Dating volcanic eruptions with tree-ring chemistry

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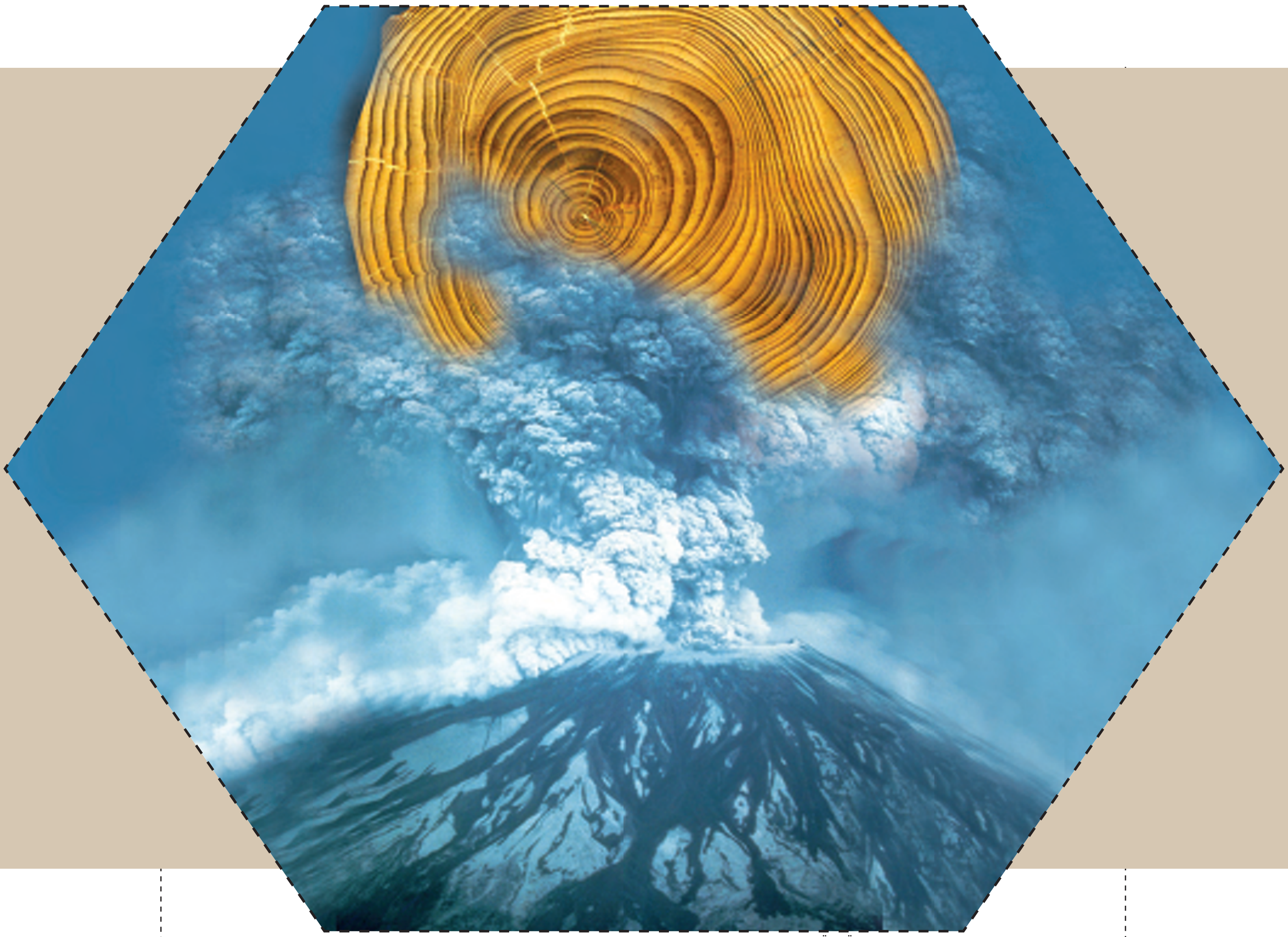

VOLCANO: THE U.S. GEOLOGICAL SURVEY, TREE RING: KENAN ÜNLÜ, CHARLOTTE PEARSON, DANIELLE K. HAUCK, AND PETER I. KUNIHOLM

Dendrochemical analyses of absolutely dated, overlapping sequences of tree rings allow identification of temporally-conscribed, volcanically-influenced periods of environmental change. Dendrochemistry, or the study of tree-ring elemental composition, is a promising new technique for reconstructing climate/environmental history at annual resolution. In particular, dendrochemistry may be useful for identifying periods of climatically and/or environmentally effective volcanic activity. Airborne pollution from major volcanic eruptions in the form of increased environmental acidity from sulfur dioxide can cause changes in availability and concentration of certain elements, and can increase the availability of those elements in the soil, resulting in increased uptake by trees from the substrate or direct from the atmosphere. In particular, spikes, dips, or major changes in trace element concentration may be an indication of changes in soil or atmospheric chemistry (e.g., Padilla and Anderson 2002). Although there are other records of past volcanism (especially from ice-cores—e.g, Vinther et al. 2005), tree-ring based work (e.g., Salzer and Hughes 2007) offers several important advantages: first, tree-ring series are available with

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wide spatial coverage from most of the globe, and second, they are datable with annual and even subannual resolution on a fixed (absolute) calendar timescale (whereas even the best ice-core work has errors of several years or more beyond the last few hundred years).

Many trees in temperate zones grow one visible tree-ring per year, forming an annual record or ring pattern that reflects climatic conditions. The widths of these rings are dependent on the quality of the yearly growth season. Wood samples are dated by creating a timeline series of tree-ring widths, called a chronology. Often, climatically significant years are readily apparent in a chronology as having anomalously large or small ring widths or varying wood densities. Trees of the same or similar species that grew in the vicinity of each other will exhibit similar ring-width patterns that may be matched against each other, a method called cross-dating. Environmental events that are large may affect trees worldwide, providing a hemispheric or even global dating marker. By creating chronologies for trees where a growth ring can be placed in a known year (through sampling of living or recently felled trees), it is then possible to match the growth patterns and unusual events backwards to date premodern tree-rings and to build long absolutely dated tree-ring chronologies (e.g., Briffa et al. 1998).

The capacity for annual resolution analysis is an essential requirement when attempting to trace volcanic eruptions in dated tree-ring sequences. Major explosive volcanic events tend to be of short duration, with any potential elemental impact limited to a particular year or growth season, or, in the case of the more major eruptions of the Holocene, to a few years immediately following the event. Analytical techniques which necessitate multi-elemental analysis on a decadal or lower resolution scale are therefore unsuitable for this particular research question. Potential volcanic signatures would be lost within the bulk sample, and the overriding aim of tracing eruptions in tree rings to provide annual resolution dates for key events could not be met.

Over the last few years, exciting new dendrochemical applications have been made by a team of researchers at the Radiation Science and Engineering Center (RSEC) at the Pennsylvania State University and the Malcolm and Carolyn Wiener Laboratory for Aegean and Near Eastern Dendrochronology at Cornell University. Neutron activation analysis (NAA), solution or laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS/ICP-MS) and synchrotron scanning x-ray fluorescence microscopy (SXFM) have all been utilized to determine volcanic signatures in tree rings.

NAA and solution ICP-MS are two analytical techniques that have been used with varying degrees of success in attempts to trace volcanic eruptions at annual resolution in tree-ring sequences. In each case the resolution of the data collected is limited only by lengthy sample preparation procedures involving the manual dissection of individual tree rings, and the thickness of the rings in question which determines the accuracy of dissection possible. The most likely tree rings from which an elemental signature may eventually be derived are those that come from trees growing in marginal, stressed environments, potentially more responsive to subtle changes in elemental chemistry. The downside of this is that growth rings from such trees are likely to be extremely narrow and therefore difficult to extract in sufficient quantity and with sufficient accuracy to deliver exact annual resolution by these techniques. For tree-ring series where rings are typically under 500 µm, SXFM is the only technique that offers the potential for annual resolution analysis. Where rings are too small to dissect, SXFM can be used to map elemental intensities in each ring or to analyze a transect through the individual rings at as low as 50 µm (annual or sub-annual) resolution.

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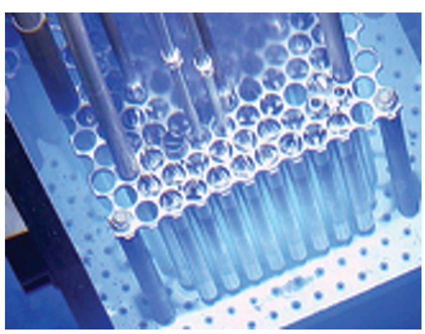

Fig. 1 The Core of the Penn State Breazeale Reactor at RSEC.

Facilities and resources

The Malcolm and Carolyn Wiener Laboratory for Aegean and Near Eastern Dendrochronology at Cornell is one of the world's leading dendrochronological laboratories. Over the past three decades researchers have worked on the construction of high precision dated tree-ring sequences extending from the present back over 8,000 years across the archaeologically significant Aegean region (e.g., Kuniholm et al. 1996; Manning et al. 2001). The Wiener laboratory archives over 40,000 individually-dated tree samples with 4.5 million rings from 109 forests and several hundred medieval monuments and archaeological sites in the eastern Mediterranean and former Soviet Union. These samples span most of the period from 7000 B.C. to the present. The Wiener laboratory also has an extensive collection of recent material from a variety of sites in eastern North America. All tree-ring samples mentioned in this study were obtained from this remarkable archive.

Dendrochronologically-dated samples for this study were cut from each ring using sterile dissection tools in a clean environment and placed in heat-sealed polyethylene bags. For NAA, sealed samples were put together in a large polyethylene vial and irradiated at 1-MW reactor power for four hours in the Penn State Breazeale Reactor (PSBR) irradiation facilities at the RSEC. A picture of the core at PSBR at RSEC is shown in Fig. 1. Thermal neutron flux at sample irradiation location is 1.5×10^{13} n/cm^2/s. Each run is accompanied by a gold wire for flux monitor. After irradiation, samples are taken from the heat-sealed bags, weighed, placed in small vials, and labeled. Fig. 2 shows a wood sample, cut samples from rings, samples after irradiation, and archived samples. Each sample is counted for one hour.

An automated sample handling system is used to handle the volume of samples in a reliable, consistent manner and to reduce errors. The system consists of a rotary sample table, pneumatic horizontal-transfer arm, pneumatic motion slide,

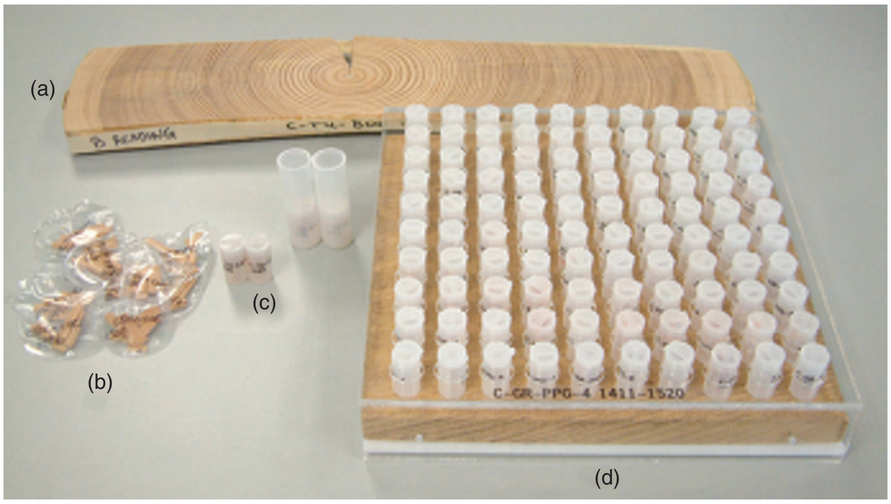

Fig. 2 (a) typical wood sample, (b) samples cut from single rings and heat-sealed in bags, (c) samples after irradiation and weighing, but before counting, (d) archived samples.

gripper actuator, sample nest, and logic control system. Fig. 3 shows the system, detector, and shielding. The automatic sample handling system provides sequential presentation of samples to the detector for analysis. Cycle time is less than 10 seconds. A state-of-the-art gamma spectroscopy system is used for NAA data acquisition and analysis. The system includes a 40% efficient HpGe detector in a well-shielded cave, a digital spectrum analyzer, and a computer with Genie2000 software. Samples are rchived after counting is completed. Samples showing elevated signature elements are re-analyzed to determine other trace elements. A Compton suppression system, which provides a tool to suppress the unwanted background, was used for some critical data. An appropriate multi-element standard reference material is used for quantifying multiple analytes.

Dendrochemical analysis with NAA

Three major irradiation and counting schemes have been employed for analyzing tree-rings with NAA. Each scheme is designed for identifying isotopes with a different range of half-lives. Certain isotopes or elements are observed in order to identify promising environmental markers. Only the results obtained using a long irradiation schedule are briefly discussed below with an emphasis on the applicability of the identified isotopes to environmental monitoring.

Much of the work completed for the dendrochemistry project has focused on quantifying gold in tree-rings with irradiations suited to identify the ^{198}Au isotope which has a 2.7 day half-life. Following this approach, four sets of tree-ring samples, each over 300 years in length, have been analyzed for the dendrochemistry project. Each batch was allowed to decay overnight and then each tree-ring was counted for one hour. This procedure allowed for the identification of ^{24}Na, ^{42}K, ^{56}Mn, ^{82}Br, ^{69}Zn, ^{140}La, and ^{198}Au. In one tree La was not detected consistently because it was present at or below the detection limits and Au was near the detection limits in all trees. Mn was detected but has a short enough half-life (2.6 hours) that it had decayed away before the counting of each batch of samples was completed. All other elements (Na, K, Br, and Zn) were quantified well above the detection limits. The tree-ring samples analyzed in this way and the average concentration results are provided in Table 1. The concentration values are in parts-per-million except where specified otherwise. The values in parenthesis are the number of rings that had been identified for each element and were used as part of the calculation of the average.

Gold behaved in a unique manner compared to the other elements listed in Table 1. Au showed no anomaly at the heartwood boundary (HWB) and has large peaks in single tree-rings. In most cases the other identified elements had either peaks or increases in the continuum level at the HWB. Au concentrations observed are shown in Fig. 4 for four different samples analyzed.

Another series of measurements performed for the dendrochemistry project was the analysis of four small tree samples (30–60 years in age) from the Çatacık forest in Turkey. The period of analysis covered at least the years 1980–2002. The four Çatacık tree samples (C-TU-CAT32, C-TU-CAT21, C-TU-CAT16 and C-TU-CAT28) were analyzed with longer count time in order to increase statistical accuracy. The four tree samples that were analyzed from the Çatacık forest are listed in Table 2. A typical gamma spectrum for two samples analyzed is shown in Fig. 5.

Three major irradiation and counting schemes have been employed for analyzing tree-rings with NAA. Each scheme is designed for identifying isotopes with a different range of half-lives.

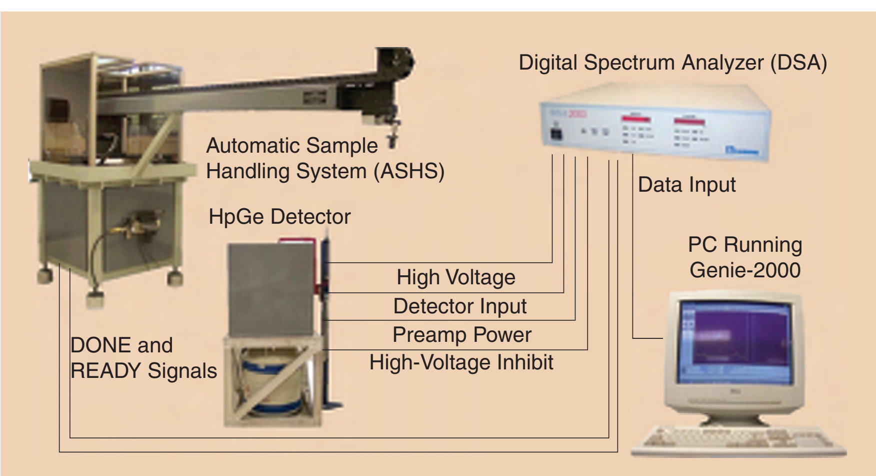

Fig. 3 Automated sample handling system, HpGe detector with shielding, and data acquisition system at RSEC-NAA Facility.

Large peaks in Au concentration were observed and each ring was reanalyzed with the new procedure to check the reproducibility of the gold peaks. The results in Table 2 for C-TU-CAT16 are the average of the results from the two sections and the results for C-TU-CAT32 represent the average of the results from the first and second irradiations. At a later time, each of the tree-rings from C-TU-CAT32 and C-TU-CAT21 was recounted for one day each. A wealth of new isotopic data was obtained. Longer counting time resulted in the identification of seven additional metals including ^{46}Sc, ^{59}Fe, ^{60}Co, ^{65}Zn, ^{110m}Ag ^{124}Sb and ^{134}Cs. The concentrations of these long-lived isotopes in C-TU-CAT32 and C-TU-CAT21 are presented in Table 3. The Ag and Au concentrations in C-TU-CAT32 were correlated with each other. Ag and Au are the only elements that had the sharp peaks above the level of their continua. It was hypothesized that the unique noble metal characteristics of these elements made them suitable for documenting events in the life of the tree. In

Table 1. Average isotopic concentration in four conifer samples analyzed by NAA.

Tree Sample	C-GR-PPG4A*	C-TU-KLK10B	C-TU-CAT14C	P-SW-SEQ2
Years Analyzed	1411–1979	1628–1999	1500–1979	1446–1948
Country	Greece	Turkey	Turkey	USA (CA)
Au**	8 ppb (467)	7 ppb (314)	4 ppb (445)	8 ppb (360)
Br	.08 (470)	.07 (355)	.06 (459)	.4 (368)
K	10 (548)	22 (369)	18 (459)	37 (370)
La	.003 (42)	.006 (203)	.02 (428)	.2 (369)
Mn***	7.4 (47)	11 (67)	21 (237)	14 (200)
Na	69 (557)	60 (369)	37 (459)	86 (374)
Zn	.67 (197)	1.6 (331)	1.5 (456)	1 (307)

* PPG4A is *Pinus leukodermis* from Grevena, Greece; KLK10B is *Pinus nigra* from Kalkım, Çanakkale, Turkey; CAT14C is *Pinus nigra* from Çatacık, Eskişehir, Turkey; and SEQ2 is *Sequoia sempervirens* from the Humboldt Grove, California.

** Rows 4–10 contain the average isotopic concentrations in ppm units. The values in parenthesis are the number of concentration values used for calculating the average.

*** The Mn concentration was measured in a small percentage of the tree-ring samples due to its short half-life.

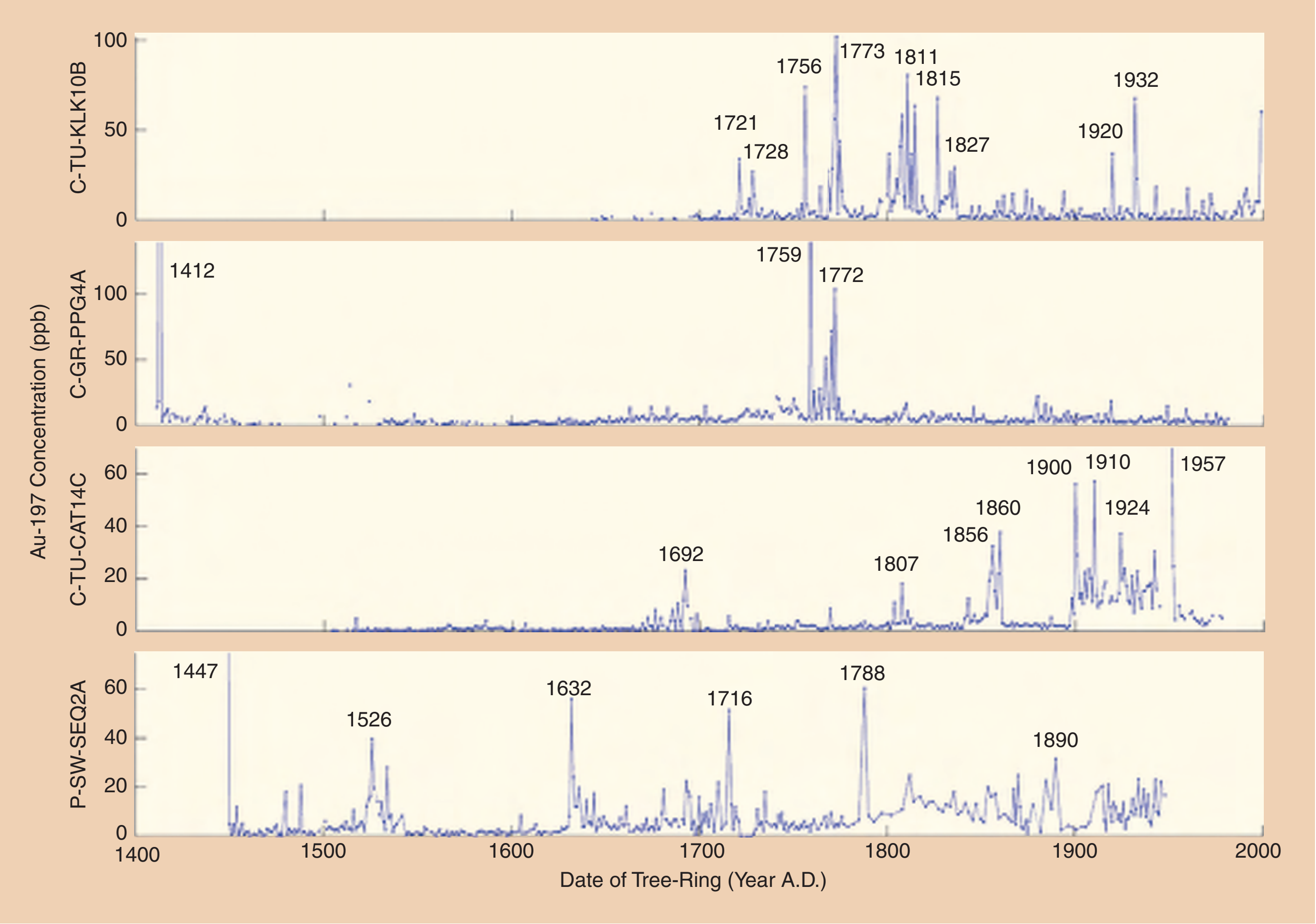

Fig. 4 Gold concentrations in four different tree samples. Observed gold peaks at the date of tree rings are marked.

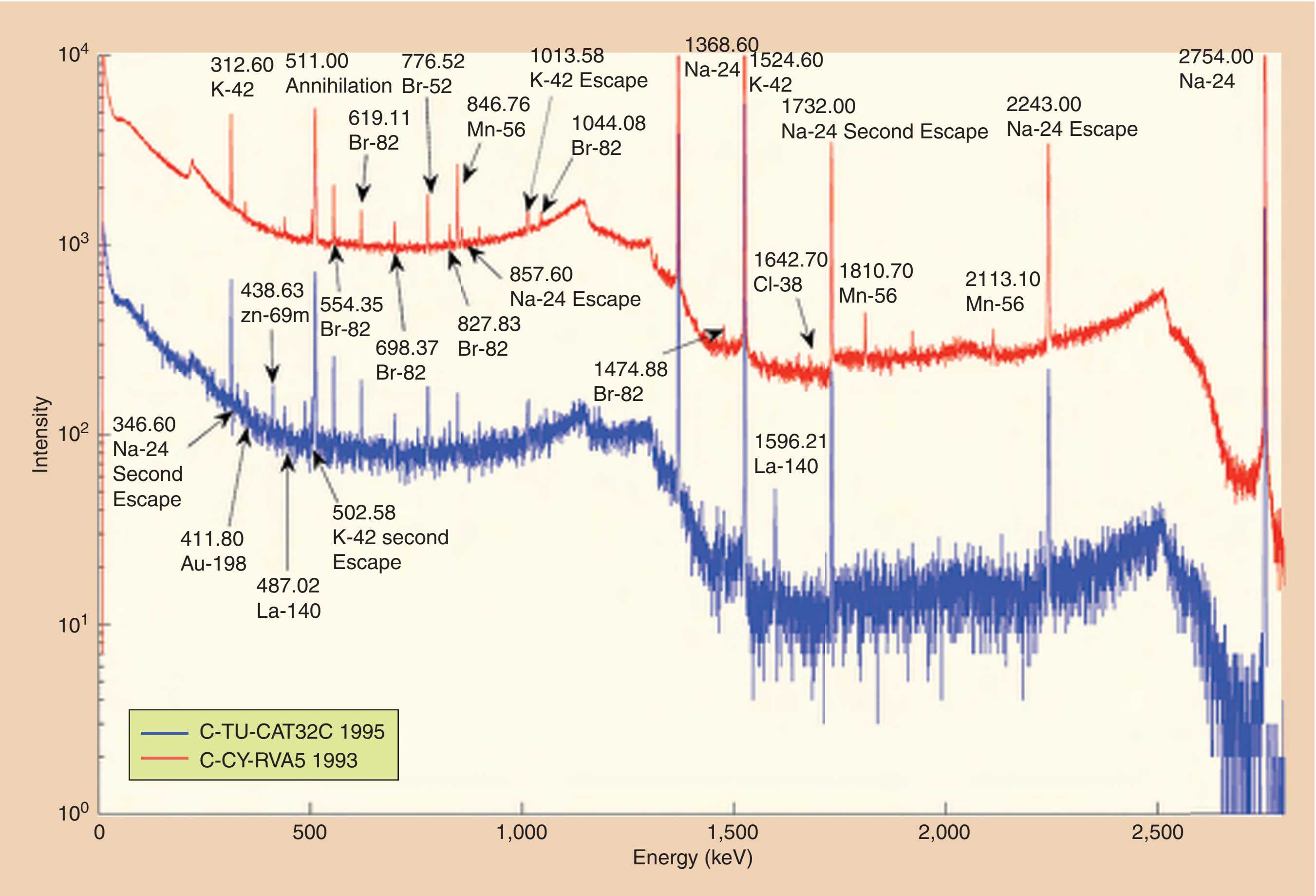

Fig. 5 Typical gamma spectrum from C-TU-CAT32C (1995) and C-CY-RVA5 (1993) Tree-ring samples for long irradiation and 1 hour count after 24 hours of decay.

particular, the Ag and Au compositions may have been documenting the severity of environmental effects such as volcanic activity and/or Mediterranean dust storms, another candidate for such an effect on a tree in this region. Ag has a short lived isotope (^{108}Ag) which might be identified with short irradiations of tree rings. This study is being pursued to determine if environmentally significant data, especially the concentration of Ag in tree rings, could be determined in a more efficient manner through short irradiations.

Table 2. Average isotopic concentrations obtained in four tree samples from the Çatacık Forest using RSEC-NAA with longer counting time.

Tree Sample	C-TU-CAT32	C-TU-CAT21	C-TU-CAT16	C-TU-CAT28
Years Analyzed	1940–2002	1969–2002	1972–2002	1971–2002
Au	1.3 (61)	.19 ppb (30)	.42 ppb (30)	.14 ppb (16)
Br	27 ppb (62)	18 ppb (33)	18 ppb (30)	43 ppb (31)
K	41 (62)	20 (33)	19 (30)	30 (31)
La	27 ppb (62)	15 ppb (33)	19 ppb (30)	16 ppb (31)
Mn	51 (62)	44 (33)	42 (30)	29 (30)
Na	3.1 (62)	5.1 (33)	6.2 (30)	2.5 (31)
Zn	.78 (62)	.81 (33)	.36 (30)	.86 (31)

The great advantage of ICP-MS over other analytical techniques is that it combines ultra-low detection limits with the capacity for near simultaneous analysis of the majority of elements in the periodic table.

ICP-MS and tree-rings—preliminary results

The great advantage of ICP-MS over other analytical techniques is that it combines ultra-low detection limits with the capacity for near simultaneous analysis of the majority of elements in the periodic table, including the rare earth elements, thus providing the greatest possible range of elemental data from which to evaluate a whole suite of possible volcano—environmental—growth increment connections. The disadvantages, as previously stated are a lengthy sample preparation process which is destructive and holds a great deal of potential for contamination, and, in the case of LA-ICPMS a lack of matrix matched calibration standards and higher detection limits for most elements.

Analyses via ICP-MS and LA-ICPMS at the NERC ICP-MS Facility at Kingston-upon- Thames, UK has nevertheless yielded some exciting results (Pearson et al., 2005, 2006). Samples from the Aegean Dendrochronology Laboratory were dissected at annual resolution, dry ashed and brought into solution with nitric acid. They were diluted to appropriate concentrations for standard ICP-MS analysis. In addition, sub-samples of each core were mounted to fit the dimensions of a laser sampling chamber for LA-ICP-MS. Analysis was carried out using a quadrupole, Thermo Elemental PlasmaQuad ICP-MS in conjunction with a Cetax LSX-100 laser (Nd:YAG pulsed with Q-switch) operating at 266 nm. These were driven by PQVision version 4.1.2 and Cetac laser system version 1.20, with a high resolution CCD camera system for observation of the sample during analysis. Due to the lack of matrix matched calibration standards, alternative calibration steps using NIST SRM 610 glass were put in place.

Of the two techniques, the best results and potential were demonstrated by the LA-ICPMS analysis (Pearson et al., 2005)—an example is provided below showing an elemental response to environmental acidification following the largest volcanic eruption of the last 200 years—Tambora, in 1815 A.D. (Fig. 6).

SXFM and tree-rings—preliminary results

SXFM has also been demonstrated to have exciting potential for tree-ring analyses. Using the Cornell High Energy Synchrotron Source (CHESS) SXFM maps have been made for a variety of elements in different species of wood. It is anticipated that spatially resolved analysis in particular, (the capacity for annual resolution analysis of tree rings which are to small to be analyzed by other methods is also a significant advantage) will play a significant role in the interpretation of future series of NAA data and elemental time series generated by other analytical methods. It offers a means by which to understand the physiological associations of certain elements within the tree rings at high resolution—offering a vital insight into the question of exactly how volcanic acidity might impact tree-ring chemistry. An example of spatially resolved micro-analysis with SXFM is shown in Fig. 7.

SXFM has also been used more specifically to trace volcanic eruptions, as illustrated by the following two examples.

A key tree-ring record with regards dating volcanic eruptions using

Table 3. Average concentrations of long-lived Isotopes in two tree samples.

	C-TU-CAT32	C-TU-CAT21
Years Analyzed	1962–2000	1969–2000
^{46}Sc	1.6 ppb (39)	2.6 ppb (33)
^{59}Fe	16 ppb (33)	26 ppb (33)
^{60}Co	78 ppb (39)	32 ppb (33)
^{65}Zn	1.8 ppm (39)	1.7 ppm (33)
^{110m}Ag	6.7 ppb (37)	4.3 ppb (33)
^{124}Sb	2.8 ppb (38)	1.8 ppb (29)
^{134}Cs	1.9 ppb (34)	2.0 ppb (30)

* Rows 3–9 contains the average isotopic concentrations. The values in parenthesis are the number of concentration values used for calculating the average.

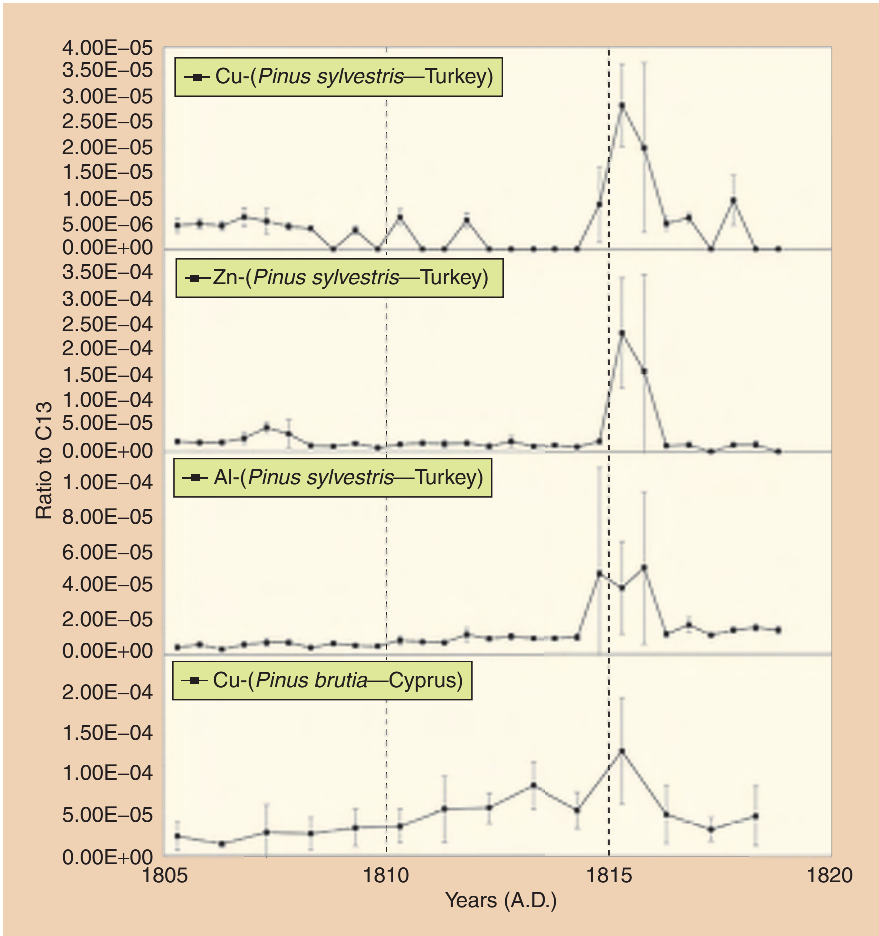

Fig. 6 Zinc, copper, and aluminum all show an increase in and after the year 1815 A.D. (Tambora) in two different trees from Turkey and Cyprus (Pearson and Manning 2009). Data are in ratios to ^{13}C, generated using LA-ICP-MS.

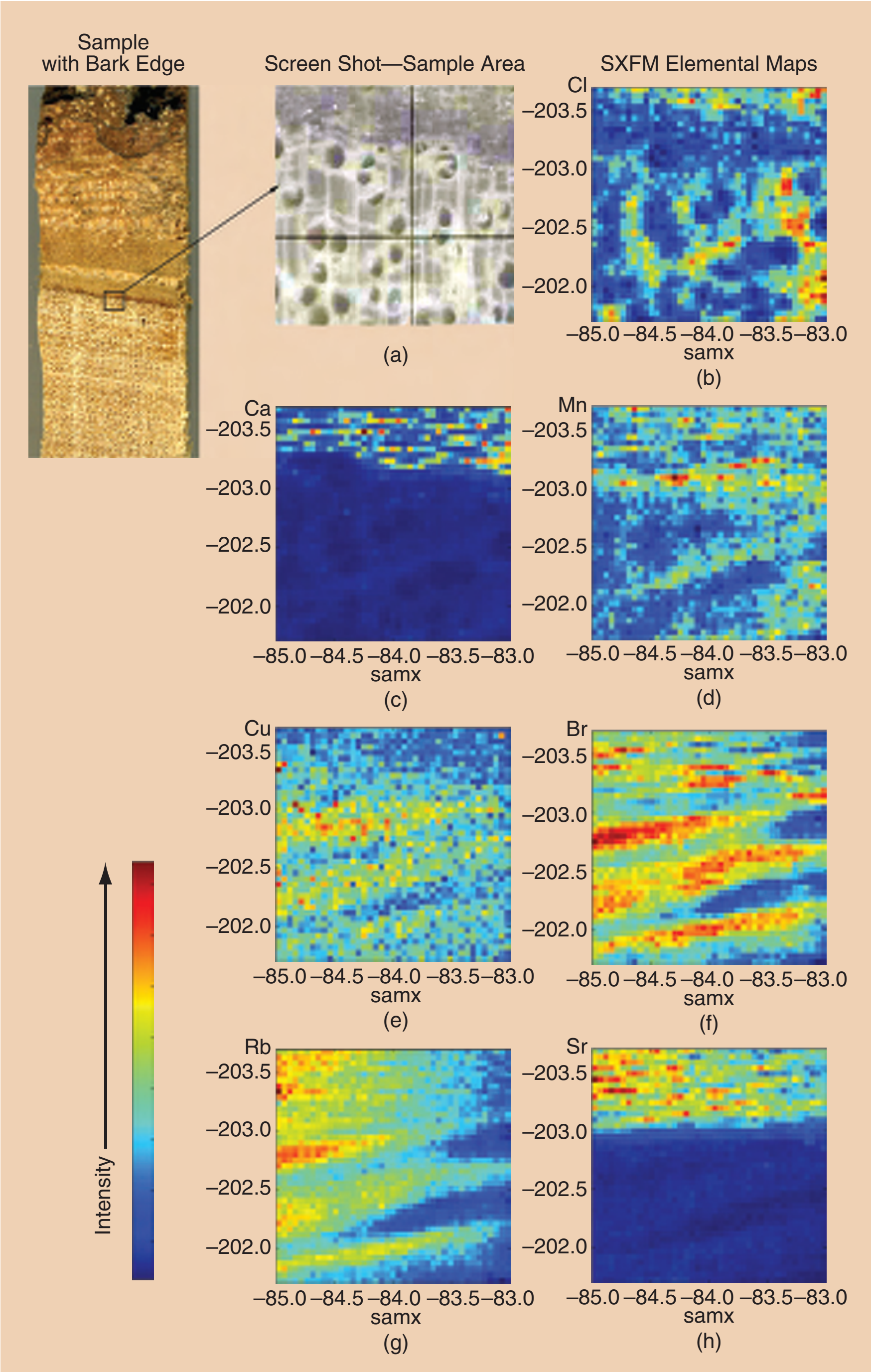

Fig. 7 Multi-elemental maps for *Avicennia* sp. (Mangrove genus) for a 2 mm by 2 mm square at the cambial boundary, shows distribution of the various elements according to wood structure and physiology. Significant changes in intensity were shown between the bark and the xylem tissue. Ca (c) and Sr (h) are higher in the bark. Mn (d) and Cl (b) show depletion at the cambium. Ca (c) and Cl (b) show general incorporation in the woody structure, with Mn (d), Cu (e), Sr (h), and Rb (g) showing some form of periodicity in the wood structure. Data were collected at F3 Beamline–Cornell High Energy Synchrotron Courtesy of Darren Dale and Charlotte Pearson.

anatomical proxy indicators for the climatic impact of volcanism (frost and/or micro rings), is that of the North American bristlecone pines (*Pinus aristata* Engelm. and *Pinus longaeva* D.K. Bailey) (Salzer and Hughes 2007). Within the 10,000-year record from the bristlecone pines, distinct correlations have been proven between modern volcanic eruptions of known date and damage to cells due to unseasonable frost events caused by cooling as solar radiation was backscattered by volcanic sulfur dioxide. Rings of typically less than 500 μm, and

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the fact that any analysis of these samples must be nondestructive means that multi-elemental analysis has until recently been largely impossible. SXFM was used to map a frost ring in a sample of *Pinus longaeva* from the White Mountains of California in order to ascertain whether any elemental signature for volcanism could be detected in addition to the physical response (frost ring) associated with the eruption of Huaynaputina, Peru in c. 1600 A.D. Increases of several elements were demonstrated at the frost ring. Some appeared to reflect a change associated specifically with the frost ring itself, but others extended over several years following the frost event. Fig. 8 shows an example for strontium (Sr). Analysis of other rings in trees from the same site which were not damaged by frost in 1600 A.D. indicated that observed elemental responses were due to a physiological reaction within the tree rather than an external impact from volcanic chemistry.

The most marked tree-ring growth anomaly in the Aegean dendrochronological record over the last 9,000 years occurs in the mid 17th century B.C. and has been speculatively correlated with the impact of the Late Bronze Age eruption of Thera (Santorini). If such a connection could be proved it would be of major interdisciplinary significance. It would open up the possibility of a precise date for a key archaeological, geological and environmental marker horizon, and offer a direct tie between tree-ring and ice-core records some 3,600 years ago. Charcoal and wood samples that have survived since the Bronze Age require analysis via a nondestructive method, so SXFM was carried out on growth-ring series from four trees displaying the anomaly which begins with relatively-dated ring 854. Fig. 9

shows increased calcium (Ca) from the onset of the growth anomaly, which may be indicative of a rise in environmental acidity at the time of the eruption.

Conclusion

The three techniques illustrated here all have strong advantages for the analysis of tree-ring sequences to identify elemental markers for volcanic eruptions. Once samples have been dissected, NAA is nondestructive so the same samples can be analyzed by a variety of additional methods—a clear advantage in terms of replication. ICP-MS via laser ablation offers fast, low destructive sampling at high resolution but there are difficulties with turning data into accurate concentrations, and the range of elements that can be detected in wood is greatly limited in comparison with the solution induction method. Solution ICP-MS can be used to successfully generate an annual resolution archive of environmental chemistry from long sequences of tree rings, for over 40 different elemental concentrations (including volcano-specific Rare Earth Elements), however, samples are destroyed in the process and the preparation protocol is prohibitively time and labor intensive. SXFM has exciting potential for use in combination with NAA and/or ICP-MS to provide complimentary data for a more informed interpretation of annual data series, or to analyze tree rings at annual resolution where dissection is not possible due to the thinness of the growth increment.

Trace element concentrations in dendrochronologically-dated (absolute calendar age) wood samples could provide access to an ultra-high-resolution record of past climatically-significant atmospheric pollution events, and, in particular, major volcanic eruptions, for example, in defining years of high atmospheric SO_2 and decreased soil pH. It may also be possible to demonstrate a connection between signature trace element concentrations in dendrochronologically-dated wood and environmental change. By correlating elemental fluctuations in absolutely dated tree-ring samples with environmental effects, it may be possible to provide environmental modelers with a several thousand-year record of change and give archaeologists exact dates for significant volcanic events in prehistory.

For future work attempting to trace volcanic events in tree rings we foresee a combined approach using NAA to create long annual resolution sequences of elemental change, solution ICP-MS analysis of selected events to broaden the range of elements detectable and SXFM to understand the physiological context of those associations. It is through such a combined approach along with further in-depth research into tree-ring physiology that we hope complex hypotheses can be developed to explain the elemental record so that tree-ring chemistry may one day be used as a Holocene-long archive of absolutely dated volcanism.

Trace element concentrations in dendrochronologically-dated (absolute calendar age) wood samples could provide access to an ultra-high-resolution record of past climatically-significant atmospheric pollution events.

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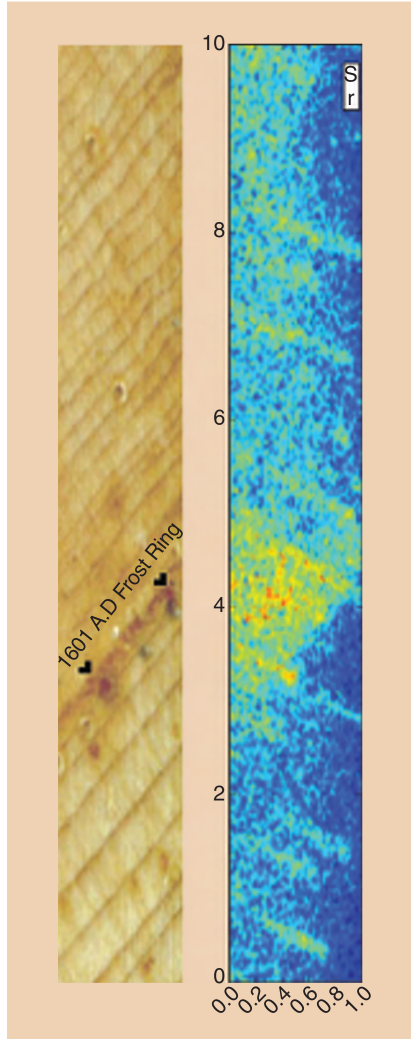

Fig. 8 Bristlecone pine dendrochemistry (Pearson *et al.,* 2009a) shows an increase in Sr following the 1601 A.D. frost ring.

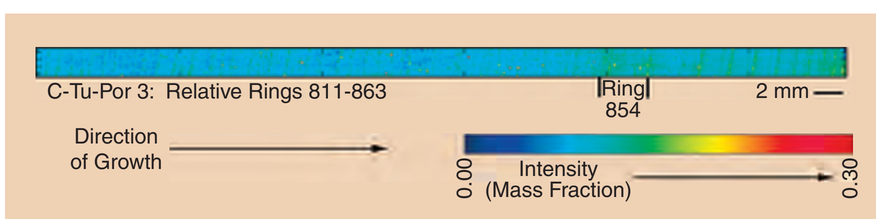

Fig. 9 Dendrochemistry of the Porsuk growth anomaly (Pearson *et al.* 2009b). An increase in elemental intensity for Ca at the growth anomaly may indicate increased environmental acidity related to volcanic activity at this time.

leading role in the development of SXFM for tree-ring analysis.

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Read more about it

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