Radiocarbon (2024), **66**, pp. 806–815
doi:10.1017/RDC.2024.83

CAMBRIDGE UNIVERSITY PRESS

TECHNICAL NOTE

A tree-ring cellulose extraction device adapted to radiocarbon analysis

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Received: 27 October 2023; **Revised:** 15 April 2024; **Accepted:** 29 April 2024; **First published online:** 26 September 2024

Keywords: cellulose extraction; dendrochronology; radiocarbon analysis; tree-ring analysis

Abstract

Tree-ring cellulose is a commonly used material for radiocarbon analysis. Extracting cellulose is labor-consuming and several devices that enable batchwise extraction have been developed. However, these devices bear the risk of sample contamination. The present study describes a new device which improves upon two aspects of currently available devices. First, to prevent cross-sample-contamination, we redesigned the drainage module to enable independent removal of chemical waste from each individual sample funnel. Second, we added covers to the sample funnels to reduce the risk of external contamination. Cellulose purity (i.e., holocellulose) was confirmed by Fourier Transform Infrared (FTIR) Spectroscopy. Furthermore, accuracy of the radiocarbon analysis was confirmed by results of ^{14}C-blank samples and samples of known age. In conclusion, while maintaining labor-saving, our modified device significantly reduces the risk of sample contamination during extraction of tree-ring cellulose.

Introduction

Radiocarbon (^{14}C) analysis by high-precision accelerator mass spectrometry (AMS) is an increasingly active area of research in dendrochronology and the Earth sciences. Radiocarbon analysis of tree-ring time series is used to construct ^{14}C calibration curves (Hogg et al. 2020; Hua et al. 2021; Reimer et al. 2020), which enable determination of the calendar age of a sample (e.g., a prehistory artifact) from its ^{14}C age. Furthermore, tree-ring radiocarbon analysis can provide information about solar activity during the pre-telescope period, such as long solar cycles, and therefore contributes to a better understanding of the physical basis of the sun's activity (Damon and Sonett 1991; Suess 1980; Stuiver and Braziunas 1993; Usoskin et al. 2021). In addition, exceptional increases in tree-ring ^{14}C abundance, as observed for events in the years 774 AD and 993 AD, may indicate past occurrences of solar super-flares (Büntgen et al. 2018; Miyake et al. 2012, 2013; Usoskin et al. 2013; Uusitalo et al. 2018).

Cellulose is commonly utilized in tree-ring based isotope analysis. As a major structural component of xylem cell walls, cellulose does not migrate between tree rings and is therefore unambiguously linked to the year of tree-ring formation (Leavitt and Danzer 1993). Cellulose composition is comparatively simple, thus avoiding potential bias due to varying proportions of constituents between tree-rings (Rinne et al. 2005). Early studies recommended the use of α-cellulose for radiocarbon analysis since α-cellulose only contains D-glucose monomers (Head 1979; Hoper et al. 1998; Stuiver and Quay 1981). However, more recent studies have shown that ^{14}C abundance of holocellulose is not systematically different from α-cellulose (Capano et al. 2018; Hajdas et al. 2017; Lange et al. 2019; Michczyńska et al. 2018; Southon

and Magana 2010; Staff et al. 2014). Since holocellulose extraction from wood is less labor-intensive than α-cellulose extraction, holocellulose has become the preferred analytical substrate for reconstructing interannual atmospheric ^{14}C content (Büntgen et al. 2018; Brehm et al. 2021, 2022; Güttler et al. 2013; Wacker et al. 2014).

To extract cellulose (either holocellulose or α-cellulose) from wood materials is a central step in the preparation of tree-ring samples for radiocarbon analysis. Cellulose extraction involves the removal of resins and lignin via organic solvent extraction and chlorination (Green 1963; Leavitt and Danzer 1993), or alkaline extraction and chlorination (Rinne et al. 2005; Wieloch et al. 2011). The cellulose extraction process requires repeated addition and removal of reagents as well as washing and is, therefore, a highly labor-intensive process. Consequently, dendro-scientists have developed various devices to facilitate batchwise extraction of cellulose. These include filter bags (Cullen and Macfarlane 2005; Leavitt and Danzer 1993), filter tubes or funnels with or without polytetrafluorethylene (PTFE) drainage blocks (Andreu-Hayles et al. 2019; Harada et al. 2014; Loader et al. 1997; Fogtmann-Schulz et al. 2021; Wieloch et al. 2011), and plate splines with PTFE punching sheets (Kagawa et al. 2015; Li et al. 2011). With regard to radiocarbon analysis, however, all currently available devices carry the potential risk of external contamination and internal cross-sample-contamination due to either direct contact between tree-ring samples (plate splines) or their interconnected storage spaces (filter bags and funnels). For instance, Santos et al. (2020) report that the extraction procedure designed by Andreu-Hayles et al. (2019) can result in modern carbon contamination and suggest it is unsuitable for analyses of preglacial samples. This is a significant concern, especially because Sookdeo et al. (2020) recommend that, for high-precision radiocarbon analysis, cellulose extraction for tree-ring samples should be performed simultaneously with blank samples. Thus, a device for high-throughput extraction of tree-ring cellulose suitable for radiocarbon analysis (i.e., mitigating the risk of external and internal contamination) is currently unavailable.

To fill this gap, we adapted the batchwise cellulose extraction device developed by Wieloch et al. (2011), known as "multiple sample isolation system for solids" (MSISS) to radiocarbon analysis. First, to prevent cross-sample-contamination, we redesigned the drainage module to enable independent removal of chemical waste from each individual sample funnel. Second, to reduce the risk of external contamination, we added covers to the sample funnels. In the following sections, we start with a detailed illustration of the modified device, followed by an evaluation of its suitability for tree-ring cellulose extraction for radiocarbon analysis.

Description of the device

Two key modifications were implemented to adapt the original MSISS:

1. Independent drainage channels: In the original MSISS, the drainage channels were interconnected. This bears the risk of transferring chemicals and contaminants among tree-ring samples. However, in the modified version, each tree-ring sample has its own separate drainage channel, which prevents cross-sample-contamination (Figure 1a).
2. Funnel covers: In the original MSISS, the funnels are uncovered during entire experiment. This bears the risk of external contaminants entering the funnels. To address this issue, PTFE covers have been added to each funnel in the modified device (Figure 1a–d). These covers effectively prevent contaminants from falling into the funnel during the extraction process (Figure 1e). Additionally, the covers minimize volatilization of ClO_2 and Cl_2 gases produced during the acid $NaClO_2$ treatment step. Each cover is equipped with a short handle for effortless lifting and replacing.

The modified device has an inverted T-shaped drainage module with staggered L-shaped drainage channels inside. The upper part of the drainage channel functions as an outlet (Figure 1a-d). On both sides of the drainage module, 18 funnels are arranged with PTFE covers. Numbers 1 to 18 are engraved

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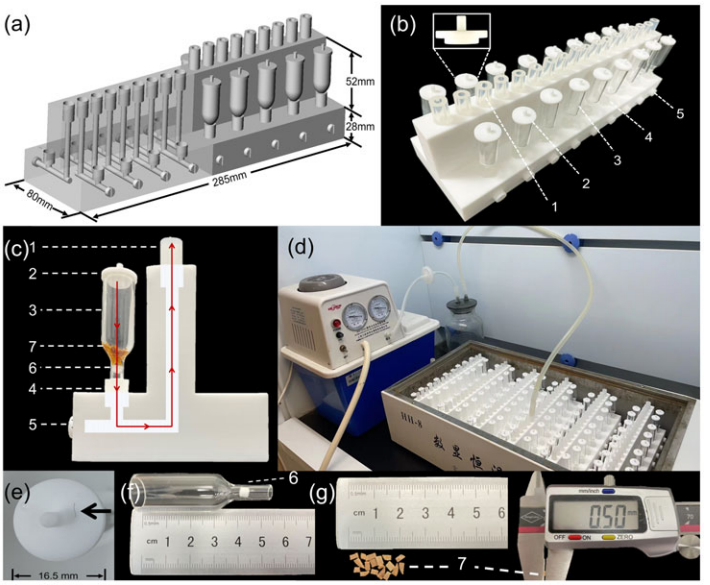

Figure 1. *An illustration of the device and sliced samples used to extract tree-ring cellulose for radiocarbon analysis. (a) The three-dimensional structural diagram of the modified device. The left part of the diagram shows the internal drainage channels. (b-c) An image and side view of the device: 1. silicone tubing outlet used to remove chemical waste; 2. PTFE cover used to prevent external contamination with its side view shown at the top left; 3. glass funnel used for loading tree-ring sample; 4. silicone tubing used to connect funnels to the drainage module; 5. PTFE screw; 6. sintered glass filter disc inside the funnel (similarly, hereinafter); 7. sliced wood samples inside the funnel (similarly, hereinafter). The red curve with arrows indicates the flow path of chemical waste during the pumping process. (d) Experimental setup used here for batchwise extraction of tree-ring cellulose. For chemical treatments, six devices containing 108 tree-ring samples were heated in a water bath (600 mm × 300 mm). With a vacuum pump, chemical waste can be pumped through the outlets at the top of PTFE block. (e) Contaminants (black arrow) that fell on the cover during the experiment. (f) The funnel. The filter disc at the base of the funnel has an aperture size of ca. 30–50 μm. (g) The wood samples that were sliced into pieces around 3×3×0.5 mm^3 in size.*

next to the corresponding funnel for easy identification. Prior to chemical treatment, wood samples are placed into the funnels. Subsequently, chemical reagents are manually added to the funnels using a pipette gun. Once the chemicals are added, the funnels must be covered immediately to prevent contaminants from entering the system. Removing chemical waste from funnels can be achieved using a vacuum pump to create a negative pressure environment inside the drainage module. The silicone tube is attached to the outlet, allowing chemical waste to flow automatically into a collecting bottle. The covers do not need to be adjusted while chemical waste is pumping out because they are not air-tight seals. A filter disc (ca. 30–50 μm aperture size) is fixed at the base of each funnel, keeping the wood samples inside funnels during pumping (Figure 1c, f). The process of pumping chemical waste from each funnel just takes 3 to 5 seconds when using a vacuum pump with a suction rate of 10 L/min. With six drainage modules in a water bath (length: 600 mm; width: 300 mm; height: 130 mm), 108 tree-ring samples can be processed simultaneously (Figure 1d). The entire procedure must be conducted under a fume hood, since harmful gases are released during cellulose extraction.

Overall, these modifications are shown to significantly reduce the risk of contamination during cellulose extraction while maintaining the high efficiency of the existing device. For more information on the device design and availability, please refer to Text S1.

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Table 1. *General information about the wood samples used for assessing the suitability of our modified tree-ring cellulose extraction device for radiocarbon analysis*

Sample ID	Age	Species	Type	Site
IAEA-C9	Infinite*	Kauri (*Agathis australis*)	Subfossil wood	New Zealand
NF142	Infinite	Pine (*Pinus uniseriate*)	Fossil wood	Xianfeng Basin, Yunnan, China
KB	Infinite	Kauri (*Agathis australis*)	Subfossil wood	New Zealand
LWQ1796	1796 AD	Spruce (*Picea likiangensis*)	Logged wood	Riwoche, Xizang, China
AD1515	1515 AD	Oak (*Quercus* sp.)	Architecture wood	Windsor Castle, Berkshire, UK

*Infinite ages denote ages that lie beyond the ^{14}C dating limit (Taylor and Bar-Yosef 2016). The ^{14}C abundance of an infinite-aged sample is indistinguishable from background values.

Materials and methods

Wood samples

Five wood samples of different ages were used to assess the suitability of the modified device for radiocarbon analysis (Table 1). The samples are IAEA-C9, a subfossil kauri wood from peat bogs in New Zealand (Hogg et al. 1995); NF142, a fossil pine wood from the late Miocene layer in Xianfeng Basin, Yunnan, China (Wang et al. 2017); KB, a kauri wood from Marine Isotope Stage 7 in New Zealand (Sookdeo et al. 2020); LWQ1796, a tree-ring sample of known age (1796 AD) from an absolutely dated spruce tree-ring-width chronology (LWQ; Figure S1; Chen et al. 2019) from Riwoche County in Xizang, China; AD1515, an architecture oak wood with a known age (1515 AD) from A. Bayliss of Historic England (Bayliss et al. 2023).

Holocellulose extraction

The protocol for holocellulose extraction was conducted according to the revised Base-Acid-Base-Acid-Bleaching procedure as shown in Figure 2 (Němec et al. 2010; Sookdeo et al. 2020). Prior to chemical processing, the glass funnels and 10 mL glass vials were heated at 500°C for 4 hr in a Muffle oven (Lindberg/Blue M™, Thermo Scientific™, USA) to remove carbon contaminants. To attain the desired level of cleanliness, the glass funnels, PTFE drainage modules, silicone tubes, and covers were immersed for 24 hr in 1M HNO_3 and ultrapure water respectively. To ensure an efficient chemical extraction of cellulose, all wood samples were sliced into grains of ca. $3\times3\times0.5$ mm^3 (Figure 1g). To reduce the corrosive effects of alkaline solution on the sintered glass filter discs inside the funnels, the first step was executed in the glass vials inside an oven (DHG-9123A, SANFA®, China). Subsequent steps were performed in a water bath environment using our modified extraction devices. The procedure is carried out under a fume hood, due to the release of harmful gases during cellulose extraction. In general, the second base step and the bleaching step should be repeated 3 times. However, fossil wood samples typically require more repetitions than other samples. The samples need to be rinsed in ultrapure water 3 times after the base steps and the first acid step, and 6 times after the bleaching step.

After chemical processing, the treated samples were transferred to clean glass vials, homogenized using an ultrasonic cell crusher (JY96-IIN, LICHEN®, China) and then subjected to lyophilization for 48 hr using a freeze dryer (Alpha 1-4 LSCbasic, Christ™, Germany). Subsequently, FTIR (Nicolet™ iS50, Thermo Scientific™, USA) was used to test the holocellulose purity (Kagawa et al. 2015;

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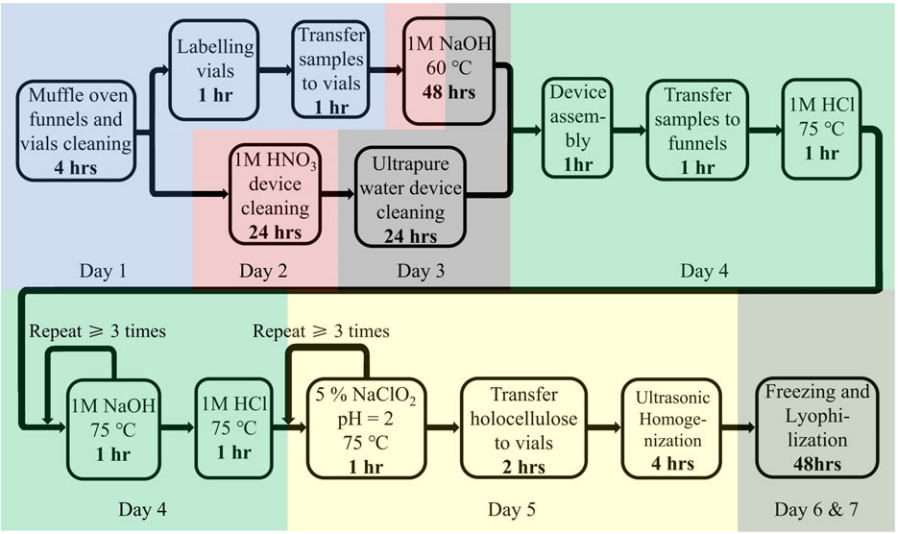

Figure 2. *Protocol for holocellulose extraction used in this study. This protocol is a modified version of the Base-Acid-Base-Acid-Bleaching protocol (Němec et al. 2010; Sookdeo et al. 2020). Day 1 (blue): The glass funnels and vials are heated at 500°C in a Muffle oven for 4 hr. After cooling down, the vials are labelled and loaded with wood samples. Day 2 (red): Wood samples are treated with 1M NaOH in the vials at 50°C inside an oven for one day. Meanwhile, all parts of the device including glass funnels, PTFE drainage modules, silicone tubes, and covers are immersed in 1M HNO_3 for 24 hrs. Day 3 (gray): Wood samples continue to be treated with 1M NaOH for one day. Meanwhile, all parts of the device are rinsed and immersed in ultrapure water for 24 hr. Day 4 (green): The device is assembled. Wood samples are transferred to funnels followed by ultrapure water rinsing for 3 times. Wood samples are treated with 1M HCl at 75°C inside a water bath for 1 hr then rinsed for 3 times. Subsequently, the samples are treated with 1M NaOH for 1 hr. This step should be repeated until the supernatant remains colorless. After being rinsed 3 times, wood samples are treated with 1M HCl again. Day 5 (yellow): The wood samples are bleached for 1 hr with HCl-acidified 5% $NaClO_2$ ($pH = 2$). This step should be repeated until the wood transforms into holocellulose and the color turns into pure white. After being rinsed 6 times, the holocellulose samples are transferred to vials and then homogenized using an ultrasonic cell crusher. Day 6 & 7 (brown): The holocellulose samples are frozen and lyophilized.*

Schollaen et al. 2017). Extracting holocellulose from 108 samples was completed in less than a week. Since the system is scalable, higher sample throughputs are possible.

Radiocarbon analysis

Each treated sample (2.4 ± 0.1 mg) was wrapped in a tin foil vessel. The sealed samples were combusted at 920 °C in an organic elemental analyzer (vario ISOTOPE SELECT, ELEMENTAR™, Germany) and graphitized at 580 °C with hydrogen gas and iron powder in an automated graphitization system (AGE3, IONPLUS™, Switzerland). $F^{14}C$ (Reimer et al. 2004) measurements were performed on graphitized samples at the Laboratory of AMS Dating and the Environment at Nanjing University on a compact radiocarbon AMS system (MICADAS, IONPLUS™, Switzerland; Wacker et al. 2010a). Fractionation and background correction was conducted automatically using the BATS program (Wacker et al. 2010b) based on results of standard (OXII, NIST SRM 4990C) and ^{14}C-blank (Phthalic

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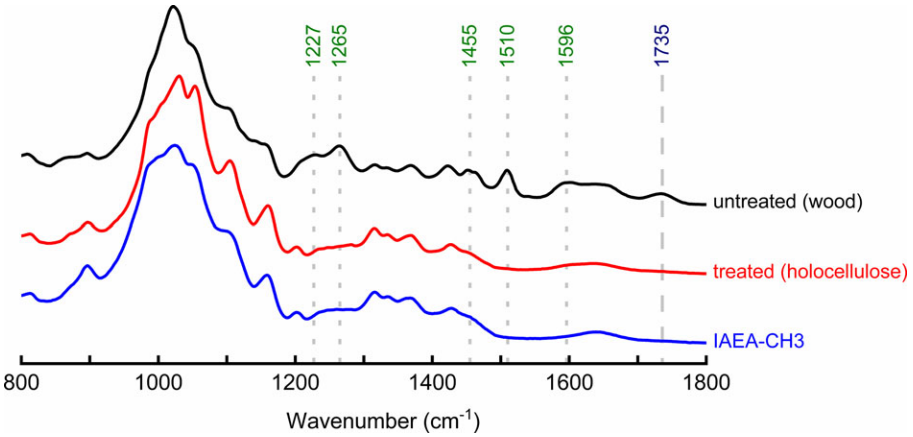

Figure 3. *The normalized FTIR absorbance spectra of untreated LWQ1796 sample (wood, black line), chemically treated LWQ1796 sample using our modified cellulose-extraction device (holocellulose, red line), and the reference sample IAEA-CH3 (cellulose, blue line). The dotted and dashed vertical lines denote the absorption bands related to lignin and hemicellulose, respectively (Pandey and Pitman 2003; Schollaen et al. 2017).*

Acid, PhA, Sigma Aldrich®, PN-320064-500g) samples. $\Delta F^{14}C$, which is defined as the deviation between the measured $F^{14}C$ of each replicate and their weighted mean (Santos et al. 2023) was calculated to demonstrate scatter of the replicates. Reduced χ^2 (χ^2_{red}, i.e., MSWD; Wendt and Carl 1991) was used to check the consistency of replicates.

Results

The FTIR results of both untreated and treated wood sample (LWQ1796) along with the international standard cellulose reference sample IAEA-CH3 are shown in Figure 3. In contrast to the wood spectrum, absorption peaks caused by resin lignin (dotted lines, 1596 cm^{-1}, 1510 cm^{-1}, 1455 cm^{-1}, 1265 cm^{-1} and 1227 cm^{-1}; Pandey and Pitman 2003; Schollaen et al. 2017) are absent in the spectrum of treated sample. The spectrum of treated sample agrees with that of IAEA-CH3, confirming that lignin had been removed using our modified device. Notably, the absorption peak related to hemi-cellulose (dashed line, 1735 cm^{-1}; Schollaen et al. 2017) is not obvious in the spectrum of treated sample, indicating a very low content of hemicellulose in our chemically treated sample. Similar result was produced by Khumalo et al. (2024). However, we referred to the chemically treated sample as holocellulose because it did not undergo the standard α-cellulose extraction procedure involving alkaline treatment with 17.5% (w/v) NaOH solution (Anchukaitis et al. 2008; Gaudinski et al. 2005; Němec et al. 2010).

The results of the radiocarbon analysis on treated ^{14}C-blank wood samples are shown in Figure 4a–b (the data are listed in Table S1). NF142 and KB, which currently lack reference values, are compared directly with PhA without background correction (Figure 4a). The $F^{14}C_{wm}$ values (weighted mean of $F^{14}C$ values) for NF142 and KB are 0.0023 ± 0.00003 (n = 10) and 0.0019 ± 0.00005 (n = 4), respectively, which are comparable to PhA (0.0021 ± 0.00004, n = 6). The $F^{14}C_{wm}$ value of IAEA-C9 is 0.0021 ± 0.0002 (n = 8; i.e., 0.21 ± 0.02 pMC; Figure 4b), which concurs with previously reported reference values (0.12 to 0.21 pMC, Hogg et al. 1995; 0.2 ± 0.05 pMC, Scott 2003). The $\Delta F^{14}C$ of IAEA-C9 varies in the range −0.0006 to 0.0004. The χ^2_{red} value of the IAEA-C9 replicates is 0.5, indicating that the replicated radiocarbon measurements are consistent. These results demonstrate that the ^{14}C-blank wood samples were not influenced by other samples and confirm that no modern contamination was introduced during cellulose extraction.

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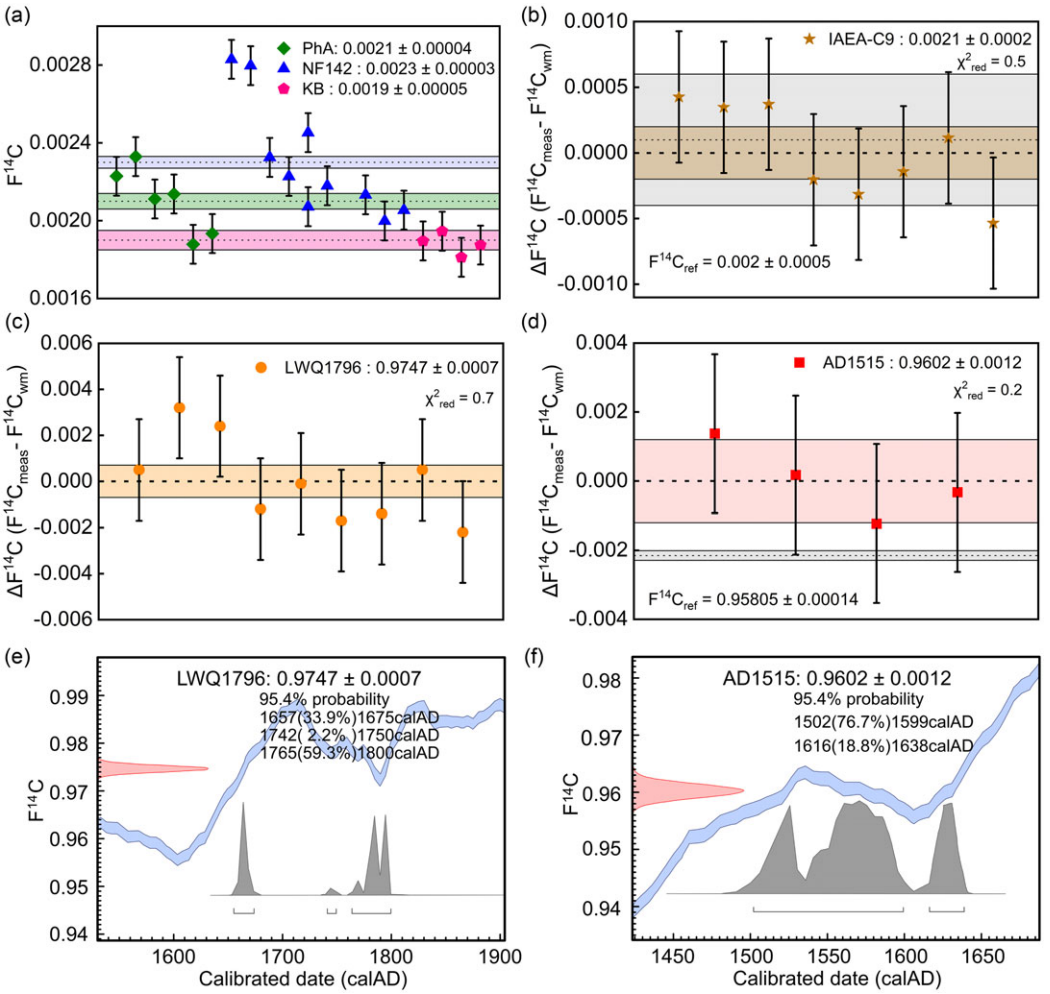

Figure 4. *Radiocarbon analytical results of the wood samples. (a) Comparison of measured $F^{14}C$ values of the ^{14}C-blank (PhA, green diamond), NF142 (blue triangle) and KB samples (pink pentagon). Weighted means of individual $F^{14}C$ values ($F^{14}C_{wm}$) are given in the legend (similarly, hereinafter; Table S1). Dotted lines represent $F^{14}C_{wm}$, and colored bands represent the $\pm 1\sigma$ interval (similarly hereinafter). (b) $\Delta F^{14}C$ of the IAEA-C9 samples and the associated χ^2_{red} value. The thinner dotted line with the gray band represents the reference value ($F^{14}C_{ref}$; Scott 2003; similarly, hereinafter). (c–d) $\Delta F^{14}C$ of the LWQ1796 and AD1515 samples, respectively. $F^{14}C_{ref}$ is from Brehm et al. 2021. (e–f) Calibrated $F^{14}C_{wm}$ results of the LWQ1796 and AD1515 samples derived from the program Oxcal 4.4 (Bronk Ramsey 2009) with IntCal20 used as reference (Reimer et al. 2020).*

Figures 4c–f show the results of our radiocarbon analysis on wood samples of known age. The $\Delta F^{14}C$ of LWQ1796 and AD1515 varies within the range −0.0022 to 0.0032 (n = 9; Figure 4c) and −0.0012 to 0.0014 (n = 4; Figure 4d), respectively. The χ^2_{red} values of LWQ1796 and AD1515 replicates is 0.7 and 0.2, respectively. According to $\Delta F^{14}C$ and χ^2_{red}, the replicates of both LWQ1796 and AD1515 are not significantly different. Although all AD1515 replicates have higher $F^{14}C$ values than the reference ($F^{14}C_{ref} = 0.95805 \pm 0.00014$; Brehm et al. 2021), they are still in statistical agreement. The calibrated ages of LWQ1796 and AD1515 correspond to their known calendar ages (Figure 4e–f). These results indicate that our adapted device and methods are suitable for high-precision radiocarbon analysis.

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Discussion

Exogenous contamination, possibly arising from inadequate cleaning of devices or use of inappropriate reagents, can impact high precision radiocarbon dating (Gaudinski et al. 2005; Anchukaitis et al. 2008). According to Santos et al. (2020), the use of current batchwise devices for tree-ring cellulose extraction yielded sub-optimal results for blank samples. This implies that substances originating from introduced contaminants and/or other wood samples inside the device may taint the blank samples. In this study, the original MSISS is adapted to extract holocellulose for radiocarbon analysis by converting the drainage channel from interconnected to independent, and adding funnel covers. The FTIR results show that our modified device can produce pure holocellulose, while the radiocarbon analysis revealed that there are no effects of contaminates on the blank or reference samples.

Pumping chemical waste out for each sample individually with our modified device requires more operations compared to the original MSISS, which has the capability of pumping dozens of samples simultaneously (Wieloch et al. 2011). However, pumping out chemical waste for individual samples is still labor-saving compared to pipetting chemical waste from test tubes (Santos et al. 2023; Southon and Magana 2010). Fogtmann-Schulz et al. (2021) designed a system that is comparable to ours but contains more intricate and bigger funnels with valves. The smaller funnels of our device enable the simultaneous extraction of more samples. Meanwhile, our modification enables a laboratory currently equipped with MSISS to manufacture the new drainage modules and covers, while the glass funnels, silicone tubes, and vacuum pump remain unchanged. This is more convenient and economical than ordering and designing new devices.

In summary, the adapted tree-ring cellulose extraction device has considerable benefits in reducing contamination and saving labor in the removal of chemical waste. Although the adapted device was initially designed for radiocarbon analysis, considerable benefits can be conducted to ascertain its applicability to cellulose extraction for other plant tissues and stable isotope analysis.

Conclusions

An adapted device for cellulose extraction was developed at the Laboratory of AMS Dating and the Environment at Nanjing University. It is particularly suitable for radiocarbon analysis of large numbers of tree-ring samples based on the following innovations:

(a) Independent drainage channels prevent cross-contamination among samples.
(b) Funnel covers prevent sample contamination from the outside and enhance the efficiency of the $NaClO_2$ treatment step.

The suitability of the adapted device for radiocarbon analysis is confirmed by FTIR and a number of radiocarbon test analyses. Researchers in related disciplines, such as stable isotope analysis, should find the device useful, particularly when there is an increased risk of error due to sample contamination.

Supplementary material. Information about the device design and availability is referred to Text S1. The LWQ tree-ring chronology and the radiocarbon data in Figure 4 are shown in Figure S1 and Table S1.
For supplementary material accompanying this paper visit https://doi.org/10.1017/RDC.2024.83

Acknowledgments. We thank Dr. Irka Hajdas (ETH Zurich, Switzerland) for her valuable comments as well as supplying the wood samples KB and AD1515. We are also grateful to Prof. Tao Su (Chengdu University of Technology, China) for supplying the wood sample NF142. We feel thankful to Prof. Michael Meadows (Nanjing University, China) for his contribution to improve the language. We would like to thank Editor-in-Chief, Prof. Timothy Jull, Associate Editor Prof. Steven Leavitt and two anonymous reviewers for their insightful comments. This research is supported by the National Natural Science Foundation of China (42171155; 42011530119; 42201014), the "GeoX" Interdisciplinary Research Funds for the Frontiers Science Center for Critical Earth Material Cycling, Nanjing University (2023300297) and "Formas - a Swedish Research Council for Sustainable Development" (2022-02833).

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Cite this article: Lin P, Zhao Y, Zhang H, Wieloch T, Gu Y, Liang C, Chen F, and Lu H (2024). A tree-ring cellulose extraction device adapted to radiocarbon analysis. *Radiocarbon* **66**, 806–815. https://doi.org/10.1017/RDC.2024.83