent across publications as well as between the publications where the accession numbers are published and the records available in *GenBank* and *ENA*. To add complexity, the names given to genetic samples sometimes are the museum catalogue numbers (with their own inconsistencies across publications), while in many publications authors report an interval of genetic accession numbers (*first to last*) with no one-to-one match between individual accession numbers and individual samples or specimens[82].

Locality

We report the name of the *Site* (column AD), current geopolitical *Region* (column AE), *Country* (column AF), and *Geolocation* (column AG) for each date. The seven geolocations serve as a general guide and include:

(i) Europe: European countries and the part of Russia west of the Ural Mountains.
(ii) Russia/Siberia: from the eastern side of the Urals in Russia to the western flank of the Lena River.
(iii) Arabia/Asia: Armenia, Azerbaijan, Beirut, China, Georgia, Iran, Iraq, Israel, Japan, Jordan, Kazakhstan, Kyrgyzstan, Mongolia, Saudi Arabia, Tajikistan, Turkey, Uzbekistan and the Primorsky Krai in Russia.
(iv) Western Beringia (W_Beringia): from the eastern flank of the Lena River and northern flank of the Aldan River to the Russian coastline of the Bering Strait including Russian islands.
(v) Eastern Beringia (E_Beringia): Alaska and Yukon to the western flank of the Mackenzie River including Canadian and USA islands.
(vi) Canada: Greenland and Canada excluding Yukon.
(vii) USA: Belize, Mexico and the USA excluding Alaska.

For each date, we also reported the WGS84 latitude and longitude (columns AH to AU) of the site where the dated fossil was found. If not documented in the literature, we obtained exact or approximate latitude/longitude

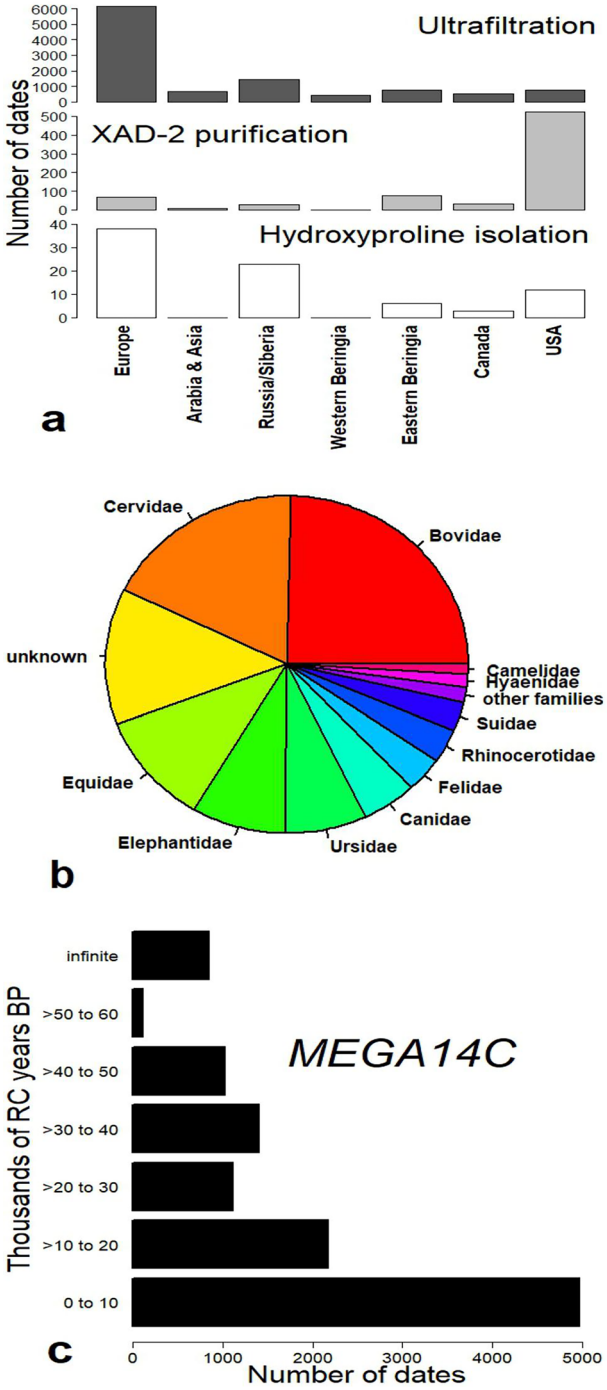


Fig. 2 Summary of 11,715 accelerator-mass-spectrometry radiocarbon dates in the MEGA14C dataset for late Quaternary Eurasian and North American mammal records. Top (**a**): Frequency of dates by pretreatment method, grouped by fossil provenance across seven Holarctic regions, with bars roughly ordered east to west. American AMS facilities produced all dates on XAD-2 purified collagen, while ultrafiltration has been more widely adopted by European AMS laboratories. Middle (**b**): Frequency of dates across 23 mammal families, from Bovidae to Camelidae, where 'other families' includes taxa each contributing <1%, and 'unknown' includes records with uncertain family-level taxonomy. Bottom (**c**): Frequency of dates by 10,000-year temporal bins (with 'infinite' indicating ages beyond radiocarbon detection).

(1) from authors or museums, (2) by matching maps from the publications of ^{14}C dates (or references therein) with aerial views of the study regions in *Google Earth* or *Google Maps*, and (3) from a variety of internet sources. The exact coordinates of the same site frequently varied among publications. In the case of single caves (e.g., Natural Trap Cave, Niedźwiedzia Cave) or discrete human settlements, monuments or burial grounds (e.g., Çatalhöyük, Menga, Varna Cemetery) we standardized all coordinates per site to a single set of latitude and longitude values and reported the source of those coordinates. For islands (e.g. Aylon, Wrangel), lakes (e.g., Baikal,

Taymyr) and complexes of sites (e.g., Dutchess Quarry Caves, Kostënki, creeks and rivers in Alaska and Yukon), we endeavoured to report the exact coordinates of each fossil find whenever possible.

For many fossil finds, the exact coordinates were not published, are often not public (mostly in North America), and therefore could not be estimated with absolute precision[82]. Therefore, we have used a total of 12 columns to track how we estimated latitude and longitude per fossil and ^{14}C date. This approach allows us to capture and communicate the exact level of precision available for each record (e.g., degrees and minutes but not seconds) and facilitates refinement in future versions of the dataset. The first six columns (AH to AM) are raw coordinates (as extracted from the literature) broken down by latitudinal and longitudinal degrees, minutes and seconds (*NA* used when minutes and/or seconds were unavailable), the following four columns are raw decimal coordinates (AN = *Lat* [latitudinal estimate] and AO = *N* [Northern Hemisphere], AP = *Long* [longitudinal estimate] and AQ = *East/West* differentiating *E* [Eastern] from *W* [Western] coordinates). The following two columns are the final decimal coordinates with positive or negative longitudinal coordinates indicating Eastern or Western geolocation, respectively (AR = *Latitude*, AS = *Longitude*). Finally, column AT (*Lat_band*) groups records into 5° longitude bands, from >15° to 20° to >80° to 85° North; and column AU (*Lon_band*) groups records into 5° latitude bands, ranging from >0° to 5° East to <5° to 0° West. These bands cover approximately an east-to-west distance of ~557 km at 0° latitude (Equator), ~278 km at 60° latitude and 0 km at 90° latitude (North Pole). Together, *Lat_band* and *Lon_band* enable users to filter records into square or rectangular cells, each covering an area from ~40,000 km² (northernmost records) to ~300,000 km² (southernmost records).

Bibliography. We cited the publication source of each ^{14}C date as the primary reference, including the first author along with the journal/book name, volume, pages, year of publication (column AV = *Primary_Reference*), and the digital object identifier or, if absent, a webpage (e.g., *ResearchGate*, *Academia.edu*) giving access to the publication (column AW = *Primary_Access*). The citation of an Honors, Masters or PhD thesis shows the author, year of publication and the university conferring the degree. For the majority of records, we also cite one or several additional references (column AX = *Secondary_Reference*) because we deemed them important in terms of data quality, chronology or cultural importance of the site, source of isotopic and/or genetic data or, in a few cases, because that secondary reference was likely to be the publication in which a ^{14}C date was first published but which we could not access as a full text. Regardless of whether the publication was a peer-reviewed research paper or secondary literature (theses, reports, books), we always chose the publication where a date was first published *and* we had access to its full content as the primary reference because first-publication year is a reliable proxy for the state of the art of ^{14}C pretreatment at the time of publication.

Data Records

We have deposited the MEGA14C dataset in *figshare*[123], together with two *R* scripts for subsetting records and calibrating ^{14}C dates. The scripts are summarized in Usage Notes. The dataset has been stored in a Microsoft™ Excel workbook containing two worksheets: (1) *MEGA14C_raw*, which is free of any formatting, and (2) *MEGA14C_formatted*, which includes the dataset with column colours to distinguish information fields and cell formulas (e.g., for estimating latitude and longitude in decimal degrees, see above).

Data Overview

MEGA14C contains 11,715 AMS ^{14}C dates from mammal skeletal fossils across Eurasia and North America, obtained through high-quality chemistry. Of these, unpublished collagen pretreatment details come from personal communications (2,495 records, 21% of dataset), the ORAU online repository (2,195 OxA records: c14.arch.ox.ac.uk/dataset), or references in original publications (435 records). 93% (10,858 dates) derive from collagen samples pretreated with ultrafiltration, while 743 use XAD-2 purification and 86 use hydroxyproline isolation (Fig. 2a). 81% of ultrafiltered and 75% of hydroxyproline-isolated samples are from Eurasian mammals, and 86% of XAD-2-purified samples are from North America (Fig. 2a). Up to 2,207 records include combined ^{14}C and genetic data.

Within MEGA14C, 83% of records correspond to taxa exceeding 44 kg in maximum adult body mass, and 27% represent extinct taxa. Together, these include 134 species and 29 subspecies, 76 genera (Table 1), 23 families (Fig. 2b), and 8 orders (Artiodactyla, Carnivora, Cingulata, Hyracoidea, Perissodactyla, Pilosa, Proboscidea, Rodentia). The following genera each contribute over 300 ^{14}C dates (Table 1): *Equus* (1,211), *Bos* (894), *Mammuthus* (857), *Rangifer* (844), *Bison* (784), *Ursus* (756), *Cervus* (692), *Canis* (399), *Coelodonta* (338) and *Sus* (332). Among domesticated taxa, the dataset is dominated by *Bos taurus* (627 records), followed by *Equus caballus* (402), *Ovis aries* (218), *Canis familiaris* (200), *Sus domesticus* (164), *Capra hircus* (39), and *Felis silvestris catus* (4). Eurasian records constitute 76% of the dataset, while the 912 dates from Eastern Beringia (Alaska and Yukon) are twice as many as those from Western Beringia. Overall, 62% of the mammal fossil dates fall within the Holocene and terminal Pleistocene (20,000 RC years BP to present); an additional 30% date to 20,000–50,000 RC yeasrs BP, 1% exceed 50,000 RC years BP and 7% are infinite (Fig. 2c).

Technical Validation

The dataset is complemented with related work providing a detailed assessment of the chemical effectiveness of our three target pretreatments[65], and the companion study[82] (Fig. 1) that examines challenges in collagen pretreatment reporting and in accessing unpublished ^{14}C data from researchers and geochronological laboratories. Together, these studies highlight the need for improved documentation of published ^{14}C measurements and propose solutions to ensure high-quality datasets and, ideally, the development of a global ^{14}C data repository. Nevertheless, a brief summary is provided here to guide users of our dataset, while Fig. 1 illustrates the sequence

of enquiries that unfold when a data curator searches for the unpublished pretreatment details of one or more ^{14}C dates. We used this approach to validate unpublished information across records in the dataset.

The three target pretreatments of collagen are not chemically equivalent, particularly when collagen contamination is present. In such cases, both chemical reliability and ^{14}C dating accuracy are expected to improve with increasing purification stringency, progressing from ultrafiltration (substrate = collagen protein molecules) to XAD-2 purification and finally hydroxyproline isolation (substrate = free amino acids). Irrespective of the minimum or maximum molecular size of the dated fraction (e.g., 3, 5, 30 or 100 kDa), ultrafiltration does not fully eliminate non-collagenous and <30 kDa material[124–126], nor humic substances cross-linked with collagen such as through Maillard reactions[108]. XAD-2 purification and hydroxyproline isolation, however, entail higher costs and reduced yields of datable material relative to ultrafiltration[65].

Those records in our dataset bearing some type of uncertainty were assigned to seven categories of reliability (DATED_FRACTION, UNCONFIRMED_PRETREATMENT, INFINITE, CAUTION, UNQUESTIONED, UNRELIABLE). DATED_FRACTION dates (281 records) were obtained through 3–30 or 5–100 kDa ultrafiltered fractions (Rancho La Brea fossils) or from total amino-acid content isolated from samples not gelatinized nor purified with XAD-2 resins. UNCONFIRMED_PRETREATMENT are dates (61 records) potentially obtained from ultrafiltered or XAD-2 purified gelatin but pretreatment could not be ascertained. INFINITE dates (37 records) are ^{14}C measurements that gave finite dates with errors but authors consider them to be infinite. UNQUESTIONED dates (245 records) were treated as accurate by authors despite a range of caveats, e.g., samples out of depositional order, from uncertain localities, in batches of samples with inaccurate dates, with borderline quality indicators (e.g., high Carbon/Nitrogen ratio) and/or outliers in chronological models. CAUTION dates (293 records) were treated as potentially or tentatively inaccurate by authors due to similar caveats as those described for UNQUESTIONED dates. UNRELIABLE dates (827 records) are considered inaccurate by authors and/or have been confirmed to originate from carbon-contaminated samples. These dates should be abandoned and no longer included in future geochronological studies because they might mislead chronological results and inferences. For instance, the entire series of ORAU's dates obtained from skeletal remains in the intervals OxA-9361 to OxA-11851 and OxA-12214 to OxA-12236 are 'unreliable' due to demonstrated carbon contamination from glycerol in the ultrafilters[127,128], a contamination now eliminated by ultrasonicating the membranes in ultrapure water[127].

Critically, accurate chronological reconstructions depend not only on the number of available ^{14}C dates but also on the chemical reliability of each measurement. To validate the data included in MEGA14C, when pretreatment information was not reported in the literature, we sought verification through direct correspondence with data owners, research teams, ^{14}C laboratories, museums, government agencies and even fossil collectors[82] (Fig. 1).

Finally, we note that some authors round ^{14}C dates and errors in RC years BP to the nearest 10 or 100, e.g.[24,129–132], following the conventions adopted at the 9th International Radiocarbon Conference (Los Angeles and La Jolla, 1976): "…The magnitude of the standard error determines the rounding off of a conventional ^{14}C age… In reporting the standard error, the first two digits should be retained. For instance, 8,234 ± 256 and 42,786 ± 2,322 are rounded, respectively, to 8,230 ± 260 and 42,800 ± 2,300. When the standard error is less than 100 yrs [years], rounding off to the nearest multiple of ten is recommended between 50 and 100 yrs, and rounding off to the nearest multiple of five below 50 yrs"[133]. The application of this rounding convention is inconsistent in the literature and might add minute variation to the calibration of ^{14}C dates. Such a variation will be fully procedural in nature, hence without geochronological meaning.

Usage Notes

Along with the full dataset, we provide two scripts in language *R*[134] and an associated manual for users to manipulate the dataset in two forms — these materials are all deposited in *figshare*[123]. Firstly, users can subset a given number of records of the dataset by selecting a specific taxonomic *Order*, *Family* or *Genus* and/or *Site*, *Country* or *Geolocation*. This subsetting also allows for excluding or including fossils with uncertain taxonomy and dates within the seven categories of reliability described in the previous section [Technical Validation]. Secondly, users can calibrate all records in the dataset or a chosen subset of records and fossils using the latest version of the *IntCal* (Northern Hemisphere)[135] and *Marine* (marine)[136] calibration curves as implemented in the *R* package *Rextinct*[137]. The *R* scripts comprise the files: (*i*) "MEGA14C_Function" (*.R): the function hosting the subsetting/calibrating functionality, and (*ii*) "MEGA14C_Main" (*.R): the lines of code through which users can install the required *R* packages, load the dataset, select subsetting/calibrating criteria, load and run the function (MEGA14C_Function), and generate output files simultaneously in both Microsoft™ Excel and Text formats. Running the scripts is simple and user-friendly (e.g., in *RStudio*[138]), requiring no advanced bioinformatics knowledge. The file "MEGA14C_Rscript_HowToManual" (*.pdf), also deposited in *figshare*, provides detailed instructions on how to use these scripts.

Data availability

The dataset is available at https://doi.org/10.6084/m9.figshare.27826200.

Code availability

The *R* scripts developed for manipulating the MEGA14C dataset [Usage Notes/Calibrating 14C dates in MEGA14C] are available on *figshare*[123]. *R* is a programming language in a free software environment distributed under the terms of the GNU General Public License (www.R-project.org/Licenses).

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Author contributions

S.H.P. conceived the idea, reviewed the literature, collated and curated the data, communicated with researchers, curators, technicians and AMS facilities, built the dataset, and wrote the first draft of the manuscript and the *R* scripts. T.W.S. contributed archival data from 1988 to present, contacts for researchers and institutions, discussion and details on ^{14}C protein chemistry and edited preliminary and final drafts of the manuscript. J.R.S. contributed sample processing details for Lawrence Livermore National Laboratory, Livermore (to 2001; CAMS) and University of California, Irvine (2001 to present; UCIAMS), contacts for researchers and institutions and chemical discussions. K.J.M. advised on selection and formatting of dataset metadata fields and categories, provided feedback on early iterations of the dataset design, participated in discussions about megafauna chronologies, revised the *R* script manual and contributed to the writing of the manuscript. C.S.T. contributed funding for the project, gave advice on the dataset and contributed to the writing.

Competing interests

The authors declare no competing interests.

Additional information

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