ENVIRONMENTAL ARCHAEOLOGY
https://doi.org/10.1080/14614103.2025.2561284

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Radiocarbon Dating Insect Samples: New Data and Recommendations

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ABSTRACT

Radiocarbon dating insects is difficult because insects are small, fragile, and rarely found in sufficient quantities. Interactions between the chitin in the insect body and sources of carbon contamination are less well understood than for collagen and cellulose-based organic materials such as bone and plant remains. Thus there is as yet no single radiocarbon pretreatment that reliably removes all contaminating carbon across all types of insect remains. Various studies have highlighted important information concerning the practicalities and applicability of different approaches to radiocarbon dating insect remains. However, there are no papers that synthesise findings across different studies. Here, we present a review of previous work alongside new data to investigate different chemical approaches to sample processing and long-term storage and their impact on the chemistry of samples and contaminants. This confirms the difficulty of removing contamination from older samples while retaining sufficient pretreated material for subsequent measurement. It also shows that Fourier transform infrared (FTIR) spectroscopy does not always provide sufficient resolution to detect carbon contamination from non-insect sources with confidence. Thus, insect samples that have been in contact with paraffin or stored in ethanol for an extended period of time should not be selected for radiocarbon dating.

ARTICLE HISTORY
Received 22 April 2025
Revised 29 August 2025
Accepted 8 September 2025

KEYWORDS
Radiocarbon dating; insects; chitin; environmental archaeology; chemical pretreatment; fourier transfrom infrared spectroscopy (FTIR)

Introduction

Radiocarbon dating of archaeological sites is usually based on bone, wood or other plant materials (Bayliss and Marshall 2022). However, it may sometimes be desirable to date fossil insects for environmental sequences where little or no other material survives or to precisely determine the timing of environmental changes seen only in the fossil insect record (e.g. Nakazawa et al. 2015; Holden and Southon 2016). Tripp and Higham (2011) p. 63 state that: 'Direct dating of insects is desirable because it removes any uncertainty of association between the insects and other dated objects, providing a more accurate placement of the insects upon a chronological scale'. Insects have a chitinous exoskeleton and less is currently understood about how chitin interacts chemically with carbon-containing compounds from both the depositional environment and radiocarbon dating protocols than for bone (collagen) or wood, seed and charcoal (cellulose). Thus, whilst there is detailed guidance available for sample collection and storage for these (Bayliss and Marshall 2022) and well-established pretreatment protocols (e.g. Brock et al. 2010), the same is not true for insects.

This is particularly problematic given the use of various organic chemicals (e.g. paraffin, ethanol, industrial methylated spirits) for both processing and storage of insect environmental samples (e.g. Rousseau 2011). These substances are usually petroleum based and generally assumed to be radiocarbon-dead. Paraffin is not water soluble, either as solid wax, which can be used to preserve or embed fragile plant or charcoal specimens, or in its liquid form (also known as kerosene, often containing additives), which is used for flotation of insect remains from sediments. There are also indications from previous research that chitin may sometimes be chemically altered during storage in ethanol (Panagiotakopulu et al. 2015) or be particularly effective at absorbing atmospheric CO_2 under particular chemical conditions (e.g. Eftaiha et al. 2016; Foong et al. 2023), although an acid wash step within the pretreatment should remove this (Table 1). The chemical processes through which these changes may occur are complicated by changes in the chemical structure and composition of insect remains on degradation. A further problem is that there are almost always very few insect sclerites retrieved, leading to a very limited number of securely dated samples because there is often not enough carbon left following pre-treatment to produce a radiocarbon age. The lack of insect material means that a choice must

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Table 1. Sources of potential contaminants when dating fossil insects and approaches / pretreatments applied to remove them after Tripp and Higham (2011) and Bayliss and Marshall (2022).

Contamination from the environment – before sample arrives in laboratory	Contamination during laboratory preparation and storage	Approaches / pretreatments required to remove contamination
Older carbon		
Inorganic carbonates from soils in arid zones		Acid wash with HCl
	Paraffin flotation	Repeated washing with volatile organic solvents
	Storage in petroleum-derived ethanol or industrial methylated spirits	Avoiding contact where practical. Complete evaporation of any liquid storage media before proceeding. Thorough washing with pure water and/or other reagents.
Younger carbon		
Fulvic acids		Acid wash with HCl
Humic acids		Base wash with NaOH
	Materials that physically adhere to the sample due to storage/handling procedures (e.g. wood, hair, paper)	Avoiding contact between the sample and material that may transfer contaminants. Physical cleaning and visual inspection.
	Materials that physically or chemically adhere to the sample via the storage medium (e.g. grain alcohol)	Avoiding contact where practical. Complete evaporation of any liquid storage media before proceeding. Thorough washing with pure water and/or other reagents.
	Storage materials (wood, hair, paper)	Avoiding contaminant use. Physical cleaning.
	Direct CO_2 capture from air during base step	Second acid wash with HCl after base step*

*Hatté et al. (2001) describes how modern atmospheric CO_2 can be incorporated into samples during the base stage of pre-treatment which cannot be removed by a subsequent acid wash; the materials most likely to be affected are wood, peat, and palaeosols.

often be made if the researcher wants to undertake both radiocarbon dating and other destructive techniques such as isotopic or DNA analyses (e.g. Hodgins et al. 2001; Mullin et al. 2023). In addition, the rigorous proof-of-concept studies available for other radiocarbon dated materials are consequently not available. Methods are therefore applied (by necessity) with less supporting evidence than for methodologies for most other radiocarbon dated materials. Additionally, the limited number of published studies cover a range of different settings, from glaciers to organic lake sediments and peat (Table 2), reducing the potential for direct comparison of contamination sources and thus making it hard to generalise.

This paper summarises previous research on radiocarbon dating insect samples from multiple contexts, presents new experimental data from both radiocarbon dating samples of known age with multiple pretreatments and testing the use of Fourier Transform Infrared Spectroscopy (FTIR), previously used on other archived samples, to detect contamination in insect samples. It also provides some recommendations for archaeological practitioners considering radiocarbon dating insect samples, both when planning sampling protocols for current research, and using samples that have been in long-term chemical storage.

Background – Contamination and Pretreatment Chemistry

Insect cuticles are composed of chitin, with additional scleroproteins which have caused the exoskeleton to harden or sclerotize (Elias 2010). The chitin itself is a polymer, comprised of *N*-acetyl-d-glucosamine units $(C_8H_{13}NO_5)_n$ (Elias 2010). It can be broken down chemically into its constituent parts or deacetylised using sodium hydroxide to the related compound chitosan. It is generally assumed that the protein elements of the insect skeleton are the most sensitive to contamination during post-depositional degradation or diagenesis (chemical transformation), thus many radiocarbon pretreatments for fossil insects have aimed to date only the non-protein parts of the structure to reduce the danger of contamination (Tripp and Higham 2011). However, the processes by which contaminants are physically and chemically incorporated into chitin are as yet poorly understood and likely to vary based on the chemistry of each contaminant and the timing when they are introduced (e.g. during deposition, extraction, storage and/or radiocarbon pretreatment). There is also currently no accepted test for contamination of samples, although Kessler et al. (2023) effectively used FTIR to detect successful removal of paraffin wax used for preservation in museum charcoal samples before radiocarbon dating.

As with all radiocarbon samples, care should be taken in the first instance to only date material in which the ^{14}C at the time of death was in equilibrium with the atmosphere. For insects, this means avoiding any potential freshwater reservoir effect by not selecting insects that feed on aquatic vegetation in life because the water in which they grew may contain dissolved older carbon which has an additional 'apparent' radiocarbon age in addition to the true age. Detritus feeders may also take up older carbon if vegetation

Table 2. Summary of results gained from radiocarbon dating insect samples of various ages using different pretreatments.

Study	Sample context and laboratory treatment / storage	Experiments / pretreatments / findings	Recommendations for future work
Elias et al. (1991)	Late glacial / early Holocene basal lake sediments. Not paraffin floated.	Pretreatments not reported. Insect ages c. 1000 years younger than wood and c. 4–5000 years younger than moss and believed to be the most reliable ages from the site.	None
Walker et al. (2001)	St Bees – late glacial kettlehole infill. Probably not paraffin floated.	Acid wash only. Insufficient insect material despite very large samples (1 kg). Most of the insect-derived dates were younger than stratigraphically associated material. Different beetle species gave different ages.	Avoid multi species aggregated samples because they give different ages. Concern over post-depositional diagenesis of chitin and introduction of contaminants.
Hodgins et al. (2001)	Late glacial age peats at St Bees, Cumbria and Redkirk Point, Dumfrieshire. Pooled into Carabids (terrestrial scavengers) and Dytiscids (aquatic carnviores). Not paraffin floated.	Aimed to isolate d-glucosamine: • Sequential solvent washes (methanol, methanol:chloroform 2:1, chloroform) • Freeze dry • Acid (1 N HCl, 3 h, room temperature) • Water wash, freeze dry • Base (1 N NaOH, 3–12 h, 100°C) • Acid (1 N HCl, 30 m) • Water wash, freeze dry Low recovery of insect sample mass (c. 5%). Insect ages still younger than seeds – incomplete purification.	Insects unsuitable for dating without new pretreatments able to purify without reducing yield
Tripp, Higham, and Hedges (2004)	Roman era sites in Cambridgeshire – Priors Gate in Eaton Socon and at Godmanchester. Samples cannot be matched with published reports (Robinson n.d.; Jones 2003) therefore paraffin flotation status unknown.	Method A (to remove carbonate): • Sequential solvent washes (acetone, methylene chloride, acetone) • Acid (0.5 M HCl, 3 days) • Water wash, freeze dry • Method B: (partial deacetylation to try and isolate chitin – solvent plus acid, base, acid – ABA): • Method A • Base (50% NaOH, 30 min, heated) • Acid (6 m HCl) • Freeze dried Method B results in the loss of c. 85% of mass	Both methods work well to give accurate ages for young (<3000 years) samples. Sample yield low.
Porch and Kershaw (2010)	Last glacial age peats and organic silts (all older than 27,000 years). Not paraffin floated.	Acid-base-acid pretreatment. Three insect ages: one beyond the detection limit (older than seeds and pollen ages), one infinite (older than seeds and pollen ages), one c. 28,000 years old, similar to seeds and c. 4000 years older than pollen age.	None – more research needed.
Tripp and Higham (2011)	Review paper	N/A	All pretreatments aiming for compound-specific isolation have very low sample yield. No suggested alternative.
Nakazawa et al. (2015)	Plant and insect fragments from a glacier.	No pretreatments used because collected directly from ice. Insect ages are younger than plant ages by up to 1000 years – attributed to plant reworking.	No clear recommendations.
Panagiotakopulu et al. (2015)	Various locations with different independent control all younger than 12,000 years • No chemicals • Paraffin flotation and storage in ethanol • Paraffin flotation and ethanol sorting but water storage Sorting in water without flotation frequently fails to yield enough sample to date.	Tripp, Higham, and Hedges (2004) Method A (solvent + acid) • Samples stored in water yielded accurate ages even with paraffin flotation (all <2000 years old) • Young samples (<2000 years old) with long-term storage in ethanol yield ages that are too old using method A, but with no consistent offset. • Late-glacial samples (c. 11,000 years old) with paraffin flotation and long term storage in ethanol were in agreement with other radiocarbon ages from the site, but these were too young compared with optically-stimulated luminescence ages.	Short term immersion in paraffin and alcohols during processing probably has no impact. Chitinous material for dating should be stored in acidified distilled water, particularly for young samples. Tripp, Higham, and Hedges (2004)'s Method B is too destructive to use routinely.

(*Continued*)

Table 2. Continued.

Study	Sample context and laboratory treatment / storage	Experiments / pretreatments / findings	Recommendations for future work
Holden and Southon (2016)	Insects from within various skulls at La Brea Tar Pits. Some contaminated with asphalt, others not.	Solvent + ABA: • Sequential solvent washes (chemicals not stated, but ending with 2:1 toluene/methanol) • 1N HCl, 1N NaOH, 75°C, repeated as necessary every 30 m until solutions clear • 1N HCl • Water wash, vacuum dry All samples were >30,000 years old and gave ages consistent with bone collagen ages. Yields ranged from 30 to 77% – surprisingly high; possibly because prolonged exposure to the tar pits biogeochemical environment had already removed some of the cuticle protein, leaving a chitin-rich residue.	No general suggestions because highly specific depositional setting.

has persisted in the landscape for 10s or 100s of years, but this is likely to create a significant offset only for the youngest of samples. In addition, care should be taken that only the insects themselves are submitted for dating, not additional unrelated material. These constraints will further reduce the sample size of insect material available for dating.

The successful radiocarbon dating of fossil insect samples is also dependent on the identification and removal of any potential carbon-containing contaminants which may be of a different age to the insects. The two extremes of contamination are 'dead' (containing no ^{14}C) or 'modern' carbon, making radiocarbon ages older and younger respectively (Figure 1). However, contamination can also be of any ^{14}C value on the continuum between these two extremes, either because it is a mixture of carbon with multiple different 'ages', or because it is carbon of a single intermediate 'age'. It can be seen in Figure 1 that the scale and significance of any error introduced is dependent not only on the percentage contamination but on the original radiocarbon age of the sample, e.g. a 1000-year offset will be more significant for a sample that is 10,000 years old than one which is 25,000 years old.

Contaminants can enter the sample either during burial in the environment or during excavation/collection, laboratory processing and storage (Table 1). Sources of contamination from the depositional environment include inorganic carbonates from soils in arid zones and organic carbon from the

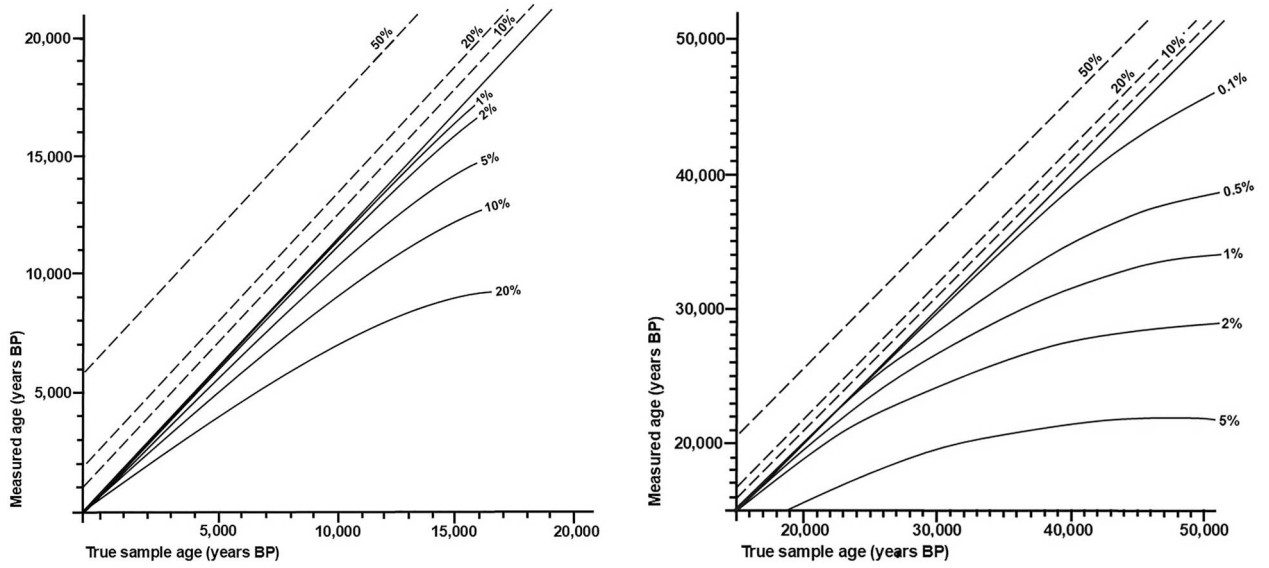


Figure 1. Theoretical relationships between true and measured radiocarbon ages for samples contaminated with 'modern' and 'dead' carbon, for the a. 0–16,000 year range and b. the 12–50,000 year range. Redrawn from Gillespie (1984). The thick solid line shows the relationship between true sample age and measured sample age for uncontaminated samples. The thin solid lines show the effect of contamination by 'modern' carbon (130% of the modern reference standard, about the atmospheric level in 1984). The dotted lines show the effect of contamination by 'dead' carbon (containing no ^{14}C).

surrounding sediments, e.g. organic acids such as humic and fulvic acids. Less commonly, carbon may be introduced from other organisms such as microbes, macrofauna, plants (roots etc), fungi, or unusual materials such as tar (Holden and Southon 2016). Whilst there are standard approaches to the removal of a number of contaminants that can also be applied to insect remains (Table 1), the strength and length of acid and base steps varies significantly and even the strongest acid-base-acid washes are not always sufficient to remove all contamination in older samples where ^{14}C concentrations are naturally very low (Briant et al. 2019). There are also concerns that there may be unintended reactions during processing and storage (Hatté et al. 2001; Panagiotakopulu et al. 2015).

For insect remains particularly, there is less consensus about the impact of laboratory chemicals such as paraffin and ethanol used to extract the insects from bulk sediment samples and in their long-term storage. Rigorous solvent washes have been shown to effectively remove paraffin in the case of samples in which it is abundant due either to use in conservation of artefacts (Bruhn et al. 2001) or long-term preservation of seeds (e.g. Zazula et al. 2009) or charcoal (e.g. Kessler et al. 2023). There is less consensus about whether a solvent step is needed when paraffin has been used for the standard flotation required to time-efficiently separate insects from bulk samples (Rousseau 2011). It is important to note in this context that the number of pretreatment steps for dating ideally needs to be limited only to those which are necessary both because sample mass is lost each time it is handled and insect samples are usually already small, and to limit the number of steps which could introduce contaminating carbon.

The pretreatments listed in Table 1 have been used in various combinations in studies aiming to date insect samples. The aims of each study were different, with some aiming to isolate a specific chemical fraction and others simply removing contamination (Table 2), but all aimed to determine the minimum number of necessary pretreatment steps. The depositional settings were also different. It should also be noted that there has been a standardisation of general laboratory preparation procedures since the first papers were published in the late 1990s. However, all the studies cited in Table 2 found that a rigorous pretreatment using a solvent wash and acid-base-acid (ABA) steps experienced extremely low recovery of insect sample mass when applied to insect remains (e.g. 5–15% – Hodgins et al. 2001; Tripp, Higham, and Hedges 2004), due to destruction of much of the sample. Where a solvent wash followed by acid only (Tripp et al.'s 2004 Method A) was used, this successfully yielded dates in line with independent age control for younger samples (<3000 years old) as long as they had not experienced long term storage in ethanol. This is in concordance with the theoretical understanding from Figure 1 that younger samples are most affected by older carbon contamination from substances such as paraffin and ethanol and thus more likely to need a solvent wash and less likely to need the base step that removes the younger carbon contamination of humic acids.

For older samples, results are mixed, but interestingly, the issue when ages do not accord with expectation does not ever seem to be that the samples are 'too old', suggesting that the presence of residual paraffin and petroleum-derived ethanol contamination are less problematic, again as expected from Figure 1. Hodgins et al. (2001) applied a solvent wash and ABA to insects from Late-glacial age peats in Cumbria, UK which had not been paraffin floated or stored in ethanol. Insect ages were younger than those from associated seeds, suggesting that younger carbon contamination had not been completely removed despite the use of a base step. In contrast, Panagiotakopulu et al. (2015) found that a solvent wash with acid was sufficient to yield an age concordant with dates on other organic materials from Late-glacial age samples where paraffin and ethanol were used, although they did note that all radiocarbon ages at this site were younger than the associated optically-stimulated (OSL) ages (Bateman 1995), so it is possible that the base wash was insufficient to remove younger carbon. For the two studies on older samples, Porch and Kershaw (2010), using ABA pretreatment only reported an age of c. 28,000 ^{14}C BP which was in line with seed ages (older samples did not yield finite ages). Holden and Southon (2016) found radiocarbon ages of c. 29–44,000 ^{14}C BP in accordance with bone collagen using a solvent and ABA pretreatment but the depositional setting in a tar pit is not representative of most environmental archaeology samples. In addition, the rigorous solvent extraction required to efficiently remove the asphalt from the samples would have likely also removed any residual kerosene used to extract the insect remains from the tar.

A further concern in radiocarbon sample pretreatment is the absorption of carbon from the atmosphere into the sample during laboratory pretreatment. Each additional step in sample processing in the laboratory risks exposing the sample to contamination and there is a trade-off to be made between removal of previous contaminants and potential introduction of new ones at each stage. For example, a base wash should only be used where absolutely necessary because Hatté et al (2001) raised the absorption of CO_2 during the base wash as a concern in relation to particular sample types (wood, peat and palaeosols, which contain sulphur, nitrogen or other functional groups that can be rearranged, permitting modern carbon introduction during the base wash). However, Dee et al.

(2011) showed experimentally using a silica substrate rather than environmental samples that whilst significant amounts of carbon contamination built up during the base wash they were removed by the following acid wash. It is possible that chitin's ability to act as a 'CO_2 scrub' (e.g. Eftaiha et al. 2016; Foong et al. 2023) may lead to the incorporation of younger carbon. Whilst there are no detailed studies of this phenomenon in the context of radiocarbon dating of insects a similar pattern of the sufficiency of an acid wash to remove atmospheric CO_2 seems likely.

In this paper we address some of the ongoing uncertainties in the best methods for processing and pretreatment of insect radiocarbon samples by reporting additional data from:

- Insect radiocarbon dating experiments on archived older samples. The aim of these experiments was to assess whether modern contamination could be removed from archived insect samples using methods that are currently standard in radiocarbon laboratories (Table 1). It was decided to keep the number of pretreatment steps to a minimum, to reduce both the risk of introduction of contaminants and unnecessary sample loss. A solvent wash was not included because these samples did not show evidence of contaminants that would require this (e.g. waxes or oils added for preservation). We tested only acid and base pretreatments because the potential contaminants fit a set of 'normal' categories.
- Fourier Transform Infrared Spectroscopy (FTIR) of modern samples subjected to wet sieving or paraffin flotation and storage in various media, including ethanol and tap water for up to six months, after Kessler et al. (2023). The aim of our FTIR experiments was to assess whether this technique could corroborate the assertion of Panagiotakopulu et al. (2015) that processing of samples using liquid paraffin would not generate contamination unless samples were then stored in ethanol for an extended period. The modern samples were chosen from locations which had been sampled previously by one of the authors (Smith et al. 2010). They were known to be dredged regularly so that bottom samples were only c. 10–15 years old. It was also known that a sufficient volume of insect material would be retrieved.

Materials and Methods

Archived Radiocarbon Samples

Two sites were chosen for testing different pretreatments on archived insect samples – one of Late-glacial age and one suggested by independent age control to be beyond the limit of radiocarbon dating. Both yielded sufficient insect material to undertake multiple radiocarbon dates. No other available archived samples were large enough to do this. All insect samples available were originally collected by Russell Coope and archived either at Royal Holloway University of London or the Lapworth Museum in Birmingham.

The Late-glacial sequence from Norton Farm pit at Condover in Shropshire, UK, came from a kettle hole fill above the sand and gravel deposits of the Late Devensian glacial Stockport Formation (Scourse et al. 2009), with mammoth remains likely from Unit C1 at the base of the kettle hole fill. A sample named Condover 6 was retrieved from storage at Royal Holloway University of London. This comprised dung beetle remains (*Aphodius sphlatus / prodromus*) from the skull (lower jaw and sinuses) of a juvenile mammoth. The sample was paraffin floated and then stored in industrial methylated spirits (IMS) from 1986 to 2019, followed by ethanol from 2019 until radiocarbon dating between 2020 and 2021. Dung beetles from the sinuses of this juvenile mammoth were radiocarbon dated (OxA-1591) in 1987 to 11,880 ± 130 ^{14}C BP (14,009 to 13,434 cal BP) following pretreatment with dilute alkali (0.1M NaOH) and acid (0.5M HCl), both at room temperature. The juvenile mammoth cranium was radiocarbon dated using collagen extraction with ultrafiltration in 2009 (OxA-19903) to 12,375 ± 50 ^{14}C BP (14,747 to 14,101 cal BP).

Asample from Isleworth, Middlesex, UK, came from a single organic silty clay bed directly overlying the London Clay and underlying a fluvial gravel sequence (Kempton Park Gravel (Coope and Angus 1975; Bridgland 1994). The organic silty clay contained fossils of mammoth, woolly rhinoceros, reindeer and bison. The insect sample is remarkable because it provides evidence for a temperate yet treeless episode during the last glacial period (Coope and Angus 1975) with reconstructed maximum temperatures of between 16°C and 18°C and minimum temperatures between 0°C and −5°C (Coope 2000). A sample named Isleworth 4 was retrieved from storage at the Lapworth Museum, University of Birmingham. This comprised unidentified beetle remains aggregated in order to provide enough material for dating. The sample was originally extracted from a sediment sample by paraffin flotation in 1956 and then stored in industrial methylated spirits (IMS) from 1956 to 2019, followed by ethanol from 2019 until radiocarbon dating in 2022. Initial radiocarbon dating of sorted plant material from the same organic lens pretreated in 5% HCl followed by 2% NaOH gave an age of 43,140 ± 1400 ^{14}C BP (Birm-319; Shotton and Williams 1973). However, later amino-acid dating of *Bithynia tentaculata* opercula suggested an age beyond the radiocarbon dating limit, possibly in the later parts of MIS 5 or 4 (c. 70–100;000 years BP – Penkman et al.

2011). The sample was chosen to represent an insect sample from beyond the radiocarbon limit, because none of the insects from last interglacial sequences archived at either Royal Holloway or Birmingham contained sufficient material.

Radiocarbon Pretreatments

Condover 6 was larger than Isleworth 4 and was split into four aliquots, whilst Isleworth 4 was split into three. The three comparable measurements comprised one subsample measured without chemical pretreatment (raw, Table 2) and two measured after different standard chemical pretreatments (acid-only and acid-base-acid [ABA], Table 2) (Ascough et al. 2024). The 'acid-only' samples were digested in 0.5M HCl (80°C, 2 h), then washed free from mineral acid with deionised water, dried and homogenised. The mild ABA samples were digested in 1M HCl (80°C, 30 min), washed free from mineral acid with deionised water then digested in 0.2M KOH (80°C, 5 min). The residue was rinsed free of alkali, digested in 1M HCl (80°C, 1 h) then rinsed free of acid, dried and homogenised. A strong ABA pretreatment (1M strength at each stage) was also attempted on the fourth subsample of Condover 6, but mass loss was too great to proceed to radiocarbon dating for this sample. For all samples, the total carbon in a known weight of the pre-treated sample was recovered as CO_2 by heating with CuO in a sealed quartz tube. The gas was converted to graphite by Fe/Zn reduction.

The result is reported as conventional radiocarbon years BP (relative to AD 1950) and % modern ^{14}C, both expressed at the ± 1 σ level for overall analytical confidence. $^{13}C/^{12}C$ ratio was measured by AMS during ^{14}C determination and used to correct for isotopic fractionation according to the conventions of Stuiver and Polach (1977) i.e. to normalise ^{14}C data to $\delta^{13}C\text{-VPDB‰} = -25$.

Where the laboratory code has the prefix SUERC the sample was prepared to graphite at the NEIF Radiocarbon (Environment) laboratory in East Kilbride, Scotland and passed to the Scottish Universities Environmental Research Centre AMS laboratory for ^{14}C analysis. Where the laboratory code has the prefix UCIAMS, the sample was very small in size. It was therefore prepared to graphite at the NEIF Radiocarbon (Environment) laboratory and passed to the Keck Carbon Cycle AMS Facility, University of California, Irvine, USA for ^{14}C at low current, requiring the expertise of Dr Xiaomei Xu. Preparation of additional standards was required to match the sample carbon weight and the data were corrected for modern carbon contamination after Santos et al. (2007). For samples <100 μgC an additional correction for old carbon was made based on small NIST Oxalic acid II. The uncertainty reported takes into account the overall analytical uncertainty, including the corrections applied and observed scatter in process standards of comparable size to the sample analysed.

Modern Insect and Plant Remains and Chitin Powder

Samples were taken from locations used in a previous study of modern insect and plant remains (Smith et al. 2010) in July 2022 at Bears Rails Pond, Windsor Great Park, Berkshire, UK (SU 97382 73665; WGS84 51.453603 latitude, 0.599881 longitude) and Smithy Pool, Dunham Massey, Cheshire, UK (SJ 73806 87390; WGS84 53.382703 latitude, 2.395256 longitude). Bucket sampling of c. 10 litres of sediment captured approximately the top 10 cm, representing 1–2 decades of accumulation because the ponds are regularly dredged.

Samples were split in two, with one half wet sieved without paraffin flotation, using mesh sizes of 1 and 0.5 mm. The other half were processed using the standard method of paraffin flotation for archaeoentomological samples, as outlined by Kenward, Hall, and Jones (1980). The chemicals involved in this process are sodium carbonate (to disaggregate samples), paraffin/kerosene (for flotation), Fairy Liquid (a widely available concentrated liquid detergent, for cleaning) and finally industrial methylated spirits (IMS). Following processing, insects and plant remains were picked out of the samples and stored separately using a range of representative storage media:

- Tap water
- Ethanol (99.8%)
- Industrial methylated spirits (IMS)
- 'Kew Mix' (53% IMS (98/99% total alcohols), 37% water, 5% formaldehyde solution (38% w/w) and 5% glycerol – Natural Science Collections Association 2019).

50 mg aliquots of commercially available chitin from shrimp powder (Sigma Aldrich, UK) were placed in identical vials to those used to store the insect remains, and filled with each of the four storage media, which allowed analysis of a 'known' material after six months storage. All samples were stored at room temperature.

Fourier Transform Infrared (FTIR) Spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR) provides qualitative information regarding the nature of chemical bonds in a sample. Analysis of chitin powder and insect and plant remains was undertaken using a Bruker Alpha mid-IR with a 2 mm × 2 mm square diamond crystal and Platinum ATR (Attenuated Total

Table 3. Summary of radiocarbon results on archived samples. Both were paraffin floated during processing and stored in industrial methylated spirits since 1956 (Isleworth, Coope and Angus 1975) and 1986 (Condover, Scourse et al. 2009).

Publication code	Sample identifier	%Modern	%Modern 1 σ error	Radiocarbon age (years BP)	Radiocarbon age 1 σ uncertainty	%Carbon
Condover 6						
Scourse et al. (2009)	C6- Reference age (bone)		12,375	50		
UCIAMS-236602	C6- Raw	24.87	0.10	11,180	35	64.47
SUERC-100381	C6- Acid only	24.11	0.14	11,429	47	41.58
UCIAMS-253936	C6- ABA mild	22.86	0.10	11,855	35	15.33
Isleworth 4 – note that the actual age of this sample is believed to be beyond the radiocarbon limit at c. 70-100,000 years BP						
Coope and Angus (1975)	I4- Initial radiocarbon age		43,140	1400		
SUERC-104941	I4- Raw	22.56	0.13	11,962	46	33.38
SUERC-104943	I4- Acid only	17.04	0.12	14,218	55	40.04
SUERC-104942	I4- ABA mild	6.18	0.10	22,364	133	44.54

The 'acid-only' samples were digested in 0.5M HCl (80°C, 2 h), then washed free from mineral acid with deionised water, dried and homogenised. The mild acid-base-acid (ABA) samples were digested in 1M HCl (80°C, 30 min), washed free from mineral acid with deionised water then digested in 0.2M KOH (80°C, 5 min). The residue was rinsed free of alkali, digested in 1M HCl (80°C, 1 h) then rinsed free of acid, dried and homogenised.

Reflectance) unit. Each sample was scanned 32 times in absorbance mode between 4000 and 400 cm^{-1} wavenumbers, with 4 cm^{-1} resolution. Background scans were collected before collecting any data, and regularly throughout data collection, and were subtracted using Opus™ software v 7.2 (Bruker Optik GmbH). Individual peak positions were identified with the same software, and the chemical bonds represented by these peaks were identified through comparison with spectra of the storage and processing chemicals in the National Institute for Science & Technology (NIST) Chemistry WebBook (https://webbook.nist.gov/). Characteristic peaks for chitin were identified based on Seoudi et al. (2005) and references within. Data were plotted for publication using Excel.

To enable identification of key discriminatory peaks for each of the processing and storage materials (paraffin, ethanol, IMS, Kew solution, and local tap water), a fresh aliquot of commercially-available chitin from shrimp shells was placed on the crystal and a spectrum collected immediately after a single drop of one of the chemicals of interest was added, before it could evaporate. In this manner, the chitin acted as an absorbent substrate to allow reference spectra of the processing and storage chemicals to be collected, while also allowing the contribution of the chitin to the individual chemical spectra to be compared with spectra of archaeological insect remains.

To determine how quickly the storage media evaporated and was no longer visible on FTIR spectra, an aliquot of chitin stored in each of the storage media (ethanol, IMS, Kew solution and water) for six months was analysed after both five minutes and one hour of air drying on absorbent paper tissue. None of the organic storage media were observed in the chitin spectra after five minutes of air drying, thus all insect samples were analysed after five minutes to allow for evaporation to have occurred.

FTIR spectra were collected for insect remains extracted by paraffin flotation and sieving in water from both Windsor Great Park and Dunham Massey and stored in tap water for c. one month and after storage for a further five months. Insect samples from both sites, processed by both methods, and stored separately in ethanol, IMS and Kew solution were also analysed c. six months after processing.

Results

Radiocarbon Dating

Table 3 and Figure 2 both show that neither of the standard pretreatments applied reproduce the radiocarbon ages used as reference for the archived samples, even within the range of 2σ error bars. The offset in all cases is clearly younger than the age control, meaning that the contaminant carbon is of a younger age than the sample itself. These results show that the insect material contains contamination that was not removed using the acid only or mild acid-base-acid pretreatments after Ascough et al.

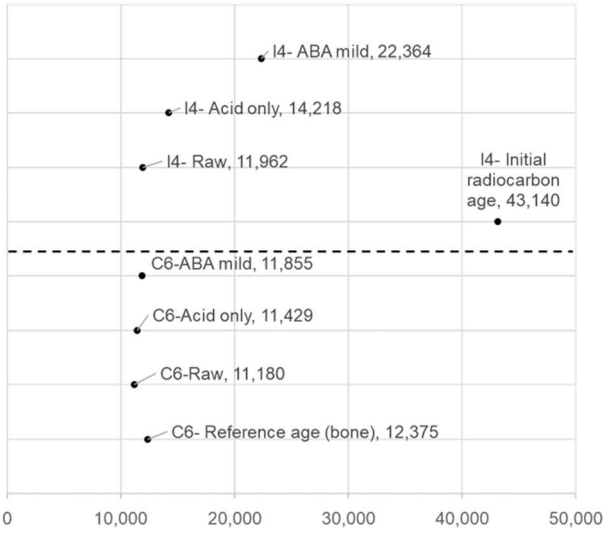


Figure 2. Measured radiocarbon ages for samples paraffin floated and then stored in industrial methylated spirits since 1956 (Isleworth, Coope and Angus 1975) and 1986 (Condover, Scourse et al. 2009). C6 is Condover 6; I4 is Isleworth 4. Note that the actual age of sample I4 is likely beyond the radiocarbon limit at c. 70–100,000 years BP. Sample details in text.

(2024). Since even these pretreatments yielded some samples that were too small for measurement at SUERC, it is not practicable to apply stronger pretreatments. This is frequently the case with insect samples, as shown in the previous studies summarised in Table 2.

It is not possible from these results to determine whether this contamination occurred in the environment / during initial laboratory preparation or during archive storage. For the Condover 6 sample, the 1987 insect radiocarbon age from the same sample matches the ages reported here, suggesting that this contamination occurred during burial and/or subsequent time in the depositional environment and was not successfully removed during the dating from 1987 either. For the Isleworth 4 sample, the initial radiocarbon age was not on insects and is also believed to be unreliably young, so no conclusions can be drawn as to the exact source, age, or nature of the carbon contamination.

FTIR Spectroscopy

Reference Spectra

FTIR spectroscopy identifies functional groups based on vibrations/stretching of molecular bonds, hence it's potential use to detect chemical contamination of radiocarbon samples. Figure 3 shows reference spectra of each of the chemicals used in the processing and storage of insect remains collected using chitin as a substrate. It should be noted that these chemicals and the archaeological insect remains are all hydrocarbons and therefore their spectra contain many of the same common peaks, including C–H bonds (c. 3000–2800 and 1450–1300 cm^{-1}), C-O stretching (~ 1376, 1082, 1046 cm^{-1}), and a broad O-H stretching peak in the 3200–3400 cm^{-1} region (region A on Figure 3). The spectra for chitin include large, often broad, NH peaks at ca. 3270 and 3100 cm^{-1} (Seoudi et al. 2005), which can overlap with the broad O-H peak. Additional smaller NH (amide) peaks appear at ca. 710 and 690 cm^{-1} (Seoudi et al. 2005). To detect the presence of contaminating chemicals within the archaeological specimens, key peaks were identified from the reference spectra produced in this study for comparison with sample spectra.

The key spectral signature for paraffin, as also noted by Kessler et al. (2023), is the presence of three peaks in the 2950–2850 cm^{-1} region (the highest being the middle of the three at ~ 2922 cm^{-1}) with distinctly different peak heights to those observed in the same spectral region for ethanol, IMS and Kew solution (region B on Figure 3). FTIR spectra of (modern) insect remains also typically include 3 distinct peaks in this region, attributed to alkyl chain bonds from epicuticular lipids, protein and chitin (Johnson and Naiker 2020 and references therein). However, since the insects in this study were sampled from pond sediments where they had been present for up to 10–15 years direct comparison with spectra of modern insects was done with caution. Spectra were only considered to indicate the presence of paraffin where the relative proportions of the three peaks in this region were in agreement with the reference chitin and paraffin spectrum and in contrast to those presented by Johnson and Naiker (2020).

The spectra for ethanol and IMS appear identical (Figure 3), suggesting that the additives in IMS to

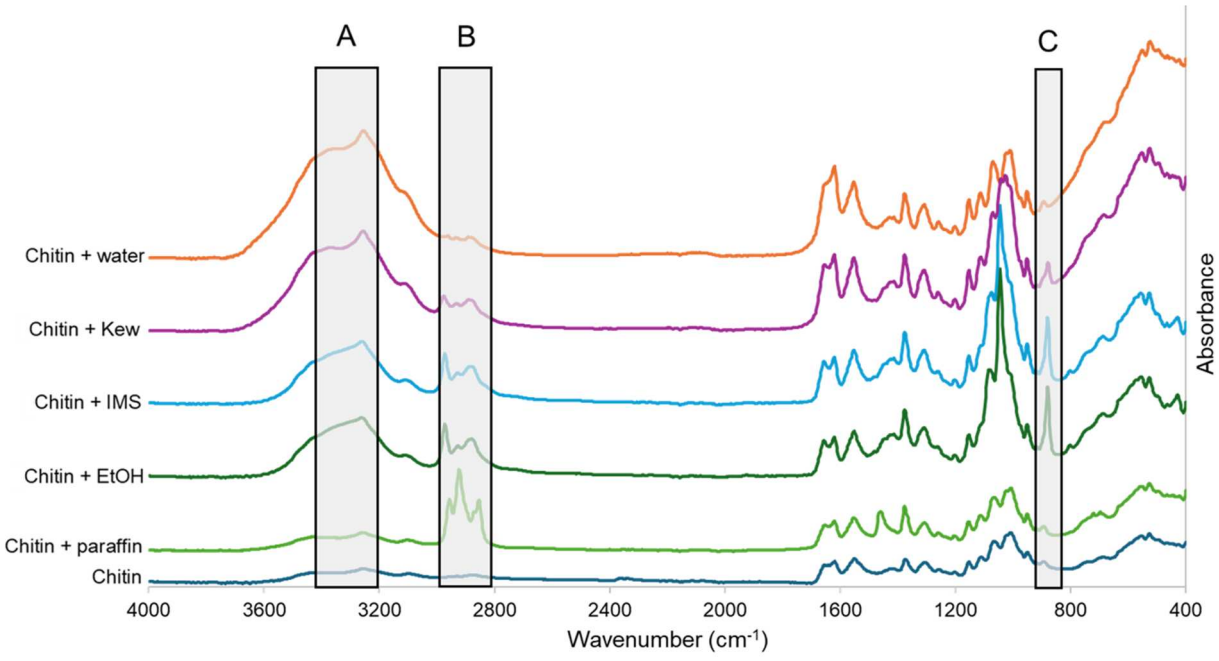


Figure 3. FTIR spectra (staggered for ease of viewing) of paraffin and other storage media on commercially-available chitin from shrimp. Region A (3400–3200 cm^{-1}) is dominated by a broad OH peak; Region B highlights key peaks associated with organic storage media or paraffin between 2950–2850 cm^{-1}; Region C highlights key peak at c. 880 cm^{-1}.

discourage human recreational consumption were either of similar organic molecular composition to the ethanol and chitin and/or below the detection limit of FTIR. Peaks chosen to identify the potential presence of ethanol or IMS in spectra of insect remains are shown in Figure 3 (sharp peak at 2970 cm^{-1} and two smaller C–H peaks at 2927 and 2880 cm^{-1} highlighted in region B; sharp peak at 880 cm^{-1} highlighted in region C).

The spectrum for Kew mix is similar to that of ethanol, but the presence of both formaldehyde (5%) and glycerol (5%) reduce the relative peak heights between 3000 and 2800 cm^{-1} (Figure 3 region B) and at 880 cm^{-1} (Figure 3 region C). Additional glycerol peaks are present at c. 1026 cm^{-1} and c. 1153 cm^{-1}. The addition of water to chitin adds a broad O-H stretching band at c. 3400–3200 cm^{-1} (Figure 3, region A).

Paraffin Detection Using FTIR

Figure 4 shows a representative selection of FTIR spectra obtained from the modern samples from Dunham Massey (DM) and Windsor Great Park (WGP). The spectrum at the bottom represents a reference insect spectrum, as it was not in contact with either paraffin or any storage media other than water. This reference spectrum is similar to that from the commercially-available chitin and water (Figure 3), with many peaks lacking definition and a significant broad OH peak in the c. 3400–3200 cm^{-1} range (region A). Other samples that had only been in contact with water showed a similar lack of definition in the spectra, but also significant natural chemical heterogeneity of specimens, likely reflecting differences in preservation/degradation state and/or species (see Johnson and Naiker 2020 and references therein). Note that the OH peak is not impacted by the NH stretching peak at ca. 3270 cm^{-1} observed on all the chitin reference spectra in Figure 3.

Of the spectra for the insect remains that had been extracted by paraffin flotation and stored in water for one month, one sample (out of three) from each site clearly showed three peaks in the 2950–2850 cm^{-1} region (region B, samples shown on Figure 4) similar to those in the reference spectra of paraffin on chitin (Figure 3).

None of the FTIR spectra for paraffin-floated insect samples from Windsor Great Park stored in ethanol, IMS, water or Kew solution for six months gave clear indication of the presence of paraffin (two example spectra shown on Figure 4). In comparison, the spectra of at least four of the 15 insect samples from Dunham Massey (all stored for 6 months) clearly indicated the presence of paraffin (not shown on Figure 4). Paraffin presence occurred in combination with various storage media, with no clear relationships. A further six Dunham Massey samples were inconclusive (as in the spectrum shown on Figure 4). It is not clear why the presence of residual paraffin should be different between sites, and most likely reflects heterogeneity between insect samples since, although paraffin can be chemically variable, in this study the brand of paraffin used was the same in all cases.

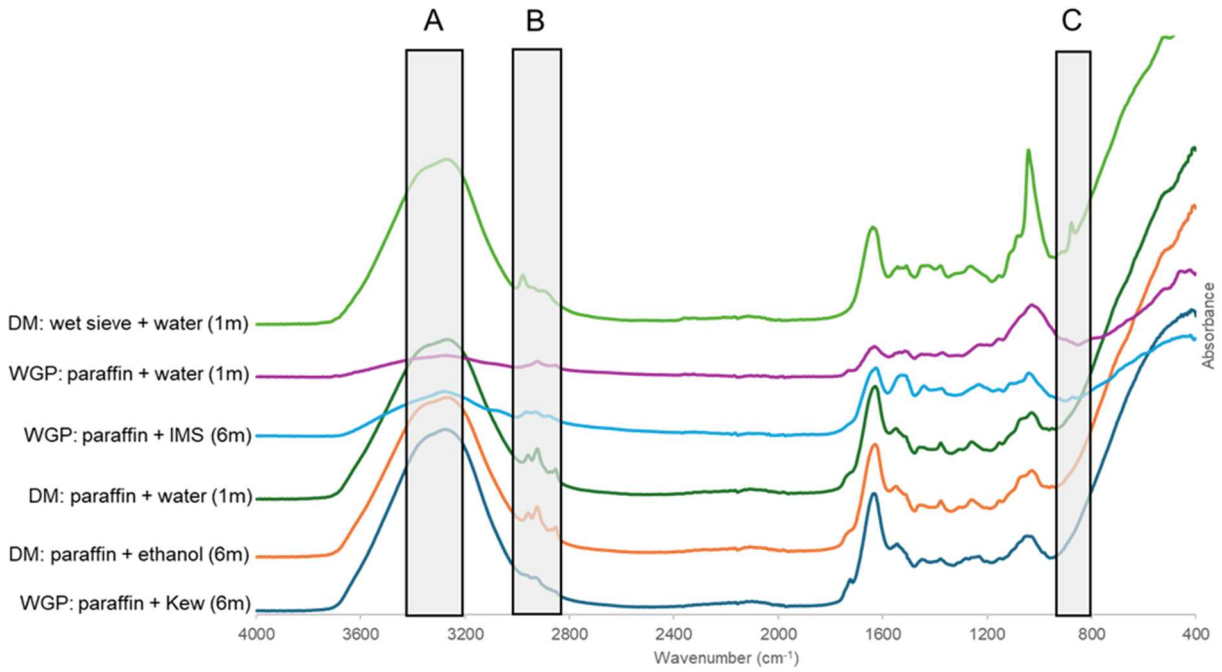


Figure 4. Representative FTIR spectra of modern insect samples from Dunham Massey (DM) and Windsor Great Park (WGP) exposed to paraffin and various storage media for zero or six months. Region A (3400–3200 cm^{-1}) is dominated by a broad OH peak; Region B highlights key peaks associated with organic storage media or paraffin between 2950–2850 cm^{-1}; Region C highlights a key peak at c. 880 cm^{-1}.

Storage Media Detection Using FTIR

Confirmation of the presence of organic storage media in any of the insect remains after six months was challenging, as many of the discriminatory peaks are also observed in the spectra of the remains themselves because the compounds contain many of the same chemical bonds and functional groups. In some cases, the key peaks are in the same region as the paraffin peaks and require interpretation of relative height differences. Contaminants present in low concentrations may also be present below the limit of detection, or 'hidden' by larger, more dominant peaks. Whilst initial chitin analyses showed that both ethanol and IMS evaporated within five minutes of exposure to air, IMS was potentially present in the spectrum of one of three insect remains from Windsor Great Park after six months of storage. Similarly, ethanol appeared present in one sample of insect remains from Dunham Massey. The presence of Kew solution was harder to identify with confidence than ethanol or IMS but may have been present in all three insect remains from Dunham Massey and one from Windsor Great Park (see peak in Region C, top spectrum on Figure 4).

A Note on FTIR on Plant Remains

The spectra of the plant remains sieved and stored in water from both sites showed more variation in peak size and location than the insect remains, likely due to variation in species and element (e.g. seed, nut shell) as well as degradation state. The significant variation between spectra then made any contamination with paraffin, ethanol, IMS or Kew mix in the paraffin-floated samples difficult to detect because degraded plant material consists of many of the same basic chemical functional groups, and hence both sample and storage media produce spectra with many shared peaks. Tentative interpretation of these spectra suggested that for those which had been paraffin floated and stored in water for one month, none of the spectra indicated the presence of residual paraffin. For those paraffin floated and stored in various storage media for six months, none of the spectra of seeds from Windsor Great Park indicated the presence of residual paraffin, irrespective of storage medium. In contrast, at least five of the eleven samples from Dunham Massey analysed contained paraffin, with the spectra of four other samples inconclusive.

Discussion

Removal of Extraneous Carbon Contamination from Insect Samples

Examination of the literature on previous radiocarbon dating of insect samples suggested that carbon contamination could be successfully removed using standard acid and acid-base-acid washes for younger samples, but that samples from the Late-glacial (c. 11,000 ^{14}C BP) and older were often younger than associated plant remains, particularly for the Late-glacial samples. The results reported in this paper confirm these findings for older samples, reflecting the statistically increased effect of contamination with younger carbon on samples of this age (Figure 1; Grant and Marshall 2025) and the difficulty of removing all contamination from insect remains when they are so small and fragile. These results suggest that standard acid-only or ABA pretreatments seem to be insufficient to completely remove contaminating carbon from insect remains in these samples. However, more robust treatments are not possible because of excessive sample loss. Whilst exact yields could not be calculated from the measurements taken, we observed that any treatment led to mass losses from the material. These were greatest during the alkali step of ABA processing, to the extent that even if a relatively large sample was used (50–100 mg of dry insects), a 1M NaOH treatment led to almost complete loss of the sample within 1 h. It is not clear how (or even if) a balance can be found for the effective removal of humic acid contaminants with sodium hydroxide, as is standard in radiocarbon dating of other archaeological materials, without resulting in (complete) deacetylisation – and hence loss – of the chitin. We therefore advise that if other organic material is available, this should be radiocarbon dated in preference, even in cases where the ability to directly date insects would be useful (e.g. in the last glacial period, where only insect fossils can pick up the peak warmth of key interstadial periods). If there is no other suitable organic material, the pretreatment strategy for insect remains needs to consider the problem of likely significant sample loss before proceeding and particular care should be taken when radiocarbon dating samples suspected to be older than c. 10,000 ^{14}C BP.

Detection of Carbon Contamination Using FTIR

In this paper, FTIR analysis demonstrated the natural variation in composition of both archaeological insect and plant remains, reflecting both the difference in composition of the original material (e.g. different species, insect wings versus exoskeleton or different parts of the exoskeleton, and seeds versus nut shells) and likely differing states of preservation. Beyond this, the results were equivocal, suggesting that the technique is less useful for detecting the small quantities of liquid paraffin used here than for detecting large quantities of paraffin wax as reported by Kessler et al. (2023). This difference in clarity of results also reflects the differences in chemistry between chitin and the charcoal used in the Kessler study. In this paper, despite the

challenges of conclusively identifying the presence of paraffin in the samples using FTIR, these results suggest that it might be prudent to assume the presence of paraffin when radiocarbon dating insect samples that have been extracted from sediment by paraffin flotation. This potentially older carbon is particularly important for archaeological samples, which are often younger than 10,000 ^{14}C BP and thus more susceptible to contamination (Figure 1). If such samples are to be dated, a series of solvent washes as detailed in Table 2 (e.g. Tripp, Higham, and Hedges 2004's Method A) should be used.

The presence of organic storage media (ethanol, IMS and Kew solution) was also difficult to confirm using FTIR. Given that many of these substances will evaporate if allowed to air-dry (as observed with the commercial chitin samples dried for 1 h) and are soluble in water and likely to be removed during standard radiocarbon pretreatment (Table 1), this is less problematic for short term storage. However, the long-term effects of storage in these media on the chemical composition of partially degraded insect and plant remains is not known, a concern raised by Panagiotakopulu et al. (2015) who suggest that ages they report from Roman and Norse sites are too old due to ethanol storage. They note studies which showed enrichments or depletions of δ^{13}C after long term storage in ethanol of turtle tissues (Barrow, Bjorndal, and Reich 2008) and fish tissues (Kaehler and Pakhomov 2001), although no effect on deep sea corals (Strzepek et al. 2014). There was no systematic study at that time of such an effect. The FTIR data reported in this paper is unable to shed any further light on this issue.

Conclusions / Recommendations for Radiocarbon Dating Insects in Environmental Archaeology

(1) Significant care must be taken if attempting to radiocarbon date insect remains suspected to be older than c. 10,000 ^{14}C BP because the standard acid-only and ABA pretreatments do not seem to reliably remove contamination in all cases and sample loss is high. These problems are particularly pronounced because insect samples are smaller, harder to handle and more likely to lose material during pretreatment than more robust material such as wood or bone.

(2) FTIR cannot be reliably used for pre-screening or quality assurance of pretreatment protocols on insect samples. These findings show that whilst it can potentially show the presence of paraffin, it cannot categorically confirm the absence of paraffin or ethanol because insect remains have very similar spectral signatures to both paraffin and ethanol and partial degradation of these samples increases the variability of the signal enough to further obscure any such evidence. The small amount of material available also limits the potential for FTIR analysis.

(3) We suggest that residual paraffin is likely to be present in many insect remains, and also some plant remains, that have been paraffin-floated. Thus, radiocarbon dating of younger insect remains which have been paraffin-floated should be avoided, given their greater susceptibility to such contamination (Figure 1), unless there is sufficient material to undertake a full solvent wash as outlined in Tripp, Higham, and Hedges (2004).

(4) Short term storage of insects (and likely also plant remains) in ethanol may not affect radiocarbon ages because it evaporates quickly, but there is some evidence, which has yet to be fully investigated, that longer term storage may change the chemical structure of insects (Panagiotakopulu et al. 2015). There is no clear evidence from the literature what length of storage counts as long term, nor how ethanol storage may affect plant material, which is often radiocarbon dated in preference to insects.

Acknowledgments

This research was funded by Birkbeck, University of London and the Quaternary Research Association. We are also grateful to Danielle Schreve for access to archived samples at Royal Holloway University of London; Jon Clatworthy for access to archived samples at the Lapworth Museum University of Birmingham; the Crown Estate for access to and support during fieldwork at Windsor Great Park and the National Trust for access to and support during fieldwork at Dunham Massey. FTIR analysis was undertaken in the Centre for Defence Chemistry, Cranfield University, Defence Academy of the United Kingdom. Paul Rostron (Cranfield University) is thanked for helpful discussions on sample chemistry.

Disclosure Statement

No potential conflict of interest was reported by the author(s).

Funding

This work was supported by Quaternary Research Association.

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2025-09
Taylor & Francis
https://doi.org/10.1080/14614103.2025.2561284
Briant RM, Ascough P, Brock F, Smith DN. (2025) Radiocarbon dating insect samples: new data and recommendations. Environmental Archaeology, Available online 24 September 2025

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