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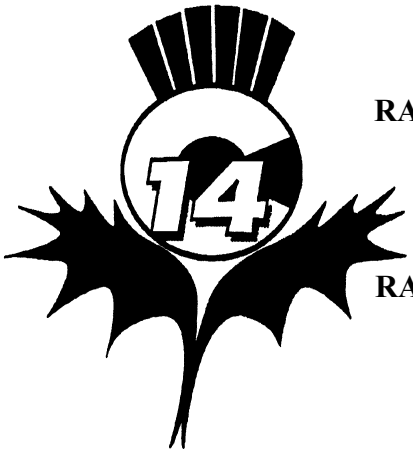

The THIRD INTERNATIONAL RADIOCARBON INTERCOMPARISON (TIRI)

and

The FOURTH INTERNATIONAL RADIOCARBON INTERCOMPARISON (FIRI)

1990–2002

Results, Analyses, and Conclusions

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The FIRI organizers gratefully acknowledge the generous support of the NSF, University of Arizona AMS lab.

FROM THE GUEST EDITOR

Reliable, precise, and accurate radiocarbon age measurements are essential. Such measurements also require traceability to international standards' activities which are known exactly by independent means and also to reference materials' activities which are estimated and typically accompanied by associated uncertainty statements. Within the ^{14}C community, there has been an increasing realization of the need for adequate reference materials. Long and Kalin (1990) stressed that it was incumbent upon individual ^{14}C laboratories to engage in a formal program of quality assurance (QA). Polach (1989) noted that the opportunity for internal checking by individual laboratories involved in producing routine ^{14}C measurements was hampered by a lack of suitable quality control (QC) and reference materials.

Since the early days of applied ^{14}C measurement, it has been common practice for laboratories to exchange samples in attempts to improve and sustain analytical confidence. With time, this practice tended to give way gradually to a succession of more formal group intercomparison exercises. Within the ^{14}C community in just under 20 years, there have been a number of significant and very extensive interlaboratory trials organized by individual laboratories and the International Atomic Energy Agency to the benefit of the ^{14}C community, both laboratories and users (Otlet et al. 1980; ISG 1982; Rozanski et al. 1992; Scott et al. 1990, 1992; Gulliksen and Scott 1995; Scott et al. 1998; Bryant et al. 2000; Boaretto et al. 2003).

These comparisons have varied widely in terms of sample type and preparation, but all (with one exception) have had as their primary goal the investigation of the comparability of results produced under possibly quite different laboratory protocols. However, in reaching this goal, a number of these studies have also created reference materials. As methods and instrumentation have developed and new laboratories are formed, the reference materials created as a result of the intercomparisons, have been widely used for checking procedures and performance. Users have been reassured by the existence of regular comparisons that the laboratories are striving to ensure highest quality results while at the same time, the laboratories have been able to identify any systematic offsets and additional sources of variation. Indeed, in studies which have used representative samples requiring pre-treatment, chemical synthesis and counting, it has been possible to identify the procedure at which problems have arisen and to quantify their relative contributions to the overall variation in the results. Thus, participation in a laboratory intercomparison has been seen to be a part of a formal QA program and the resulting reference materials to form a community resource for the benefit of all.

This special issue of *Radiocarbon* brings together, for the first time, all the experimental results and their analysis from the last two major ^{14}C intercomparison exercises (Third International Radiocarbon Intercomparison [TIRI] and Fourth International Radiocarbon Intercomparison [FIRI]).

The impetus for its production has been two-fold, the need for transparency in the work and the dissemination of the results beyond the participating laboratories to a wider community of laboratories and users.

As can be seen from the lists within the issue of participating laboratories, the ^{14}C community has embraced these intercomparisons with a great deal of enthusiasm, and commitment since the experimental effort involved is not inconsiderable and has usually taken place over a relatively short period of time. In the 20 years during which the Glasgow group has been involved in their organization, the participation rate in the intercomparisons has reached over 75% of operational ^{14}C laboratories worldwide and the reference materials now reach all parts of the globe, so truly an

international effort. A suite of reference materials (all natural) and spanning the applied ^{14}C timescale has been created for the benefit of the ^{14}C dating community.

ACKNOWLEDGEMENTS

A number of individuals and organizations should be thanked for their financial and other support (most notably those individuals who have provided sample material, often in amounts of tens of kg).

FOR TIRI:

Professor Mike Baillie, Queen's University of Belfast	wood
Mr Steinar Gulliksen, ^{14}C laboratory, NUST, Trondheim	whalebone and doublespar
Dr Andrew Dugmore and Mr Anthony Newton, Edinburgh University	Icelandic and Hekla Peat
Dr John Thomson, Institute of Oceanographic Sciences	turbidite
Dr Svein Jakobssen, Natural History Museum, Reykjavik	doublespar
Dr Anne Crone, AOC, Edinburgh	Crannog wood
Glengoyne Distillers	barley mash
Ms Ellen Ostvik, Flatanger Council, Norway	whalebone
Dr Kazimiercz Rozanski, IAEA, Vienna	cellulose
Drs Adrian M Hall, Graeme Whittington, Neil J Alexander	Fuglaness wood and Ellanmore peat
Dr R Preece, University of Cambridge	Tufa

FOR FIRI:

Professor Mike Baillie, Queen's University of Belfast	wood
Mr Steinar Gulliksen, ^{14}C laboratory, NUST, Trondheim	mammoth tusks
Dr Kh Arslanov, St Petersburg	mammoth tusks
Dr John Thomson, Institute of Oceanographic Sciences	turbidite
Dr Alan Hogg	Kauri wood
Dr Marco Spurk, Hohenheim	wood
Glengoyne Distillers	barley mash
Dr Ganna Zaitseva, St Petersburg	Dogee Barrow wood, leather
Dr Roy Switsur	wood

TIRI was only possible as a result of the financial support of the Natural Environment Research Council, UK. TIRI was organized by Marian Scott, Gordon Cook, Doug Harkness, and Philip Naysmith.

Funding for FIRI was provided by the Natural Environment Research Council, UK (Grant GR9/03389) and the European Commission under the SMT program of FPIV (SMT-CT98-2265).

A number of individuals were involved in the development and implementation of the FIRI program, including:

Gordon Cook and Philip Naysmith	SUERC ^{14}C laboratory, East Kilbride
Eddie McGee	^{14}C laboratory, University College Dublin
Israel Carmi and Elisabetta Boaretto	^{14}C laboratory, Weizmann Institute
Steinar Gulliksen	NTNU, Trondheim
Goran Possnert	Tandem accelerator laboratory, Uppsala
Mark van Strydonck	KIK ^{14}C laboratory, Brussels
Hans van der Plicht	^{14}C laboratory, University of Groningen
Jan Heinemeier	AMS laboratory, University of Aarhus
Doug Harkness and Charlotte Bryant	NERC ^{14}C laboratory, East Kilbride
Marian Scott and John McClure	University of Glasgow, Glasgow

FIRI was organized by Marian Scott, Gordon Cook, Doug Harkness, Philip Naysmith, and Charlotte Bryant.

THE FUTURE

Will there be a Fifth International Radiocarbon Intercomparison (VIRI)? The historical progression of ^{14}C laboratory intercomparisons from the Third (TIRI, completed in 1995) and Fourth (FIRI, completed in 2000) suggests that a Fifth (VIRI, completed in ??) should also be expected.

Criticisms of the design of TIRI and FIRI have included the need for the measurements to be made over a relatively short period of time (hence the workload within the laboratory is compromised), the fact that they provide only a snapshot in time and that the samples are not anonymous but that laboratories are. Can we do better?

A new program, VIRI, is being planned to address some of these criticisms while retaining some of the important features of TIRI and FIRI. One proposal being considered is that VIRI becomes a rolling and ongoing program, with a small number of samples being dispatched to participating laboratories each year. However, the frequency, number of samples, and their type within VIRI are still to be finalized after consultation with the community. The Glasgow group is committed to implementation of VIRI, which should commence in 2004.

On a personal note, first TIRI and then FIRI evolved from two earlier intercomparisons which I co-ordinated, and I would like to take this opportunity to thank two people especially who have been instrumental in this work.

Murdoch Baxter, as my doctoral supervisor, first introduced me to the world of ^{14}C dating and to my first experience of laboratory intercomparisons. That first intercomparison (ISG 1982) was small, involving only 20 laboratories, but with their support and help, the program developed. Today, 20 years later, those same 20 laboratories (almost) are still participating.

In the later intercomparisons, one other person played an important role and I would also like to thank Doug Harkness (now enjoying a well-earned retirement in Forfar) for sharing his knowledge of ^{14}C dating with me, and for playing a pivotal role in keeping the program on track.

I much appreciate all the support and trust which the ^{14}C community has placed in me. Without their willingness to participate, the intercomparison program would not be as strong as it is today.

E Marian Scott

Glasgow, July 2003

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Malcolm John Head

DR. JOHN HEAD IS VERY NEAR TO US

We suddenly heard the sad news that John Head had passed away, which made all of us in our institute deeply grief-stricken. We cannot believe it is true because I had just phoned him three days earlier. He told me that he had been well, except for high blood pressure. He was going to Xi'an, China with me after the 2003 Radiocarbon Conference.

The death of John means I have lost a good teacher and a helpful friend. It is even more of a great loss, not only for the State Key Laboratory of Loess and Quaternary Geology of China, but also for the whole radiocarbon community. He had cooperated with us for 19 years. His every word and action often appears before me and stays in my mind. It was in 1984 when I first met him in Guiyang. He gave a report about the chemical pretreatment of bone fossils and demonstrated it himself in the laboratory. Though he looked a little shy, he worked very carefully and hard. I knew at that time that he came from a well-known university in the Southern Hemisphere and he had been a radiocarbon expert with much practical experience over 20 years. However, I was a student at that time; I thought he was very far from me…

Actually, John is very near to us. When I first went abroad after the opening of China, I collaborated with the Australian National University in 1987 on the Loess chronology. John himself went to the Sydney airport to meet me, and then flew with me back to Canberra. I was deeply moved by this and have never forgotten, since he could have easily have just met me at Canberra airport. As soon as he met me at the Sydney airport, he excitedly told me that his laboratory had been successful in dating small samples with 100–200 mg carbon using a Quantulus counter and he suggested that it was necessary for Xi'an to build a method to date small samples using a liquid scintillation counter.

With the help of John, the method of small sample preparation for liquid scintillation counting was systematically built in our laboratory. This method was important in solving difficulties of small

sample dating in geology and archaeology, particularly as there was no AMS radiocarbon facility in those days.

The cooperation with John benefited us a great deal. It was he who introduced for the first time to Chinese scientists that the key to the reliability of ^{14}C dating depends on the physical and chemical pretreatment of samples. It was he who helped our laboratory to set up chemical methods of pretreatment for different samples. The methods included organic separation from paleosol, the separation of primary and secondary carbonate, and the extraction of wood cellulose and bone collagen. This was a great step forward in ^{14}C dating in China.

In recent years, John joined us in studying the characteristics of the Younger Dryas in monsoonal China and its spatial variations. We proposed that the Younger Dryas precipitation fluctuation was contributed to not only by the summer monsoon, but also by paleo ENSO. This gave a picture of the Younger Dryas pattern from high latitude to middle-low latitude areas, and provides a historic analogue for future prediction. This scientific work will always remind us of John.

He also made an important contribution to our laboratory in the education of the younger generation. All of our students graduated with degrees in geology and experimental laboratory work was their common weakpoint. He tirelessly, conscientiously, and meticulously taught them himself, step by step**,** until they learned and did well. He still spent a lot of spare time in helping Chinese scientists both in and out of our laboratory to revise their English manuscripts, including scientists such as Prof Chen Jun (Vice President of Nanjing University), Prof Peng Zhicheng (China University of Science and Technology, Chinese Academy of Sciences), and many others.

We remember not just a geochemist but a generous and kind personality and an unselfish scientist. Though a lot of frustrations and difficulties filled his life, he leaves a free, easy, and high-spirited figure for us to remember.

As an experienced expert in radiocarbon dating, he left our young people innovative scientific ideas, strict experimental protocols, and an enthusiasm to continue in the field.

John lives in our hearts forever. Let us turn the grief into strength and make more scientific contributions for the good of all mankind.

Weijian Zhou

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SECTION 1: THE FOURTH INTERNATIONAL RADIOCARBON INTERCOMPARISON (FIRI)

1.1 INTRODUCTION AND BACKGROUND TO FIRI

Radiocarbon dating is universally used as an essential dating tool in the archaeological and earth (Quaternary) sciences. The technique has enjoyed considerable success with ongoing developments in both the sophistication of experimental practice and an ever-widening range of applications. Most recently (since the 1980s), a new generation of laboratories has been created, based on the exploitation of accelerator mass spectrometry (AMS) for the differentiation and measurement of carbon isotope abundances in natural materials. Worldwide, there are over 100 ^{14}C laboratories now operational in universities, research organizations, museums, and as commercial enterprises. There is an inevitable diversity of experimental approaches and applied priorities within these facilities. Some are well established, while others are relatively recent members of the international ^{14}C community. Consequently, as a group, the laboratories reflect to varying extents the progress achieved over several decades of experience and methodological options. Furthermore, since progress in archaeology and related earth sciences cannot respect geographical and/or present political boundaries, there has been, and continues to be, an inevitable consequence that sample materials from specific cultural contexts are submitted to different laboratories and at different times. In this situation, the issue of comparability between results and amongst laboratories becomes paramount. Users of the results from ^{14}C dating are also concerned with the comparability and quality of laboratory results and the quality assurance programs that laboratories undertake are thus important in ensuring user confidence. The harmonization of measurements and the traceability of results to internationally recognized standards are also major goals of the program of work described in this special issue.

The need for a quantitative assessment of the comparability of ^{14}C dates from diverse laboratories is well recognized and a laboratory intercomparison gives laboratories an opportunity to perform an independent check on their internal quality assurance procedures. In particular, the Fourth International Radiocarbon Intercomparison (FIRI) aimed to define and improve the overall level of confidence in the direct comparison of information obtained from necessarily different measurement systems. This was achieved through a program that focused on assessing and establishing consensus protocols to be applied in the identification, selection, and sub-sampling of materials for subsequent analysis. A large-scale intercomparison then produced direct evidence on precision and accuracy. The intercomparison provided a quantification of some of the main sources of variation associated with the measurements. Finally, as a result of the intercomparison, a further series of ^{14}C reference materials has been created.

An intercomparison is generally considered to be the best scientific tool to determine the current status of laboratory comparability. In the first instance, this approach presents an invaluable opportunity to individual laboratories for checking procedures and results (i.e., it functions at an individual laboratory level as a sound foundation for formal quality control). Most importantly, however, it fosters a harmonization amongst laboratories while simultaneously providing an independent and verifiable measure of interlaboratory comparability for the user. From the statistical analysis of the pattern of results, individual laboratories are able to identify any systematic offsets and to quantify any additional sources of variation (above those already quantified from the intrinsic random nature of radioactive decay). Secondly, the intercomparison also offers vital information in the evaluation of the associated uncertainty statements with each analysis, since all sources of variation are incorporated and through appropriate design, it is possible to quantify the

source and magnitude of different contributors. Finally, dissemination of the outcome of the intercomparison and the highlighting of perceived issues build towards a better understanding between the provider laboratories and the multi-disciplinary user community.

In keeping with the principles of analytical science, ^{14}C laboratories have always been conscious of the importance of accuracy and precision for their reported results, i.e., the ethic of analytical quality control (QC), which in turn is the foundation for the wider concept of quality assurance (QA). This concern for good quality management within the ^{14}C community is exemplified by the care and effort given to establishing and maintaining primary standards and reference materials.

The internationally recognized primary standard is oxalic acid (NBS-OxI and subsequently NBS-OxII [now NIST]). While oxalic acid has all the physical and chemical attributes of a primary standard (e.g., homogeneity, high purity, stability in storage, a constant and known gram molecular weight, etc.), the quantitative and fractionation free recovery of its component carbon (initially as CO_2) has been problematic and the calibration of a material that is compositionally closer to the vast majority of samples submitted for ^{14}C analysis would be advantageous.

International efforts have resulted in the creation of a second tier of materials, so called secondary standards or reference materials. These include materials such as ANU-sucrose and the IAEA quality control reference series (Rozanski et al. 1992). They have 2 main functions: i) for calibration, to demonstrate traceability; and ii) for quality control, to verify the performance of a laboratory.

For quality control purposes, a reference material is commonly a natural material, so that it behaves as similarly as possible to the samples being measured. Therefore, most reference materials must be certified on the basis of measurement by several laboratories, using different methods and using an independent calibration. Certification is only possible when agreement among laboratories performing the measurements can be demonstrated, usually in an interlaboratory comparison.

As early as 1989, Long and Kalin (1990) stressed that it was incumbent on individual ^{14}C laboratories to engage in a formal program of quality assurance (QA), while Polach (1989) noted that the opportunity for internal checking by individual laboratories in routine ^{14}C measurement was hampered by a lack of suitable quality control (QC) and reference materials. Over an extended period of time, the ^{14}C dating community has created a set of reference materials, resulting from a series of voluntary international interlaboratory comparisons. The work in this issue builds on the previous laboratory intercomparisons that have taken place over the last 20 years (ISG 1982; Scott et al. 1991; Rozanski et al. 1992; Gulliksen and Scott 1995), and has created a further series of additional reference materials that are available to the ^{14}C community.

1.2 GENERAL OUTLINE OF THE SPECIAL ISSUE

This special issue is devoted to the design and analysis of FIRI (and to a lesser degree, to the Third International Radiocarbon Comparison—TIRI—see "Part II" section) and has 4 main components:

1. Description of the selection, collection, and preparation of samples for use in the intercomparison.
2. Design and organization of the laboratory intercomparison;
3. Analysis and interpretation of the results;
4. Discussion of the future needs in the community and how the further tier of reference materials created can be used.

1.3 THE PRINCIPLES IMPLICIT IN THE DESIGN AND ORGANIZATION OF A LABORATORY INTERCOMPARISON

1.3.1 Selection of Samples for Reference Materials to be Characterized in an Intercomparison

For quality control purposes, a reference material is commonly a natural material so that it behaves as similarly as possible to the samples being measured. To ensure the widest possible practical advantages, the materials should be representative of routinely dated materials and their ages should span the full range of the applied ^{14}C timescale. These materials are typically certified on the basis of a laboratory intercomparison; therefore, when selecting samples for an intercomparison, their dual purpose must be considered. Given the importance of ^{14}C dating in chronology construction, ideally some of the samples should be independently dated. The most appropriate material for this purpose is dendrochronologically-dated tree-ring sequences, which are already used to underpin the absolute calibration of the conventional ^{14}C timescale back to approximately 9000 yr BP. Also, because of the considerable use of ^{14}C dating within routine archaeological investigations, several samples should be of archaeological significance. Therefore, several samples were included of particular compositional or contextual interest to satisfy the large archaeological user community. Further, given the long history of intercomparisons in the ^{14}C community, it is also important that samples should link any new intercomparison to past studies. In this way, continuity of laboratory performance can be assessed. Available materials in this category include a marine turbidite, Southern Hemisphere wood, and grain. In this way, a catalog of possible samples can be compiled (Section 1.4.3), the samples collected, and a final decision concerning their use made only after appropriate testing.

1.3.2 Preparation and Testing of Material: General Principles

An essential requirement when using a naturally occurring material as the basis for analytical intercomparison is homogeneity. In some instances, this may require that the material be chemically homogenized. However, in the case where the question of interest concerns the variation in the sample results, the raw material can also be provided to allow direct quantification of the natural variation within a typical raw sample and the extent, if any, to which the preselection procedures influence this.

Within FIRI, dendrochronologically-dated wood samples, by their nature, typically required no preparatory treatment, other than cutting into identical (in tree-ring terms) components. For any non-dendro-dated wood, the material was finely chopped and the cellulose component extracted. This was finely ground, homogenized, and pelleted.

For other materials, the degree of preparation varied from a thorough physical mixing (e.g., a turbidite), to grinding and mixing (a whole peat), to complete chemical homogenization (a humic acid extraction from peat). The materials were then packaged and archived.

All bulk materials, after being homogenized, should be checked by replicate analyses on randomly selected aliquots before distribution to the intercomparison participants. Materials should also be tested at different sub-sample sizes (reflecting one of the key differences between AMS and radiometric measurement). In FIRI, all materials were prepared in one batch and a number of randomly selected sub-samples were taken for testing.

1.4 THE PLANNED LABORATORY INTERCOMPARISON (FIRI)

The planned analytical laboratory intercomparison was similar in design to 2 previous ^{14}C studies (TIRI: Gulliksen and Scott 1995, Scott et al. 1998; and IAEA: Rozanski et al. 1992) and included existing reference materials.

1.4.1 Number of Samples and Study Design

The number of samples had to be balanced between the requirements for meaningful statistical analysis of the data and, of course, the practical commitments of the participating laboratories. It was intended that the study should include a degree of replication (with the identity of replicates withheld from the participating laboratories) to allow a direct assessment of within-lab variation (or repeatability). A final figure was agreed of 10 samples to be analyzed within a 1-yr period. A detailed protocol for the reporting of results was also prepared for distribution to all participants. Specific requests to laboratories, including details of how they calculate the error term associated with a ^{14}C age, were made at this time.

1.4.2 Specific Aims and Objectives for FIRI

The fundamental aims and objectives of FIRI can be simply summarized:

- Demonstration of the comparability of routine analyses carried out by both AMS and radiometric laboratories;
- Quantification of the extent of, and sources of, any variation;
- Investigation of the effects of sample size, pretreatment, and precision requirements on the results.

The design structure was rather simple: the intercomparison included core samples, which all laboratories will measure, and optional samples, representing "typical" materials.

The sample selection criteria were relatively simple to express, but more difficult to satisfy due to the quantity of material required. The criteria were the following:

i. Homogeneity in ^{14}C activity, either as a natural property or artificially induced;
ii. The samples' activities should span the activity range from "modern" to "close to background";
iii. Some duplicates should be incorporated;
iv. Some of the samples should form a link to past exercises;
v. Samples should be available in sufficient quantity to enable excess material to be retained for archiving as new reference materials;
vi. Most materials should be suitable for measurement by both AMS and radiometric laboratories
vii. All samples should be natural and several should be dendrochronologically-dated wood.

These criteria translated into samples which included the following: (i) dendrochronologically-dated wood samples with a limited number of rings or drawn from a plateau on the calibration curve; (ii) samples with only a short growing period, and (iii) samples that could be chemically treated and physically homogenized in bulk.

1.4.3 Catalog of Potential Samples for FIRI

A set of potential samples was first identified and collected. The intention after this stage was to identify a subset of these materials that would form the core samples. Any unused samples could be archived or used for optional samples.

1.4.3.1 Dendro-Dated Belfast Wood I

Professor M Baillie of the Queen's University of Belfast Dendrochronology laboratory provided 11.8 kg of dendro-dated wood. The sample was from a Scots pine tree from Garry Bog, Co. Antrim, Northern Ireland with sample identification number of Q7780. The grid reference for the site is C930074, latitude 54°54′N, longitude 6°33′W.

Age: The sample had 40 annual growth rings dating from 3239 BC to 3200 BC.

1.4.3.2 Dendro-Dated Hohenheim Wood

The dendrochronology laboratory of the University of Hohenheim (Dr M Spurk) provided 9.6 kg of dendro-dated oak. The sample identification number was Pettstadt 262.

Age: The sample had 20 annual growth rings dating from 313 BC to 294 BC.

1.4.3.3 New Zealand Kauri Wood

A sub-fossil Kauri wood sample was obtained from Dr A Hogg of the University of Waikato. A previous Kauri wood sample was used by the IAEA (IAEA-C4) (Rozanski et al. 1992), but this sample was not identical. It was expected that this sample would function as a "close to background" sample (Hogg et al. 1995), being at least 40 kyr. Seventy kg of the sample was received as 4 slabs.

Approximate age: close to infinite age with respect to ^{14}C.

1.4.3.4 Russian Wood from Dogee Barrow

A wood sample (part of a log) of approximately 10 kg covering around 190 annual rings from the burial mound of Dogee Barrow, grave 8, (the Tuva king barrows from Scythia) was provided by Dr G Zaitseva of the Institute of the History of Material Culture. The material was excavated in 1998 and was very degraded. Its approximate age was 2300–2400 BP (Sementsov et al. 1998). The sample had not been dendro-dated, and would require careful homogenization since the calibration curve has a steep slope at this time.

Approximate age: less than 1 half-life.

1.4.3.5 Cambridge Modern Wood

A sample of oak (*Quercus robur*) was obtained from Dr R Switsur of the Godwin Institute for Quaternary Research. The tree was planted around AD 1722 and the material corresponding to the period AD 1820–1880 (a relatively flat area on the calibration curve) was removed to provide a sample of 10.4 kg.

Age: modern

1.4.3.6 Belfast Wood II

A further bulk sample of a similar age to Belfast I was also made available. In fact, the second Belfast sample spanned a contiguous set of rings. This sample was used for pretreatment investigation. The sample, which had a finite 40-yr ring span, was again supplied by Prof M Baillie, The Queen's University of Belfast. The sample was 16.3 kg of Scots pine from the Garry Bog, Co Antrim, Northern Ireland, with a sample identification number of Q7780 was provided.

Age: The dendrochronologically determined age span was 3299 BC–3257 BC.

1.4.3.7 Turbidite Carbonate

This sample was supplied by Dr J Thomson, Southampton Oceanography Centre. The sample was mainly coccolith calcite from a single distal turbidite emplaced on the Maderia Abyssal Plain. A remarkable feature of these turbidites is their homogeneity. The basal layers are graded and inhomogeneous, but are overlain by relatively thick deposits. The material used in this study was derived from the middle ungraded deposit and was considered homogeneous. This turbidite was used in the Third International Radiocarbon Intercomparison (TIRI) as the optional Sample K and 25 laboratories had measured it.

Approximate age: 3 half-lives

1.4.3.8 Ellanmore Peat

This sample was again prepared for TIRI (Sample H) but was not used as a core sample. Twenty-seven laboratories chose to analyze it.

It is finely-ground peat from a well-defined stratigraphic section. The Ellanmore peat occurs as an about 50 cm thick horizon intercalated with glacial diamicts and is exposed in a stream bank section of the Reisgill Burn, Ellanmore, Caithness, Scotland (58°18′N, 3°17′W; Natl Grid Ref ND 237 370).

Approximate age: 2 half-lives

1.4.3.9 St Bees Head (Whole Peat) and St Bees Head (Humic Acid)

This sample is from a coastal cliff deposit at St Bees Head in Cumbria, northwestern England (54°29.5′N, 3°37.5′W; NGR NX 9472 1196), which had been exposed by erosion. The apparently well-humified felted peat deposit is approximately 0.5 m thick, and is overlain by several meters of lacustrine material of Holocene age that is largely mineral in nature. Approximately 20 kg of peat were collected and taken back to the laboratory for pretesting. The site was subsequently re-sampled and approximately 30 kg of peat from a slightly different elevation was collected.

Approximate age: 2 half-lives

1.4.3.10 Modern Barley Mash

A modern sample of barley mash provided in the previous exercise (TIRI) was available. Additionally, a new sample was collected from Glengoyne Distillery, just outside Glasgow. The sample comprises a barley grain residue that is left after fermentation and, as such, is a by-product from the manufacture of malt whiskey. The sample represents a single year's growth (1998) and we collected 20 kg of the sample.

Age: modern

1.4.4 Proposed Treatment of Samples Before Testing and Dispatch

Pretesting of the samples was carried out in the Scottish Universities Environmental Research Centre (SUERC) Radiocarbon Laboratory and at the Natural Environment Research Council (NERC) ^{14}C Laboratory.

1.4.4.1 Kauri Wood

For the FIRI samples, 2 slabs were taken and sliced into sub-samples of approximately 50 g. All participating radiometric laboratories would receive samples of 50 g. For AMS laboratories, 10 of

the 50 g sub-samples, were selected at random and sub-divided into samples of approximately 5 g. Each AMS laboratory received 5 g samples. No pretreatment was carried out on this material. The 2 remaining slabs were sent to the IAEA for archiving.

Both the SUERC and NERC laboratories carried out pretesting of this sample (Table 1.1).

Table 1.1 Pretesting results (pMC ± 1σ) for the Kauri wood sample

Sample	Test 1	Test 2	Test 3	Test 4
Kauri (A and B)	0.15 ± 0.27	0.11 ± 0.26	0.14 ± 0.26	0.12 ± 0.25
	Test 5	Test 6	Test 7	Test 8
	0.11 ± 0.11	0.20 ± 0.11	0.22 ± 0.11	0.30 ± 0.11

A total of 8 analyses were made on this sample and a weighted mean value of 0.2 ± 0.05 pMC was determined. The 8 samples were selected at random from the bags, which were waiting to be sent out to participating laboratories.

1.4.4.2 Turbidite Carbonate

The sample had been stored in a sealed air-tight container since the last intercomparison study (TIRI), to limit interaction with atmospheric CO_2. The sample was remixed thoroughly before bagging. Approximately 100-g samples would be sent to each radiometric laboratory and 10 g to each AMS laboratory. No pretreatment was carried out on this sample and laboratories were advised not to pretreat it in any way.

Four test analyses were carried out on this sample. Test 4 was an AMS result in which the graphite target was prepared at SUERC and the final measurement was made at the NSF-AMS facility in Arizona. The consensus value for the turbidite sample from the TIRI study is 18,155 BP, while the weighted mean of the 4 results presented here is 18,150 ± 90 BP (Table 1.2).

Table 1.2 Pretesting results (yr BP ± 1σ) for turbidite Sample C

Sample	Test 1	Test 2	Test 3	Test 4
Turbidite (C)	18,305 ± 180	18,010 ± 180	18,220 ± 165	18,050 ± 190[a]

[a]indicates an AMS result

1.4.4.3 Belfast Dendrochronologically-Dated Wood I

The sample was chopped with a clean chisel to give approximately 40-g sub-samples for radiometric laboratories and 4-g sub-samples for AMS laboratories. No further pretreatment was undertaken on this sample. It was felt that there was no need to carry out any pretesting on this sample because of its provenance.

1.4.4.4 Humic Acid

A sub-sample of St Bees peat was pretreated to produce humic acid and humin. Four tests were carried out on this sample and the fractions were dated radiometrically. Table 1.3 shows the results. It can be noted from the results that there were indications of age differences between the humic acid and the humin. The weighted mean value for humic acid age = 11,180 ± 50 BP, while for humin, the weighted mean age = 11,500 ± 115 BP.

Such differences had already been observed in results from a nearby profile (Doug Harkness, personal communication). Therefore, it was decided not to use the whole peat sample as a core sample because different pretreatments that laboratories employ could lead to an additional source of variation in age. A second peat sample was collected from the site for humic acid extraction to form Sample E.

Table 1.3 Pretesting of humic acid and humin fractions (yr BP ± 1σ) from the first St Bees Peat sample

Sample	Test 1	Test 2	Test 3	Test 4
St Bees humic	11,220 ± 90	11,270 ± 110	11,190 ± 100	11,040 ± 100
St Bees humin	11,600 ± 200	11,450 ± 300	11,610 ± 250	11,350 ± 210

The pretreatment for humic acid extraction was as follows:

- The sample was digested in 2% KOH at 80 °C to solubilize the humic acid. The sample was then filtered and re-extracted. A total of approximately 150 liters of humic acid solution were extracted from the peat.
- The humic acid was precipitated by adjusting the pH to below 3 by stirred additions of 2 M H_2SO_4. The humic acid was recovered by filtration through glass fiber filter papers and then washed with cold distilled water and dried.
- The humic acid was then re-dissolved in KOH, re-precipitated, washed, and dried to produce a crystalline humic acid material. This was then sub-sampled to 10 g for radiometric laboratories and 1 g for AMS laboratories. (The first peat sample was subsequently archived.)

1.4.4.5 Barley Mash

In the laboratory, the sample was force dried and physically mixed. It was then sub-sampled, giving 50 g for radiometric laboratories and 5 g for AMS laboratories.

This sample was pretested by selecting 2 bags randomly from the sub-samples. The results of the analyses are presented in Table 1.4. The weighted mean value is 110.3 ± 0.8 pMC.

Table 1.4 Pretesting results (pMC ± 1σ) for the barley mash, Samples G and J

Sample	Test 1	Test 2
Barley mash (G and J)	111.0 ± 0.6	109.5 ± 0.6

1.4.4.6 Hohenheim Dendrochronologically-Dated Wood

The sample was chopped with a clean chisel to produce pieces of approximately 40 g for radiometric laboratories and 4 g for AMS laboratories. This sample was not pretested because it was of a known age.

1.4.4.7 Belfast Wood II and Cambridge Cellulose, Pretreatment/Preparation

For both samples, fine shavings were produced by planing the wood with a power plane. The samples were then pretreated using a standard acid/alkali/alkali/acid scheme, followed by bleaching with a solution of sodium chlorite in hydrochloric acid. The bleaching process was repeated and the samples washed with distilled water until they were white in color. The samples were dried at 40 °C to leave a white cellulose fraction. The samples were then mixed and sub-sampled to 10 g for

radiometric laboratories and 1 g for AMS laboratories. No pretesting was carried out since these samples were of known age.

The remaining samples which had been identified, although included in the catalog, were not pretested since, *a priori*, they did not meet all the selection criteria. They were considered as potential optional samples.

1.4.4.8 Dogee Barrow Wood, Pretreatment/Preparation

This sample was milled, acid/alkali washed, and further physically homogenized and mixed before testing. Homogenization was demanding due to the nature of the calibration curve corresponding to its approximate age.

1.4.4.9 St Bees Head Whole Peat, Pretreatment/Preparation

A bulk peat sample (~30 kg) was cut from a 10-cm-depth increment from within the 0.5 m deposit. The sample was broken up roughly and dried in an oven at approximately 50 °C. Upon drying, the sample was further broken up and then ground to a fine powder. The sample was then thoroughly mixed several times to produce a homogeneous sample.

1.4.4.10 Ellanmore Whole Peat, Pretreatment/Preparation

The peat was air dried at room temperature, ground to a fine powder, and thoroughly mixed to produce a homogeneous sample. This material, as provided, contains about 40% by weight of carbon.

1.4.5 Optional Samples

A series of other materials were also gathered and are described below (their provenance and expected age). Two sets were considered: those for AMS laboratories only (due to the limited quantities) and those suitable for AMS and radiometric laboratories. These samples were not considered potential core samples.

1.4.5.1 Mammoth Tusk (Supplied by Mr S Gulliksen, NUST, Trondheim)

The total weight of the sample was approximately 265 g, (the carbon content was 3.2% of the bone weight). Although the dentin looked very dense and probably would be hard to contaminate to any depth, there were longitudinal cracks that could carry contaminants to attack the "inner" surfaces.

The geological setting of the find was published by Mangerud et al. (1999): *Boreas* (28):46–80. The tusk was found in 1997 by Herbjørn Heggen and Jan Mangerud in a diamicton in a section at the locality of Byzovaya in Pechora, Republic of Komi, in the northern part of Russia.

Approximate age: >5 half-lives.

1.4.5.2 Mammoth Tusk (Supplied by Prof Kh A Arslanov, St Petersburg University, Pechora Project)

This mammoth bone, (Pechora 98-2092), was collected by Valery Astakhov from the right bank of Ute-Yaha river (tributary to More-Yu river). The sample was picked up close to a section of thick aeolian sand covering the local till at 67°12′N, 59°45′E.

Approximate age: close to background.

1.4.5.3 Mammoth Tusk (Supplied by Prof Kh A Arslanov, St Petersburg University, Pechora Project)

A mammoth bone collected by V Makeev from the left bank of the Balyktarkh river, Kotelny Island, Novosibirsk Ostrova.

Approximate age: >2 half-lives.

1.4.5.4 Leather (Supplied by Dr G Zaitseva, Institute of History of Material Culture)

This sample comprises the remains of leather clothes found on a mummified skeleton in the Dogee Barrow (grave 6). Textiles from the same burial have previously been dated. The leather has not undergone any preservation treatment.

As well as these samples, a number of other samples were also available but did not formally form part of FIRI. These included parchment (donated by Asher Jacbob, Jerusalem), woollen fabric (Coptic textile), a textile from a late Scythian barrow, as well as other mammoth tusks.

1.5 HOMOGENEITY TESTING

The key sample requirement in an intercomparison is that of homogeneity, which simply means that any sub-sample taken randomly from the bulk material is representative of that material, and that when dealing with trace element assay, that the trace element is uniformly distributed throughout the bulk material. Individual sub-samples should, therefore, have the same properties. Clearly, these are stringent requirements and we describe briefly the testing of samples for inclusion in the intercomparison.

It was decided that not all samples (in particular, the whole wood samples) would require to undergo full homogeneity testing, but all others would be tested. Those samples that were not included in the full homogeneity testing still underwent screening by 2 laboratories (SUERC and NERC).

1.5.1 Test Design

It was agreed that each material would be independently tested in 2 laboratories (where possible a radiometric and accelerator mass spectrometric laboratory), and that a homogeneity testing protocol would accompany each sample. It was agreed that a minimum of 8 analyses was required for homogeneity testing of each core material. Aliquots were labelled "1 of 8", "2 of 8", and so on. This same convention was used by the testing laboratory when returning results. The next section details the material testing protocol and the laboratories which dated the materials

1.5.2 Homogeneity Testing Laboratories

Eight laboratories were involved in the homogeneity testing. They are listed below:

Table 1.5 Homogeneity testing laboratories

Name	Method
SUERC ^{14}C laboratory, East Kilbride	Liquid scintillation counting (LSC)
^{14}C laboratory, University College Dublin	LSC
^{14}C laboratory, Weizmann Institute	LSC
NTNU, Trondheim	Gas proportional counting (GPC)
Tandem accelerator laboratory, University of Uppsala	Accelerator mass spectrometry (AMS)
KIK ^{14}C laboratory, Brussels	LSC
^{14}C laboratory, University of Groningen	AMS
AMS laboratory, University of Aarhus	AMS
NERC ^{14}C laboratory	LSC

1.5.3 Sample Testing Protocols

1.5.3.1 Glengoyne Barley Mash

Homogeneity testing was to be carried out by radiometric analyses at the SUERC ^{14}C laboratory, East Kilbride, and the ^{14}C laboratory, University College Dublin.

Eight aliquots, each of 100 g, were provided. Each aliquot contained sufficient material for a number of analyses. Instructions included that a sub-sample, representative of the whole, was to be taken and measured and that the samples were not to be pretreated.

1.5.3.2 Belfast Cellulose

Homogeneity testing was to be carried out by radiometric analyses at the SUERC ^{14}C laboratory, East Kilbride, and at the ^{14}C laboratory, Weizmann Institute.

Aliquots of 15 g were provided. If necessary, a sub-sample, representative of the whole, should be taken and measured. The samples were not to be pretreated.

1.5.3.3 Turbidite Carbonate

Homogeneity testing was to be carried out by radiometric and AMS analyses at the National C-14 laboratory, NUST, Trondheim, andthe Tandem accelerator laboratory, University of Uppsala.

For radiometric laboratories, each aliquot contained sufficient material for a single analysis, while for AMS laboratories, each aliquot contained sufficient material for a number of analyses. In this latter case, a sub-sample, representative of the whole, was to be taken and measured. The samples were not to be pretreated.

1.5.3.4 Cambridge Cellulose

Homogeneity testing was to be carried out by radiometric analyses at the KIK ^{14}C laboratory, Brussels, and by AMS at the ^{14}C laboratory, University of Groningen.

Aliquots were provided of approximately 13 g for radiometric measurement and 2 g for AMS. If necessary, a sub-sample, representative of the whole, was to be taken and measured. The samples were not to be pretreated.

1.5.3.5 St Bees Humic acid

Homogeneity testing was to be carried out by radiometric and AMS analysis at the NERC ^{14}C laboratory, East Kilbride, and the AMS laboratory, University of Aarhus.

For radiometric laboratories, each aliquot (10 g) contained sufficient material for a single analysis, while for AMS laboratories, each aliquot (1 g) contained sufficient material for a number of analyses. In this latter case, a sub-sample, representative of the whole, was to be taken and measured. The samples were not to be pretreated.

1.5.4 Reporting Results

Each laboratory was instructed as follows:

Results for each analysis were to be reported as the measured enrichment relative to the NBS oxalic acid standard ($\delta^{14}C$), the conventional age BP, and % modern, each with its 1σ error. The $\delta^{13}C$ should also be reported (if estimated, then this should be noted). For the purposes of homogeneity

testing, we requested that the laboratories measure the samples to as high a precision as reasonably achievable.

1.5.6 Homogeneity Results

1.5.6.1 Pretesting

The prescreening analyses indicated that the samples had ages spanning the timescale required and showed no signs of gross in-homogeneity. Thus, the samples, other than the whole wood, went forward for full homogeneity testing as detailed in the protocols in Section 1.5. The full set of homogeneity testing results are given in Table 1.6.

Table 1.6 Homogeneity results[a]

Sample	1	2	3	4	5	6	7	8
Turbidite (radiometric)	18,069 ± 96	18,093 ± 69	18,109 ± 102	18,245 ± 90	18,120 ± 100	18,314 ± 94	18,087 ± 96	18,219 ± 57
$\delta^{13}C$	1.3	1.2	1.2	1.35	1.48	1.3	1.3	1.3
Turbidite (AMS)	18,745 ± 80	18,555 ± 95	18,655 ± 85	18,505 ± 85	18,510 ± 75	18,765 ± 85	18,655 ± 85	18,500 ± 90
$\delta^{13}C$	0.9	1.0	1.0	0.9	1.1	0.9	0.7	1.0
Humic (radiometric)	11,855 ± 50	11,855 ± 50	11,870 ± 50	12,020 ± 50	—[b]	11,875 ± 50	11,925 ± 50	11,975 ± 50
$\delta^{13}C$	–29.0	–28.9	–29.4	–29.4	—	–29.0	–29.0	–29.0
Humic (AMS)	11,790 ± 55	11,857 ± 55	11,925 ± 60	11,875 ± 60	11,940 ± 70	12,005 ± 55	11,895 ± 60	11,910 ± 75
$\delta^{13}C$	–28.81	–28.79	–28.80	–28.81	–28.82	–28.85	–28.85	–28.87
Barley mash (1) (radiometric)	111.0 ± 0.34	110.5 ± 0.36	110.4 ± 0.36	110.3 ± 0.36	111.4 ± 0.30	111.2 ± 0.27	110.6 ± 0.35	111.1 ± 0.32
$\delta^{13}C$	–29.2	–29.2	–29.1	–29.3	–29.1	–29.2	–29.2	–29.2
Barley mash (2) (radiometric)	111.1 ± 0.5	111.0 ± 0.5	111.2 ± 0.5	111.0 ± 0.5	110.7 ± 0.5	110.9 ± 0.5	110.9 ± 0.5	110.8 ± 0.5
$\delta^{13}C$	–28.9	–28.9	–28.9	–28.9	–28.9	–28.9	–28.9	–28.9
Bcellulose (1) (radiometric)	4540 ± 45	4410 ± 45	4450 ± 50	4520 ± 45	4495 ± 50	4475 ± 40	4340 ± 40	4525 ± 40
$\delta^{13}C$	–23.4	–23.2	–22.6	–22.1	–23.0	–23.0	–23.0	–22.8
Bcellulose (2) (radiometric)	4430 ± 60	4400 ± 70	4480 ± 60	4350 ± 80	4390 ± 70	4510 ± 60	4470 ± 60	4510 ± 60
$\delta^{13}C$	–23.6	–23.5	–23.5	–23.6	–23.7	–23.6	–23.6	–23.6
Cellulose (AMS)	99.92 ± 0.6	99.30 ± 0.6	99.62 ± 0.6	98.87 ± 0.6	98.52 ± 0.4	97.65 ± 0.55	98.91 ± 0.38	99.06 ± 0.39
$\delta^{13}C$	–23.2	–24.5	–25.8	–25.1	–25.2	–18.3	–25.5	–25.2
Cellulose (radiometric)	98.36 ± 0.16	98.55 ± 0.19	98.01 ± 0.60	98.31 ± 0.39	98.11 ± 0.95	98.38 ± 0.86	97.94 ± 0.30	98.25 ± 0.25
$\delta^{13}C$	–23.81	–24.72	–24.14	–25.25	–25.1	–25.02	–25.02	–24.84

[a]barley mash and cellulose given in pMC, all others in age (BP)
[b]sample lost

1.5.6.2 Analysis of the Results

The results of the homogeneity testing are summarized in Table 1.7, which shows the summary statistics for each series.

Table 1.7 Basic descriptive statistics

Variable	Mean	Median	Standard deviation
Turbidite (R)[a]	18,157 BP	18,114	90
Turbidite (A)[b]	18,611 BP	18,605	109
Humic(R)	11,905 BP	11,875	72
Humic (A)	11,902 BP	11,902	62
Barley (R1)	110.8 pmC	110.8	0.40
Barley (R2)	110.95 pmC	110.95	0.16
Cellulose (A)	99.106 pmC	99.18	0.761
Cellulose (R)	98.239 pmC	98.28	0.205
Bcellulose (R1)	4469 BP	4485	67.6
Bcellulose (R2)	4442 BP	4450	59.2

[a]*R* indicates radiometric
[b]*A* indicates AMS measurement

From the table, it can be seen that the mean age for each sample pairing with the exception of the turbidite is in good agreement. For the turbidite sample, there appears to be a difference, on average, of approximately 500 yr.

The $\delta^{13}C$ results were also measured by the participating laboratories and showed consistent results.

A test of homogeneity was carried out for each series separately based on the sum of the squared standardized residuals about the mean value. Under the hypothesis that the set is homogeneous, each test statistic should have a χ^2 distribution with (n–1) degrees of freedom, where *n* is the number of observations. The results of this test are shown in Table 1.8. For all samples, except the humic, the critical value for the test statistic is 14.07 (for humic it is 12.6). It is clear that all individual laboratory sets are homogeneous, with the exception of the Belfast cellulose (R1). The non-homogeneity in this series is likely due to the relatively small errors quoted for each measurement in that set.

Table 1.8 Homogeneity test

Sample	T(R)	T(A)	H(R)	B(R1)	B(2)	C(A)	C(R)	BC(R1)	BC(R2)
Test statistic	7.51	11.9	12.5	8.6	0.7	13.7	4.5	18.3	5.4
Result	H[a]	H	H	H	H	H	H	Non-H[b]	H

[a]H: homogeneous
[b]non-H: inhomogeneous

The summary values—mean and median difference, the standard deviation (StDev), standard error (Semean), the minimum (Min), and maximum (Max)—for the differences between the duplicate pairs are shown in Table 1.9

Table 1.9 shows evidence of a significant difference between the 2 sets of turbidite analyses (95% confidence interval for the average difference of –573.6 to –334.9) and the modern cellulose sets (0.212–1.523). All others are in agreement within error.

Table 1.9 Descriptive statistics for differences between the matched aliquots

Variable	Mean	Median	StDev	Semean	Min	Max
Turbidite	–454.3 BP	–456.3	142.27	50.4	–676	–260
Humic	8.6 BP	30.0	94.3	35.7	–130	145
Barley	–0.15 pmC	–0.20	0.524	0.185	–0.8	0.7
Cellulose	0.868 pmC	0.890	0.784	0.277	–0.73	1.61
Bcellulose	26.9 BP	12.5	96.8	34.2	–130	170

Reasons for these apparent differences were then sought.

1.5.7 Issues Raised as a Result of the Testing

1.5.7.1 Turbidite Sample

The turbidite sample had been pretreated in one laboratory and not in the other; thus, it became clear that the sample showed a statistically significant difference dependent on the pretreatment. However, it was encouraging that within each laboratory's procedure, there was no evidence of inhomogeneity. It was decided to carry out further analyses on this sample, which are reported in the following. This analysis focused on the small sample properties, and so was solely carried out by AMS.

The total chemistry and measurement background were checked by leaching an Icelandic carbonate (doublespar) before and after the 8 turbidite samples. The results, representing the 2 graphitization reactors used, were 45,490 ± 980 BP; 44,455 ± 950 BP; and 45,770 ± 880 BP; 50,010 ± 1115 BP, respectively.

The quality assurance (QA) was also performed by simultaneous preparation and measurement of a reference humic acid sample with a consensus value of 3352 ± 6 BP. The result was 3325 ± 55 BP.

Both QA checks confirmed that the laboratory procedures were stable.

In the 2nd test, 1 large turbidite sample was leached with 0.5 ml HCl. This outermost fraction was dated to 14,290 ± 135 BP. One sample was totally leached with 2M HCl and dated to 18,070 ± 100 BP. A 3rd sample was leached in 2 separate steps and the results for the 1st and 2nd fraction gave 17,820 ± 95 BP and 18,445 ± 105 BP, respectively.

In a 3rd test, the 50% inner fraction, as well as the total sample, were analyzed as:

Sample	Inner	Total
1	18,495 ± 75	18,020 ± 95
2	18,500 ± 85	18,150 ± 110

These results are consistent with the former measurements and give consensus values for total sample and inner 50% fractions of 18,073 ± 58 BP and 18,579 ± 26 BP, respectively.

The turbidite was also analyzed by x-ray diffraction; 80–85% consisted of calcite (aragonite and dolomite were <1%), 10–15% of quartz, and a few % of feldspar.

An attempt to mechanically separate different grain size fraction by vibration and sliding indicated that the material was too finely powdered to make such a partitioning possible in a simple way (normal sample handling).

Thus, it was concluded that the apparent large age differences reflected purely a pretreatment effect, and that consistent application of the no pretreatment instruction would avoid any problem with this sample.

A further detailed study of this sample also revealed the large radon (^{222}Rn) content of the gas released from this sample. Interim counting of one of the samples had indicated that the gas required storage for a minimum of 60 days to reduce the age shift due to radon contamination to less than 30 yr.

1.5.7.2 Modern Cellulose

The most likely cause of the apparent small difference in the results was due to the use of an in-house modern reference material by the AMS laboratory, which was slightly offset relative to the primary modern standard of NIST OXII.

1.5.7.3 Conclusions

Finally, the sets of results from the 2 laboratories for each material were then combined for homogeneity testing. The humic acid, Belfast cellulose, and barley mash were all homogeneous, but the other materials did not comply, as a result of the significant interlaboratory differences that were identified. The dendrochronologically-dated wood sample (modern cellulose) comes from a well-defined and limited time span, so that the lack of homogeneity observed in this sample likely relates to interlaboratory differences, rather than an intrinsic property of the material.

Notwithstanding these difficulties, the results of the homogeneity testing had indicated that all of the samples—when laboratories complied with specific instructions concerning sample handling and pretreatment—could be considered as homogeneous and, thus, suitable for use in the intercomparison. The homogeneity testing had demonstrated some of the difficulties that could be expected in the full exercise, particularly, the importance of the laboratory calibration to the modern primary standard.

1.6 FINAL SAMPLE LIST

A short list of 7 materials was identified as core samples having met the criteria previously identified and is given in Table 1.10. Three sets of duplicate samples were provided blind (Kauri, Belfast wood, and barley mash). The optional sample list then included Cambridge cellulose (Sample K), Dogee Barrow wood (Sample L), St Bees whole peat (Sample M), 3 mammoth tusk samples (Samples N, O, and P), and leather (Sample Q).

Table 1.10 Core sample descriptions

Core sample description	FIRI code	Age/Activity
Kauri wood	A, B	Near background
Marine turbidite	C	~3 half-lives
Belfast dendro-dated wood	D, F	~1 half-life
Humic acid	E	~2 half-lives
Barley mash	G, J	modern
Hohenheim wood	H	<1 half-life
Belfast dendro-dated cellulose	I	~1 half-life

1.6.1 Instructions and Information Sent to All Participating Laboratories

The samples were then sent to laboratories in August/September 1999. A brief description of the pretreatment undertaken in the laboratory before dispatch and instructions concerning sample

pretreatment (if any) before dating were provided to the participating laboratories. The duplicate samples were not identified and laboratories were asked to treat the samples using their routine laboratory procedures.

A total of 120 sets of samples were dispatched.

For Samples A and B, the laboratories were told that these samples should be considered as close to, or beyond, the limit of ^{14}C detection.

For Sample C, laboratories were told that this sample should be fully hydrolyzed and no fractions should be measured. It was emphasized that this sample required no further pretreatment and that pre-etching had been shown to produce small, but significant, age differences. The laboratories were also informed that this sample should be stored in a sealed container.

1.7 ADDITIONAL INVESTIGATIONS

During the planning of the exercise, it was suggested that 2 separate investigations be undertaken relating to laboratory precision and sample size. Specific samples were identified as being appropriate for these exercises which are described below.

1.7.1 Laboratory Precision

It was suggested that Sample D should be dated to the highest precision possible within the laboratory. We recommended that cellulose be extracted for this study.

1.7.2 Sample Size

Typically, in intercomparisons, samples are provided in sufficient quantity and well-preserved; however, this does not always reflect the "real-life" laboratory situation. Therefore, it was suggested that Sample E, which is approximately 40% carbon, should be dated (where possible) using different sample sizes, and that the sample size (mass of carbon) should be clearly stated when the results were reported. It was suggested that laboratories report the result for Sample E based on their "optimal" sample size and one further result for a sub-sample of the raw material, which was at the minimum sample size for the laboratory to report a meaningful result.

This exercise would allow us to address the question of sub-sampling and the effect of sample size on the final result.

Both investigations were optional, but we believed they were valuable in providing important information.

1.7.3 Result Reporting

Report forms were included, detailing the information requested when returning results. Additional information could be added on separate sheets, if required. The deadline for the return of results was 31 August 2000, but this was subsequently extended to December 2000.

SECTION 2: THE RESULTS

2.1 Distribution of Samples

The sets of core samples were distributed to over 120 laboratories that had returned an original questionnaire seeking expressions of interest in participation. A reporting format for the results was also agreed and distributed to the laboratories at the same time. This is shown in Table 2.1. Laboratories were originally given 1 yr (i.e., to August 2000) to complete the analyses and return the results, but this was later extended to December 2000. In this section, we briefly describe the laboratory characteristics and the overall response rate of the participating laboratories.

Table 2.1 The agreed reporting format

1. Contact details
Laboratory name:
Contact person:
E-mail address:
Number of analyses routinely performed per yr (please tick appropriate box):
❑ less than 100
❑ between 100 and 200
❑ between 200 and 500

2. Sample details
Material:
FIRI sample code (A–J):
Your laboratory code for the sample:

3. Measurement technique (please tick appropriate box):

AMS	GPC	LSC
❑ graphite target	❑ CO_2	❑ benzene
❑ other	❑ other	❑ other

4. Preparations Procedures
Sample pretreatment procedures (prior to carbon isotope analysis):
Mass of carbon used in the measurement:
Modern standard material used in the measurement (please tick):
❑ NBS OXI
❑ NBS OXII
❑ other
Please specify other:
Conversion factor to primary standard of activity of 1890 wood:
Background reference material used:

5. Results
5.1 $\delta^{13}C$ (‰) wrt VPBD, stating whether measured or estimated:
The $\delta^{13}C$ measurement (if measured) represents the isotopic ratio in (please tick appropriate box):
❑ the raw material
❑ the material after pretreatment
❑ the actual sample measured
5.2 Age (conventional yr BP ±1):
5.3 Percent modern (defined as: normalized sample activity/normalized standard activity expressed as a percentage.) Note: this should not be decay corrected.

6. Additional comments:

2.2 THE BASIC LABORATORY DEMOGRAPHICS

2.2.1 Laboratory Completion Rate

By the extended deadline of December 2000, sets of results from 85 laboratories had been received. The list of participating laboratories, as well as the technique used, are shown in Table 2.2. This represents a completion rate of 75%, which is extremely successful and exceeds that recorded in the previous intercomparison (TIRI). The reported results are provided in Appendix 1.

Table 2.2 Participating laboratories

Laboratory name	Laboratory type	Country
LATYR, La Plata	LSC	Argentina
Pabellón INGEIS	LSC	Argentina
CSIRO, Glen Osmond	Direct Absorption	Australia
ANTARES AMS Centre, ANSTO	AMS	Australia
Arsenal Research	LSC	Austria
VERA, Universität Wien	AMS	Austria
VRI, Institut für Radiumforschung und Kernphysik	GPC	Austria
IRPA, KIK	LSC	Belgium
IGSB, Minsk	LSC	Belarus
Environmental Isotope Lab, University of Waterloo	LSC	Canada
AECL, Chalk River	Direct Absorption	Canada
Geological Survey of Canada (GSC)	GPC	Canada
EHPL-Env, Ontario Hydro	Direct Absorption	Canada
IOEE Chinese Academy of Sciences	LSC	China
Ruđjer Bošković Institute	GPC	Croatia
Institut für Fysik, University of Aarhus	AMS	Denmark
Institute of Geology, Tallinn	LSC	Estonia
Geological Survey of Finland, Espoo	GPC	Finland
University of Helsinki	GPC	Finland
IPSN/LMRE, Orsay	LSC	France
HIGL, Paris-Sud University	AMS (GIF)	France
Tandetron-Gif	AMS	France
Université Claude Bernard, Lyon	LSC	France
Umweltforschungzentrum Leipzig-Halle	LSC	Germany
Leibniz, Universität Kiel	AMS	Germany
IUF, Universität Köln	GPC	Germany
UFZ-CER, PRG, Halle	LSC	Germany
Institut für Bodenkunde, Universitat Hamburg	LSC	Germany
Heidelberg University	GPC	Germany
DAI, Berlin	GPC	Germany
IGR, NLB, Hannover	GPC	Germany
Universität Erlangen, Nürnberg	AMS	Germany
LOIH, Institute of Physical Chemistry, Demokritos	LSC	Greece
LOA, Institute of Materials Science, Demokritos	GPC	Greece
Institute of Nuclear Research, HAS	GPC	Hungary
Physical Research Lab, Earth Sciences Div, Ahmedabad	LSC	India

Table 2.2 Participating laboratories *(Continued)*

Laboratory name	Laboratory type	Country
Physical Research Lab, Radiocarbon Dating Lab, Ahmedabad	LSC	India
Birbal Sahni Institute, Lucknow	LSC	India
CRDIRT, JCPJ, Jakarta	LSC	Indonesia
University College Dublin	LSC	Ireland
Kimmel Center, Weizmann Institute	LSC	Israel
RDL, University of Rome, La Sapienza	GPC and LSC	Italy
Kyushu Environmental Evaluation Association	LSC	Japan
Institute for Advanced Science, Osaka	LSC	Japan
Palynosurvey Co	LSC	Japan
CCR Nagoya University	AMS	Japan
Gakushuin University, Tokyo	GPC	Japan
Kyoto Sangyo University	GPC	Japan
Seoul National University	AMS	Korea
Institute of Geology, Vilnius	LSC	Lithuania
RJ van de Graaff Lab, Utrecht	AMS	Netherlands
Center for Isotope Research, Groningen	GPC/AMS	Netherlands
Rafter Lab, Institute of Geological Sciences	AMS	New Zealand
University of Waikato	LSC	New Zealand
Radiological Dating Laboratory, Trondheim	GPC	Norway
Silesian Technical University, Gliwice	GPC	Poland
Archaeological and Ethnographical Museum, Łódź	LSC	Poland
Instituto Technológico e Nuclear, Sacavém	LSC	Portugal
Geological Institute, RAS	LSC	Russia
Geographical Research, St. Petersburg State U.	LSC	Russia
Institute of Geography, RAS	LSC	Russia
Institute of Ecology and Evolution, RAS	LSC	Russia
Institute of History of Material Culture, RAS	LSC	Russia
Instituto de Química-Fisíca Rocasolano, Madrid	LSC	Spain
University of Granada	LSC	Spain
Facultad de Química, Universitat de Barcelona	LSC	Spain
Tandem Lab, University of Uppsala	AMS	Sweden
Universitat Bern	GPC	Switzerland
ETH, Zurich	AMS	Switzerland
Department of Geology, NTU	LSC	Taiwan
Office of Atomic Energy for Peace	Direct Absorption	Thailand
School of Geosciences, Queen's University, Belfast	LSC	UK
Research Lab for Archaeology, Oxford	AMS	UK
SUERC, East Kilbride	LSC and AMS (AA)	UK
NERC Radiocarbon Lab	LSC/AMS (AA)	UK
Lab of Radioecology, KIEV	LSC	Ukraine
USGS, Reston	AMS (LLNL)	USA
Beta Analytic Inc, Florida	LSC and AMS (LLNL)	USA
NSF, Arizona	AMS	USA
Geochron Labs, Cambridge, Massachusetts	LSC/GPC/AMS (LLNL)	USA

Table 2.2 Participating laboratories *(Continued)*

Laboratory name	Laboratory type	Country
CAMS/LLNL	AMS	USA
NOSAMS WHOI	AMS	USA
INSTAAR, University of Colorado at Boulder	AMS (WHOI)	USA
University of California, Riverside	AMS (LLNL)	USA
ISGS, Illinois	LSC	USA

In summary, the broad geographical distribution for the laboratories is shown in Table 2.3 below.

Table 2.3 Geographical distribution

Broad geographical description	Number of laboratories
Europe (EU)	35
Europe (non EU)	17
North America and Canada	13
South America	2
Asia and the Far East	13
Australia and New Zealand	4

The summary of the numbers of laboratories using the different techniques is shown in Table 2.4.

Table 2.4 Laboratory type

Laboratory type	Number
LSC	44
GPC	19
AMS	17
Target feeder for AMS	8
Direct absorption and LSC	4

Thus, almost half of the participating laboratories use liquid scintillation. Virtually all operational AMS facilities participated.

Although we have a total of 85 identified participating laboratories, several laboratories operate different independent measurement systems; thus, the total number of submitted sets of results (92) exceeded this figure. Eight laboratories submitted results for AMS, through target preparation and then measurement in a remote facility. In 2 such cases, these samples were measured at the NSF Arizona facility; in 4, the analyses were performed at CAMS/LLNL; while 1 was measured in Tandetron-Gif and 1 measured at NOSAMS WHOI. These sets of results were treated as independent. Some laboratories also submitted more than 1 set of results for a given sample.

2.3 MODERN STANDARD AND BACKGROUND MATERIAL

Other potentially useful general information, which was collected at the time of the submission of results, concerned the background and modern standard materials used by the laboratories, the method of pretreatment applied (if any), the number of routine analyses performed per yr, and information about the measurement of $\delta^{13}C$. Not all laboratories provided all of this ancillary

information. The background and modern standard materials used are surprisingly diverse, but have been broadly categorized to allow a simple summary shown in Tables 2.5 and 2.6 below.

Table 2.5 Classifications used for background and modern standard

a) Background

Original description	Coding for analysis
Anthracite	Anthracite (Anth)
Benzene	Benzene (Benz)
Calcite	Calcite (calc)
Charcoal	Charcoal (char)
Bituminous coal	Coal (coal)
Graphite	Graphite (graph)
Doublespar/IAEA C1	Marble
IAEA C4/wood/limestone	Other

b) Modern standard

Original description	Coding for analysis
ANU sucrose	ANU sucrose (ASUC)
Benzene	Benzene (Benz)
NIST OxI	NBS1
NIST OxII	NBS2
GIN/HD-95,C-3	Other
NIST 1/II	NBS12

Table 2.6 Numbers of laboratories using the identified background and modern standard materials

a) Background material

Classification of material	Number of laboratories using this material
Anthracite	12
Benzene	17
Calcite	3
Coal	4
Graphite	3
Marble	25
Other	27

b) Modern standard materials used

Analysis classification	Number of laboratories using this material
ANU sucrose	9
Benzene	5
NBS1	30
NBS2	29
NBS12	9
other	5

It is clear that there is a wide diversity of background materials, but marble and benzene are common and popular choices.

We can see that the NIST Oxalic acids predominate, but that there are still a few laboratories (19) that do not make use of these materials. In addition, we considered whether the distribution of materials was associated with the different measurement techniques.

Table 2.7 Numbers of laboratories of each type by background and standard material used

a) Background material used

	Background material							
Laboratory type	Anth	benz	calc	coal	graph	Marble	other	**All**
AMS	3	0	2	0	2	15	8	**30**
GPC	6	0	0	3	1	4	3	**17**
LSC	3	17	1	1	0	6	6	**34**
All	12	17	3	4	3	25	17	**81**

b) Modern standard material used

	Standard material						
Laboratory type	ASUC	Benz	NBS1	NBS12	NBS2	other	**All**
AMS	1	0	16	9	5	1	**32**
GPC	1	0	7	0	7	3	**18**
LSC	7	5	7	0	17	1	**37**
All	9	5	30	9	29	5	**87**

c) Modern standard by background material used

	Background							
Standard	Anth	benz	calc	coal	graph	Marble	other	**All**
ASUC	1	1	1	1	0	3	2	**9**
Benz	0	4	0	0	0	0	0	**4**
NBS1	4	2	1	0	0	10	11	**28**
NBS12	0	0	1	0	0	6	0	**7**
NBS2	7	8	0	2	2	5	3	**27**
Other	0	1	0	1	1	1	1	**5**
All	12	16	3	4	3	25	17	**80**

There appears to be no strong evidence of an association between the background and modern standard material used with the measurement technique. It is clear that there are a number of commonly used background materials including, anthracite, benzene (only LSC), and marble (predominantly AMS). The NIST modern standards are widely used, but some laboratories do not make use of these materials and rely on ANU sucrose, benzene, and other materials.

2.4 HOW BUSY ARE THE LABORATORIES?

When submitting their results, laboratories were also asked to provide an approximate figure of the number of analyses they performed per yr. It was thought that this information might be helpful in understanding any outlier distribution and also in explaining deviations from sample consensus values and variation. A brief summary of the findings is presented in the following.

2.4.1 Number of Analyses Carried Out Per Year

The are 4 levels for the "number of analyses performed":

- *1* indicates <100 analyses done per yr by that laboratory;
- *2* indicates 100–200;
- *3* indicates 200–500;
- *4* indicates >500.

First, we consider the association between laboratory type and the number of analyses performed per yr.

Table 2.8 Numbers of laboratories in each Technique/Nr-of-analyses-per-year category

	Nr of analyses per yr			
Laboratory type	1	2	3	4
AMS	0	1	5	17
GPC	1	8	3	4
LSC	14	17	4	6
All	15	26	12	27

As expected, the AMS laboratories predominantly do over 500 analyses per yr (17/23 = 74%), while radiometric laboratories predominantly do fewer than 200 analyses per yr ([1+8+14+17] / (16+41) = 70%), particularly LSC labs ([14+17] / 41 = 76%)

Table 2.9 Numbers of results returned in FIRI by laboratories in each technique categorized by Number-of-analyses-per-yr category

	Number of analyses per yr				
Laboratory type	1	2	3	4	**All**
AMS	0	10	52	298	**360**
GPC	9	92	38	41	**180**
LSC	113	185	33	64	**395**
All	122	287	123	403	**935**

We note that the number of results submitted to FIRI per laboratory tends to increase as the number of analyses per yr carried out increases from an average of 8 (122/15) per laboratory for those doing less than 100 analyses per yr to 15 (403/27) for those doing over 500 per yr.

2.5 Conclusions

These demographic summaries indicate that there is a substantial diversity in the background and the modern standard material used by the laboratories. In particular, a number of laboratories do not routinely use the NIST primary standards. The background materials used are predominantly inorganic, which may prove a factor in the analysis of the Kauri wood samples (A and B). There is a substantial variation among laboratories in the number of analyses per yr which are performed. As would be expected, the AMS laboratories are typically performing substantially more analyses than the radiometric laboratories.

SECTION 3: PRELIMINARY ANALYSIS OF THE RESULTS

3.1 INTRODUCTION

In this section, we present the exploratory analysis of the results submitted by the extended deadline of December 2000. We first deal with Samples C–J, before considering the near-background samples A and B (Kauri wood). The aims of the exploratory analysis are to discover the range of results reported for each sample and the initial evaluation of the effects of any factors that might be a source of variation in the results. For each sample, in turn, we consider the main summary statistics—the number of results reported (N), their mean or average, median, the standard deviation (StDev), the standard error of the mean (Sem), the quartiles (25th [Q1] and 75th [Q3] percentiles), and the minimum (Min) and maximum (Max)—before graphically studying the overall distribution of results in the form of a boxplot, with a view to identifying any extreme or outlying observations. The summary statistics and distribution of results for each laboratory type are also shown. Further details on the statistical methods used are contained in Appendix 3.

3.2 FIRI SAMPLE C: TURBIDITE

The sample was mainly coccolith calcite from a single distal turbidite emplaced on the Maderia Abyssal Plain. It was selected because of its provenance and age. Laboratories had been instructed not to pretreat the sample. This sample had also previously been used in TIRI.

Table 3.1 Descriptive statistics: all results (yr BP)

N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
93	17,945	18,140	693	72	14,600	18,640	17,900	18,260

Table 3.2 Descriptive statistics: all results (yr BP) by laboratory type

Type	N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
AMS	34	18,175	18,175	135	23	17,850	18,470	18,100	18,260
GPC	18	17,990	18,180	743	175	15,230	18,640	17,890	18,315
LSC	41	17,735	18,090	874	136	14,600	18,610	17,740	18,193

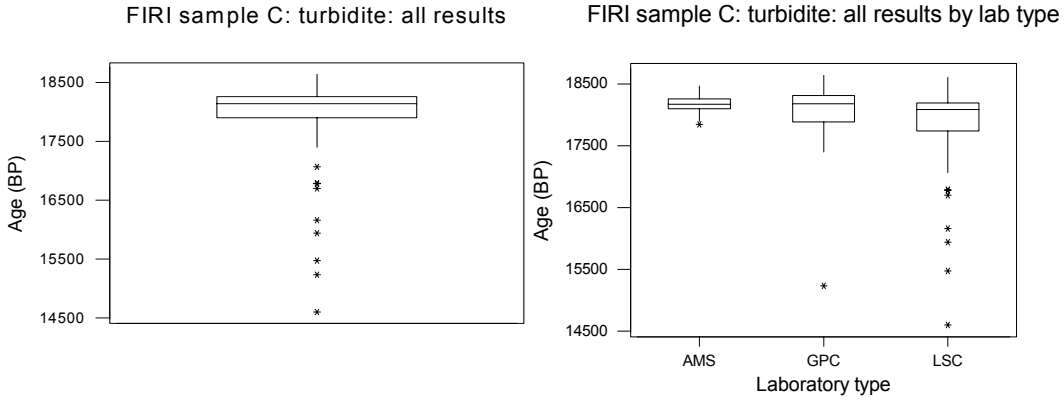

Figure 3.1 Distribution of results for Sample C by all results (left) and laboratory type (right)

3.2.1 Comments

From Table 3.1, we can see that the mean and median age are slightly different at 17,945 and 18,140 BP, suggesting that the distribution is skewed. There is a wide range of results (14,600–8640 BP), but 50% of the values lie between 17,900 and 18,260 BP (interquartile range, Q3 to Q1, of 360 yr).

Table 3.2 shows the results for the different laboratory types. There is little difference in the median age for the 3 laboratory types, but, interestingly, we see that the standard deviation for both LSC and GPC laboratories are considerably larger than that for AMS laboratories.

Figure 3.1 graphically shows the distribution of results, with any extreme values (or outliers) identified by an asterisk.

We can see that there is a long lower tail for the turbidite results. When we consider the distribution by laboratory type, we see that this tail is predominantly composed of results from LSC laboratories.

In the homogeneity testing (Section 1), significant differences had been identified between the results from the 2 laboratories, which could be explained by the effect of pretreatment. The mean non-pretreated result had been 18,157 BP.

The turbidite sample had also been used in TIRI (see Part II), where on the basis of 30 results, calculation of the TIRI consensus value gave a result of 18,155 BP with a 1 σ of 34 yr.

3.3 FIRI SAMPLE D: BELFAST DENDRO-DATED PINE

The sample was from a Scots pine tree from Garry Bog, Co. Antrim, Northern Ireland, and had 40 annual growth rings dating from 3239–3200 BC. This sample was distributed in duplicate as Samples D and F. Its ^{14}C age (from the master calibration curve) is approximately 4495 BP.

Table 3.3 Descriptive statistics: all results (yr BP)

N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
108	4494.4	4517.5	224.2	21.6	2990.0	5060.0	4471.5	4579.0

Table 3.4 Descriptive statistics: all results by laboratory type

Type	N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
AMS	41	4530.3	4520.0	52.0	8.1	4430.0	4670.0	4500.0	4550.0
GPC	20	4495.1	4504.5	75.9	17.0	4273.0	4600.0	4468.5	4522.5
LSC	47	4462.9	4535.0	331.7	48.4	2990.0	5060.0	4400.0	4590.0

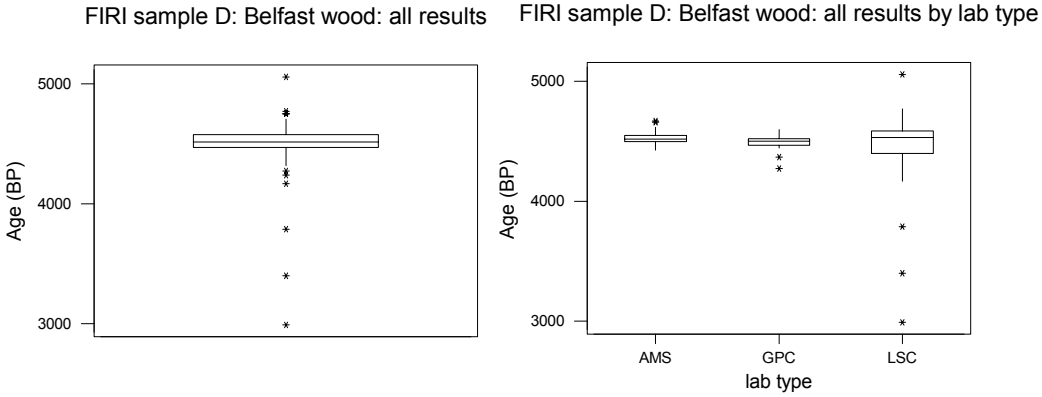

Figure 3.2 Distribution of results for Sample D by all results (left) and laboratory type (right)

3.3.1 Comments

We can see from Table 3.3 that the mean and median age are slightly different at 4494 and 4517 BP. We see a wide range (2990–5060 yr), but 50% of the values lie between 4471 and 4579 BP (i.e., just over 100 yr).

Table 3.4 shows the results for the different laboratory types. There is little difference in the median for the 3 laboratory types. Interestingly, as with Sample C, we see that the standard deviation for the results from the LSC laboratories is considerably larger than that for GPC and AMS laboratories.

From Figure 3.2, we can see that there is a lower tail for the results. When we consider the distribution by laboratory type, we see that this tail is predominantly composed of LSC results.

3.4 FIRI SAMPLE F: BELFAST DENDRO-DATED PINE

Table 3.5 Descriptive statistics: all results (yr BP)

N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
103	4521.4	4504.0	195.8	19.3	4100.0	5870.0	4460.0	4560.0

Table 3.6 Descriptive statistics: all results by laboratory type

Type	N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
AMS	37	4534.2	4534.0	62.0	10.2	4420.0	4710.0	4489.0	4570.0
GPC	21	4485.0	4470.0	120.1	26.2	4250.0	4740.0	4439.5	4528.5
LSC	45	4527.8	4500.0	279.9	41.7	4100.0	5870.0	4420.0	4555.0

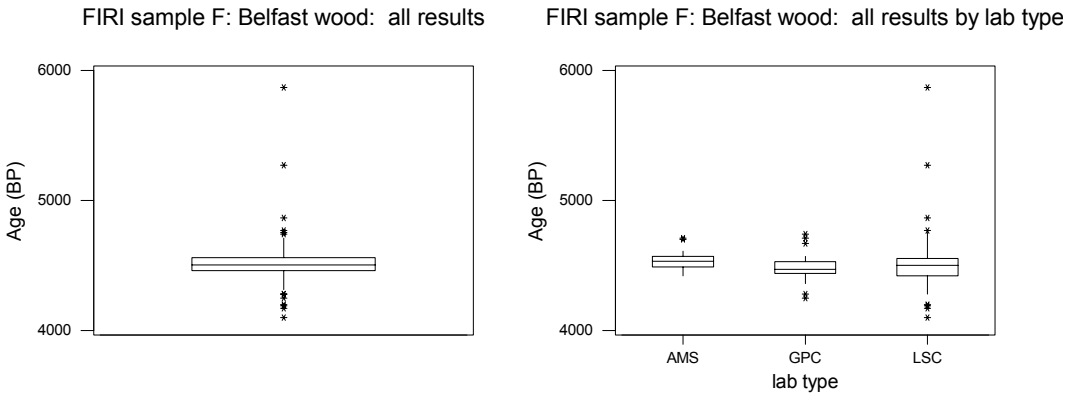

Figure 3.3 Distribution of results for Sample F by all results (left) and laboratory type (right)

3.4.1 Comments

From Table 3.5, we can see that the mean and median age are only slightly different at 4521 and 4504 BP. We also see a narrower range (4100–5870) than for Sample D and that 50% of the values lie between 4460 and 4560 BP (i.e., exactly 100 yr).

Table 3.6 shows the results for the different laboratory types. There is little difference in the median for the 3 laboratory types. Again, we see that the standard deviation for LSC laboratories is considerably larger than that for GPC and AMS.

The median and the middle 50% range for Sample F is almost identical to the results for Sample D.

From Figure 3.3, it is clear that there is both a lower and upper tail for the results. When we consider the distribution by laboratory type, we see that this tail is predominantly composed of results from LSC laboratories.

3.5 FIRI SAMPLE E: HUMIC ACID

Table 3.7 Descriptive statistics: all results (yr BP)

N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
139	11,781	11,780	545	46	7700	15,150	11,670	11,872

Table 3.8 Descriptive statistics: all results by laboratory type (yr BP)

Type	N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
AMS	65	11,822	11,800	188	23	11,430	13,000	11,765	11,870
GPC	26	11,768	11,734	240	47	11,300	12,314	11,617	11,920
LSC	48	11,731	11,726	888	128	7700	15,150	11,591	11,878

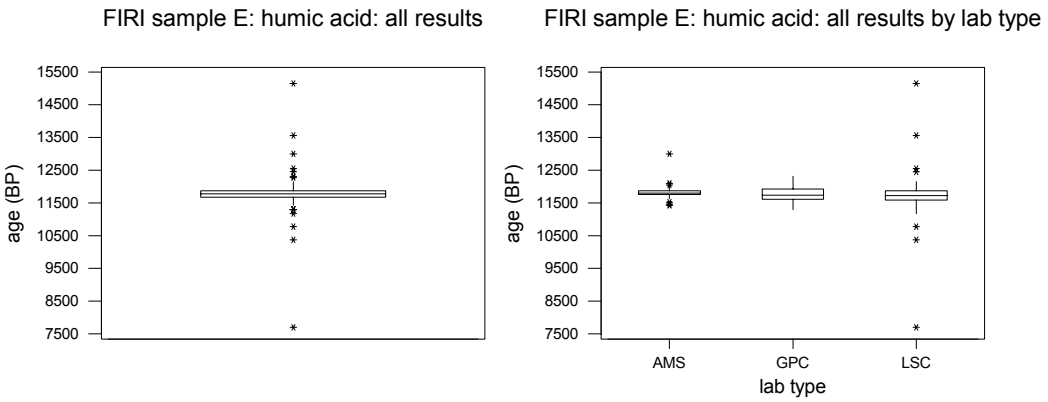

Figure 3.4 Distribution of results for Sample E by all results (left) and laboratory type (right)

3.5.1 Comments

For the humic acid, the mean and median are again in excellent agreement at 11,822 and 11,800 BP, respectively. Again, there is a wide range (7700–15,150 BP), but the interquartile range (IQR) is much narrower (11,670–11,872 BP). We see the same features (Figure 3.4) as before when we look at the summary statistics for each laboratory type with broadly similar mean/median values, but LSC laboratory results have a much larger standard deviation. The distribution of results shows the presence of some extreme values, again predominantly, but not exclusively, reported by LSC laboratories.

3.6 FIRI SAMPLE G: BARLEY MASH

This sample was provided as a duplicate sample with Sample J and reflected current atmospheric levels.

Table 3.9 Descriptive statistics: all results (pMC)

N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
99	110.08	110.50	2.86	0.29	94.47	121.00	109.71	111.08

Table 3.10 Descriptive statistics: all results by laboratory type (pMC)

Type	N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
AMS	34	110.3	110.3	0.68	0.12	109.0	111.9	109.8	110.8
GPC	19	110.6	111.0	1.36	0.31	107.0	112.6	110.0	111.4
LSC	46	109.6	110.4	4.04	0.60	94.2	121.0	108.8	111.3

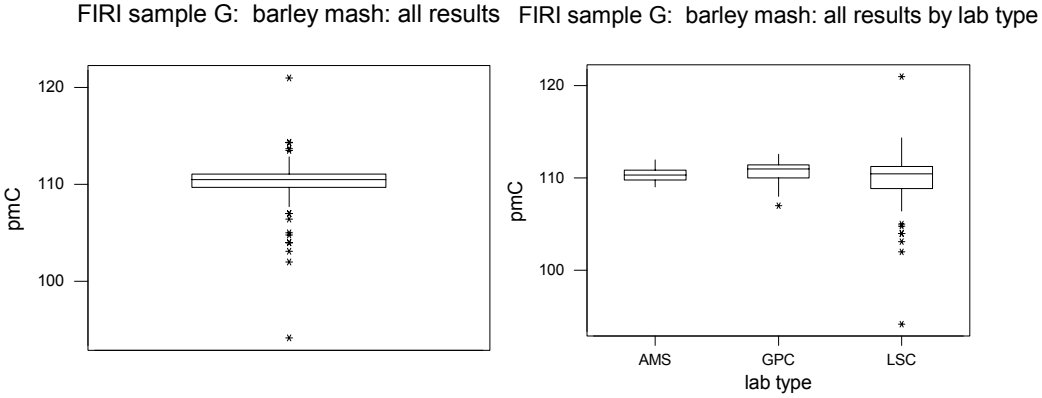

Figure 3.5 Distribution of results for Sample G by all results (left) and laboratory type (right)

3.6.1 Comments

The mean pMC value is estimated at 110.1 and 50% of the data lie in the range 109.7–111.1 (Table 3.9). It is clear, however, from the summary statistics and the graphs that again there are a number of extreme values and that these are reported predominantly by LSC laboratories (Table 3.10).

3.7 FIRI SAMPLE J: BARLEY MASH

Table 3.11 Descriptive statistics: all results (pMC)

N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
99	110.4	110.6	2.73	0.27	97.1	122.0	110.0	111.3

Table 3.12 Descriptive statistics: all results by laboratory type (pMC)

Type	N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
AMS	99	110.4	110.6	2.73	0.27	97.1	122.0	110.0	111.3
GPC	19	110.8	110.7	1.19	0.27	108.3	114.4	110.4	111.3
LSC	45	110.0	110.8	3.93	0.59	97.1	122.0	109.0	111.6

3.7.1 Comments

The mean pMC value is estimated at 110.4 and 50% of the data lie in the range 110.0–111.3. However, it is clear from the summary statistics and the graphs that again there are a number of substantial outliers and that these are reported by LSC laboratories. The distribution of results is very similar to that observed for FIRI G.

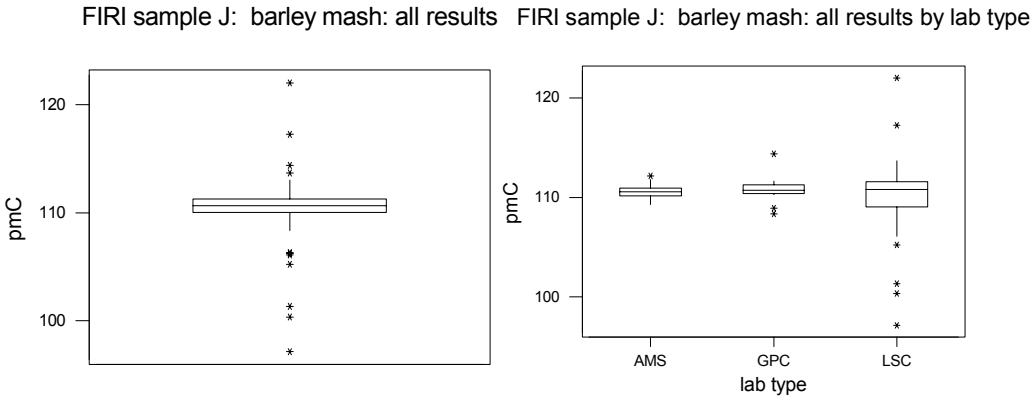

Figure 3.6 Distribution of results for Sample J by all results (left) and laboratory type (right)

3.8 FIRI SAMPLE H: HOHENHEIM DENDRO-DATED OAK

The sample had 20 annual growth rings dating from 313–294 BC, which corresponds to a ^{14}C age of 2215 BP.

Table 3.13 Descriptive statistics: all results (yr BP)

N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
99	2240.9	2230.0	165.4	16.6	1530.0	2980.0	2180.0	2290.0

Table 3.14 Descriptive statistics: all results by laboratory type (yr BP)

Type	N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
AMS	36	2228.7	2230.0	48.2	8.0	2135.0	2318.0	2202.3	2260.0
GPC	20	2259.7	2204.0	193.3	43.2	2093.0	2980.0	2180.0	2267.5
LSC	43	2242.4	2232.0	211.3	32.2	1530.0	2690.0	2160.0	2340.0

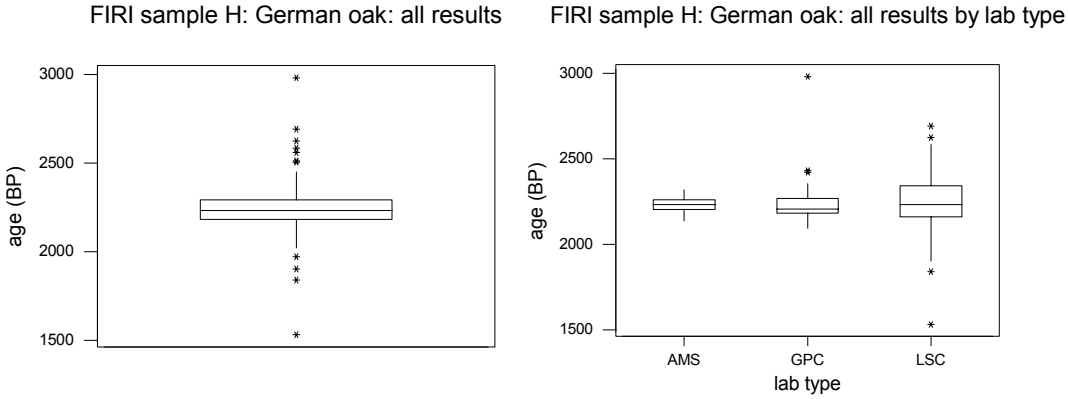

Figure 3.7 Distribution of results for Sample H by all results (left) and laboratory type (right)

3.8.1 Comments

The mean ^{14}C age is estimated as 2241 yr BP and the IQR is 2180–2290 BP (90 yr), but the full range of the data is again extended due to the presence of outliers. The mean and the median age correspond well to the master calibration value ascribed to this sample.

3.9 FIRI SAMPLE I: BELFAST CELLULOSE

Table 3.15 Descriptive statistics: all results (yr BP)

N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
96	4484.6	4490.0	218.8	22.3	3780.0	5650.0	4420.0	4560.0

Table 3.16 Descriptive statistics: all results by laboratory type (yr BP)

Type	N	Mean	Median	StDev	Sem	Min	Max	Q1	Q3
AMS	35	4499.1	4490.0	74.1	12.5	4400.0	4710.0	4450.0	4550.0
GPC	18	4498.8	4463.0	192.4	45.3	4290.0	5100.0	4399.0	4493.8
LSC	43	4466.9	4500.0	297.2	45.3	3780.0	5650.0	4380.0	4580.0

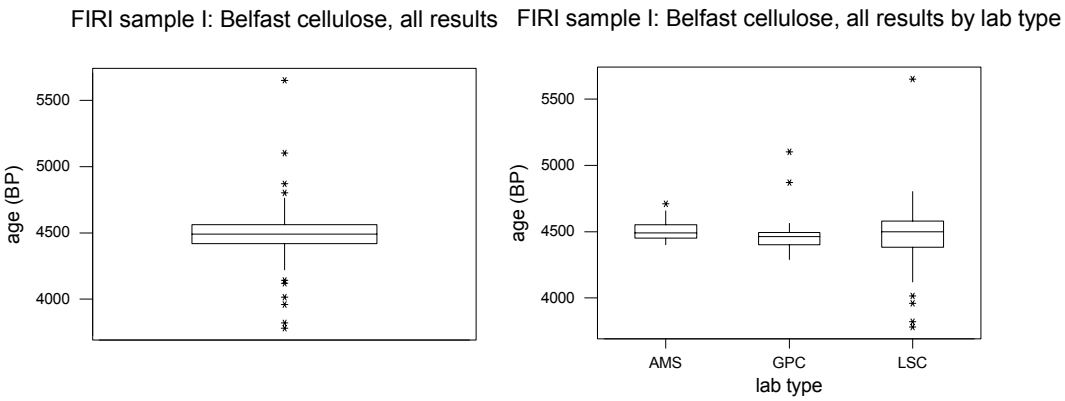

Figure 3.8 Distribution of results for Sample I by all results (left) and laboratory type (right)

The second Belfast sample spanned a contiguous set of rings to FIRI D and F. The sample, which had a finite 40-yr ring span, had a dendrochronologically-determined age span of 3299–3257 BC. This corresponds roughly to a ^{14}C age of 4471 BP.

3.9.1 Comments

The mean and median are very close together at 4485 yr BP, and approximately 15 yr younger than linked samples D and F. The IQR is 140 yr. The graphs show the presence of outliers, again predominantly from LSC laboratories.

3.10 CONCLUSIONS FROM THE PRELIMINARY DISTRIBUTION OF RESULTS

The preliminary analysis of the results for FIRI Samples C–J has shown a consistent pattern, with a reasonably tight IQR (the mid-50% of the results) but with a large range (usually determined by a small number of extreme values). The IQR is reasonably constant at around 100 yr, extending to 300 yr for the oldest sample (Sample C). In the main, although not solely, the extreme results have been reported by liquid scintillation laboratories. From the tables of summary statistics, it is also apparent that the standard deviation in all samples is much larger for LSC laboratories than for GPC or AMS laboratories. Figures A1.a to A1.j in Appendix 1 show the full distribution of results for each sample as well as the ±2 σ range for the individual results. These figures also show the same overall pattern as observed in the boxplots, but now the effect of, and relationship to, the quoted error is also

apparent. In these figures, a steeply sloping section indicates that there are a large number of laboratories with very similar results; such a feature is very striking in Figure A1.e, and to a lesser extent in A1.d and A1.f. It is also clear that the size of the quoted error does vary quite substantially amongst laboratories. This preliminary analysis has not formally used the associated laboratory quoted error and in the next section, the quoted errors are further explored. For this purpose, all results in Section 3.11 have been quoted in % modern carbon (pMC) for comparability purposes.

3.11 SUMMARY OF THE DISTRIBUTION OF QUOTED ERRORS

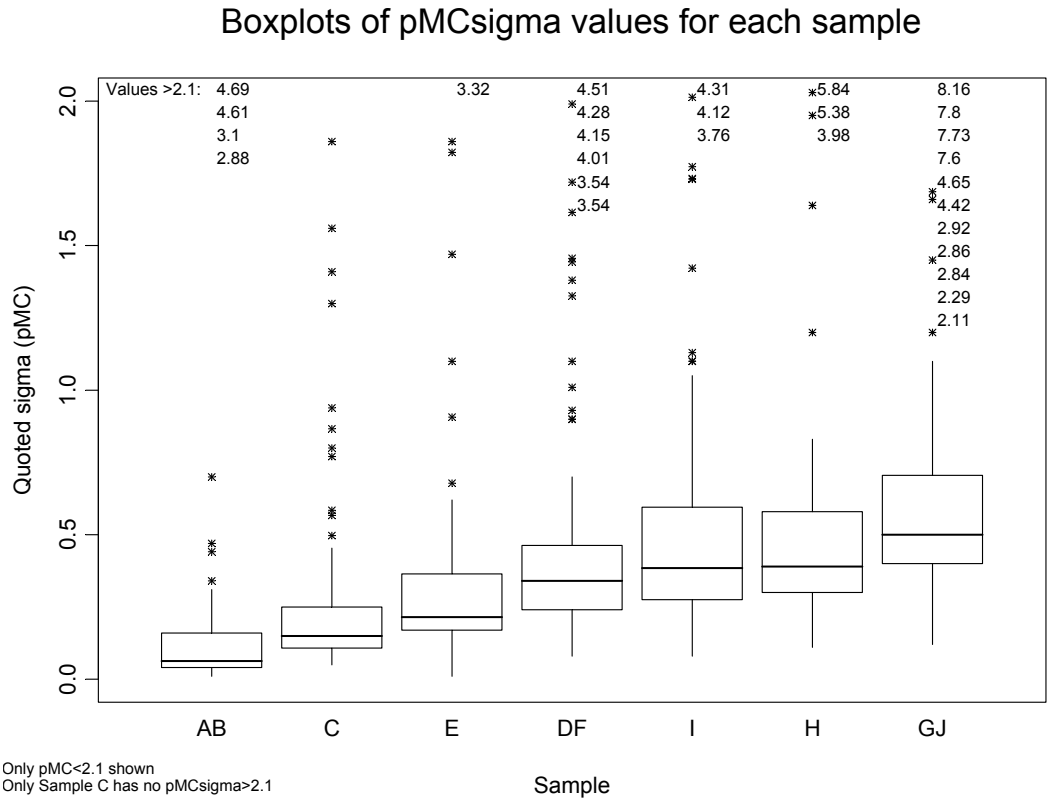

Figure 3.9 Distribution of laboratory quoted errors

Figure 3.9 shows the distribution of quoted errors (all results are given in terms of pMC) for all samples (now including Samples A and B). Extreme values (outliers) are clearly marked by the asterisks. It is clear from the figure that there is a relationship between the pMC and the quoted error, with the quoted error slowly increasing as the sample pMC increases. Similarly, from figures for the different laboratory types, it was quite clear that the quoted errors tend to be larger and more variable for LSC laboratories than for the other laboratory types, and that the AMS laboratories quoted errors tend to be smaller and for there to be much less scatter in their magnitude.

3.12 SUMMARY OF THE $\delta^{13}C$

Laboratories were asked to provide $\delta^{13}C$ values for each sample and to indicate whether these values were measured or estimated. Table 3.17 summarizes the number of laboratories providing this information. In the reporting questionnaire, laboratories were also asked to indicate the stage of the dating process to which the fractionation measure best referred.

Table 3.17 Summary of $\delta^{13}C$ reporting

Lab type	Estimated	Estimated and measured	Measured	Missing	**Total nr of labs**
AMS	0	2	22	1	**25**
GPC	2	1	14	11	**28**
LSC	8	2	29	10	**49**
All	10	5	65	12	**92**

The different parts of the process where $\delta^{13}C$ was measured were classified as:

1. The raw material;
2. The material after pretreatment;
3. The actual sample measured.

The $\delta^{13}C$ values for each sample are summarized first for all results in Table 3.18, and then by the stage of the process in Table 3.19.

Table 3.18 Summary table for $\delta^{13}C$ (all results)

Sample	N	Mean	Median	StDev	Min	Max	Q1	Q3
AB	170	–23.9	–24	1.48	–31	–20.1	–24.7	–23.3
C	82	0.51	1.1	2.84	–22.6	3.864	0.8	1.2
DF	188	–24.8	–25.0	1.36	–32.2	–21.6	–25.3	–24
E	119	–28.7	–29.1	2.2	–34.3	–12.3	–29.5	–28.4
GJ	172	–28.9	–29.1	1.34	–34.1	–24.5	–29.5	–28.6
H	87	–25.0	–24.9	1.34	–31.1	–21.1	–25.5	–24.4
I	86	–23.8	–23.7	0.85	–25.5	–20	–24.3	–23.4

Table 3.19 Summary statistics for $\delta^{13}C$ by process stage

Sample/(Stage)	**AB**	(1)	(2)	(3)	**C**	(1)	(2)	(3)	**DF**	(1)	(2)	(3)
N	153	10	62	66	72	12	22	32	165	9	70	68
Mean	–23.8	–24.2	–23.7	–23.9	0.6	0.9	0.9	1.0	–24.7	–25.1	–24.5	–25.0
Median	–23.9	–24.1	–23.8	–23.9	1.1	1.0	1.1	1.1	–24.9	–25.0	–24.3	–25.0
StDev	1.5	0.5	1.4	1.8	3.0	0.5	0.8	1.3	1.4	0.6	1.3	1.6
Min	–31.0	–25.0	–31.0	–31.0	–22.6	–0.7	–2.4	–3.4	–32.2	–26.0	–32.2	–32.2
Q1	–24.4	–24.5	–24.2	–24.6	0.9	0.8	0.9	1.0	–25.3	–25.5	–25.1	–25.5
Q3	–23.2	–24.0	–23.2	–22.9	1.3	1.2	1.1	1.4	–23.9	–25.0	–23.7	–24.1
Max	–20.1	–23.4	–20.9	–20.1	3.9	1.3	1.6	3.9	–21.6	–24.0	–21.7	–21.6
Sample/(Stage)	**E**	(1)	(2)	(3)	**GJ**	(1)	(2)	(3)	**H**	(1)	(2)	(3)
N	69	20	17	27	155	37	37	67	79	4	34	34
Mean	–29.0	–28.8	–29.1	–29.3	–29.1	–28.8	–29.0	–29.4	–25.0	–25.4	–24.7	–25.2
Median	–29.1	–28.8	–29.1	–29.3	–29.1	–29.0	–28.9	–29.3	–24.8	–25.3	–24.8	–25.0
StDev	1.3	1.5	1.1	0.6	0.9	0.9	0.6	1.1	1.4	0.6	1.5	1.4
Min	–32.9	–29.9	–32.9	–30.2	–34.1	–29.8	–30.8	–34.1	–31.1	–26.0	–31.1	–31.1
Q1	–29.5	–29.5	–29.2	–29.6	–29.5	–29.5	–29.2	–29.7	–25.5	–25.9	–25.2	–25.5
Q3	–28.8	–28.8	–28.9	–29.1	–28.7	–28.6	–28.5	–28.9	–24.3	–24.8	–24.1	–24.4
Max	–23.0	–23.2	–27.7	–27.6	–25.9	–26.0	–28.0	–25.9	–21.1	–24.8	–21.1	–23.0
Sample/(Stage)	**I**	(1)	(2)	(3)								
N	77	18	20	33								
Mean	–23.7	–23.7	–23.5	–23.8								
Median	–23.7	–23.7	–23.5	–23.9								
StDev	0.7	0.6	0.6	0.8								
Min	–25.5	–25.1	–25.0	–25.5								
Q1	–24.0	–24.0	–23.7	–24.4								
Q3	–23.4	–23.2	–23.3	–23.6								
Max	–21.7	–22.3	–21.7	–21.7								

The boxplots in Figure 3.10 show the pattern of measured $\delta^{13}C$ values for the samples, except Sample C. The barley and humic samples are comparable and lighter than the wood samples. There may be some suggestion that Sample I (cellulose) is heavier than Samples D and F. It is also of interest to consider the differences in the $\delta^{13}C$ values at the different stages and this is shown graphically in Figures 3.11 and 3.12. It should be remembered that the $\delta^{13}C$ values should not be used as the reference isotopic ratio for these samples; rather, it may prove a useful marker for the variation in measurement. The results have shown small differences in the different process stages. There is little evidence for any of the samples that there is significant variation in the fractionation incurred at the different stages. There is some variation in the $\delta^{13}C$ values quoted, but these effects are likely to be small in the overall variation of the results.

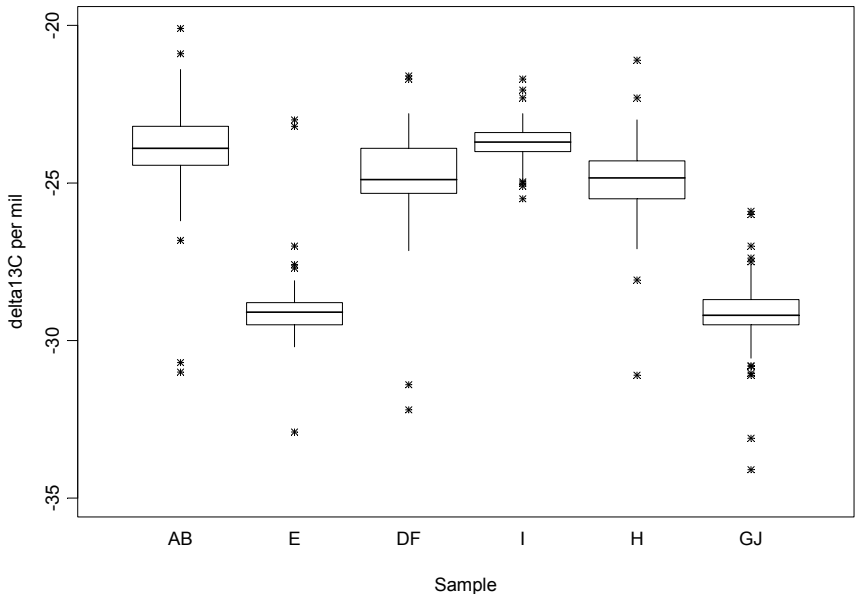

Figure 3.10 $\delta^{13}C$ for all samples (except Sample C, turbidite)

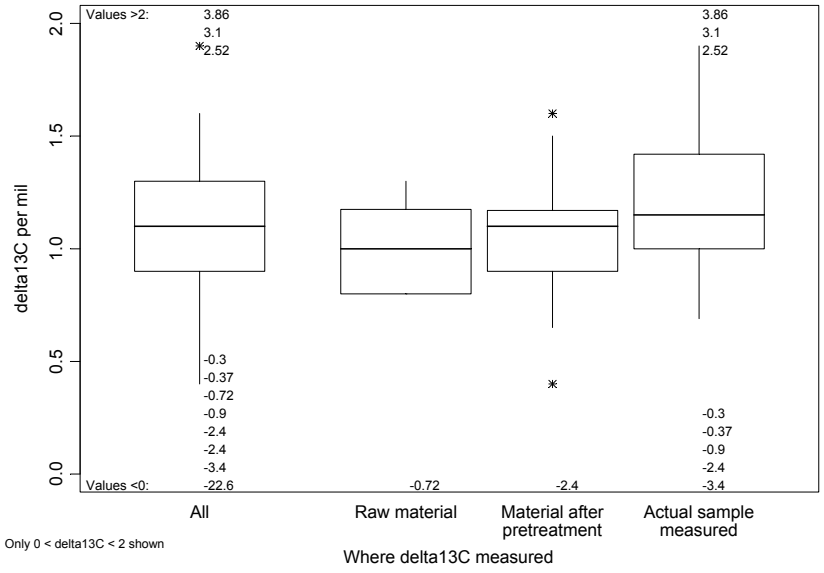

Figure 3.11 $\delta^{13}C$ for Sample C (turbidite) in process for different point of measure categories

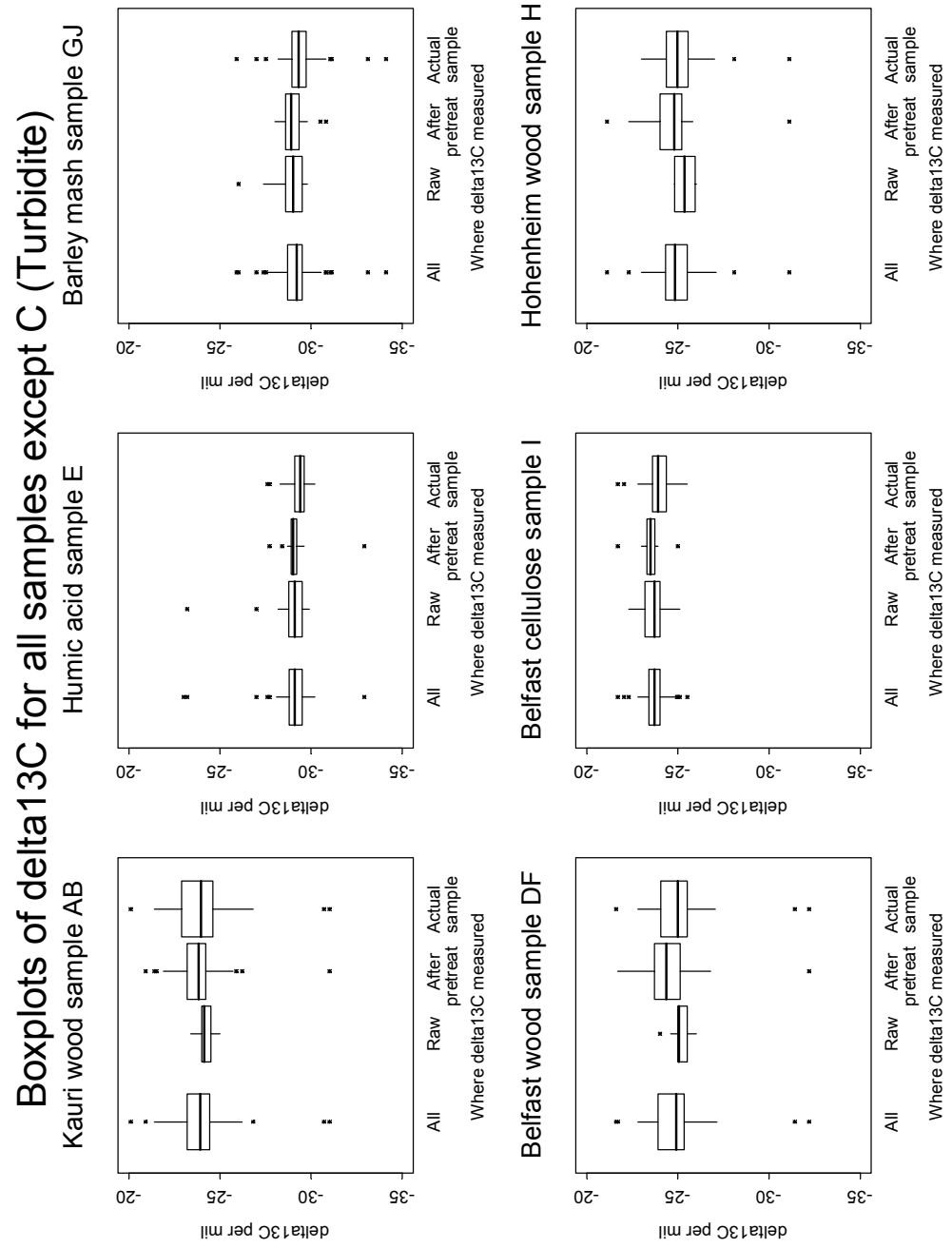

Figure 3.12 $\delta^{13}C$ for all samples by stage in process

3.13 OUTLIERS OR EXTREME VALUES

3.13.1 Outlier Definitions

There are many ways for defining outliers and no universal statistical definition. In this report, we have used a conventional definition that is widely used in the statistical software, and in particular, is used to identify outliers when producing boxplots.

For the purposes of this investigation of outliers and the similarities they exhibited, outliers were defined as all results greater or less than 1.5× IQR from the middle 50% of the results or result < Q1 – 1.5(Q1–Q3) or result > QU + 1.5(QL–QU), where *QL* and *QU* are the upper and lower quartiles, respectively.

In a previous analysis of ^{14}C results from an intercomparison, the standard consensus (Rozanski et al. 1992) calculations were used at the first stage of the calculation, a definition such that outliers were classed as those results that were more than 3 IQR from the middle 50% of the results (i.e., result < QL – 3(QL–QU) or result > QU + 3(QL–QU), where *QL* and *QU* are the upper and lower quartiles, respectively.

Using the 1.5 IQR definition, the outlier boundaries are defined below:

Table 3.20 Outlier boundaries (in pMC for Samples AB and GJ)

Limit	AB	C	DF	E	GJ	H	I
Lower	–0.5	17,362	4313	11,358	108	2004	4210
Upper	1.3	18,796	4723	12,168	113	2457	4770

3.13.2 Outlier Description

A total of 122 observations from 1056 (i.e., slightly over 10%) were identified as outliers using these definitions and here we explore the nature of these outlier observations.

Table 3.21 Percentage distribution of outliers amongst laboratory types

Laboratory type	Number of identified outliers	%
AMS	1	0.82
GPC	15	12.30
LSC	106	86.89
All	**122**	**100.00**

Thus, of the 122 outliers, 87% came from LSC laboratories.

We can also consider whether there was any association with the outlier results and the modern standard material or background material used.

Table 3.22 Number of outliers reported where laboratory used the given standard material

Modern standard material	Number of outliers	%
ASUC	15	14.02
Benz	17	15.89
NBS1	23	21.50
NBS2	45	42.06
other	7	6.54

Over half of the outliers were submitted by laboratories using NBS Ox1 and NBS Ox2.

Table 3.23 Number of outliers reported where laboratory used the given background material

Background material	Number of outliers	%
Anth	17	17.17
Benz	39	39.39
Coal	5	5.05
Graph	4	4.04
Marble	17	17.17
None	2	2.02
Other	15	15.15
All	**99**	**100.00**

The distribution of outliers is uniform over the sample; thus, no single sample contributes the majority of the outliers if we consider the joint distribution of laboratory type and standard used for those outlier results. The distribution is shown in the table below.

In terms of background material, the most common background material is benzene (scintillation-grade benzene) and over 39% of the outliers are associated with the use of benzene as the background material.

Table 3.24 Number of outliers reported where laboratory used the given background material

Sample	Number of outliers	%
A	11	9.02
B	7	5.74
C	11	9.02
D	12	9.84
E	13	10.66
F	16	13.11
G	16	13.11
H	13	10.66
I	13	10.66
J	10	8.20

Table 3.25 Numbers of outliers for given laboratory type and modern standard material

	ASUC	Benz	NBS1	NBS2	Other	**All**
AMS	0	0	0	1	0	**1**
GPC	2	0	2	11	0	**15**
LSC	13	17	21	33	7	**91**
All	**15**	**17**	**23**	**45**	**7**	**107**

There appears to be no statistical association between laboratory type and modern standard used for the outlier results.

Table 3.26 Numbers of outliers for given laboratory type and background material

	Anth	Benz	Coal	Graph	Marble	Other	**All**
AMS	0	0	1	0	0	0	**1**
GPC	4	0	4	4	1	2	**15**
LSC	13	39	0	0	16	15	**83**
All	**17**	**39**	**5**	**4**	**17**	**17**	**99**

It seems that there is a statistical association between laboratory type, background material, and outlier results.

3.13.3 Distribution of Outliers Across Labs

Of the 92 laboratories in the intercomparison, there were 39 (42%) which had at least 1 result classed as an outlier. Information about each of these is given in the following tables.

Of the 39 laboratories that had 1 or more outliers, almost 60% (23) of these had more than 1 of their results thus classed and over one-fifth (9) had 5 or more such results (see Table 3.27).

Table 3.27 Count of laboratories in different number_of_outlier_results groups

Number of outliers	0	1	2	3	4	5	6	7	9	11	**Total**
Number of laboratories	53	16	6	4	4	1	3	2	2	1	**92**

From Table 3.28, over 75% (30) of the laboratories with outliers used LSC, while all but one of the rest used GPC. Thus, a larger proportion of the outlier laboratories used LSC, compared to the LSC representation in the overall set of results, where 53% of the laboratories used LSC.

Table 3.28 Count of laboratories in different measurement method groups

Measurement method	AMS	GPC	LSC	**Total**
Number of outlier laboratories	1	8	30	**39**
All laboratories	25	18	49	**92**

From Table 3.29, we can see that just over 50% (20) of these 39 laboratories did not state that they measured the $\delta^{13}C$ for all their samples. Nine of these 20 laboratories definitely estimated $\delta^{13}C$, 3 used both measured and estimated values, while the other 8 did not specifying whether or not they did. In the overall case, only 29% (27) of the 92 did not state that they measured the $\delta^{13}C$ for all their samples.

Table 3.29 Counts of outlier and all laboratories' $\delta^{13}C$ categories

$\delta^{13}C$ measured or estimated	Estimated	Estimated and measured	Measured	Missing	**Total**
Number of outlier laboratories	9	3	19	8	**39**
All laboratories	10	5	65	12	**92**

Table 3.30 shows the types of background and modern standard materials used by laboratories with outliers and all laboratories. From this table, we can see that benzene was a far more commonly used background material in the outlier group (38% of the time) than overall (21%). This was also the case with the modern standards, where 6 out of the 7 laboratories using benzene were in the outlier group. It should be noted that the types of benzene used varied from laboratory to laboratory, unlike the other modern standards.

Table 3.30 Types and numbers of laboratories using backgrounds and modern standards

Background material			Modern standard material		
Category	Outlier laboratories	All	Category	Outlier laboratories	All
Benzene	15	19	ANU Sucrose	3	8
CO_2	1	3	Benzene	6	7
Coal	9	17	NBS OXI	6	21
Graphite	1	4	NBS OXII	17	32
Marble	2	6	NBS OXI/OXII	0	5
Natural Gas	1	3	1 NBS & 1 other	3	3
Others	4	10	Other	2	7
More than 1	1	13	Missing	2	9
Missing	5	17	**Total**	**39**	**92**
Total	**39**	**92**			

3.13.4 Conclusions

A total of 122 observations out of 1056 (i.e., slightly over 10%) were identified as anomalous (i.e., outliers). From the statistical definition of an outlier, around 5% of the results would have been expected to have been classed as outliers. Thus, approximately twice as many outliers were identified as would be expected if they were occurring purely by chance. Of the 122 outliers, 87% came from LSC laboratories. The distribution of outliers was uniform over the 10 samples; thus, no single sample contributed the majority of the outliers. Thirty-nine laboratories (42%) had at least 1 result classed as an outlier. Of the 39, almost 60% (23) of these had more than 1 of their results thus classed, and over one-fifth (9) had 5 or more such results.

Table 3.31 Operational information concerning laboratories with at least 1 outlier

Lab nr	$\delta^{13}C$ measured (M) or estimated (E)	Background material	Modern standard material	Nr of outlier results
5	E	IAEA C1	NBS OXI	7
10	—	Benzene	Benzene	6
11	M	Anthracite	OXII / ANU	4
13	M	Benzene	NBS OXII	3
15	M	Anthracite	NBS OXII	1
16	—	—	Benzene	6
17	E	—	NBS OXI	1
18	M (E & M)	Anthracite	NBS OXI	1
19	E (E & M)	Methanol	NBS OXII	1
21	E	Benzene	Benzene	2
23	M	Anthracite	NBS OXII	2
26	—	—	—	6
28	—	—	NBS OXII	2
30	M	Benzene	Benzene	1
31	M	TIRI-G CO_2	ANU Sucrose	1
32	M	Marble	NBS OXII	1
39	M	Benzene	NBS OXII	5
42	E	Benzene	Benzene	2
43	M	Anthracite	ANU Sucrose	3
44	M	Graphite	NBS OXII	4

Table 3.31 Operational information concerning laboratories with at least 1 outlier *(Continued)*

Lab nr	$\delta^{13}C$ measured (M) or estimated (E)	Background material	Modern standard material	Nr of outlier results
53	E	Marble	ANU Sucrose	9
56	M	Anthracite	NBS OXII	2
57	E	Natural Gas	OXII / C3	1
59	M	Anthracite	NBS OXII	1
63	E	Benzene	NBS OXII	2
66	M	Limestone	NBS OXI	1
67	E	Benzene	GIN	3
68	M (E & M)	Benzene	NBS OXII	1
59	M	Benzene	NBS OXII	7
70	—	IAEA C4	NBS OXI	11
71	—	Benzene	Other	3
75	—	Benzene	Other	1
76	M	Benzene	Other	1
78	—	—	—	9
80	M	Benzene	NBS OXI	1
81	E	Anthracite	NBS OXII	4
89	M	Benz/Anthracite	NBS OXII	1
90	M	Anthracite	NBS OXII	4
92	M	Benzene	NBS OXII	1

Clearly, a relatively small number of laboratories (14%) generated more than 60% of the outlying observations. The majority of these laboratories use liquid scintillation techniques (including direct absorption). However, it should be noted that there remains a substantial number of liquid scintillation laboratories with none or only 1 outlier.

Further analysis indicated that the presence of outliers was linked to the modern standard used, with some laboratories having no access to the primary standards of NIST OxI and OxII.

SECTION 4: INVESTIGATION OF POTENTIAL SOURCES OF VARIATION

4.1 INTRODUCTION

The design of FIRI is such that for each laboratory, we have some basic, though limited, information on the laboratory procedures, including the method of pretreatment applied to the samples, the modern standard, and the background material used. These can be considered as *factors* in the experiment and through statistical analysis, we can investigate whether they offer a statistically significant explanation of the observed variation. The different levels of the factors are described in Table 4.1. In addition, the laboratory type is also considered as a further factor (with 3 levels of LSC, GPC, and AMS).

Therefore, this section considers each sample, in turn, and explores the proportion of variation, which can be explained by each of the factors. For these analyses, extreme values (outliers) have been omitted (as identified in Section 3).

The structure of the section for each sample includes the summary statistics with the number of omitted values from the analysis, a boxplot showing the distribution of the results for the different levels of the factor of interest, and the output from a formal *analysis of variance* (a formal test of the hypothesis that the mean age/activity is the same for each level of the factor). This output takes the form of a table, where the key statistic is the *p-value*. Conventionally, at a 5% significance level, if the p-value is less than 0.05, then we reject that the mean age/activity is the same for all levels of the factor and conclude there are statistically significant differences. In such a case, a follow-up analysis can be used to identify the magnitude of any differences.

Table 4.1 Classifications used for modern standard and background material

a) Modern standard

Original description	Analysis classification/level
ANU sucrose	ASuc
Benzene	Benz(ene)
NIST OxI	NBS1
NIST OxII	NBS2
GIN/HD-95,C-3	Other
NIST I/II	NBS12

b) Background

Original description	Analysis classification
Anthracite	Anth(racite)
Benzene	Benz(ene)
Calcite	Calc(ite)
Charcoal	Charc(oal)
Bituminous coal	Coal
Graphite	Graphite
Doublespar/IAEA C1	Marble
IAEA C4/wood/limestone	Other

4.2 LABORATORY TYPE AS A SOURCE OF VARIATION

In this section, the analysis is focused on whether there are statistically significant differences in the mean activity/age among the different laboratory types.

4.2.1 Sample C: Turbidite

Table 4.2 Summary statistics of age for Sample C

Type	N	Nr of omitted values	Mean	Median	StDev
AMS	34	0	18,175	18,175	135
GPC	17	1	18,152	18,200	288
LSC	33	10	18,110	18,120	244
AMS	34	0	18,175	18,175	135

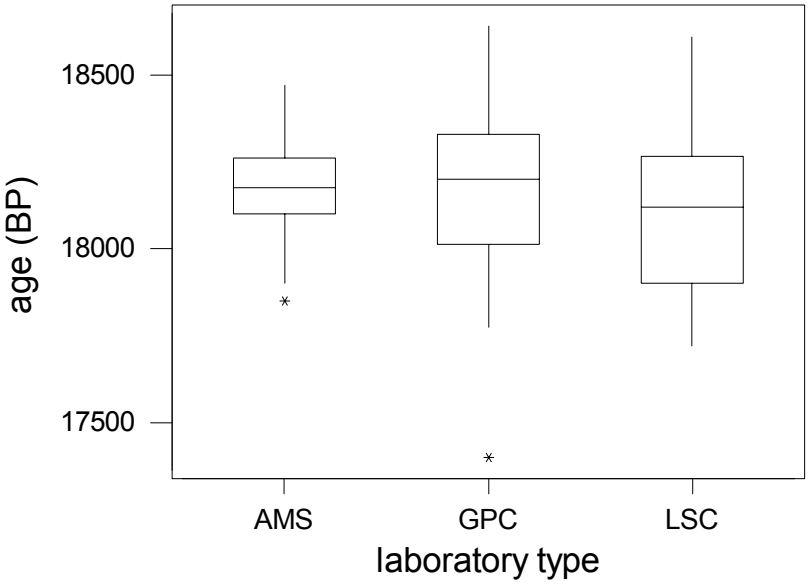

Figure 4.1 Distribution of age by laboratory type for Sample C

Table 4.3 Analysis of variance for Sample C

Source	DF	SS	MS	F	P
type	2	70472	35236	0.75	**0.478**
Error	81	3830128	47286		
Total	83	3900600			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
AMS	34	18175	135	(-----------*-----------)
GPC	17	18152	288	(----------------*----------------)
LSC	33	18110	244	(-----------*-----------)
				-----+---------+---------+---------+-
Pooled StDev =		217		18060 18120 18180 18240

4.2.1.1 Conclusion

Since the p-value is >0.05 in Table 4.3, there is no evidence of statistically significant differences among laboratory types. The results from each laboratory type broadly overlap.

4.2.2 Sample E: Humic Acid

Table 4.4 Summary statistics of age for Sample E

Type	N	Number omitted	Mean	Median	StDev
AMS	64	1	11,804	11,800	117
GPC	23	4	11,743	11,722	173
LSC	38	14	11,757	11,736	177

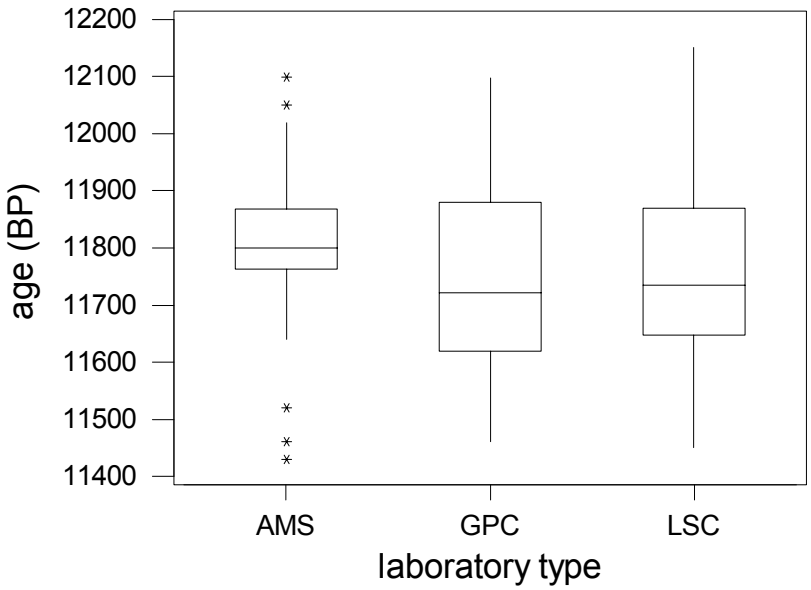

Figure 4.2 Distribution of age by laboratory type for Sample E

Table 4.5 Analysis of variance for Sample E

Source	DF	SS	MS	F	P
type	2	86788	43394	1.97	**0.143**
Error	122	2680561	21972		
Total	124	2767349			

Individual 95% CIs For Mean Based on Pooled StDev

Level	N	Mean	StDev	----+---------+---------+---------+--
AMS	64	11804	117	(-------*------)
GPC	23	11743	173	(-----------*-----------)
LSC	38	11757	177	(--------*---------)
				----+---------+---------+---------+--

4.2.2.1 Conclusion

Since the p-value is >0.05 in Table 4.5, there is no evidence of statistically significant differences among laboratory types. The results from each laboratory type broadly overlap.

4.2.3 Sample D: Belfast Wood

Table 4.6 Summary statistics of age for Sample D

Type	N	Number omitted	Mean	Median	StDev
AMS	41	0	4530.3	4520.0	52.0
GPC	19	1	4506.7	4509.0	56.5
LSC	38	12	4521.7	4537.5	106.7

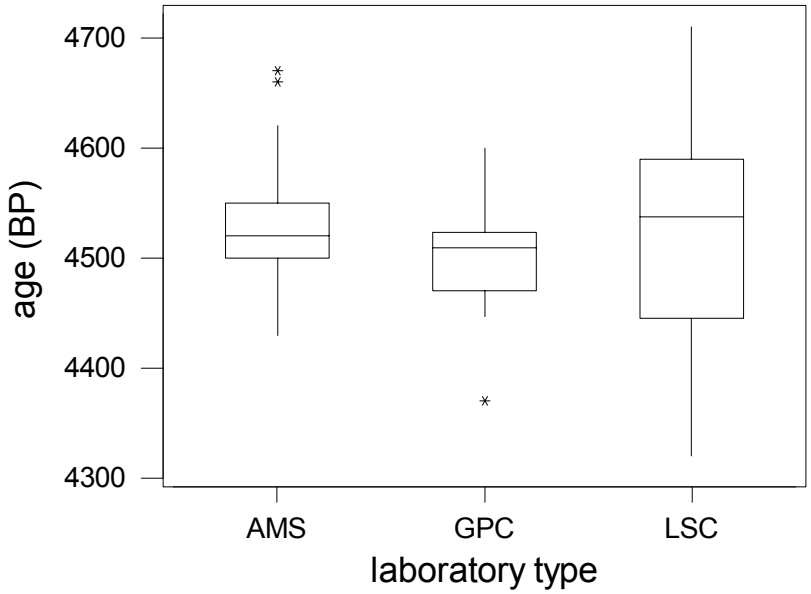

Figure 4.3 Distribution of age by laboratory type for Sample D

Table 4.7 Analysis of variance of Sample D

```
Source      DF        SS        MS        F        P
type         2      7231      3616     0.59    0.559
Error       95    586712      6176
Total       97    593943

                                    Individual 95% CIs For Mean
                                    Based on Pooled StDev
Level       N      Mean     StDev  --+---------+---------+---------+----
AMS        41    4530.3      52.0                (---------*---------)
GPC        19    4506.7      56.5  (--------------*-------------)
LSC        38    4521.7     106.7             (---------*---------)
                                   --+---------+---------+---------+----
Pooled StDev =     78.6           4475      4500      4525      4550
```

4.2.3.1 Conclusion

Since the p-value is >0.05 in Table 4.7, there is no evidence of statistically significant differences among laboratory types. The results from each laboratory type broadly overlap.

4.2.4 Sample F: Belfast Wood

Table 4.8 Summary statistics of age for Sample F

Type	N	Number omitted	Mean	Median	StDev
AMS	37	0	4534.2	4534.0	62.0
GPC	18	3	4495.3	4476.5	85.4
LSC	35	13	4493.8	4500.0	83.4

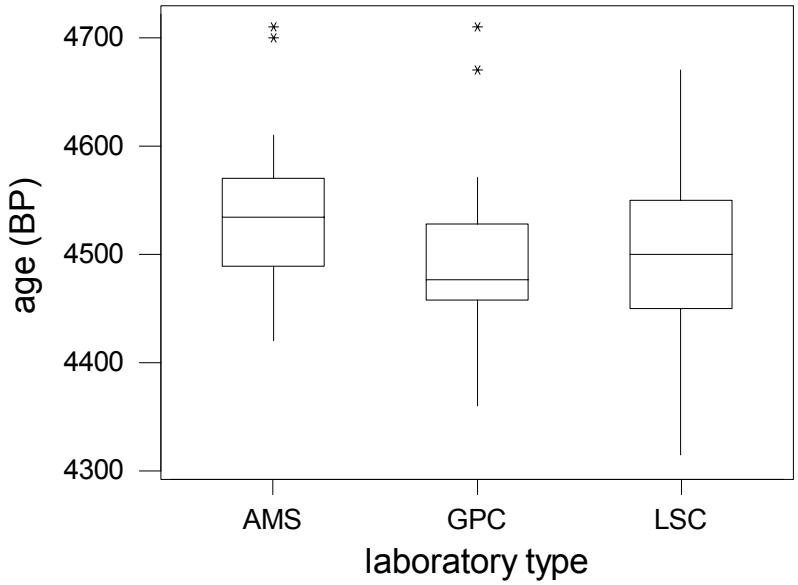

Figure 4.4 Distribution of age by laboratory type for Sample F

Table 4.9 Analysis of variance for Sample F

```
Source      DF        SS        MS        F        P
type         2     34805     17402     3.03    0.053
Error       87    499422      5740
Total       89    534227

                                  Individual 95% CIs For Mean
                                  Based on Pooled StDev
Level       N      Mean     StDev  ----+---------+---------+---------+--
AMS        37    4534.2      62.0                    (-------*--------)
GPC        18    4495.3      85.4   (----------*-----------)
LSC        35    4493.8      83.4     (--------*-------)
                                  ----+---------+---------+---------+--
Pooled StDev =     75.8              4470      4500      4530      4560
```

4.2.4.1 Conclusion

A statistically significant difference among laboratory types is detected at the 10% level (p-value = 0.053 in Table 4.9). The mean age from AMS laboratories appears older than that for either GPC or LSC laboratories.

4.2.5 Sample G: Barley Mash (pMC)

Table 4.10 Summary statistics of activity (pMC) for Sample G

Type	N	Number omitted	Mean	Median	StDev
AMS	34	0	110.33	110.35	0.68
GPC	18	1	110.85	111.00	1.06
LSC	32	15	110.53	110.60	1.10

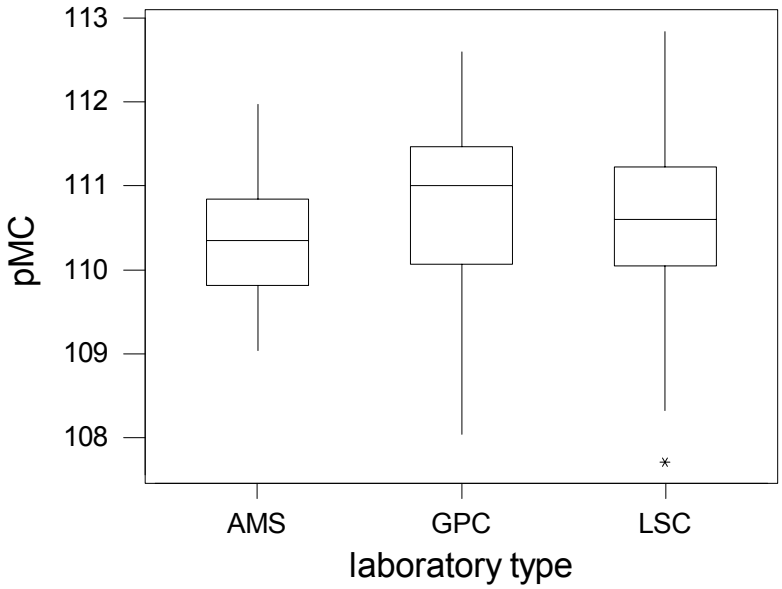

Figure 4.5 Distribution of activity by laboratory type for Sample G

Table 4.11 Analysis of variance for Sample G

Source	DF	SS	MS	F	P
type	2	3.154	1.577	1.76	**0.178**
Error	81	72.530	0.895		
Total	83	75.684			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
AMS	34	110.335	0.685	(--------*---------)
GPC	18	110.852	1.064	(-----------*-----------)
LSC	32	110.526	1.105	(---------*--------)
Pooled StDev =		0.946		110.25 110.60 110.95

4.2.5.1 Conclusion

Since the p-value is >0.05 in Table 4.11, there is no evidence of statistically significant differences among laboratory types. The results from each laboratory type broadly overlap.

4.2.6 Sample J: Barley Mash

Table 4.12 Summary statistics of activity (pMC) for Sample J

Type	N	Number omitted	Mean	Median	StDev
AMS	35	0	110.58	110.56	0.61
GPC	18	1	110.60	110.70	0.83
LSC	34	12	110.63	110.84	1.20

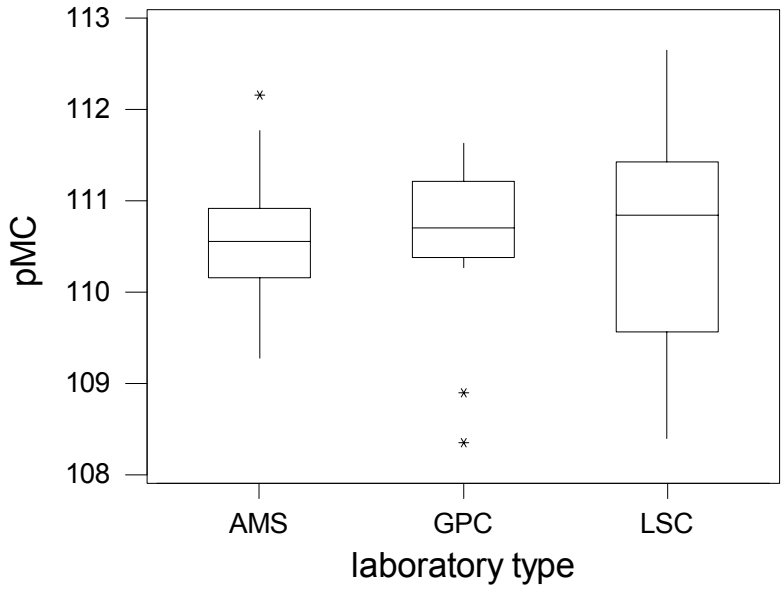

Figure 4.6 Distribution of activity by laboratory type for Sample J

Table 4.13 Analysis of variance for Sample J

Source	DF	SS	MS	F	P
type	2	0.047	0.024	0.03	**0.973**
Error	84	71.700	0.854		
Total	86	71.748			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
AMS	35	110.583	0.606	(-----------*-----------)
GPC	18	110.600	0.834	(----------------*----------------)
LSC	34	110.635	1.198	(-----------*-----------)
Pooled StDev =		0.924		110.25 110.50 110.75 111.00

4.2.6.1 Conclusion

Since the p-value is >0.05 in Table 4.13, there is no evidence of statistically significant differences among laboratory types. The results from each laboratory type broadly overlap.

4.2.7 Sample H: Hohenheim Wood

Table 4.14 Summary statistics of age for Sample H

Type	N	Number omitted	Mean	Median	StDev
AMS	36	0	2228.7	2230.0	48.2
GPC	19	1	2221.7	2200.0	95.4
LSC	33	13	2233.7	2230.0	98.1

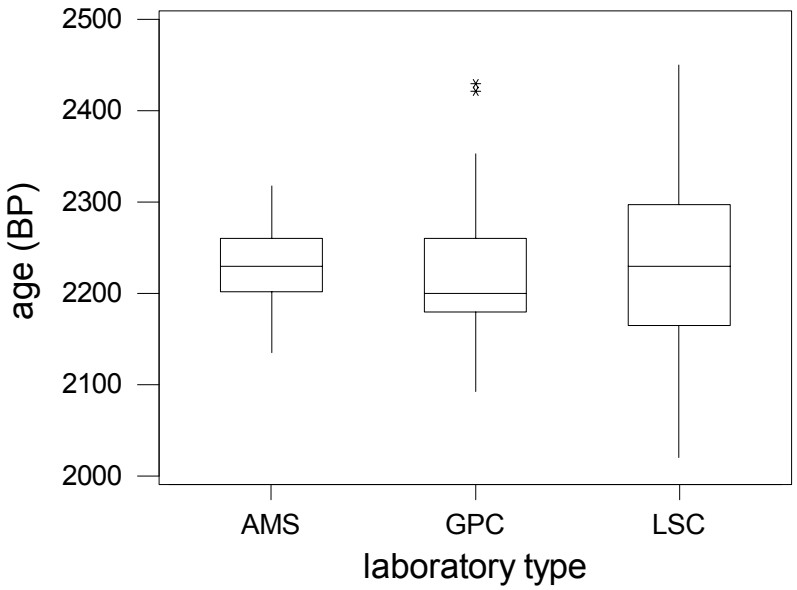

Figure 4.7 Distribution of age by laboratory type for Sample H

Table 4.15 Analysis of variance for Sample H

```
Source      DF        SS        MS        F        P
type         2      1742       871     0.13    0.875
Error       85    553268      6509
Total       87    555011

                                  Individual 95% CIs For Mean
                                  Based on Pooled StDev
Level       N      Mean     StDev  -------+---------+---------+---------
AMS        36    2228.7      48.2           (---------*----------)
GPC        19    2221.7      95.4    (--------------*-------------)
LSC        33    2233.7      98.1            (----------*-----------)
                                  -------+---------+---------+---------
Pooled StDev =     80.7               2200      2225      2250
```

4.2.7.1 Conclusion

Since the p-value is >0.05 in Table 4.15, there is no evidence of statistically significant differences among laboratory types. The results from each laboratory type broadly overlap.

4.2.8 Sample I: Belfast Cellulose

Table 4.16 Summary statistics of age for Sample I

Type	N	Number omitted	Mean	Median	StDev
AMS	35	0	4499.1	4490.0	74.1
GPC	16	3	4438.0	4450.0	68.6
LSC	35	11	4508.4	4520.0	128.1

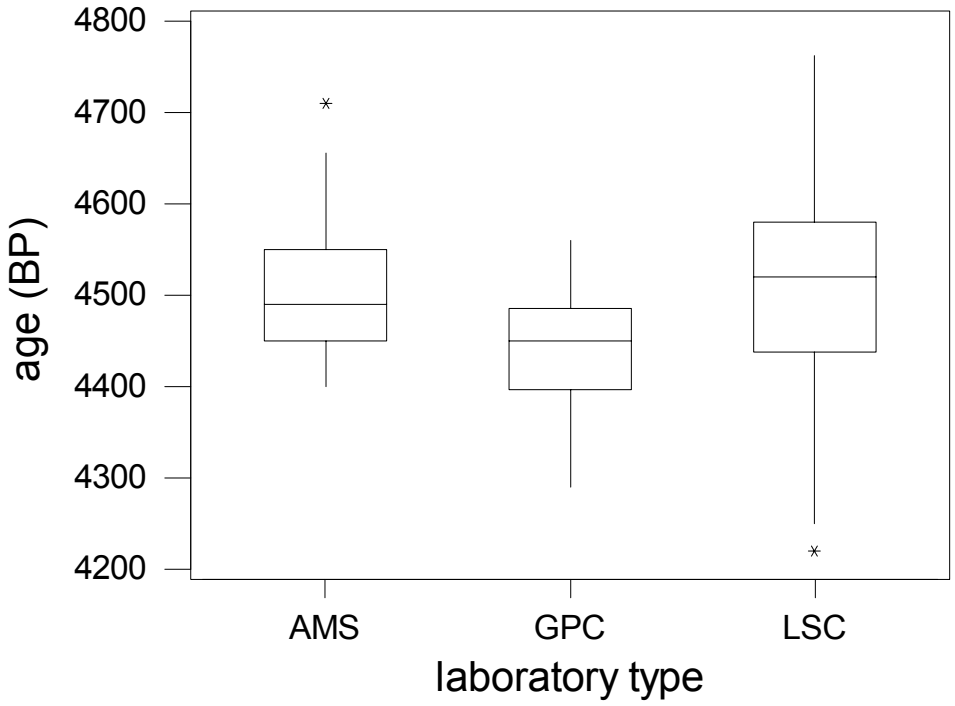

Figure 4.8 Distribution of age by laboratory type for Sample I

Table 4.17 Analysis of variance for Sample I

Source	DF	SS	MS	F	P
type	2	57840	28920	2.94	**0.058**
Error	83	815080	9820		
Total	85	872921			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
AMS	35	4499.1	74.1	(------*-----)
GPC	16	4438.0	68.6	(---------*--------)
LSC	35	4508.4	128.1	(------*-----)
				---+---------+---------+---------+---
Pooled StDev =		99.1		4400 4450 4500 4550

4.2.8.1 Conclusion

A statistically significant difference (at 10%) is found among the laboratory types. GPC laboratories quote an average age that is lower than either AMS or LSC laboratories.

4.2.9 Conclusions

In the case of Samples F and I, a statistically significant difference among the laboratory types was found at a 10% level. In general for all other samples, there is no evidence of a difference, on average, among laboratory types. We can conclude that laboratories are comparable on the average age/activity. However, where we have seen lack of comparability is in the number of outliers, with LSC laboratories (as can be seen from the preceding tables) having by far the largest number of measurements omitted. The other striking feature from the tables is the comparison of the standard deviations, which provide a measure of the scatter or variation in the population. In all cases, the AMS laboratory results have the smallest variation, in some cases by as much as a factor of 2.

4.3 MODERN STANDARD AND BACKGROUND MATERIAL AS SOURCES OF VARIATION

Two other factors of potential use in explaining the observed variation are the background and modern standard materials used. This section, thus, explores these 2 factors, with the reporting format identical to that used in Section 4.2. The classification of materials (and the analysis code used) is given below

Table 4.18 Coding for a) modern standard material and b) background material

a) Coding for modern standard material

Original description	Analysis classification/level
ANU sucrose	ASuc
Benzene	Benz(ene)
NIST OxI	NBS1
NIST OxII	NBS2
GIN/HD-95,C-3	Other
NIST 1/II	NBS12

b) Coding for background material

Original description	Analysis classification
Anthracite	Anth(racite)
Benzene	Benz(ene)
Calcite	Calc(ite)
Charcoal	Charc(oal)
Bituminous coal	Coal
Graphite	Graphite
Doublespar/IAEA C1	Marble
IAEA C4/wood/limestone	Other

4.3.1 Sample C: Marine Turbidite

Table 4.19a Descriptive statistics for age by modern standard used

Standard	N	Number omitted	Mean	Median	StDev
ASuc	8	1	18,294	18,225	217
Benz	3	2	17,918	17,820	211
NBS1	25	5	18,143	18,180	215
NBS12	9	0	18,123	18,100	188
NBS2	27	2	18,149	18,120	231
Other	5	0	18,107	18,138	248
Unknown	7	1	18,115	18,140	135

Table 4.19b Descriptive statistics for age by background material used

Background	N	Number omitted	Mean	Median	StDev
Anth	12	0	18,118	18,191	304
Benz	14	3	18,070	18,120	205
Calc	3	0	18,347	18,230	229
Coal	4	0	18,095	18,144	139
Graph	2	1	18,240	18,240	189
Marble	22	3	18,162	18,145	207
Other	14	3	18,157	18,227	216
Unknown	13	1	18,163	18,160	178

4.3.1.1 Graphical Analysis

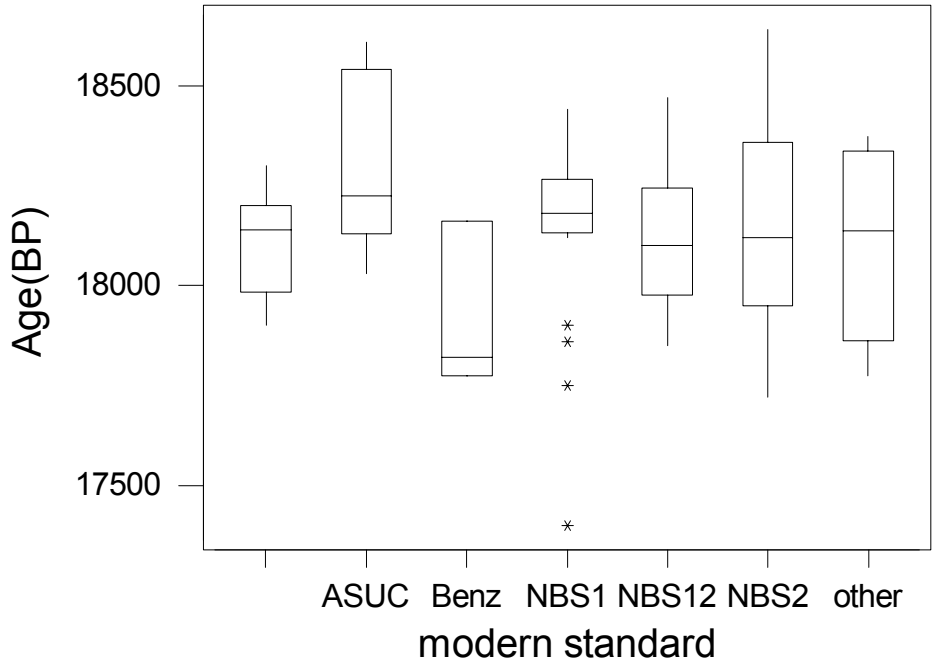

Figure 4.9a Distribution of age (yr BP) by modern standard material

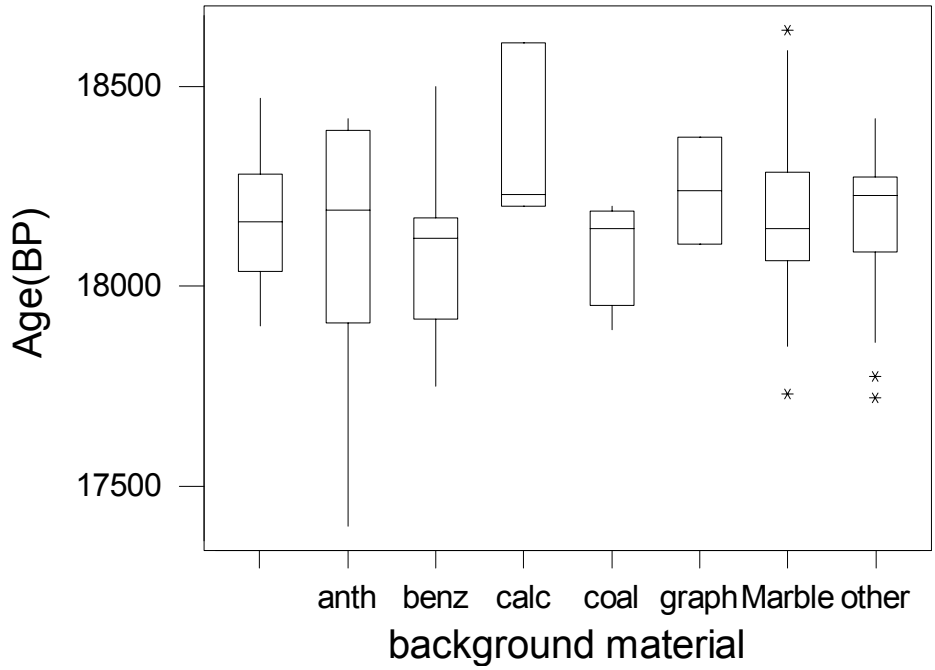

Figure 4.9b Distribution of age by background material

4.3.1.2 Formal Analysis

The formal analysis is carried out using a one-way analysis of variance (ANOVA); the hypothesis of interest is that the mean ^{14}C age is the same, irrespective of the modern standard or background material. The results are again summarized by the p-value.

Table 4.20a Analysis of variance of age by modern standard

Source	DF	SS	MS	F	P
Age	5	342707	68541	1.41	**0.230**
Error	71	3442176	48481		
Total	76	3784883			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
ASUC	8	18294	217	(-----*-----)
Benz	3	17918	211	(---------*---------)
NBS1	25	18143	215	(---*--)
NBS12	9	18123	188	(-----*-----)
NBS2	27	18149	231	(--*--)
other	5	18107	248	(-------*-------)
Pooled StDev =		220		17750 18000 18250 18500

Table 4.20b Analysis of variance of age by background material

```
Source      DF         SS         MS         F         P
Age          6     244377      40729      0.80     0.576
Error       64    3271008      51109
Total       70    3515384

                                      Individual 95% CIs For Mean
                                      Based on Pooled StDev
Level        N       Mean      StDev  -------+---------+---------+---------
anth        12      18118        304         (------*-----)
benz        14      18070        205       (------*-----)
calc         3      18347        229             (------------*------------)
coal         4      18095        139  (-----------*----------)
graph        2      18240        189     (---------------*---------------)
Marble      22      18162        207             (----*----)
other       14      18157        216           (-----*-----)
                                      -------+---------+---------+---------
Pooled StDev =       226               18000     18200     18400
```

4.3.1.3 Conclusion

For Sample C, there is no evidence that either the modern standard or background material used is a statistically significant factor in explaining the variation observed (p-value >0.05 in both cases).

4.3.2 Sample D: Belfast Wood

Table 4.21a Descriptive statistics age by modern standard material

Standard	N	Number omitted	Mean	Median	StDev
ASUC	6	1	4558.5	4565.0	95.4
Benz	3	3	4416.7	4420.0	90.0
NBS1	28	3	4505.4	4510.5	52.9
NBS12	7	0	4567.7	4550.0	51.4
NBS2	37	3	4537.8	4540.0	82.6
Other	7	1	4485.4	4482.0	35.3
Unknown	10	2	4516.8	4517.0	108.1

Table 4.21b Descriptive statistics age by background material

Background	N	Number omitted	Mean	Median	StDev
Anth	19	1	4530.3	4511.0	74.2
Benz	14	4	4515.1	4502.5	124.6
Calc	2	0	4530.0	4530.0	28.3
Charc	2	0	4525.0	4525.0	134.4
Coal	11	0	4487.9	4510.0	55.9
Graph	9	0	4513.7	4500.0	41.9
Marble	6	2	4505.5	4527.5	102.9
Other	18	3	4521.7	4519.0	56.6
Unknown	17	3	4551.8	4540.0	74.6

4.3.2.1 Graphical Analysis

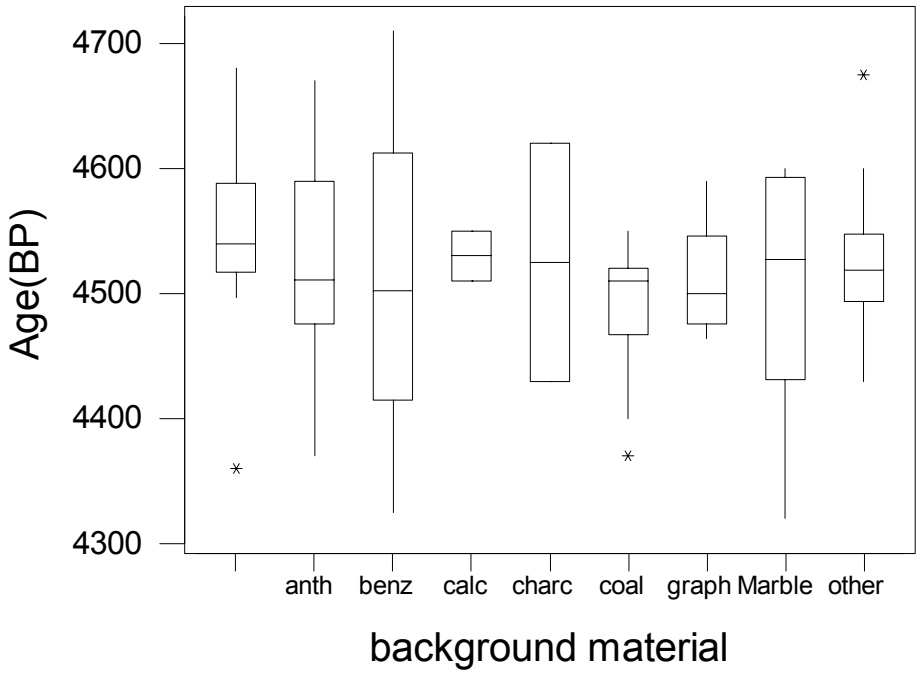

Figure 4.10a Distribution of age by background material

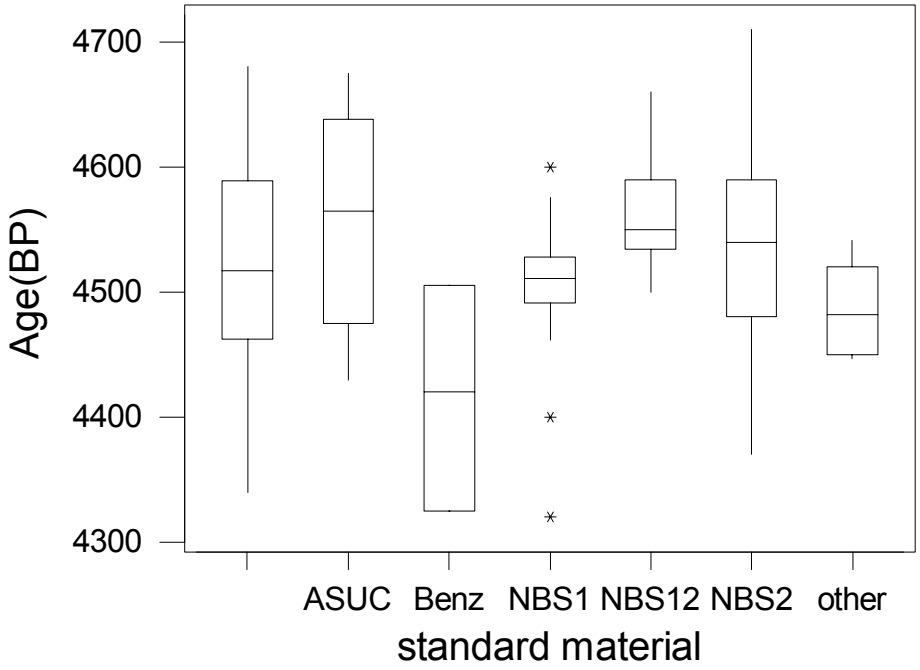

Figure 4.10b Distribution of age by modern standard material

4.3.2.2 Formal Analysis

Table 4.22a Analysis of variance of age by modern standard material

Source	DF	SS	MS	F	P
age	5	82173	16435	3.32	**0.009**
Error	82	406320	4955		
Total	87	488493			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
ASUC	6	4558.5	95.4	(------*------)
Benz	3	4416.7	90.0	(---------*---------)
NBS1	28	4505.4	52.9	(--*--)
NBS12	7	4567.7	51.4	(------*------)
NBS2	37	4537.8	82.6	(--*--)
other	7	4485.4	35.3	(------*-----)
Pooled StDev =		70.4		4400 4480 4560

Table 4.22b Analysis of variance of age by background material

Source	DF	SS	MS	F	P
age	7	14421	2060	0.32	**0.944**
Error	73	472634	6474		
Total	80	487055			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
anth	19	4530.3	74.2	(----*----)
benz	14	4515.1	124.6	(-----*-----)
calc	2	4530.0	28.3	(---------------*---------------)
charc	2	4525.0	134.4	(---------------*---------------)
coal	11	4487.9	55.9	(------*------)
graph	9	4513.7	41.9	(-------*------)
Marble	6	4505.5	102.9	(---------*--------)
other	18	4521.7	56.6	(----*----)
Pooled StDev =		80.5		4480 4550 4620

4.3.2.3 Conclusions

For Sample D, the modern standard is found to be statistically significant in explaining the observed variation. There are statistically significant differences among the mean ages for results based on the different modern standards. The modern standard material as a factor accounts for approximately 17% of the total variation observed. Laboratories using benzene as a modern standard material quote, on average, lower ages for this sample. The background material was not found statistically significant.

4.3.3 Sample F: Belfast Wood

Table 4.23a Descriptive statistics: age by background material

Background	N	Number omitted	Mean	Median	StDev
Anth	16	3	4507.9	4502.0	83.0
Benz	14	4	4507.1	4495.0	82.4
Calc	2	0	4472.5	4472.5	46.0
Charc	2	0	4525.0	4525.0	91.9
Coal	8	1	4461.8	4477.0	71.4
Graph	10	1	4554.9	4555.0	73.8
Marble	6	2	4551.8	4550.0	98.9
Other	18	2	4519.1	4511.5	66.6
Unknown	14	3	4488.9	4491.5	69.4

Table 4.23b Descriptive statistics: age by modern standard

Standard	N	Number omitted	Mean	Median	StDev
Asuc	5	3	4546.0	4550.0	63.5
Benz	5	1	4470.2	4459.0	116.0
NBS1	28	3	4502.4	4497.0	48.0
NBS12	9	0	4566.3	4550.0	63.8
NBS2	29	6	4518.4	4513.0	97.4
Other	8	0	4503.3	4487.0	42.1
Unknown	6	3	4442.8	4460.0	59.4

4.3.3.1 Graphical Analysis

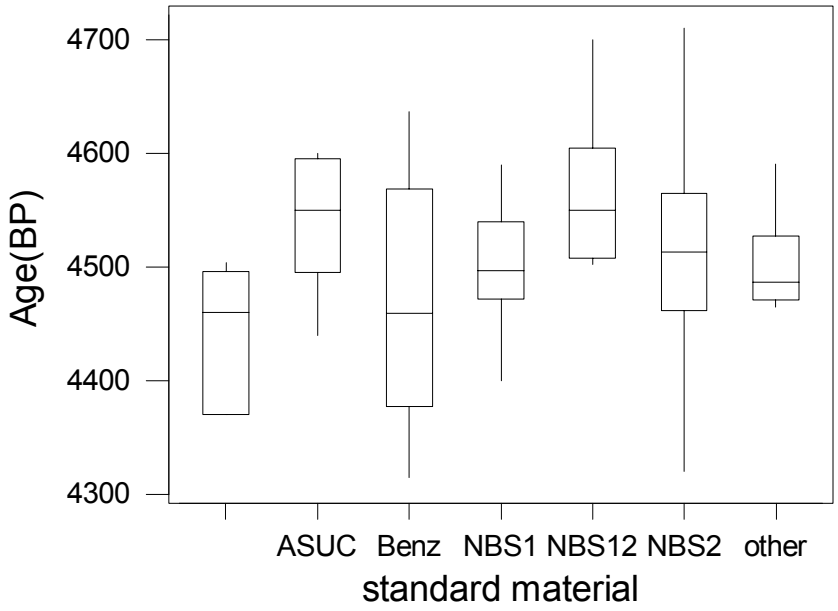

Figure 4.11a Distribution of age by modern standard material

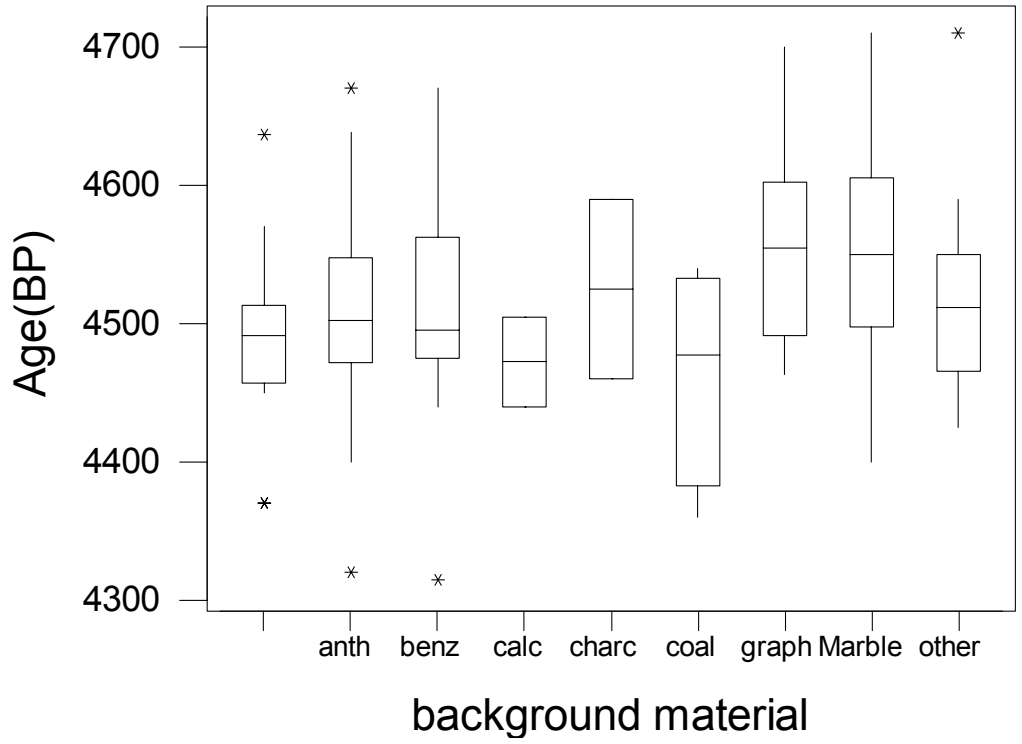

Figure 4.11b Distribution of age by background material

4.3.3.2 Formal Analysis

Table 4.24a Analysis of variance of age by background

Source	DF	SS	MS	F	P
age	7	52484	7498	1.24	**0.294**
Error	68	411176	6047		
Total	75	463660			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
anth	16	4507.9	83.0	(---*----)
benz	14	4507.1	82.4	(----*-----)
calc	2	4472.5	46.0	(-------------*-------------)
charc	2	4525.0	91.9	(-------------*------------)
coal	8	4461.8	71.4	(------*------)
graph	10	4554.9	73.8	(-----*-----)
Marble	6	4551.8	98.9	(-------*-------)
other	18	4519.1	66.6	(----*---)
Pooled StDev =		77.8		4400 4480 4560 4640

Table 4.24b Analysis of variance of age by modern standard

```
Source      DF         SS         MS         F         P
Age          5      44382       8876      1.56     0.180
Error       78     442573       5674
Total       83     486955

                                      Individual 95% CIs For Mean
                                      Based on Pooled StDev
Level        N       Mean      StDev  -------+---------+---------+---------
ASUC         5     4546.0       63.5               (-----------*----------)
Benz         5     4470.2      116.0   (----------*----------)
NBS1        28     4502.4       48.0               (---*----)
NBS12        9     4566.3       63.8                      (-------*-------)
NBS2        29     4518.4       97.4                 (----*----)
other        8     4503.3       42.1            (--------*-------)
                                      -------+---------+---------+---------
Pooled StDev =      75.3               4440      4500      4560
```

4.3.3.3 Conclusions

Neither modern standard nor background materials proved to be statistically significant in explaining the observed variation.

4.3.4 Sample E: Humic Acid

Table 4.25a Descriptive statistics: age by modern standard

Standard	N	Number omitted	Mean	Median	StDev
ASUC	8	2	11,712	11,715	130
Benz	4	2	11,681	11,682	58
NBS1	49	7	11,779	11,800	144
NBS12	5	0	11,781	11,770	87
NBS2	37	6	11,813	11,809	167
Other	14	0	11,785	11,771	175
Unknown	8	2	11,710	11,721	91

Table 4.25b Descriptive statistics: age by background material

Background	N	Number omitted	Mean	Median	StDev
Anth	20	3	11,847	11,855	139
Benz	18	4	11,761	11,700	208
Calc	2	0	11,715	11,715	78
Charc	13	0	11,832	11,800	90
Coal	18	0	11,804	11,805	141
Graph	15	0	11,734	11,772	158
Marble	9	2	11,754	11,760	176
Other	17	5	11,729	11,731	126
Unknown	13	5	11,748	11,760	103

4.3.4.1 Graphical Analysis

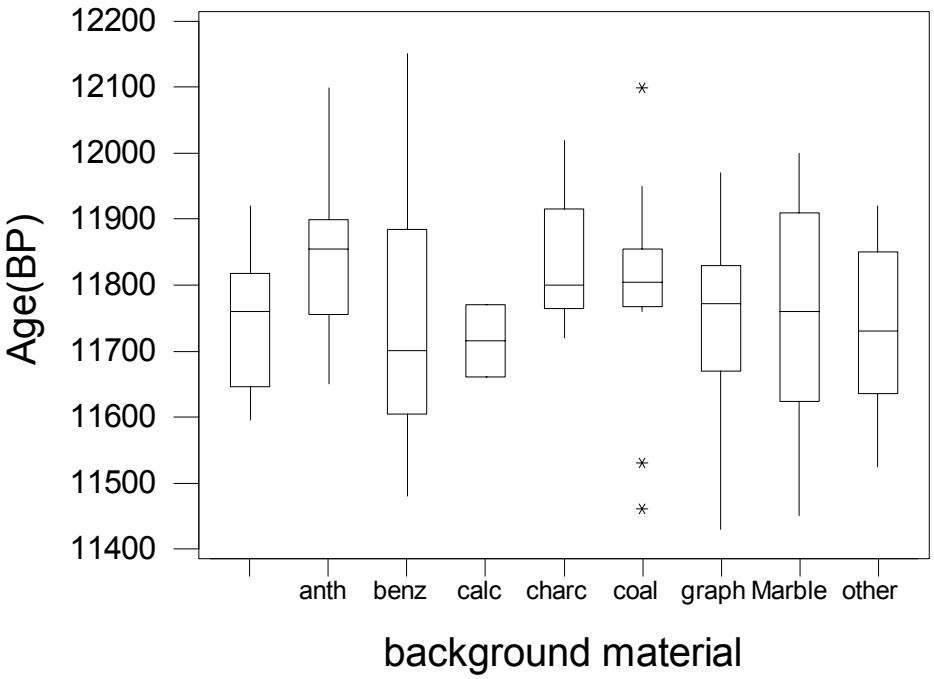

Figure 4.12a Distribution of age by background material

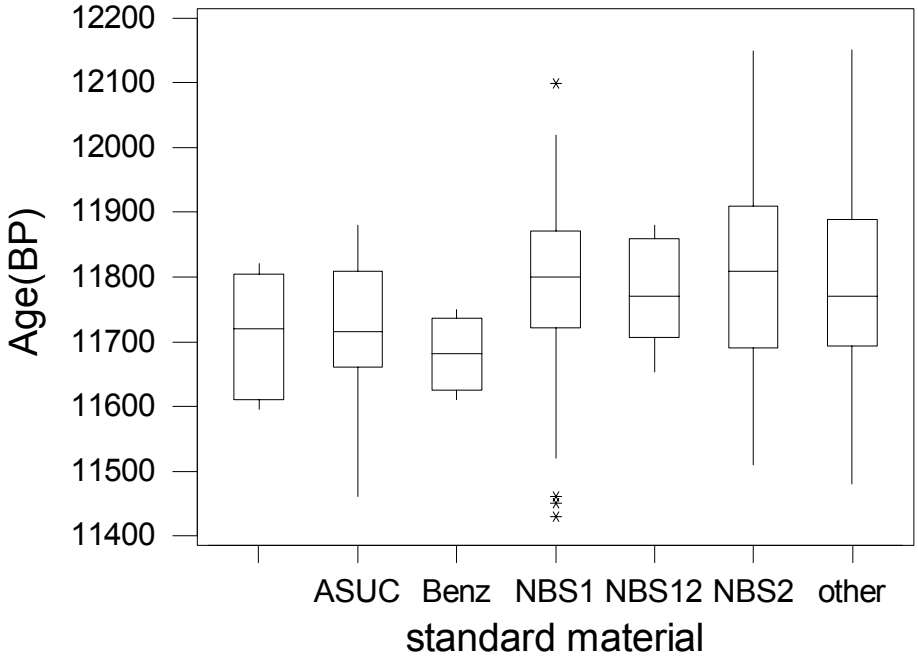

Figure 4.12b Distribution of age by modern standard material

4.3.4.2 Formal Analysis

Table 4.26a Analysis of variance of age by modern standard

Source	DF	SS	MS	F	P
Age	5	117339	23468	1.02	**0.409**
Error	111	2552299	22994		
Total	116	2669638			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
ASUC	8	11712	130	(--------*--------)
Benz	4	11681	58	(-----------*------------)
NBS1	49	11779	144	(---*--)
NBS12	5	11781	87	(----------*----------)
NBS2	37	11813	167	(---*----)
other	14	11785	175	(------*------)
Pooled StDev =	152			11640 11760 11880

Table 4.26b Analysis of variance of age by background material

Source	DF	SS	MS	F	P
background	7	234848	33550	1.46	**0.190**
Error	104	2392794	23008		
Total	111	2627641			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
anth	20	11847	139	(----*-----)
benz	18	11761	208	(-----*-----)
calc	2	11715	78	(----------*----------)
charc	13	11832	90	(------*------)
coal	18	11804	141	(-----*-----)
graph	15	11734	158	(------*-----)
Marble	9	11754	176	(-------*--------)
other	17	11729	126	(-----*------)
Pooled StDev =	152			11520 11640 11760 11880

4.3.4.3 Conclusions

For Sample E, neither modern standard nor background materials are statistically significant in explaining the observed variation.

4.3.5 Sample G: Barley Mash

Table 4.27a Descriptive statistics: activity (pMC) by background material

Background	N	Number omitted	Mean	Median	StDev
Anth	15	3	110.30	110.86	1.48
Benz	16	3	110.46	110.85	1.28
Calc	2	0	110.27	110.27	0.01
Charc	2	0	110.90	110.90	0.70
Coal	7	0	110.14	110.13	1.70
Graph	10	0	110.33	110.49	0.64
Marble	6	2	110.83	110.75	0.99
Other	16	3	110.35	110.28	0.81
Unknown	12	3	110.71	110.80	0.66

Table 4.27b Descriptive statistics: activity (pMC) by modern standard

Standard	N	Number omitted	Mean	Median	StDev
ASUC	6	1	109.39	109.75	1.32
Benz	5	1	111.10	111.03	1.10
NBS1	26	3	110.46	110.22	0.92
NBS12	7	0	110.36	110.26	0.62
NBS2	29	6	110.39	110.53	1.36
Other	7	1	110.82	110.84	0.40
Unknown	6	2	110.68	110.75	0.19

4.3.5.1 Graphical Analysis

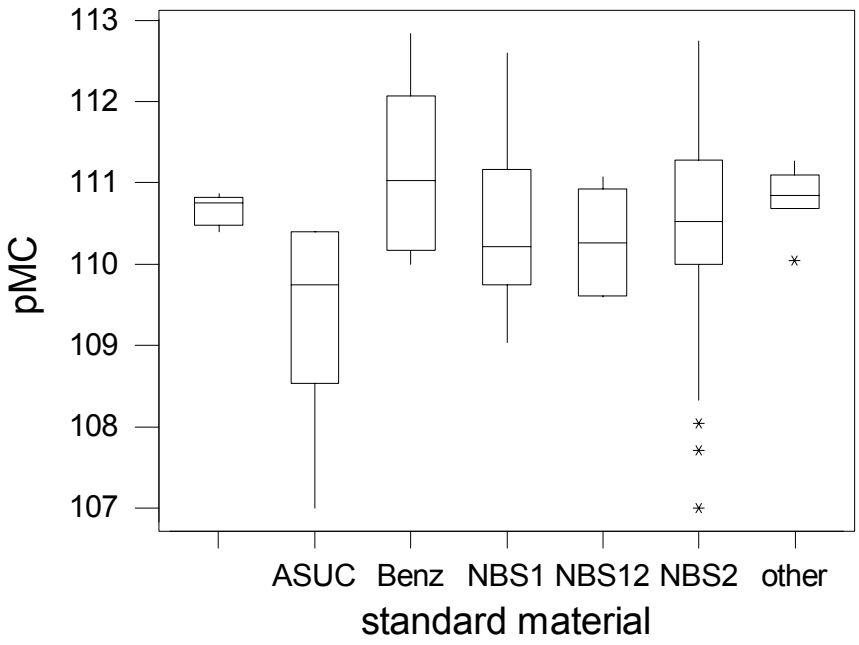

Figure 4.13a Distribution of activity by modern standard

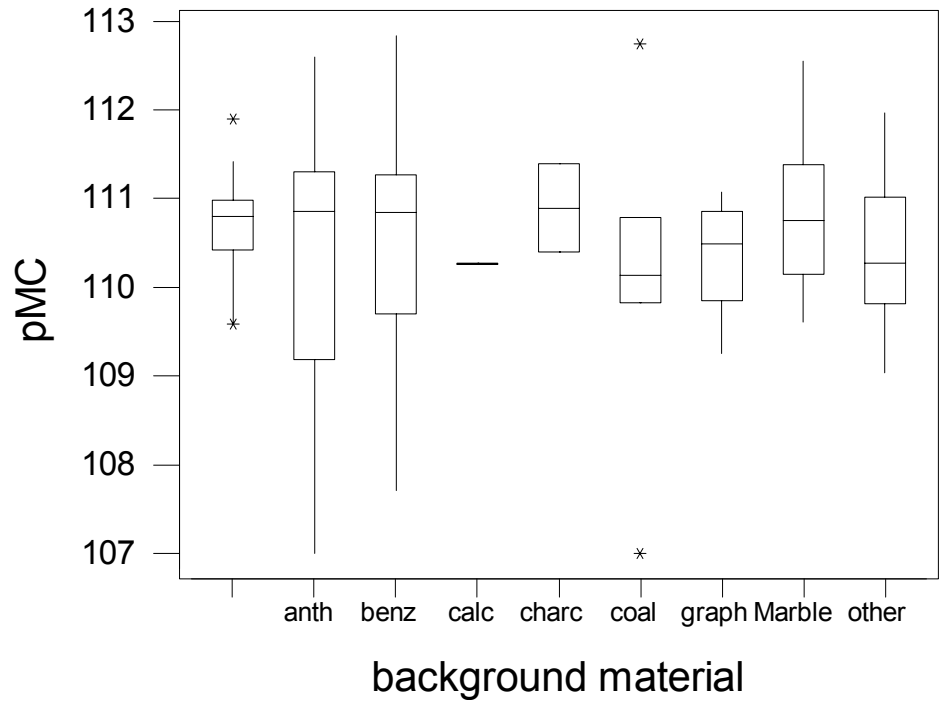

Figure 4.13b Distribution of activity by background material

4.3.5.2 Formal Analysis

Table 4.28a Analysis of variance of activity by background material

Source	DF	SS	MS	F	P
background	7	2.42	0.35	0.25	**0.971**
Error	66	91.67	1.39		
Total	73	94.09			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
anth	15	110.30	1.48	(----*----)
benz	16	110.46	1.28	(----*---)
calc	2	110.27	0.01	(----------*----------)
charc	2	110.90	0.70	(----------*----------)
coal	7	110.14	1.70	(-------*------)
graph	10	110.33	0.64	(-----*------)
Marble	6	110.83	0.99	(-------*-------)
other	16	110.35	0.81	(----*---)
Pooled StDev =		1.18		109.2 110.4 111.6 112.8

Table 4.28b Analysis of variance of activity by modern standard

```
Source      DF        SS        MS         F         P
standard     5      9.86      1.97      1.63     0.162
Error       74     89.45      1.21
Total       79     99.31

                                  Individual 95% CIs For Mean
                                  Based on Pooled StDev
Level       N      Mean     StDev  ------+---------+---------+---------+
ASUC        6    109.39      1.32   (--------*--------)
Benz        5    111.10      1.10           (---------*---------)
NBS1       26    110.46      0.92                   (----*---)
NBS12       7    110.36      0.62              (--------*-------)
NBS2       29    110.39      1.36                   (---*---)
other       7    110.82      0.40                   (-------*-------)
                                 ------+---------+---------+---------+
Pooled StDev =     1.10              109.0     110.0     111.0     112.0
```

4.3.5.3 Conclusions

Neither modern standard nor background materials are statistically significant in explaining the variation in activity.

4.3.6 Sample J: Barley Mash

Table 4.29a Descriptive statistics: activity (pMC) by modern standard

Standard	N	Number omitted	Mean	Median	StDev
ASUC	4	1	109.89	109.83	1.19
Benz	5	1	110.63	110.22	1.30
NBS1	27	2	110.52	110.50	0.77
NBS12	8	0	110.77	110.56	0.78
NBS2	28	7	110.64	110.71	0.98
Other	8	0	111.13	111.01	0.59
Unknown	7	2	110.40	110.90	1.12

Table 4.29b Descriptive statistics: activity (pMC) by background material

Background	N	Number omitted	Mean	Median	StDev
Anth	17	1	110.45	110.70	1.15
Benz	13	5	111.23	111.03	0.80
Calc	2	0	110.00	110.00	0.24
Charc	2	0	111.39	111.39	0.01
Coal	7	0	110.51	110.92	0.81
Graph	10	1	110.85	110.61	0.64
Marble	7	2	110.68	110.70	0.75
Other	16	2	110.29	110.23	0.67
Unknown	13	2	110.38	110.70	1.11

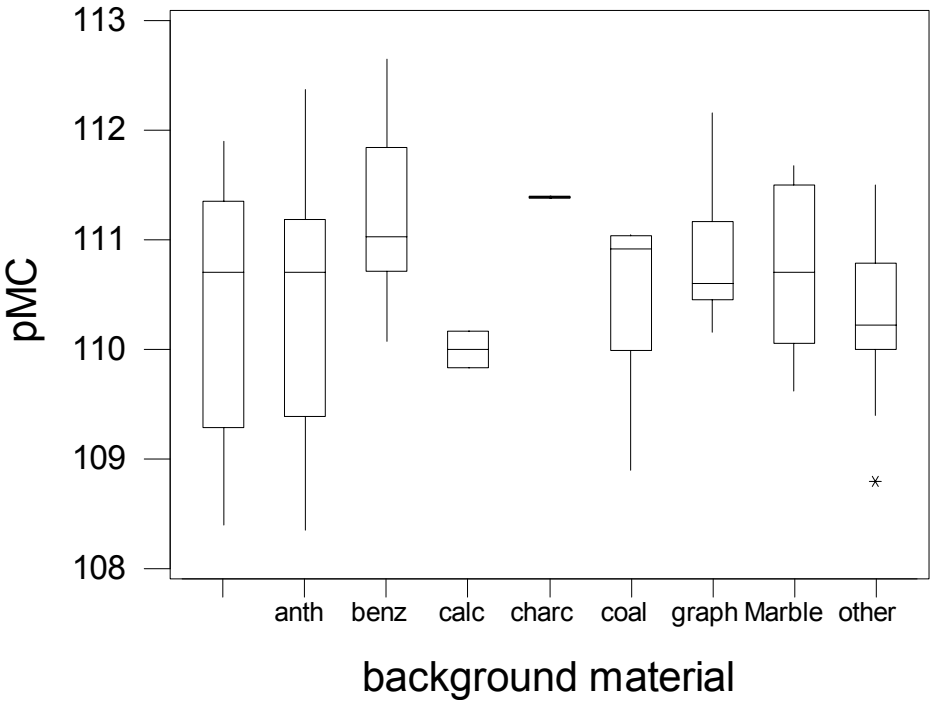

Figure 4.14a Distribution of activity by background material

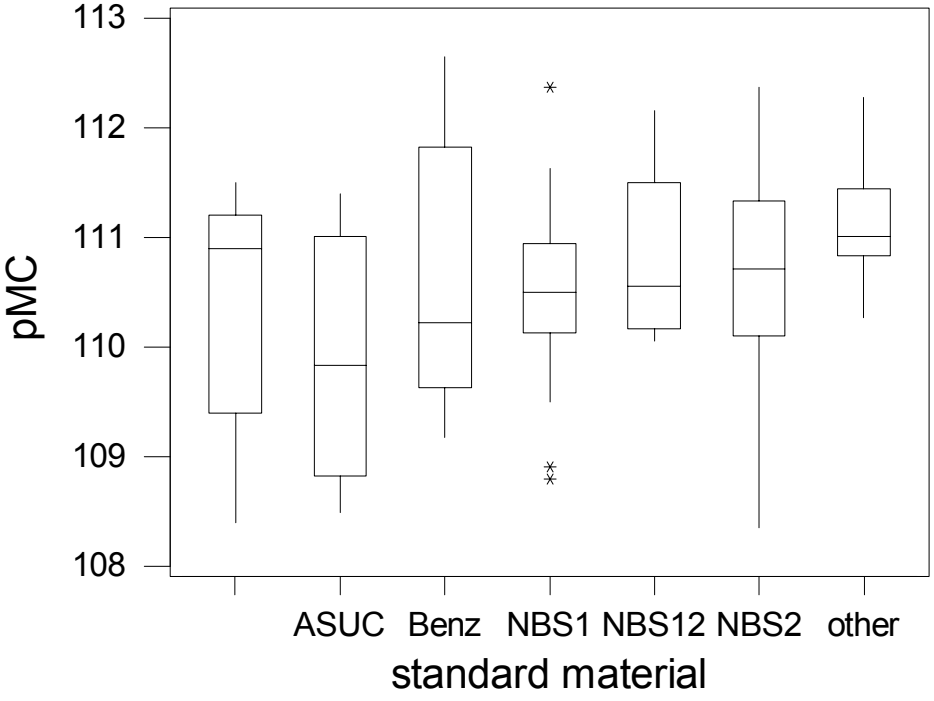

Figure 4.14b Distribution of activity by modern standard

4.3.6.2 Formal Analysis

Table 4.30a Analysis of variance of activity by modern standard

Source	DF	SS	MS	F	P
Standard	5	4.711	0.942	1.18	**0.329**
Error	74	59.228	0.800		
Total	79	63.939			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
ASUC	4	109.888	1.190	(----------*----------)
Benz	5	110.626	1.303	(---------*---------)
NBS1	27	110.520	0.771	(----*---)
NBS12	8	110.768	0.781	(-------*------)
NBS2	28	110.643	0.983	(---*---)
other	8	111.133	0.586	(-------*-------)
Pooled StDev =		0.895		109.60 110.40 111.20

Table 4.30b Analysis of variance of activity by background material

Source	DF	SS	MS	F	P
background	7	9.538	1.363	1.93	**0.079**
Error	66	46.641	0.707		
Total	73	56.179			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
anth	17	110.447	1.154	(--*---)
benz	13	111.226	0.797	(---*---)
calc	2	110.000	0.240	(---------*---------)
charc	2	111.390	0.014	(---------*---------)
coal	7	110.513	0.807	(----*----)
graph	10	110.849	0.643	(----*---)
Marble	7	110.676	0.746	(----*-----)
other	16	110.291	0.668	(--*---)
Pooled StDev =		0.841		109.2 110.4 111.6 112.8

4.3.6.3 Conclusions

The background material is statistically significant at the 10% level, but the modern standard is not a statistically significant factor.

4.3.7 Sample H: Hohenheim Wood

Table 4.31a Descriptive statistics: age by background material

Background	N	Number omitted	Mean	Median	StDev
Anth	15	4	2254.3	2240.0	74.9
Benz	12	6	2222.3	2249.0	102.6
Calc	2	0	2302.5	2302.5	53.0
Charc	2	0	2230.0	2230.0	14.1
Coal	9	0	2204.6	2210.0	52.3
Graph	10	0	2215.7	2215.0	62.9
Marble	8	0	2210.3	2205.0	118.4
Other	16	2	2233.6	2240.0	87.0
Unknown	14	2	2228.2	2209.5	66.2

Table 4.31b Descriptive statistics: age by modern standard

Standard	N	Number omitted	Mean	Median	StDev
ASUC	6	0	2276.0	2280.0	127.0
Benz	4	2	2175.8	2133.0	155.0
NBS1	27	2	2211.7	2210.0	67.5
NBS12	8	0	2219.9	2225.0	33.4
NBS2	28	7	2240.1	2240.0	77.7
Other	7	1	2282.4	2280.0	79.2
Unknown	8	2	2203.3	2190.0	43.7

4.3.7.2 Formal Analysis

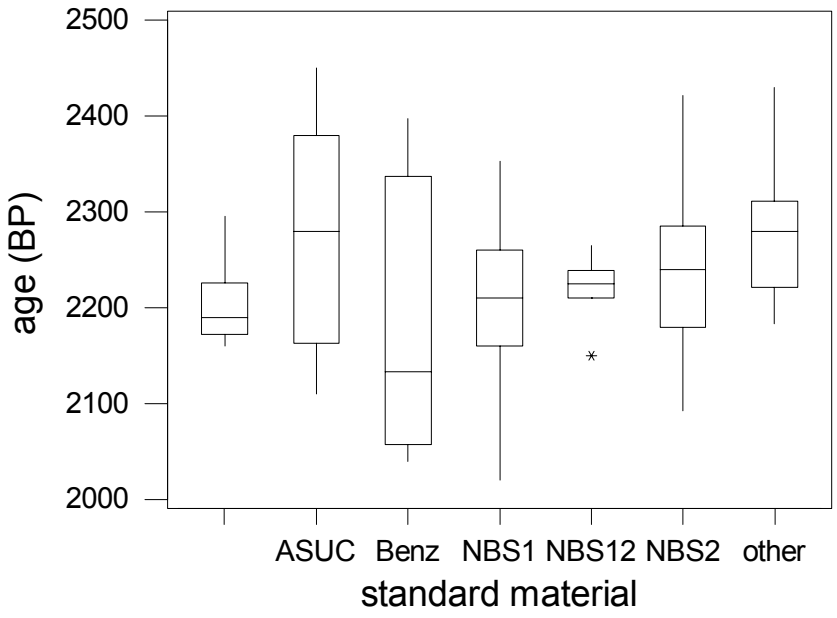

Figure 4.15a Distribution of age by modern standard

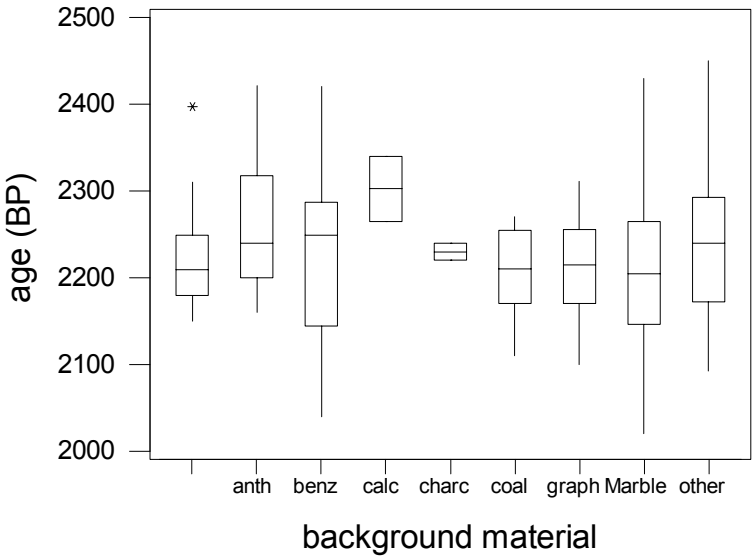

Figure 4.15b Distribution of age by background material

Table 4.32a Analysis of variance of age by background material

Source	DF	SS	MS	F	P
background	7	31284	4469	0.63	**0.728**
Error	66	466699	7071		
Total	73	497983			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
anth	15	2254.3	74.9	(---*----)
benz	12	2222.3	102.6	(----*----)
calc	2	2302.5	53.0	(----------*----------)
charc	2	2230.0	14.1	(----------*----------)
coal	9	2204.6	52.3	(----*-----)
graph	10	2215.7	62.9	(-----*----)
Marble	8	2210.3	118.4	(-----*-----)
other	16	2233.6	87.0	(---*----)
Pooled StDev =		84.1		2200 2300 2400

Table 4.32b Analysis of variance of age by modern standard

Source	DF	SS	MS	F	P
standard	5	56175	11235	1.73	**0.138**
Error	74	479615	6481		
Total	79	535790			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
ASUC	6	2276.0	127.0	(--------*--------)
Benz	4	2175.8	155.0	(-----------*----------)
NBS1	27	2211.7	67.5	(---*---)
NBS12	8	2219.9	33.4	(-------*-------)
NBS2	28	2240.1	77.7	(---*---)
other	7	2282.4	79.2	(--------*--------)
Pooled StDev =		80.5		2100 2170 2240 2310

4.3.7.3 Conclusions

Again, neither standard nor background materials are statistically significant.

4.3.8 Sample I: Belfast Cellulose

Table 4.33a Descriptive statistics: age by modern standard

Standard	N	Number omitted	Mean	Median	StDev
ASUC	6	2	4568.7	4555.0	120.9
Benz	4	1	4495	4530	201
NBS1	25	3	4473.5	4468.0	73.2
NBS12	8	0	4490.8	4490.0	53.9
NBS2	31	3	4500.8	4500.0	116.7
Other	6	2	4500.5	4455.5	88.6
Unknown	6	3	4431.7	4430.0	51.3

Table 4.33b Descriptive statistics: age by background material

Background	N	N*	Mean	Median	StDev
Anth	16	1	4483.3	4490.0	111.7
Benz	15	4	4523.5	4520.0	156.3
Calc	2	0	4540.0	4540.0	56.6
Charc	2	0	4420.0	4420.0	14.1
Coal	6	2	4485.0	4490.0	53.9
Graph	11	0	4458.6	4461.0	87.5
Marble	6	2	4474.0	4480.0	76.8
Other	16	3	4513.0	4495.0	82.4
Unknown	12	2	4480.2	4485.0	75.9

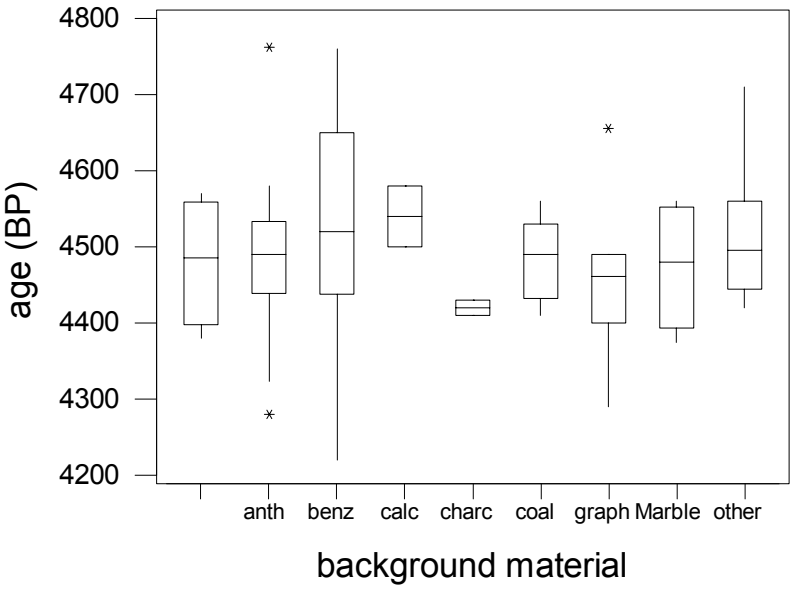

Figure 4.16a Distribution of age by background material

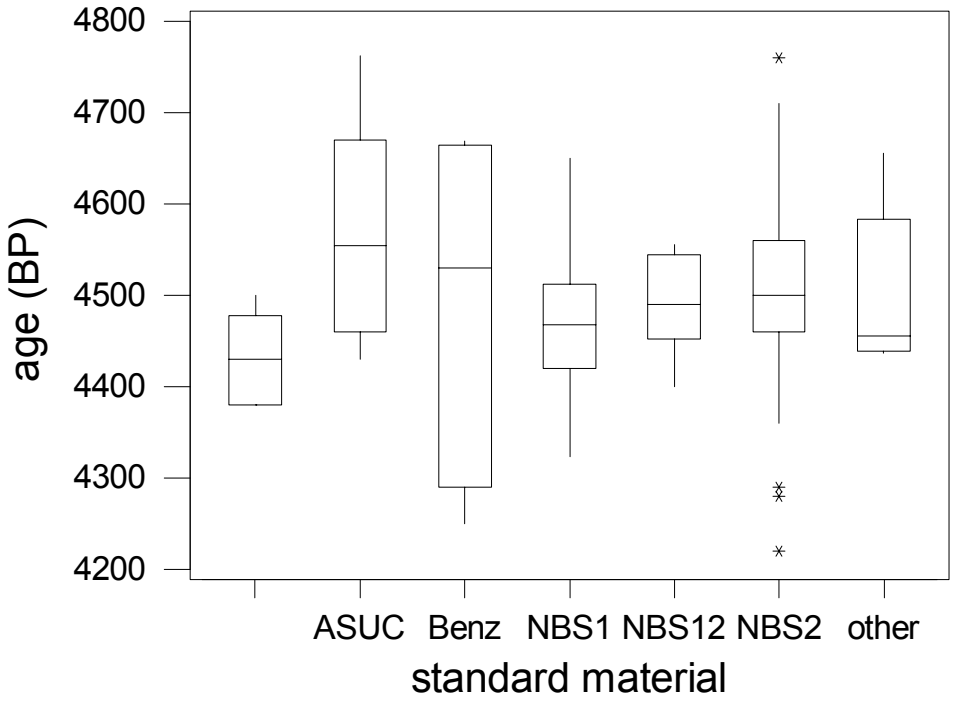

Figure 4.16b Distribution of age by modern standard

4.3.8.2 Formal analysis

Table 4.34a Analysis of variance of age by modern standard

Source	DF	SS	MS	F	P
standard	5	45428	9086	0.85	0.519
Error	74	791207	10692		
Total	79	836635			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
ASUC	6	4568.7	120.9	(---------*---------)
Benz	4	4494.8	201.3	(----------*----------)
NBS1	25	4473.5	73.2	(----*----)
NBS12	8	4490.8	53.9	(--------*--------)
NBS2	31	4500.8	116.7	(----*---)
other	6	4500.5	88.6	(----------*---------)
Pooled StDev =		103.4		4400 4480 4560 4640

4.3.8.3 Conclusions

The background and standard materials are not statistically significant factors in explaining the observed variation.

Table 4.34b Analysis of variance of age by background material

Source	DF	SS	MS	F	P
background	7	52502	7500	0.66	**0.709**
Error	66	755202	11442		
Total	73	807704			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
anth	16	4483.3	111.7	(----*---)
benz	15	4523.5	156.3	(----*----)
calc	2	4540.0	56.6	(-----------*-----------)
charc	2	4420.0	14.1	(-----------*-----------)
coal	6	4485.0	53.9	(-------*------)
graph	11	4458.6	87.5	(-----*----)
Marble	6	4474.0	76.8	(------*------)
other	16	4513.0	82.4	(---*----)
Pooled StDev =		107.0		4320 4440 4560 4680

4.4 SUMMARY FINDINGS OF BACKGROUND AND STANDARD MATERIAL EFFECTS

The information from this analysis is summarized in the table below. With few exceptions, after omission of outliers, the background and standard material is not a significant factor in explaining the observed variation. A preliminary analysis, before omission of the outliers, had however shown that standard was often a significant factor. Thus, one inference from this is that the modern standard is an important factor, but that it may be an indirect measure of the laboratory capability and experience and may be related to the presence of outliers.

Table 4.35 Summary of analysis of variance findings[a]

FIRI sample	Background	Standard
A (pmC)	S	S
B (pmC)	S (10%)	S
C	NS	NS
D	NS	S
E	NS	NS
F	NS	NS
G	NS	NS
H	NS	NS
I	NS	NS
J	S (10%)	NS

[a] S = significant; NS = not significant

4.5 PRETREATMENT AS A FACTOR IN EXPLAINING THE VARIATION

Pretreatment may also be a source of variation, but it should only be appropriate in a few samples, specifically the whole wood and the turbidite. The following tables enumerate the numbers of laboratories using a specific or general method for the samples. It also contains information pertinent to the issue of the explanation for outliers. Not all laboratories reported this information, so the table is incomplete.

Table 4.36 Number of laboratories (and laboratories with outliers) using specified pretreatment methods for Samples D, F, H, and I

Samples D, F, and H (Wood)			Sample I (Cellulose)		
Method	Non-outlier laboratories	All laboratories	Method	Non-outlier laboratories	All laboratories
AAA	40	41	AAA	1	1
AAAA	2	2	Missing	10	13
Cellulose	14	14	None	56	62
Missing	11	11	Other	9	11
Other	4	5	**Total**	**76**	**87**
None	4	4			
More than 1	8	8			
Total	**83**	**85**			

Table 4.37 Number of laboratories (and laboratories with outliers) using specified pretreatment methods for Sample C and Sample E

Sample C (Turbidite)			Sample E (Humic acid)		
Method	Non-outlier laboratories	All laboratories	Method	Non-outlier laboratories	All laboratories
Acid leaching	5	7	AAA	1	1
Missing	10	11	Missing	6	11
None	54	69	None	53	59
Other	1	2	Other	8	11
Total	**70**	**79**	**Total**	**68**	**82**

Table 4.38 Number of laboratories (and laboratories with outliers) using specified pretreatment methods for Samples G and J

Sample G and J (barley mash)		
Method	Non-outlier laboratories	All laboratories
AAA	5	7
Missing	10	11
None	54	69
Other	1	2
Total	**70**	**79**

Table 4.39 Description of pretreatment methods classed as "other" for each sample type

Sample	Wood (D, F, and H)	Turbidite (C)	Humic acid (E)	Barley mash (G and J)	Cellulose (I)
Other methods	Acid only "Routine"	AAA "Routine"	Burning AAA "Routine"	AAA "Routine"	Burning AAA "Routine"

A variety of pretreatment methods have been used, even for Sample C (turbidite) where the instructions stated that the sample should not be pretreated. AAA was the most commonly used method, with 14 laboratories extracting cellulose from the wood samples.

4.5.1 Sample C (Turbidite)

Table 4.40 Descriptive statistics: age by pretreatment method

Pretreatment	N	Mean	Median	StDev
Acid leach	9	18,037	18,090	150
Missing	10	18,166	18,165	153
None	64	18,166	18,174	209
Other	1	18,359	18,359	*

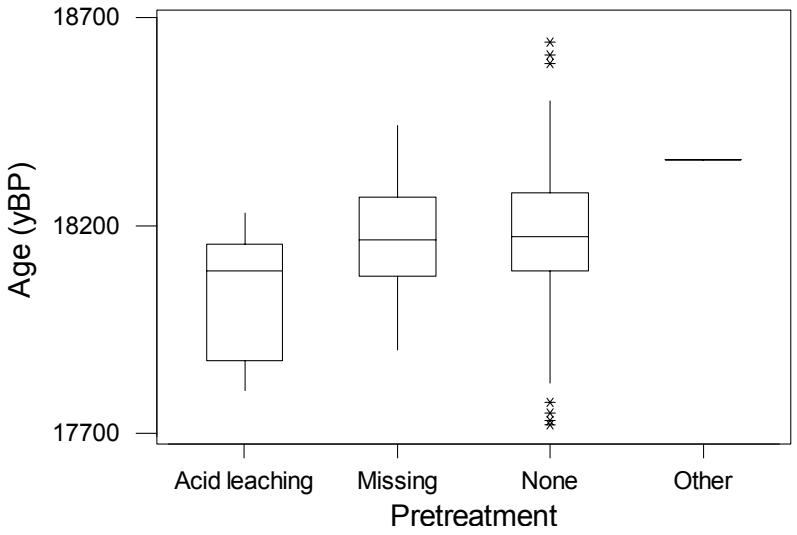

Figure 4.17 Distribution of age by pretreatment

A formal analysis to compare the results of the acid leaching and not pretreating gave a p-value of 0.04, indicating that there was a significant difference between the acid-leached and non-pretreated results. Such a difference had already been seen in the homogeneity testing and was the reason for the instruction that this sample should not be pretreated. In this case, the acid-leached results are younger.

4.5.2 Sample H (Hohenheim dendro-dated wood) and Samples D and F (Belfast)

Table 4.41 Descriptive statistics: age for Sample H

Pretreatment	N	Mean	Median	StDev
AAA	50	2230.3	2230.0	86.4
AAAA	2	2207.5	2207.5	46.0
Cell. ex	17	2246.6	2248.0	77.9
Missing	11	2230.7	2209.0	66.4
None	4	2157.5	2170.0	33.0
Other	4	2215.5	2195.0	72.4

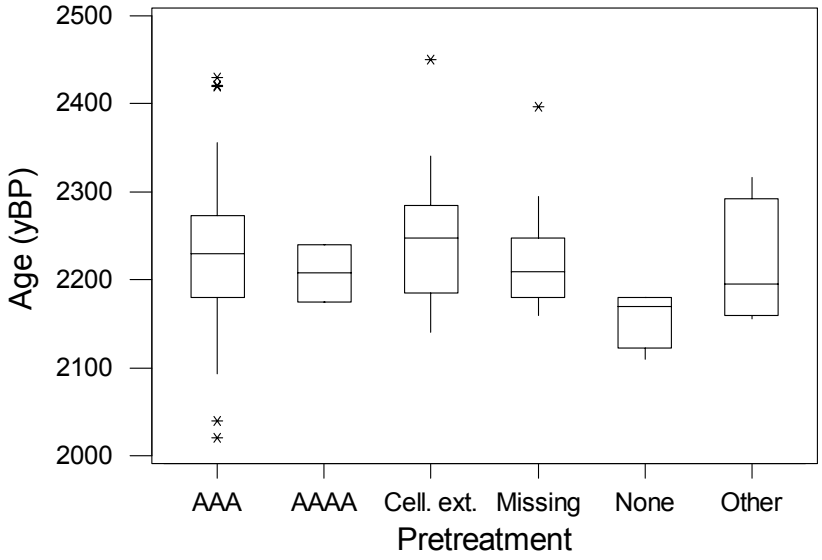

Figure 4.18 Distribution of age by pretreatment

Table 4.42 Descriptive statistics for age of Samples D and F

Pretreatment	N	Mean	Median	StDev
AAA	100	4523.3	4510.5	73.1
AAAA	4	4468.5	4472.5	16.1
Cell. ex	43	4533.1	4540.0	73.7
Missing	25	4488.1	4493.0	92.6
None	4	4468.8	4477.5	37.1
Other	9	504.1	4505.0	91.3

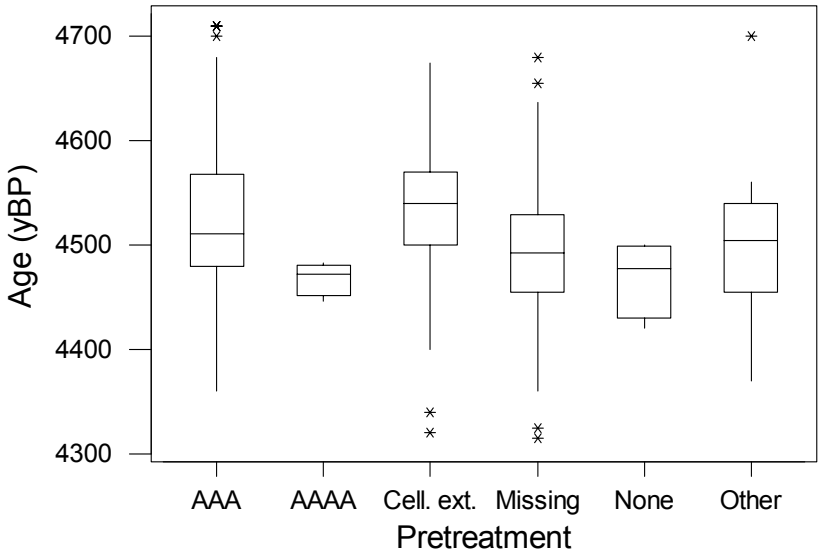

Figure 4.19 Distribution of age by pretreatment method

4.5.2.1 Samples D, F, and H (Wood)

Table 4.43 Descriptive statistics of deviation from consensus for all wood samples by pretreatment method

Pretreatment	N	Mean	Median	TrMean	StDev
AAA	150	–0.0586	–0.0387	–0.0531	0.6407
AAAA	6	0.2604	0.2663	0.2604	0.1952
Cell. ex	60	–0.1740	–0.1651	–0.1749	0.5594
Missing	36	0.119	0.125	0.135	0.643
None	9	0.447	0.506	0.447	0.358
Other	13	0.085	0.164	0.129	0.630

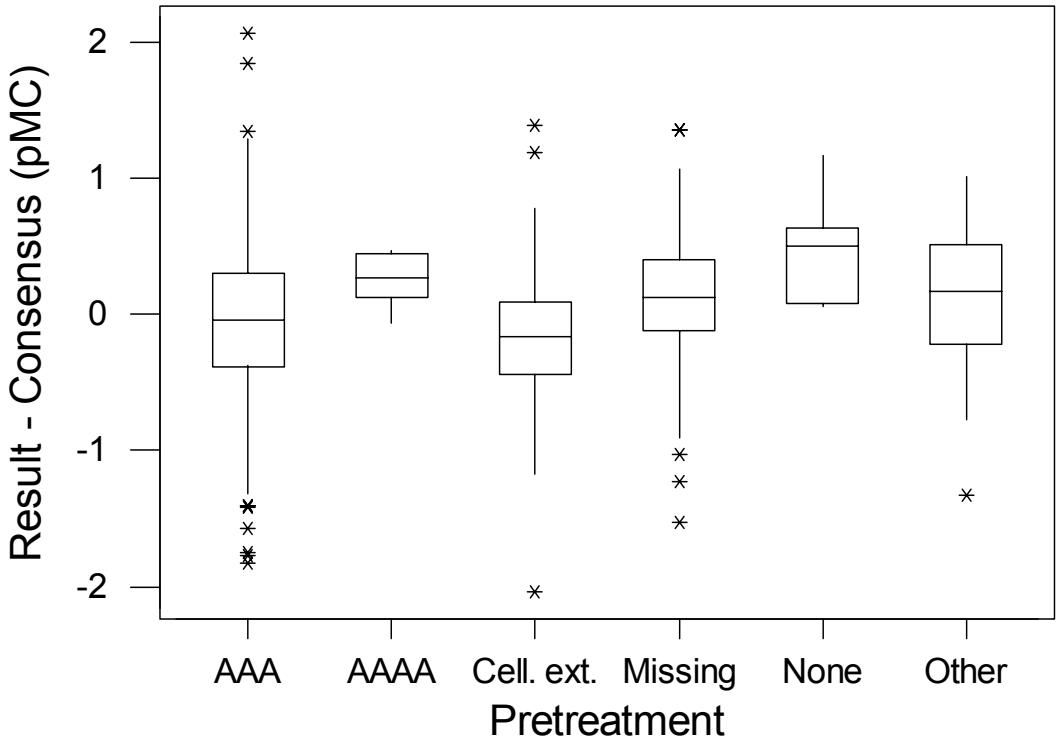

Figure 4.20 Distribution of deviation from consensus by pretreatment method

To use all the wood samples together, we have centered each sample at the consensus value (see Section 7) and investigated if method of pretreatment may be a source of variation.

4.5.2.2 Formal Analysis

Table 4.44 Analysis of variance of deviation from consensus by pretreatment method

```
Source      DF        SS        MS        F        P
Pretreat     5     4.894     0.979     2.62    0.025
Error      268   100.062     0.373
Total      273   104.956

                                    Individual 95% CIs For Mean
                                    Based on Pooled StDev
Level       N      Mean     StDev  ----------+---------+---------+------
AAA       150   -0.0586    0.6407        (-*--)
AAAA        6    0.2604    0.1952     (-------------*-------------)
Cell. ex   60   -0.1740    0.5594   (---*---)
Missing    36    0.1190    0.6426          (----*-----)
None        9    0.4471    0.3582             (-----------*----------)
Other      13    0.0851    0.6299     (--------*---------)
                                   ----------+---------+---------+------
Pooled StDev =   0.6110                    0.00      0.35      0.70
```

4.5.2.3 Conclusions

A statistically significant effect of the pretreatment method was found (p-value <0.05). There is a large amount of literature about the effects of pretreatment, and specifically for wood samples. The magnitude of the effect here, while statistically significant is, in fact, of little practical importance since any differences are very small.

4.5.3 Sample E: Humic Acid

Table 4.45 Descriptive statistics: age by pretreatment method

Pretreatment	N	Mean	Median	StDev
Missing	6	11,720	11,756	98
None	61	11,778	11,778	143
Other	9	11,742	11,720	163

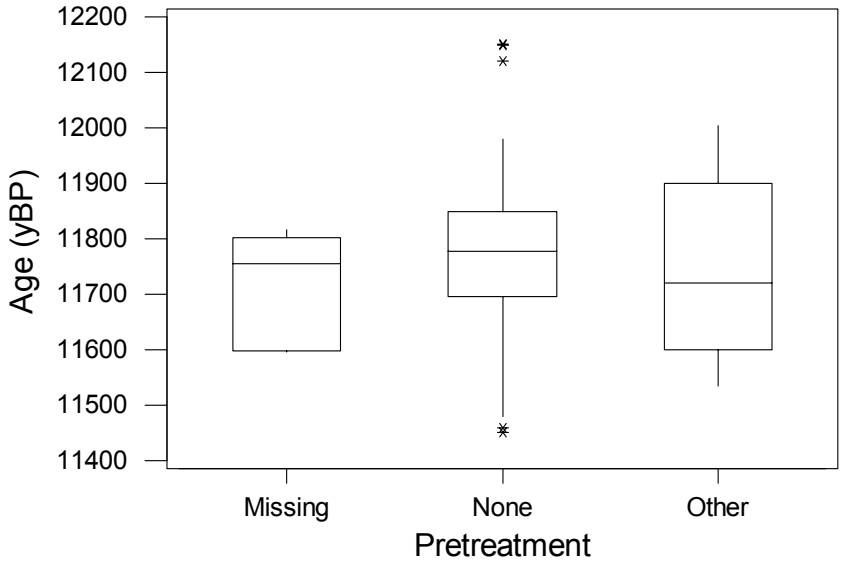

Figure 4.21 Distribution of age by pretreatment method

4.5.3.1 Formal Analysis

Table 4.46 Analysis of variance by pretreatment method

```
Analysis of Variance for Age
Source     DF        SS        MS        F        P
Pretreat    2     25789     12895     0.64    0.532
Error      73   1479517     20267
Total      75   1505306

                                 Individual 95% CIs For Mean
                                 Based on Pooled StDev
Level       N      Mean     StDev  ---+---------+---------+---------+---
Missing     6     11720        98   (---------------*----------------)
None       61     11778       143                     (-----*----)
Other       9     11742       163          (------------*-------------)
                                   ---+---------+---------+---------+---
Pooled StDev =      142            11620     11690     11760     11830
```

4.5.3.2 Conclusion

No statistically significant effect of pretreatment is observed.

4.5.4 SAMPLES G and J: Barley Mash

Table 4.47 Descriptive statistics: pMC by pretreatment

Pretreatment	N	Mean	Median	StDev
AAA	5	110.58	110.76	0.54
Missing	28	110.64	110.83	0.87
None	120	110.61	110.64	0.90
Other	18	110.52	110.50	1.24

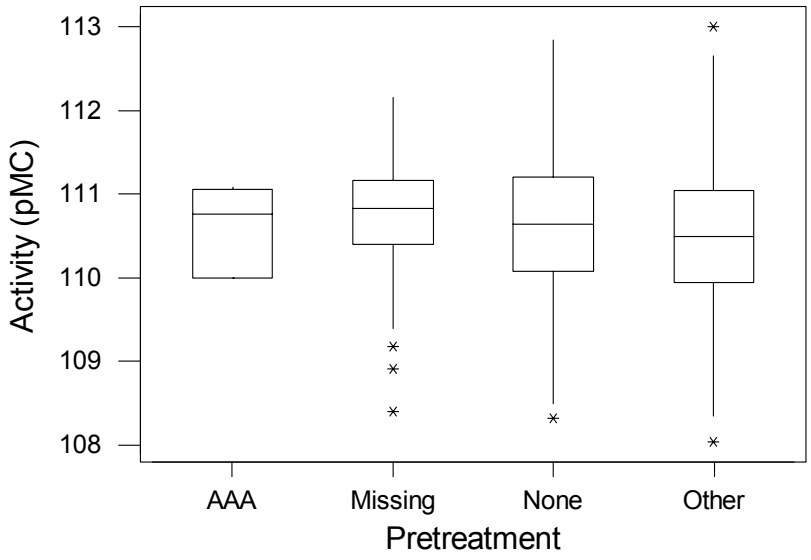

Figure 4.22 Distribution of activity by pretreatment method

4.5.4.1 Formal Analysis

Table 4.48 Analysis of variance of activity by pretreatment method

```
Analysis of Variance for pMC
Source      DF        SS        MS        F        P
Pretreat     3     0.159     0.053     0.06    0.980
Error      167   145.479     0.871
Total      170   145.638

                                  Individual 95% CIs For Mean
                                  Based on Pooled StDev
Level       N      Mean     StDev  -----+---------+---------+---------+-
AAA         5   110.576     0.540  (----------------*----------------)
Missing    28   110.638     0.873             (------*------)
None      120   110.612     0.905                (--*---)
Other      18   110.524     1.244         (-------*--------)
                                   -----+---------+---------+---------+-
Pooled StDev =    0.933             110.00    110.50    111.00    111.50
```

4.5.4.2 Conclusion

No statistically significant effect of pretreatment is observed.

4.5.5 SAMPLE I: Belfast Cellulose

Table 4.49 Descriptive statistics: age by pretreatment

Pretreatment	N	Mean	Median	StDev
Missing	13	4446.0	4452.0	54.6
None	63	4505.7	4490.0	91.8
Other	10	4463.5	4435.0	171.7

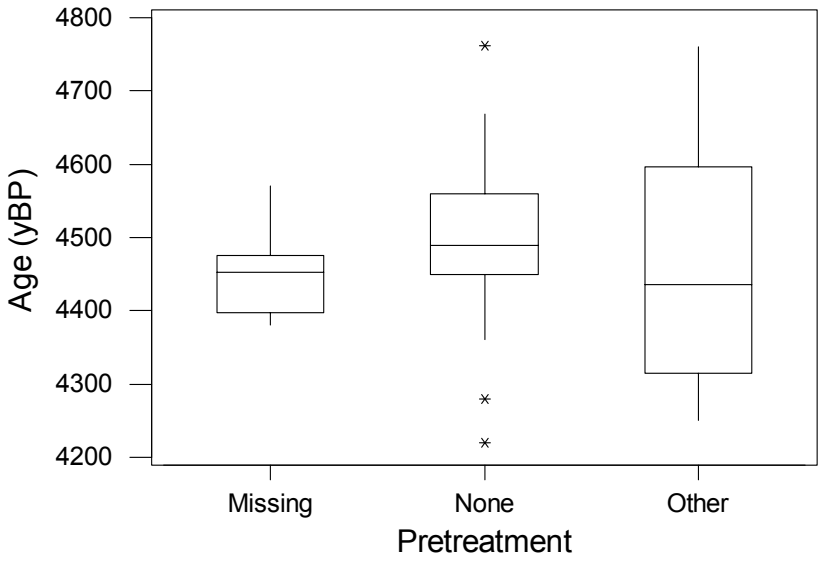

Figure 4.23 Distribution of age by pretreatment method

4.5.5.1 Formal Analysis

Table 4.50 Analysis of variance of age by pretreatment method

```
Analysis of Variance for Age
Source     DF        SS        MS        F        P
Pretreat    2     47398     23699     2.39    0.098
Error      83    823879      9926
Total      85    871278

                                  Individual 95% CIs For Mean
                                  Based on Pooled StDev
Level       N      Mean     StDev  ---+---------+---------+---------+---
Missing    13    4446.0      54.6   (-------------*------------)
None       63    4505.7      91.8                        (-----*------)
Other      10    4463.5     171.7     (---------------*---------------)
                                  ---+---------+---------+---------+---
Pooled StDev =     99.6            4400      4440      4480      4520
```

4.5.5.2 Conclusion

A statistically significant effect of pretreatment at 10% is observed, but given the insufficient information provided by the laboratories, no further conclusions can be drawn for this sample.

SECTION 5: MEASURES OF PRECISION AND REPRODUCIBILITY

5.1 DUPLICATES

The design of FIRI included 3 pairs of duplicate samples: A and B (Kauri wood) near background, D and F (Belfast wood) around 50 pMC, and G and J (barley mash) at 111 pMC. Why include duplicates? Duplicates by their nature allow us to explore the within-lab variability and to assess whether the quoted errors are representative. We can also explore the differences as a function of the sample activity. In this section, we explore the differences between the duplicates. We also consider some different graphical presentations. First, we summarize the differences, then graphically explore the boxplot (to consider the distribution of differences), then a scatterplot of the duplicate pair (to show correlation and reproducibility), and finally, a measure of agreement plot (Bland and Altman 1999). The horizontal axis in this final plot is the mean of the duplicate pair and the vertical axis is the difference in the duplicate pair. Agreement between the pairs would result in the points being randomly scattered around the horizontal zero line.

5.1.1 Summary Statistics for Duplicate Pairs

The summary statistics for the duplicates are shown below.

Table 5.1 Descriptive statistics: differences between duplicates (note: DF in yr BP)

Sample pair	N	Mean	Median	StDev	Min	Max
AB	54	0.0295	0.0000	0.2145	–0.66	0.531
GJ	71	–0.094	–0.080	1.085	–4.37	2.76
DF	79	17.4	17.0	97.3	–239	310

On average, the differences are close to zero, although it can be seen from the minimum and maximum that there is a wide scatter for sample pair GJ. For GJ, the largest difference between a pair of duplicates is just over 4 pMC, and for sample pair DF, the largest difference is 310 yr, both of which are small given the absolute activity/age of the sample. For Sample AB, the largest difference is 0.7 pMC, which is large given the near background activity for this sample. Each sample is now considered in more detail. The same pattern of analysis is repeated for the summaries by laboratory type (Tables 5.2–5.4). It is worth noting that 2 out of the 3 largest differences for the duplicates are reported by LSC laboratories.

Table 5.2 Descriptive statistics: AB differences by laboratory type

Lab type	N	Mean	Median	StDev	Min	Max
AMS	21	0.0436	0.0000	0.1234	–0.2	0.36
GPC	14	0.0662	0.0180	0.1621	–0.2	0.45
LSC	19	–0.0131	–0.0200	0.3105	–0.7	0.53

Table 5.3 Descriptive statistics: DF differences by laboratory type

Lab type	N	Mean	Median	StDev	Min	Max
AMS	25	8.7	17	68.9	–210	142
GPC	18	–2.7	5.0	96.4	–159	220
LSC	36	33.4	27.0	113.2	–239	310

Table 5.4 Descriptive statistics: GJ differences by laboratory type

Lab type	N	Mean	Median	StDev	Min	Max
AMS	25	–0.2354	–0.1000	0.47	–1.1	0.8
GPC	17	–0.104	–0.080	1.31	–4.4	1.85
LSC	29	0.034	0.110	1.32	–3.0	2.8

5.2 SAMPLES A AND B

Figure 5.1 shows that the duplicate pair differences are, on average, zero. The scatterplot and agreement plots (Figures 5.2 and 5.3) both show that the points are quite widely scattered about the line of equality and the zero line, respectively, and that the scatter of the points increases with an increasing average pMC.

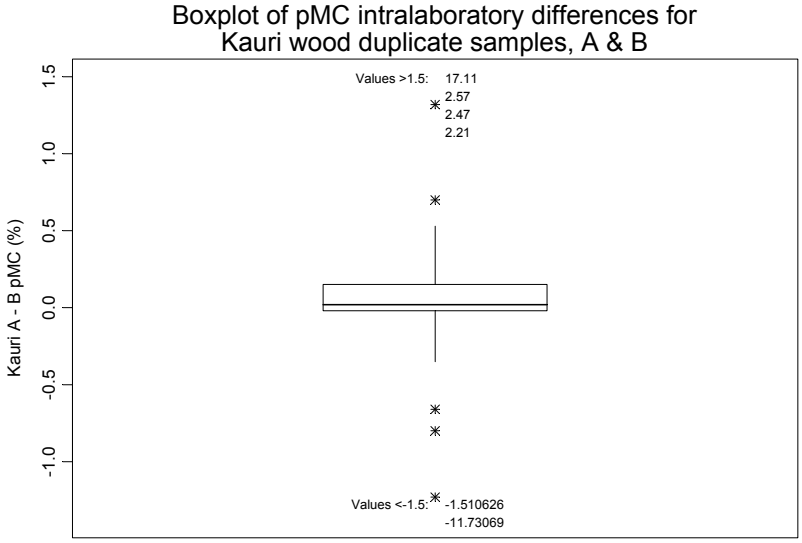

Figure 5.1 Distribution of differences (only differences <1.5 shown, uncensored results only)

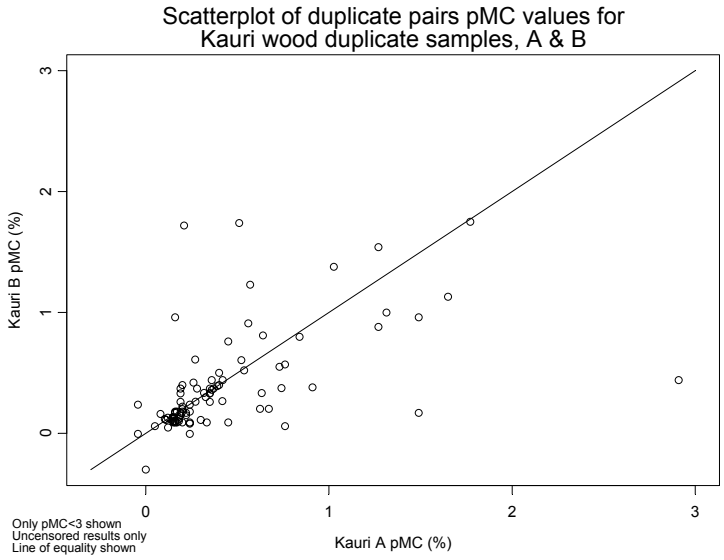

Figure 5.2 Scatterplot of duplicate pairs

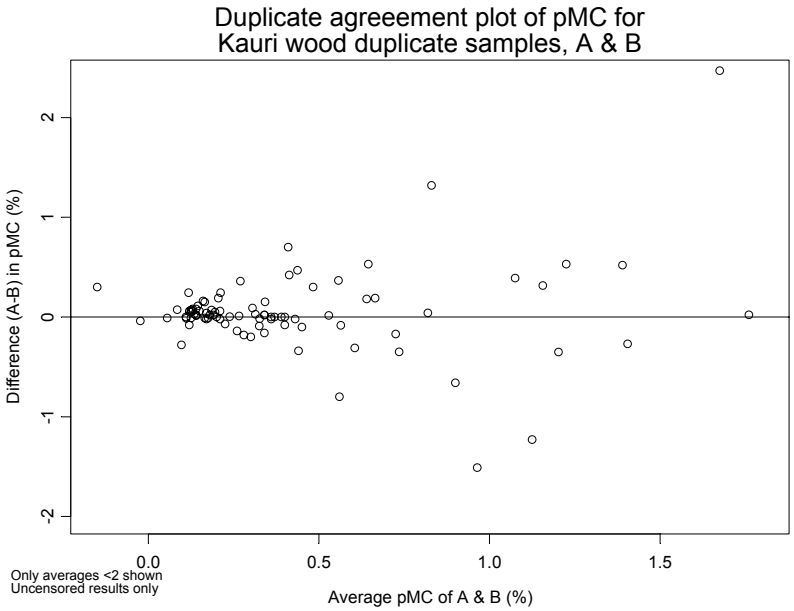

Figure 5.3 Agreement plot between duplicate pairs

5.3 SAMPLES D AND F

Figure 5.4 shows that the duplicate pair differences are, on average, zero. The scatterplot (Figure 5.5) shows that the pairs are quite widely scattered about the line of equality. Figure 5.6 shows a wide scatter around the zero line, suggesting that the difference is a function of the estimated age.

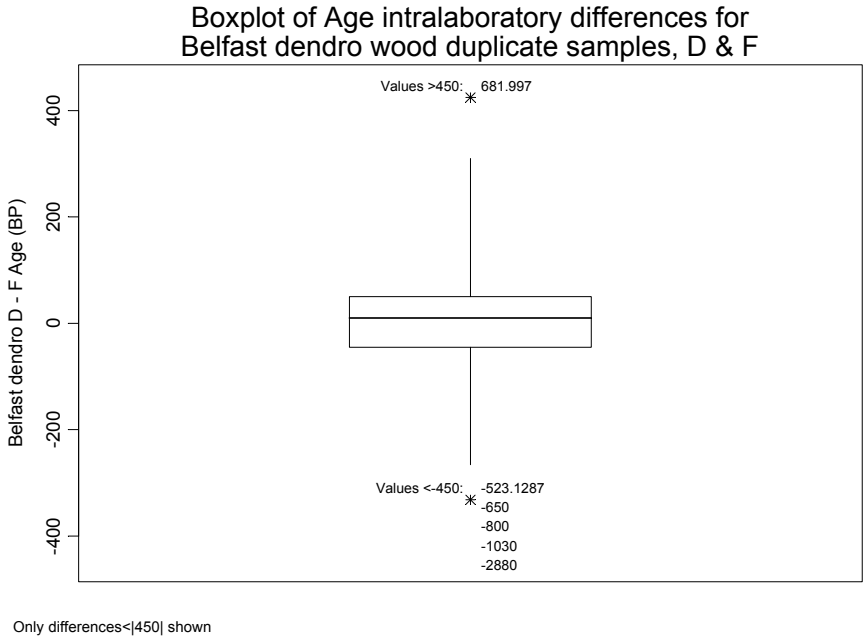

Figure 5.4 Distribution of differences

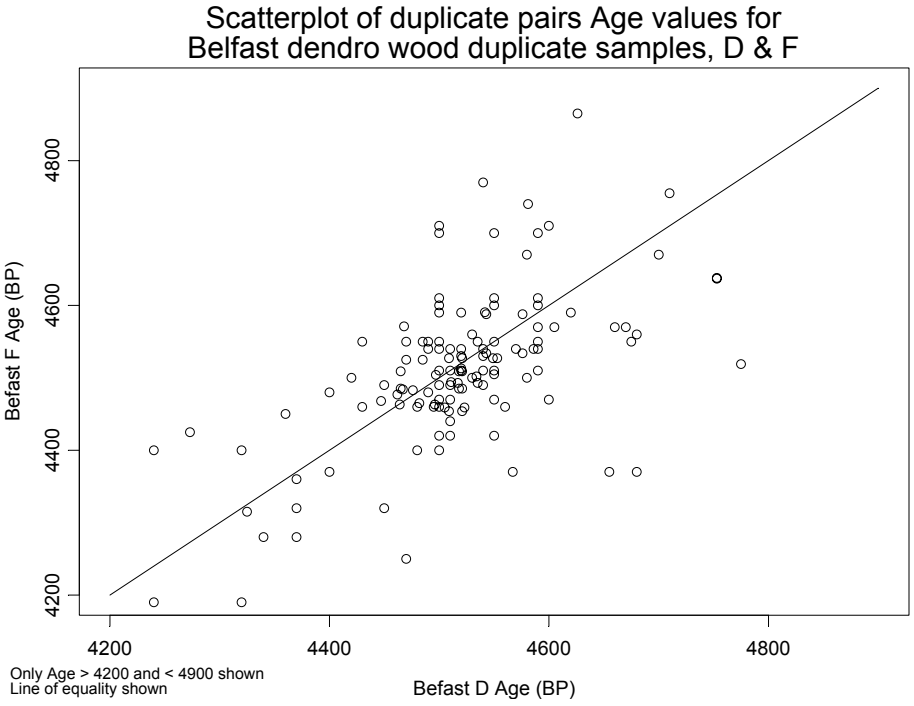

Figure 5.5 Scatterplot of duplicate pairs

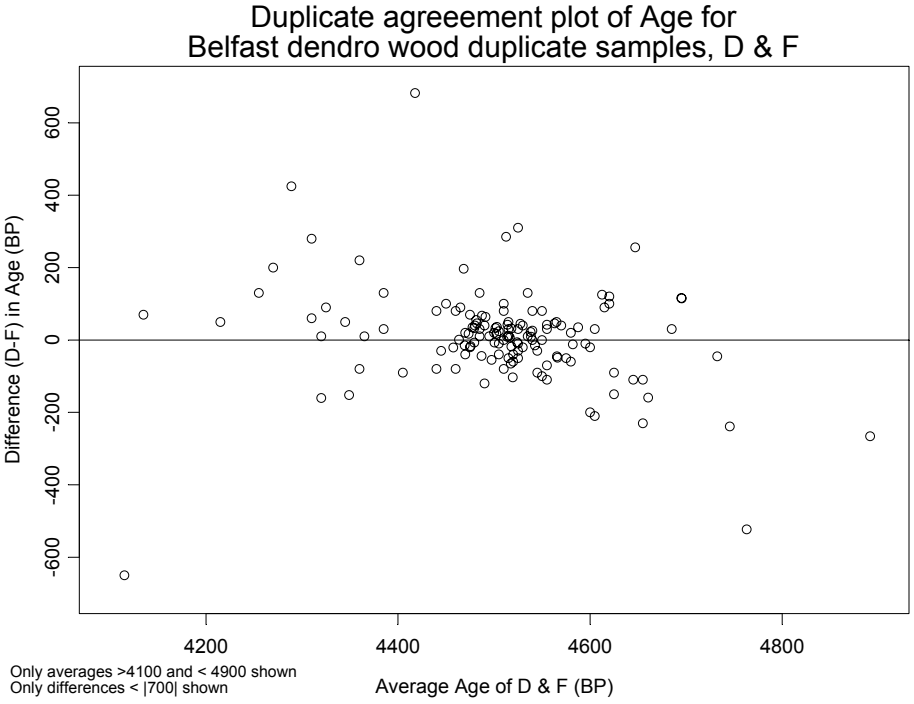

Figure 5.6 Agreement plot for duplicate pairs

5.4 SAMPLES G AND J

Figure 5.7 shows that the duplicate pair differences are on average zero. The scatterplot (Figure 5.8) shows that pairs are quite widely scattered about the line of equality. Figure 5.9 shows a wide scatter around the zero line, with a number of outliers.

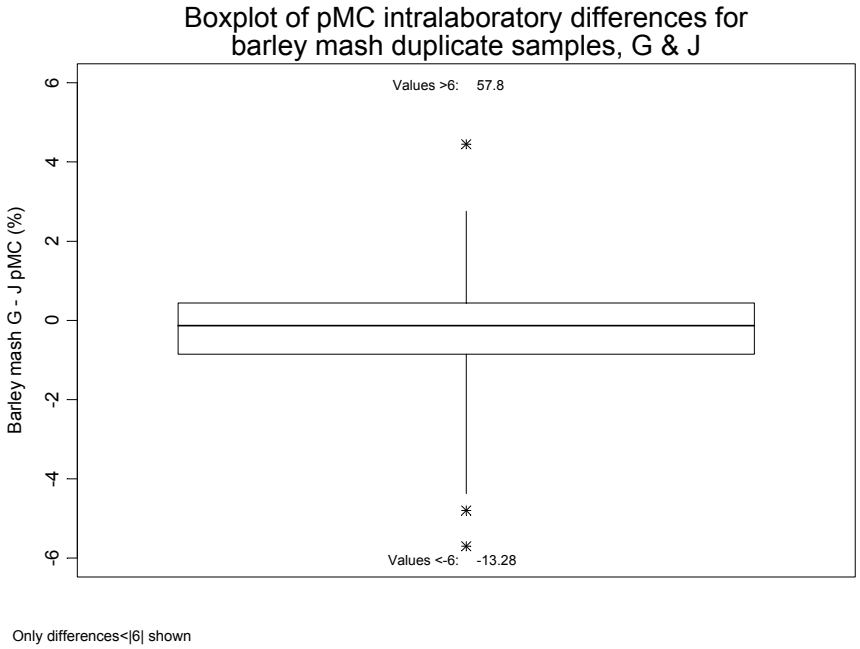

Figure 5.7 Distribution of differences

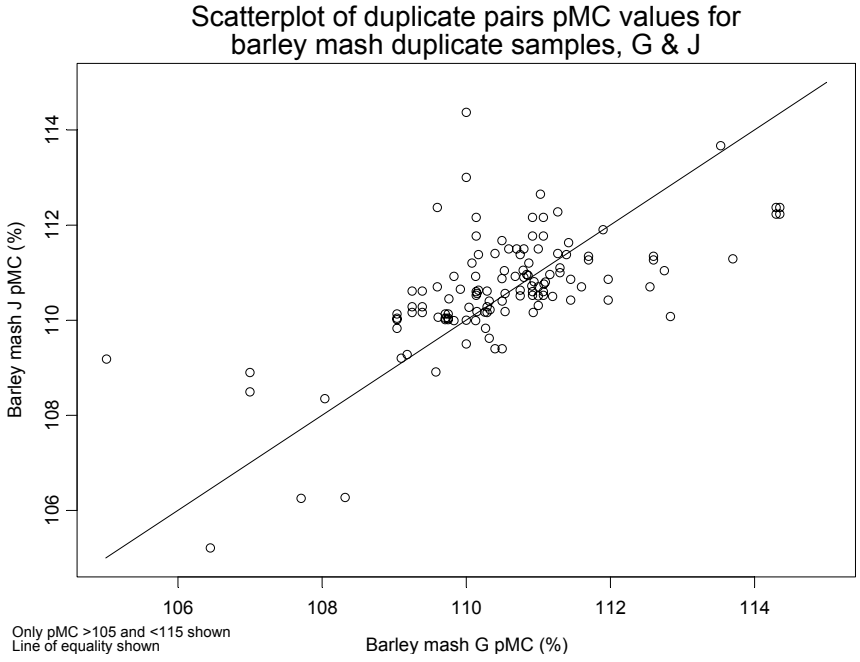

Figure 5.8 Scatterplot of duplicate pairs

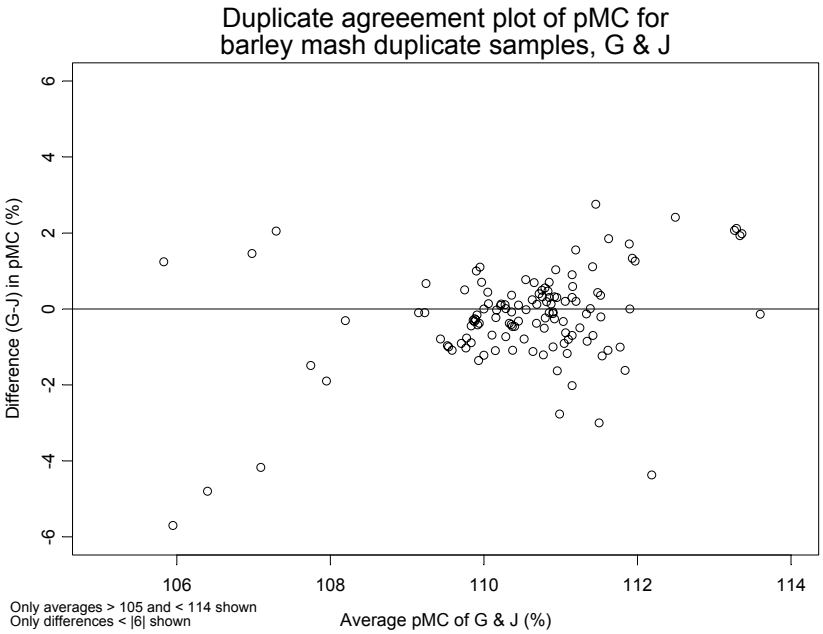

Figure 5.9 Agreement plot between duplicate pairs

5.5 QUOTED ERRORS

In addition, the duplicate results can also be used to assess the validity of the quoted errors. For each duplicate pair, the square of the difference, divided by the estimated standard deviation of the difference (deviance), should have a specific statistical distribution and name the Chi-squared distribution with 1 degree of freedom (or parameter) if the quoted errors adequately describe the uncertainty in measurement and, hence, the scatter in the differences. This theoretical distribution has a mean of 1 and a variance of 2 (standard deviation 1.4).

The tables below summarize the mean and standard deviation of the deviance each duplicate pair.

Table 5.5 Mean and standard deviation of the deviance for each duplicate pair

Sample pair	Mean	Standard deviation
AB	2.514	5.57
GJ	1.645	4.05
DF	2.220	4.76

Table 5.6a Mean and standard deviation of the deviance for duplicate pair AB by laboratory type

Sample pair AB	Mean	Median
AMS	3.36	1.19
GPC	1.54	0.185
LSC	2.220	0.27

Table 5.6b Mean and standard deviation of the deviance for duplicate pair GJ by laboratory type

Sample pair GJ	Mean	Median
AMS	0.85	0.19
GPC	2.28	0.45
LSC	2.05	0.41

Table 5.6c Mean and standard deviation of the deviance for duplicate pair DF by laboratory type

Sample pair DF	Mean	Median
AMS	2.86	0.36
GPC	1.76	0.81
LSC	2.00	0.65

5.5.1 Comments

In conclusion, these tables show clearly that the distribution of the differences between each of the duplicate pairs does not correspond to the claimed uncertainties in the measurements, since the means and standard deviations do not agree with the theoretical values. This would suggest, in general, that the differences between the duplicates are more varied than would be expected, given the quoted errors.

5.6 REPRODUCIBILITY RESULTS

5.6.1 Repeatability and Reproducibility

Analyses performed on presumed homogeneous material do not yield identical results due to unavoidable random factors inherent in every measurement method. The repeatability and reproducibility of a standard measurement method are sufficient to describe the variability in a measurement method and can be estimated from an interlaboratory test. *Precision* is considered to be the closeness of agreement between independent measurements. *Repeatability* (r) refers to measurements made under identical conditions in one laboratory, while *reproducibility* (R) refers to measurements made in different laboratories, under different conditions. Reproducibility is the closeness of agreement between test results under conditions where the same method is used in different laboratories. The reproducibility quantifies the maximum variability in results. The samples used for such experiments should thus be sub-samples taken from 1 bulk sample, as is the case with the FIRI samples. In this section, we consider the following cases: a) the method is ^{14}C dating regardless of technique, and b) where we consider LSC, GPC, and AMS as 3 different methods.

We evaluate the repeatability and reproducibility values for a) the 3 pairs of duplicates (A, B; G, J; and D, F) and b) for all samples, but in this latter case, we need to modify the calculation method since we do not have replicate results, thus, we use the quoted errors.

The *reproducibility* value (R) is the value below which the absolute difference between 2 single results obtained under reproducibility conditions may be expected to lie with a probability of 0.95. A difference larger than *R* cannot be ascribed to random fluctuations and would warrant investigation of possible sources of systematic differences.

The method used is based on BS 5497 (1), however, outliers were defined by the 1.5 IQR method and removed before the BS 5497 (1) analysis was carried out. All results were converted to pMC to unify the interpretation.

5.6.2 Statistical Models

The basic model and estimating equations for *r* and *R* are given below:

$$\text{Model: } Y = m + B + e$$

where *Y* is the ^{14}C measurement, *m* is the general average for the particular material, *B* is the between-laboratory variation, and *e* is the random error.

- *B* is assumed random in a reproducibility test and var(B) = σ^2_L
- *e* is also assumed random and within a single laboratory var(e) = σ^2_W
- We assume that σ^2_W is constant for all laboratories, with the average value σ^2_r
- The repeatability value *r* is 2.8 σ_r
- The reproducibility value *R* is 2.8 σ_R, where $\sigma_{R = \sqrt{}} (\sigma^2_L + \sigma^2_W)$

Estimation of *r* and *R* can be achieved from an intercomparison such as FIRI, where each sample can be considered as having one of *q* different levels of ^{14}C activity. The samples were sent to *p* different laboratories, which performed *n* analyses on each sample. In the case of FIRI for most samples, *n* is taken to be 1.

In the analysis for each sample separately, estimates of σ_r, σ^2_L and σ^2_R were calculated before evaluating *r* and *R*.

5.6.2 Analysis of the Duplicate Samples

The overall mean activity (m), the reproducibility measure (R), and repeatability measure (r) are shown for each material in Table 5.7.

Table 5.7 Repeatability and reproducibility

	AB	DF	GJ
m	0.348	56.991	110.603
R	0.749	1.551	2.613
r	0.451	1.047	1.728

The plots (Figure 5.10) below show the mean activity and standard deviation for the 3 pairs of duplicate samples. They show no obvious pattern between the mean and the standard deviation, but some extreme values are apparent (although they are not identified as outliers).

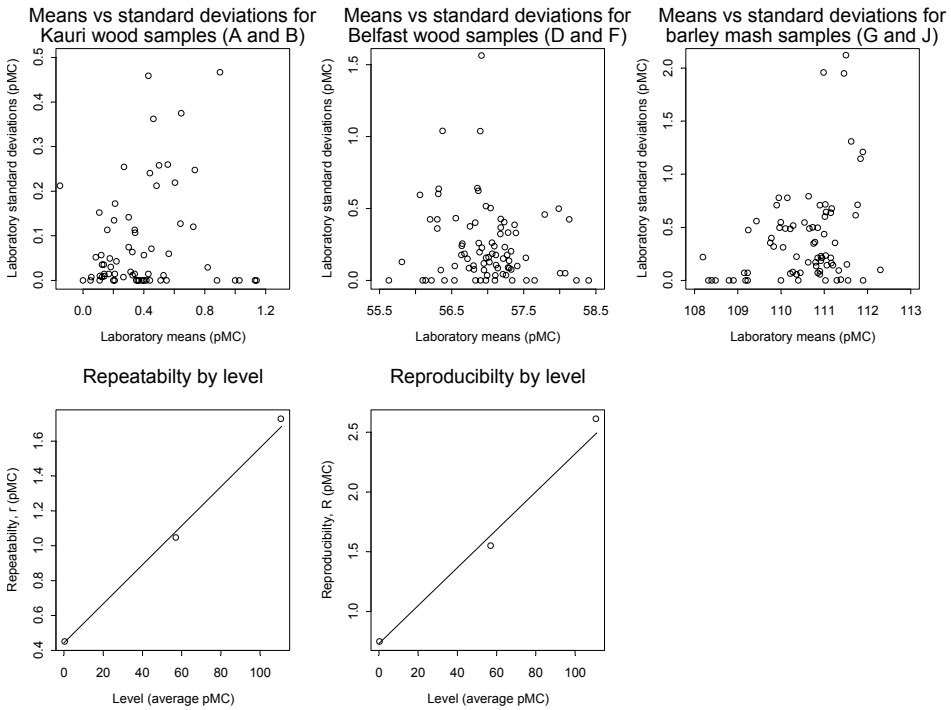

Figure 5.10 Scatterplots for duplicate samples

The last 2 plots show the strong linear relationship between *r* and *R* and the activity level.

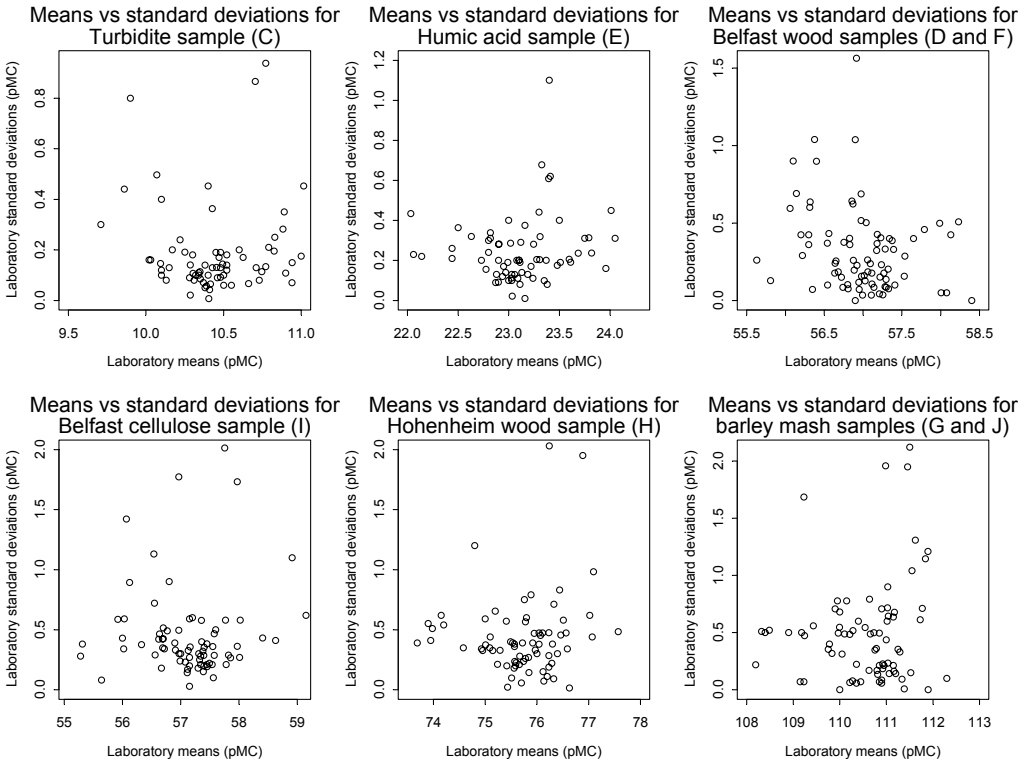

Figure 5.11 Scatterplot of means and standard deviations for all samples

The *R* values can be interpreted as the expectation that for any 2 randomly chosen measurements (i.e., laboratories), the absolute difference in their results should be less than 1.55 pMC (for a sample with an activity of 57 pMC), increasing to 2.6 for a sample with an activity of 110 pMC.

A similar analysis can be performed using all the samples (not simply the duplicate samples), however, here we need to modify the procedure such that the standard deviation previously calculated now must be estimated using the laboratory's quoted error for that sample.

5.6.3 C–J Results with Quoted Errors Used When No Replication Done

The quoted error is used as a substitute for the estimated standard deviation since we have no replicates.

Overall means (m), reproducibility measures (R), and repeatability measures (r) are given in Table 5.8.

Table 5.8 Reproducibility and repeatability for all samples

	C	E	DF	I	H	GJ
m	10.44	23.11	56.99	57.17	75.76	110.61
R	0.79	1.15	1.60	2.05	2.18	2.64
r	0.73	0.84	1.17	1.64	1.54	1.86

We can see quite clearly the dependence of *R* on the sample activity.

5.6.4 Reproducibility for the Different Techniques

In this section, a similar analysis was performed, but for the laboratory types separately. Outliers, as defined by the 1.5 IQR method, are removed and all units are pMC.

5.6.4.1 Duplicate Results

Overall means (m), reproducibility measures (R), and repeatability measures (r) for the 3 measurements techniques are given in Table 5.9.

Table 5.9a AMS repeatability and reproducibility

	AB	DF	GJ
m	0.23	56.88	110.46
R	0.41	1.14	1.84
r	0.37	0.86	1.34

Table 5.9b GPC repeatability and reproducibility

	AB	DF	GJ
m	0.28	57.09	110.78
R	0.74	1.45	2.68
r	0.47	0.88	1.48

Table 5.9c LSC repeatability and reproducibility

	AB	DF	GJ
m	0.50	57.06	110.66
R	0.83	1.91	3.22
r	0.55	1.35	2.30

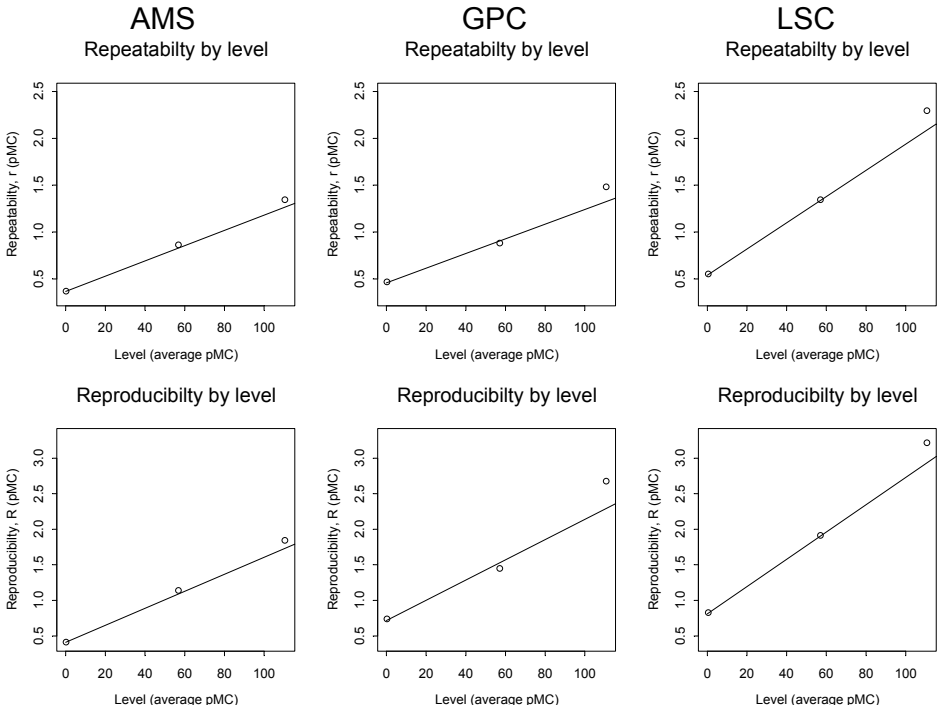

Figure 5.12 Repeatability and reproducibility for laboratory types

5.6.4.2 Comments

Large differences between techniques are observed, with AMS laboratories having lower reproducibility values compared to radiometric methods. LSC laboratories have higher repeatability values than the other techniques. Thus, for LSC, bigger differences in the results can be expected and we can expect more variation in the LSC results compared to AMS or GPC results.

5.6.5 C–J Results with Quoted Errors Used When No Replication for the Different Laboratory Types

Table 5.10a AMS repeatability and reproducibility

	C	E	DF	I	H	GJ
m	10.41	22.98	56.88	57.12	75.77	110.46
R	0.48	0.50	1.17	1.44	1.33	1.76
r	0.32	0.57	0.92	0.85	1.01	1.16

Table 5.10b GPC repeatability and reproducibility

	C	E	DF	I	H	GJ
m	10.40	23.24	57.09	57.53	75.82	110.78
R	0.90	1.28	1.47	1.28	2.64	2.68
r	0.54	1.04	0.94	1.05	1.15	1.48

Table 5.10c LSC repeatability and reproducibility

	C	E	DF	I	H	GJ
m	10.49	23.16	57.06	57.05	75.71	110.68
R	0.90	1.42	1.95	2.56	2.57	3.20
r	0.97	0.90	1.40	2.21	2.01	2.39

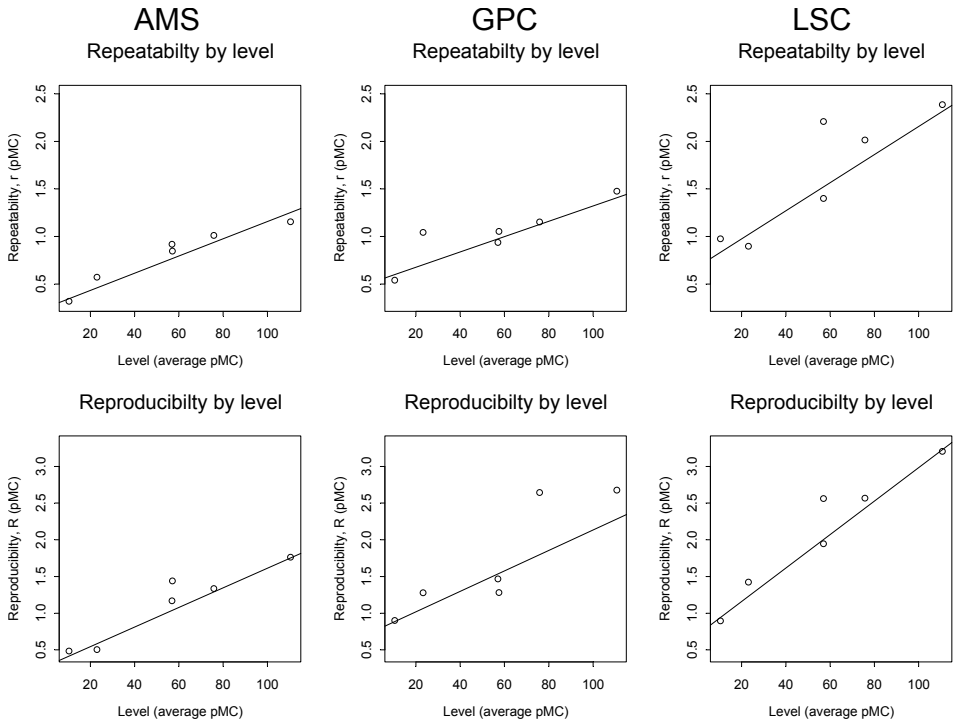

Figure 5.13 Repeatability and reproducibility for laboratory types

5.6.5.2 Comments

Differences between the measurement techniques are observed. The AMS technique has lower reproducibility values compared to radiometric methods. LSC has higher repeatability values than the other techniques. Again, based on all the materials, the LSC results would be expected to be more varied than those from AMS or GPC laboratories.

5.6.6 How Can the Reliability Figures Be Used for Each Laboratory?

In essence, each laboratory may use its reliability figure to "test" whether it is sufficiently close to the consensus value for a reference material or standard.

Comparison with a reference value for a single laboratory makes use of R. If a single determination is performed by one laboratory under repeatability conditions and yields a value y^*, which is to be compared to the reference value m_0, then the critical difference (95%) between y^* and m_0 is given by:

$$CR = R / \sqrt{2}$$

If the absolute difference exceeds this critical difference, then the determination should be considered suspect and there may be an assignable cause that should be investigated. Assuming the reproducibility values given in Table 5.8, then for each of the samples, we can calculate the critical difference (CR) for a number (n) of independent determinations.

Table 5.11 Critical differences for each sample

Number of determinations	C	E	DF	I	H	GJ
1	0.56	0.81	1.13	1.45	1.54	1.87
2	0.43	0.70	0.97	1.19	1.33	1.62
3	0.37	0.65	0.91	1.09	1.26	1.53
4	0.34	0.63	0.88	1.04	1.22	1.48
5	0.32	0.62	0.86	1.01	1.19	1.45
6	0.31	0.61	0.85	0.99	1.18	1.43
7	0.30	0.60	0.84	0.97	1.16	1.42
8	0.29	0.60	0.83	0.96	1.15	1.40

Similar calculations can also be performed for AMS, GPC, and LSC techniques separately.

Table 5.12a Critical differences for each sample for AMS laboratories

Number of determinations	C	E	DF	I	H	GJ
1	0.34	0.36	0.83	1.02	0.94	1.25
2	0.30	0.21	0.69	0.92	0.80	1.10
3	0.29	0.13	0.63	0.89	0.74	1.05
4	0.28	0.06	0.61	0.88	0.71	1.03

Table 5.12b Critical differences for each sample for GPC laboratories

Number of determinations	C	E	DF	I	H	GJ
1	0.64	0.90	1.04	0.90	1.87	1.89
2	0.58	0.74	0.92	0.74	1.78	1.74
3	0.55	0.67	0.88	0.67	1.75	1.69
4	0.54	0.64	0.86	0.63	1.73	1.66

Table 5.12c Critical differences for each sample for LSC laboratories

Number of determinations	C	E	DF	I	H	GJ
1	0.63	1.01	1.38	1.81	1.82	2.27
2	0.40	0.90	1.18	1.43	1.51	1.93
3	0.29	0.86	1.11	1.29	1.39	1.80
4	0.21	0.84	1.08	1.20	1.33	1.73

5.6.6.1 Comments and Conclusions

The critical differences decrease as the number of determinations increases; thus, the overall precision of the measurement increases as would be expected. The critical differences are a function of the material activity (an almost linear relation). We can also observe differences among the 3 measurement techniques, with AMS being more precise (given the realistic possibility of multiple determinations) than either GPC or LSC.

5.7 CONCLUSIONS

This section has mainly focused on the duplicate samples and their relationship to precision (taking account of the laboratory quoted error). On average, the difference in duplicate samples is zero, but there is some suggestion that the variation in the differences is greater than would be expected given the laboratory quoted errors. There is also a strong indication that the duplicate variation is considerably greater than would be expected in the near background Samples A and B.

Estimation of reproducibility and repeatability coefficients for firstly, the duplicate samples, and then for all materials, shows that the repeatability (measurements made under identical conditions in one laboratory) is a function of the sample activity and that the repeatability is better for the AMS technique than for the radiometric techniques. Reproducibility shows a similar pattern. Calculation of critical differences indicate that for a single determination, a relative difference from the consensus for Sample C greater than 0.05 pMC; for Sample E of 0.033 pMC; D, F, and I of 0.02 pMC; H of 0.02 pMC; and 0.017 pMC for GJ, would indicate that the measurement is aberrant.

This analysis does, however, make the assumption that the “average” quoted error is the same for all laboratories, which is clearly not the case.

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SECTION 6: KAURI WOOD, SAMPLES A AND B

6.1 INTRODUCTION

Kauri wood, a sub-fossil wood from New Zealand (which had previously been used in an IAEA exercise, IAEA-C4, in 1990), was considered to be an important sample to include in FIRI because it provided a link to previous exercises, was available in sufficient quantity, and was a "close to background" organic sample. IAEA-C4 had previously been criticized since it was believed that in its milling, some contamination had been introduced, so that a replacement sample would prove useful. The Kauri wood has a very low ^{14}C activity and, as such, is very sensitive to even small amounts of contaminant carbon. Such low-activity samples give a true test for the laboratory procedures since pretreatment and laboratory background definition become critical.

In 1994, a further Kauri wood sample was used in a small intercomparison (Hogg et al. 1995) as a potential replacement for C4. This new Kauri sample was tested in 6 laboratories and a preliminary range was quoted by the authors.

6.1.1 Preliminary Testing Results

From the earlier work on this sample (Hogg et al. 1995) involving 6 laboratories, the authors concluded:

- It was not possible to assign a definitive pMC value to the sample and the authors suggested a range of 0.12–0.21 pMC.
- The results showed some evidence of in-homogeneity (probable causes being incorrect background assessment or inadequate sample pretreatment).

Nonetheless, it was decided that this new Kauri sample should be included in FIRI, and that it should be provided in duplicate, without pretreatment.

6.2 PRELIMINARY RESULTS

A total of 83 laboratories returned results within the deadline. Due to some laboratories using more than one preparation or measurement system, this gave a set of over 90 results. It is worth noting the following:

- The basic results for the Kauri samples (A and B) were often given in 2 forms, age and pMC;
- The errors (particularly for age) were asymmetrical;
- There was a substantial number of censored observations (observations reported as "greater than");
- Some results were simply given as "background."

Thus, it is apparent that there is an important variation in how the results are reported.

6.2.1 Preliminary Analysis

The preliminary analysis focused on the distribution of results, the identification of any gross outliers (using simple graphical means such as boxplots), and the calculation of preliminary consensus values based on robust statistics (medians and interquartile ranges).

In this section, the 2 independently measured duplicate samples are reported separately and then the combined results are analyzed. Finally, a comparison of the results for AMS, GPC, and LSC laboratories is reported.

6.2.2 Sample A

Ninety-eight age results were quoted, 64 of which were finite, while 5 were simply quoted as background. For pMC, 67 finite results were quoted (not all laboratories quoted both age and pMC, and for the preliminary analysis, no conversion calculations were performed, although this was done later), and 2 laboratories simply quoted the result as background. The results came from 32 AMS, 20 GPC, and 44 LSC systems. Summary information on the results reported is shown in Table 6.1 below.

Table 6.1 Summary of the reporting format for Kauri A

a) Age				b) pMC			
Reporting format	AMS	GPC	LSC	Reporting format	AMS	GPC	LSC
>	9	7	9	<	3	2	1
Background	0	1	4	Background	0	1	1
Finite	23	12	29	Finite	27	14	26

6.2.2.1 Distribution of Results

Figure 6.1 shows a boxplot with the censored (>) observations distinguished from the finite (uncensored) results.

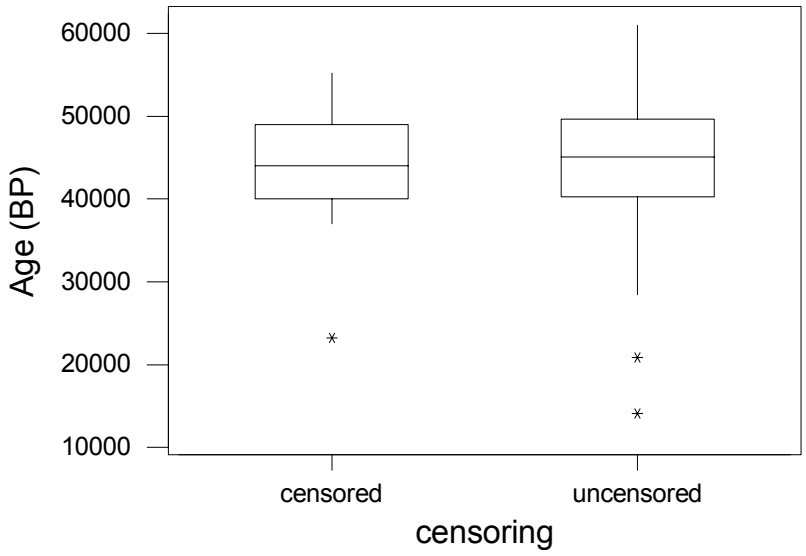

Figure 6.1 Age distribution for Kauri A

There appears to be little difference in location (activity distribution) for the censored and uncensored results. The boxplots also permit a preliminary identification of gross outliers, represented by asterisks in the figure above. Three obvious outliers with ages less than 22,000 BP are apparent.

In pMC, 3 outliers were immediately apparent with values of 7.43, 10.62, and 17.31. Figure 6.2 shows the boxplot of pMC after their removal.

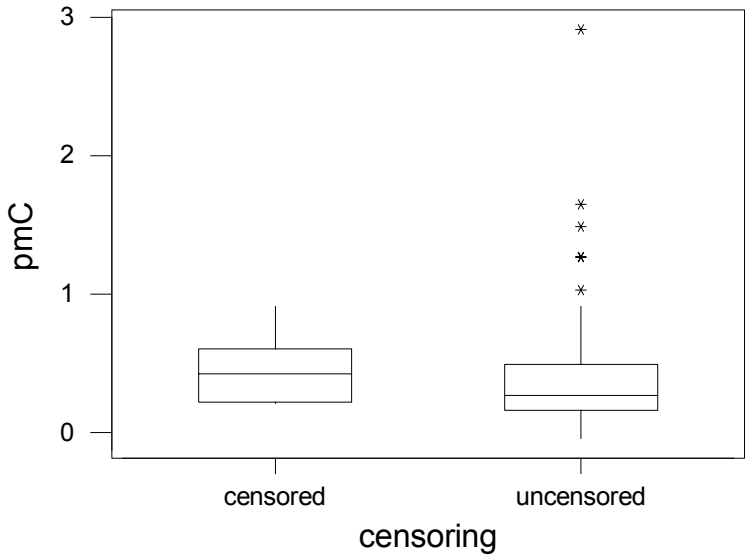

Figure 6.2 pMC distribution for Kauri A (outliers 7.43, 10.62, and 17.31 removed)

6.2.2.2 Summary Statistics (Omitting Outliers)

For finite ages, the overall mean is 44,482 yr, the median is 45,200 yr, and standard error of the mean (Semean) is 885 yr. The results for the 3 laboratory types are shown in Table 6.2 below.

Table 6.2 Summary ages for Kauri A by laboratory type

Laboratory type	Mean	Median	Semean
AMS	48,180	49,200	897
GPC	46,534	46,468	2196
LSC	40,565	41,140	1270

There are statistically significant differences in the means between LSC and both AMS and GPC laboratories.

For pMC, the overall mean is 0.4181, the median is 0.2705, and standard error of the mean is 0.0582.

Table 6.3 Summary results for pMC for Kauri A by laboratory type

Laboratory type	Mean	Median	Semean
AMS	0.2741	0.2	0.0504
GPC	0.3094	0.25	0.0636
LSC	0.653	0.45	0.135

There is a statistically significant difference in the average pMC between LSC and AMS laboratories.

6.2.3 Duplicate B

Ninety-nine age results were returned, 57 of which were finite and 7 simply quoted as background. For pMC, 64 results were finite and 2 were quoted as background. Results were received from 33 AMS, 21 GPC, and 45 LSC systems.

Table 6.4 Summary of reporting format for Kauri B

a) Age				b) pMC			
Reporting format	AMS	GPC	LSC	Reporting format	AMS	GPC	LSC
>	11	10	10	<	5	2	2
Background	0	2	5	Background	0	1	1
Finite	22	9	26	Finite	26	15	23

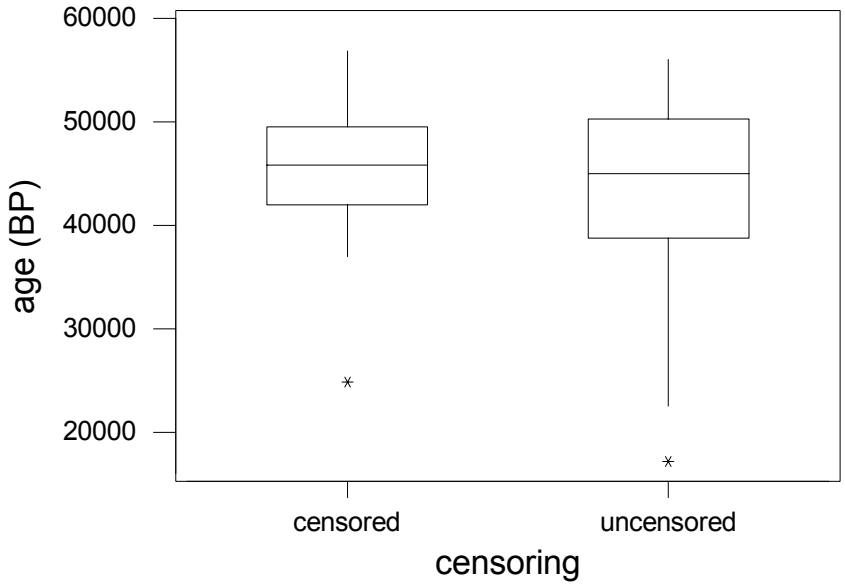

Figure 6.3 Age distribution for Kauri B

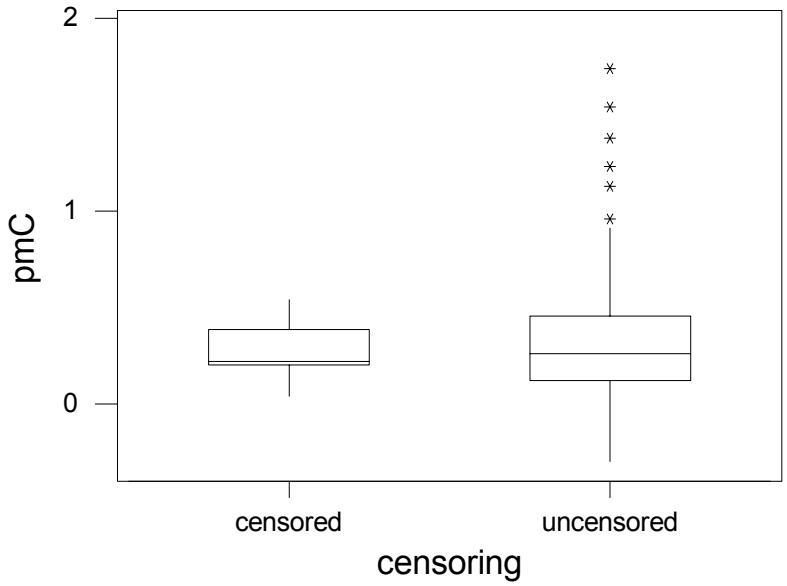

Figure 6.4 pMC distribution for Kauri B (outliers 4.86 and 8.41 removed)

6.2.3.1 Distribution of Results

Figures 6.3 and 6.4 show the boxplots with the censored and uncensored observations for age and pMC. A preliminary identification of gross outliers indicates 2 outliers with ages less than 22,000 BP and in pMC, 2 outliers were identified with values of 4.86 and 8.41.

For finite ages, the overall mean is 43,699 yr, the median is 45,000 yr, and the standard error of the mean is 1086 yr.

Table 6.5 Summary ages for Kauri B (with outliers removed)

	Mean	Median	Semean
AMS	48,942	49,350	1034
GPC	40,832	42,231	3681
LSC	40,254	41,007	1419

Statistically significant differences in age were observed between AMS and both LSC and GPC laboratories.

For pMC, the overall mean is 0.38, with a median of 0.26, and a standard error of the mean of 0.05.

Table 6.6 Summary pMC for Kauri B (with outliers removed)

	Mean	Median	Semean
AMS	0.2373	0.1750	0.037
GPC	0.348	0.237	0.122
LSC	0.5888	0.44	0.096

A statistically significant difference between LSC and AMS results was observed. Again, it is clear that the median tends to be older than the mean. Other extreme observations are also highlighted.

6.2.4 Combined Results

Since the samples were duplicates (each being split from a single block of 100 g), the results can be combined.

For age, 197 results in total were returned, 120 of which were finite and 12 simply quoted as background. For pMC, there were 125 finite results and 4 quoted as background. Overall, there were 65 AMS, 39 GPC, and 93 LSC measurements.

Table 6.7 Summary of reporting format for Kauri A and B

a) Age				b) pMC			
Reporting format	AMS	GPC	LSC	Reporting format	AMS	GPC	LSC
>	21	17	19	<	9	4	16
Background	0	3	9	Background	0	2	4
Finite	44	19	57	Finite	52	29	44

6.2.4.1 Distribution of Results

Figures 6.5 and 6.6 show the boxplots with the censored and uncensored observations marked separately for age and pMC. A preliminary identification shows 2 clear age outliers with ages of 14,090 and 17,180 BP.

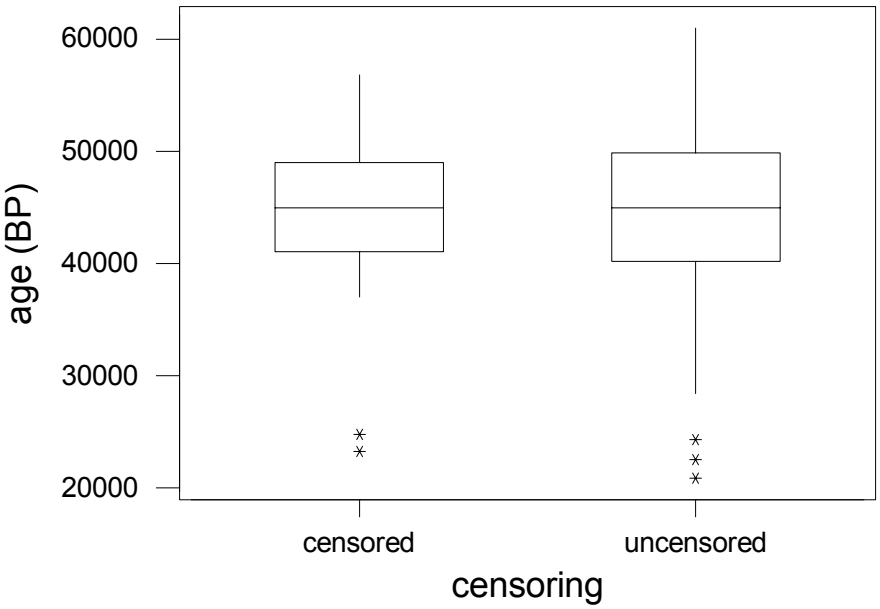

Figure 6.5 Age distribution for Kauri A and B (outliers 14,090 and 17,180 removed)

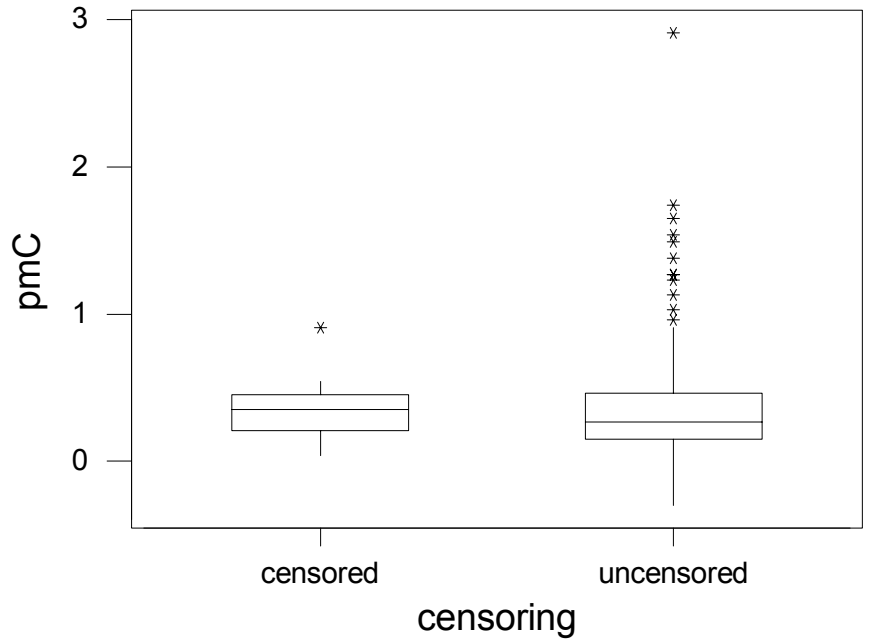

Figure 6.6 pMC distribution for Kauri A and B (outliers 4.86, 8.41, 7.43, 10.62, and 17.31 removed)

For pMC, 5 clear outliers were identified with values of 4.86, 8.41, 7.43, 10.62, and 17.31.

For finite ages, the overall mean is 44,336 BP, with a median of 45,000 BP, and a standard error of the mean of 660 yr.

Table 6.8a Summary results for Kauri A and B age (yr BP) by laboratory type

	Mean	Median	Semean
AMS	48,552	49,200	677
GPC	44,617	44,043	1495
LSC	40,789	41,013	996

A statistically significant difference between LSC and AMS results was observed.

Table 6.8b Summary results for Kauri A and B (pMC) by laboratory type

	Mean	Median	Semean
AMS	0.2561	0.19	0.0313
GPC	0.3292	0.24	0.0691
LSC	0.6225	0.445	0.0831

The overall mean pMC is 0.40, with a median of 0.26, and a standard error of the mean of 0.038.

A statistically significant difference between mean pMC for LSC and AMS systems and LSC and GPC systems was observed.

6.3 CONCLUSIONS BASED ON THE PRELIMINARY ANALYSIS

For all Kauri sample results, a preliminary analysis gives a median pMC value of 0.24 and interquartile range (IQR) of 0.15–0.44. The mean is noticeably higher (0.38) since it is non-robust and affected by extreme values. The results are also higher than those quoted by Hogg et al. (1995), but are based on a much wider group of laboratories. This analysis has only excluded the most extreme outliers. However, there is clearly some considerable variation in the results, which may be a function of laboratory background (estimation and material used) and the limits of detection. Interestingly, the analysis also appears to indicate some differences in the distribution of results between laboratory types, with AMS laboratories quoting older ages in general.

6.4 ANALYSIS OF ACTIVITY, A AND B SEPARATELY

Analysis for Kauri A and B should, in principle, follow a similar approach to that for Samples C–J, but this must be modified when considering the age of the sample and the issue of finite age reporting. By this we mean that for age, many results were simply quoted as "greater than" or indeed as "background" (described as "censored"). However, the analysis of the pMC results (since the majority of results are given in a finite form) will follow a similar pattern to the analysis for the other samples. Following the exploratory analysis, outliers have been omitted.

First, we investigate the association, if any, between whether a measurement is censored and other laboratory factors.

6.4.1 Association Between Censoring and Laboratory Factors

6.4.1.1 Kauri A

Table 6.9a Reporting status by laboratory type

	AMS	GPC	LSC	**All**
Censored	3	3	3	**9**
Uncensored	28	14	28	**70**
All	31	17	31	**79**

No evidence of an association is found; thus, one laboratory type is no more likely to report censored results than any other.

Table 6.9b Reporting status by modern standard used

	ASUC	Benz	NBS1	NBS12	NBS2	Other	**All**
Censored	1	0	2	1	3	2	**9**
Uncensored	5	4	21	5	25	6	**66**
All	6	4	23	6	28	8	**75**

Although not able to complete a formal statistical test due to the small numbers in some cells, there is no strong evidence of a statistically significant association between the reporting status and the modern standard.

Table 6.9c Reporting status by background material used

	Anth	Benz	Calc	Charc	Coal	Graph	Marble	Other
Censored	0	2	0	1	0	0	2	3
Uncensored	16	13	1	1	8	8	5	11
All	16	15	1	2	8	8	7	14

Although not able to complete a formal test, there is no strong evidence of a statistically significant association between the reporting status and the background material.

6.4.1.2 Kauri B

Table 6.10a Reporting status by laboratory type

	AMS	GPC	LSC	**All**
Censored	5	3	4	**12**
Uncensored	27	14	22	**63**
All	32	17	26	**75**

There is no statistically significant association between the laboratory type and the censoring mechanism.

Table 6.10b Reporting status by modern standard

	ASUC	Benz	NBS1	NBS12	NBS2	Other	**All**
Censored	1	0	4	1	4	2	**12**
Uncensored	5	2	18	6	23	4	**58**
All	6	2	22	7	27	6	**70**

There is no statistically significant association between the modern standard and the censoring mechanism.

Table 6.10c Reporting status by background

	Anth	Benz	Calc	Charc	Coal	Graph	Marble	Other
Censored	0	2	0	1	1	0	2	5
Uncensored	15	8	1	1	8	8	4	10
All	15	10	1	2	9	8	6	15

6.4.1.3 Conclusions

In all cases, no statistically significant association was found; thus, there is no evidence that one type of laboratory, modern standard material, or background material, is linked to whether the result is censored.

6.5 ANALYSIS OF ACTIVITY: SOURCES OF VARIATION

In this section, we now consider the effect of laboratory type, modern standard, and background material on pMC (for the purposes of this analysis, we ignore the 6 censored values and treat them as uncensored). Figure 6.7 shows the distribution of results by the 3 factors. Some variation in the results is apparent.

6.5.1 Kauri A

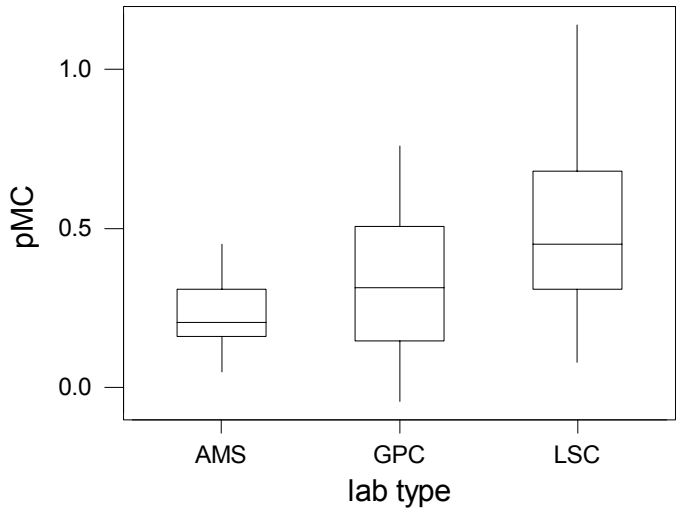

Figure 6.7a Distribution by laboratory type

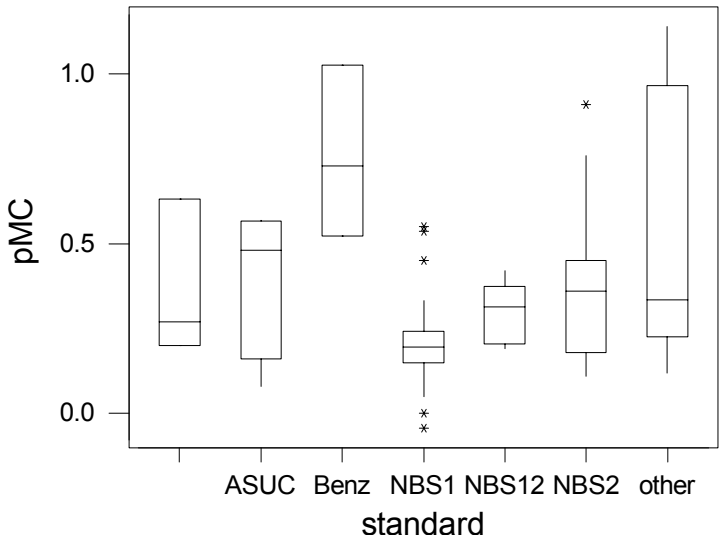

Figure 6.7b Distribution by modern standard

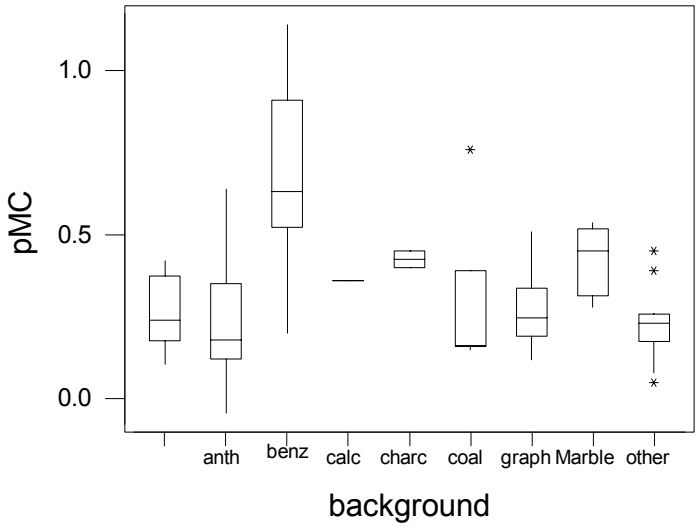

Figure 6.7c Distribution by background material

6.5.1.2 Formal Analysis

The formal analysis for each factor takes into account the hypothesis that there is no difference in the mean pMC due to the different levels of the laboratory factors. The results are shown in Table 6.11.

Table 6.11a Effect of laboratory type

Source	DF	SS	MS	F	P
type	2	0.9316	0.4658	10.52	0.000
Error	64	2.8328	0.0443		
Total	66	3.7645			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
AMS	30	0.2358	0.0999	(------*-----)
GPC	16	0.3301	0.2287	(--------*-------)
LSC	21	0.5098	0.2966	(------*-------)
Pooled StDev =		0.2104		0.24 0.36 0.48

Table 6.11b Effect of modern standard

Source	DF	SS	MS	F	P
standard	5	1.0910	0.2182	4.94	0.001
Error	58	2.5637	0.0442		
Total	63	3.6547			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
ASUC	4	0.4025	0.2287	(-------*--------)
Benz	3	0.7597	0.2538	(--------*---------)
NBS1	22	0.2163	0.1478	(---*--)
NBS12	6	0.3017	0.0906	(------*------)
NBS2	23	0.3653	0.2070	(---*--)
other	6	0.5167	0.4086	(------*------)
Pooled StDev =		0.2102		0.25 0.50 0.75 1.00

Table 6.11c Effect of background material

Source	DF	SS	MS	F	P
background	7	1.7494	0.2499	6.96	0.000
Error	53	1.9029	0.0359		
Total	60	3.6523			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
anth	15	0.2359	0.1904	(--*---)
benz	11	0.6902	0.2836	(----*---)
calc	1	0.3600	0.0000	(----------*----------)
charc	2	0.4250	0.0354	(----------*----------)
coal	7	0.3071	0.2257	(----*-----)
graph	8	0.2689	0.1211	(-----*----)
Marble	5	0.4234	0.1066	(------*------)
other	12	0.2283	0.1114	(---*----)
Pooled StDev =		0.1895		0.00 0.25 0.50 0.75

6.5.1.3 Conclusions

A significant laboratory type effect is observed, with AMS laboratories having lower mean quoted pMC. Similarly, a significant modern standard effect is observed, with NBS1 giving the lowest mean pMC. There is also a statistically significant effect of the background material with apparent differences between laboratory results based on anthracite or benzene as the background material.

In all cases for Kauri A, laboratory type (LSC laboratories have, on average, higher pMC than AMS or GPC), modern standard, and background material were all found to be statistically significant.

6.5.2 Kauri B

The same analysis is repeated for Kauri B and results presented in the same format. Figure 6.8 shows the considerable variation in the distribution of results over the factor levels.

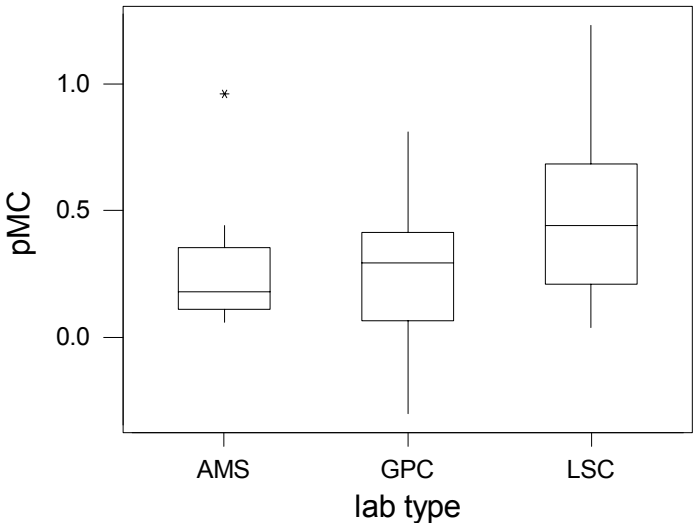

Figure 6.8a Distribution by laboratory type

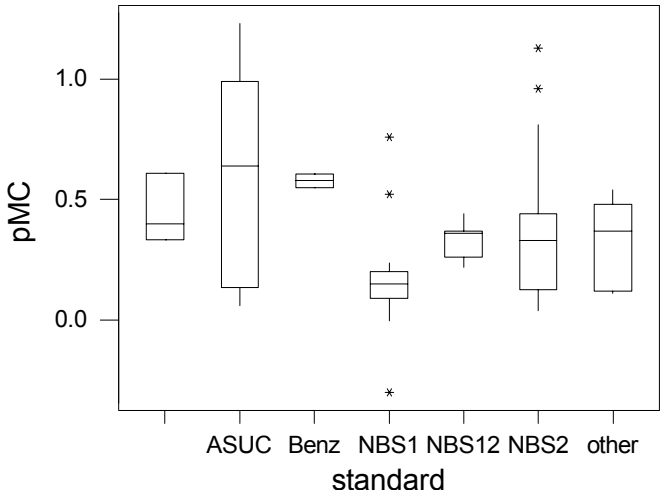

Figure 6.8b Distribution by modern standard

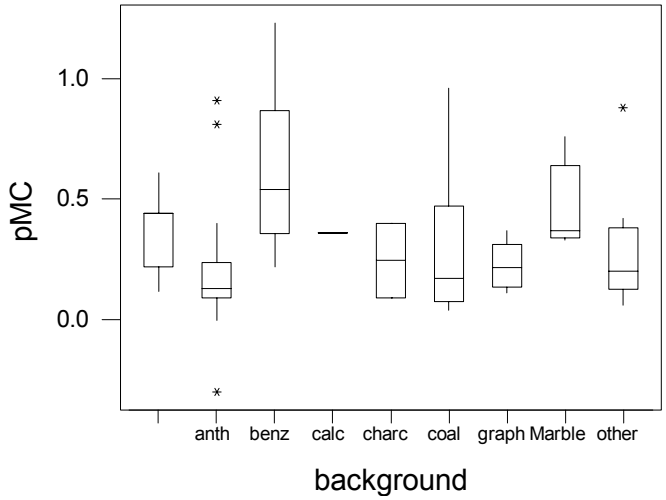

Figure 6.8c Distribution by background material

6.5.2.1 Formal Analysis

Table 6.12 shows the results of the formal analysis.

Table 6.12a Effect of laboratory type

Source	DF	SS	MS	F	P
Type	2	0.9957	0.4978	7.88	0.001
Error	66	4.1679	0.0631		
Total	68	5.1636			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
AMS	32	0.2344	0.1744	(-----*-----)
GPC	16	0.2623	0.2681	(-------*--------)
LSC	21	0.5037	0.3276	(-------*------)
Pooled StDev =		0.2513		0.15 0.30 0.45 0.60

Table 6.12b Effect of modern standard

Source	DF	SS	MS	F	P
standard	5	1.1579	0.2316	3.55	0.007
Error	60	3.9150	0.0652		
Total	65	5.0729			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
ASUC	6	0.6067	0.4683	(-------*--------)
Benz	2	0.5780	0.0396	(-------------*--------------)
NBS1	21	0.1646	0.1974	(----*---)
NBS12	7	0.3357	0.0741	(------*-------)
NBS2	25	0.3504	0.2787	(---*---)
other	5	0.3140	0.1877	(---------*--------)
Pooled StDev =		0.2554		0.25 0.50 0.75

Table 6.12c Effect of background material

Source	DF	SS	MS	F	P
background	7	1.1787	0.1684	2.39	0.033
Error	54	3.8000	0.0704		
Total	61	4.9787			

Level	N	Mean	StDev	Individual 95% CIs For Mean Based on Pooled StDev
anth	15	0.2123	0.3019	(---*----)
benz	9	0.6099	0.3459	(-----*-----)
calc	1	0.3600	0.0000	(-----------------*-----------------)
charc	2	0.2450	0.2192	(-----------*------------)
coal	9	0.2822	0.3064	(-----*-----)
graph	8	0.2226	0.0967	(-----*------)
Marble	5	0.4662	0.1807	(-------*------)
other	13	0.2638	0.2183	(----*----)
Pooled StDev =		0.2653		0.00 0.30 0.60 0.90

6.5.2.2 Conclusions

In all cases for Sample B, we have evidence of a statistically significant effect due to laboratory type, modern standard, and background material used. Again, there are apparent differences:

- ANU sucrose results give the highest average pMC;
- There is a difference in the average pMC for anthracite and benzene;
- LSC laboratories quote, on average, higher pMC values.

6.6 ANALYSIS OF AGE, KAURI A

We now use the techniques developed in the reliability analysis (see Appendix 3) to explore the age distribution, which, therefore, means that we must utilize both censored and uncensored values. A censored datum is one for which the result is expressed as "> age" BP.

In addition, given the censored nature of the data, non-parametric methods of estimation, used commonly in survival or reliability analyses (in particular, the Kaplan-Meier survival estimator),

have been used to estimate the "mean" activity of the sample. Reliability plots display the "survival" probabilities versus time, which in this context, is the probability that the sample is greater than age *t*. Each point on the plot represents the proportion of results greater than age *t* and the non-parametric reliability curve is shown graphically as a step function. In addition, common measures of the center and spread of the distribution of age are estimated. It should be noted that the mean is very sensitive to large ages, while the median, Q1 (25th percentile), Q3 (75th percentile), and interquartile range (IQR) are resistant, so they are quoted in preference.

The outlier definitions used are identical to those used in the pMC analysis.

6.6.1 Kauri A

There were 25 censored and 58 uncensored ages. Table 6.13 shows the mean and median age (estimated taking the censoring into account), the quartiles, the interquartile range, and a 95% confidence interval (CI) for the true mean age.

Table 6.13 Age of Kauri A

						95.0% normal CI	
Mean	Median	Q1	Q3	IQR	Standard error	Lower	Upper
47,007	47,935	43,900	51,530	7630	808	45,423	48,590

The mean age is estimated at 47,006 BP, with a 95% CI of 45,423–48,590 BP. The median is approximately 1000 yr older than the mean age, suggesting a tail of younger results. The 95% CI spans almost 4000 yr, indicating the substantial variation in the reported results.

6.6.2 Sources of Variation

If we now consider a similar analysis for each of the factors (laboratory type, modern standard, and background material), we can explore the differences in the age distribution of the results that also account for censoring.

For LSC laboratories, there were 26 values, 8 of which were censored; for GPC, there were 17 results, 7 of which were censored values; while for AMS, there were 32 results, 10 of which were censored.

Table 6.14 Age estimation by laboratory type

							95.0% normal CI	
	Mean	Median	Q1	Q3	IQR	Standard error	Lower	Upper
LSC	44,155	44,024	40,190	47,600	7410	1641	40,939	47,372
GPC	47,507	47,935	42,440	52,240	9800	1477	44,610	50,403
AMS	49,408	50,200	47,490	51,530	4040	569	48,293	50,524

It is clear from Table 6.14 that the AMS laboratories report a significantly older mean age for this sample (median = 50,200 yr BP) than either LSC or GPC laboratories.

6.6.2.1 Comparison of Age Distributions

A formal test comparing the age distribution can be carried out and has a p-value <0.05, showing quite clearly that there is a significant difference in the age distribution for the 3 laboratory types. Figure 6.9 shows the cumulative age distribution for the 3 laboratory types. This shows that the GPC

and AMS curves lie clearly above that for LSC. GPC and AMS laboratories are typically measuring and quoting older ages for this sample.

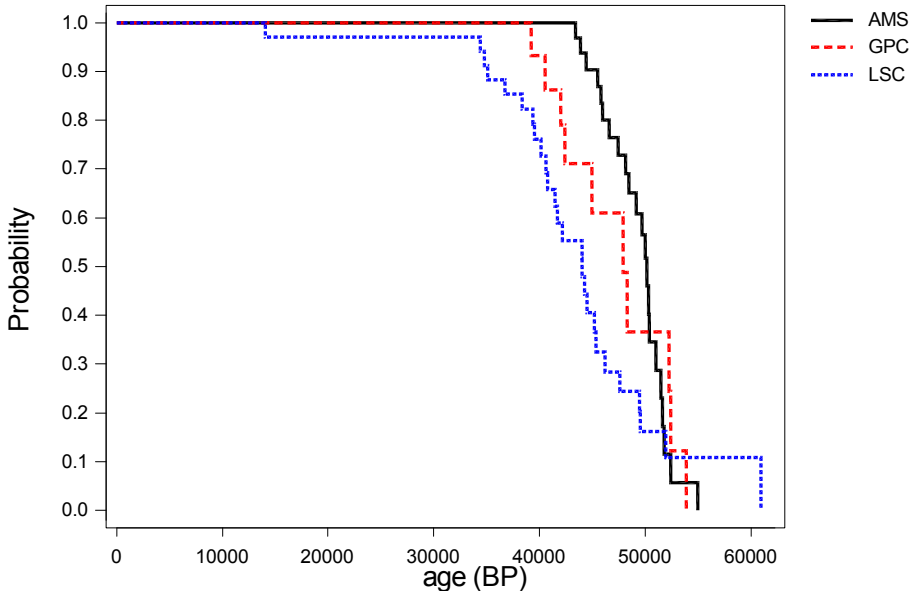

Figure 6.9 Cumulative age distribution by laboratory type

6.6.3 By Standard Material

The analysis used previously for the modern standard material is used again and shows a significant difference in the age distribution. Table 6.15 and Figure 6.10 show the age distributions.

Table 6.15 Age estimation by modern standard[a]

							95.0% normal CI	
	Mean	Median	Q1	Q3	IQR	Standard error	Lower	Upper
Other	44,800	46,610	45,000	47,935	2935	2258	40,374	49,225
NBS2	45,995	45,398	44,024	51,971	7947	1639	42,782	49,208
NBS12	48,233	50,300	45,500	50,300	4800	1436	45,417	51,049
NBS1	49,399	50,200	48,305	51,800	3495	731	47,966	50,832
Benz	40,585	39,556	36,780	42,211	5431	2574	35,539	45,631
Sucrose	40,425	*	*	*	*	1374	37,730	43,119

[a] * indicates that there were insufficient data to complete the calculation

6.6.3.1 Comparison of Survival Curves

The summary statistics of the age for each standard type are shown in the following.

The formal test of comparability of the cumulative age distribution results in p-values <0.05, so we can conclude that there is a statistically significant difference in the age distributions for the different modern standards. Figure 6.10 shows that the benzene curve is lower than all others and suggests that the NBS1 curve is the highest. This would suggest that laboratories using benzene as

their modern standard are quoting younger ages than laboratories using other modern standard materials.

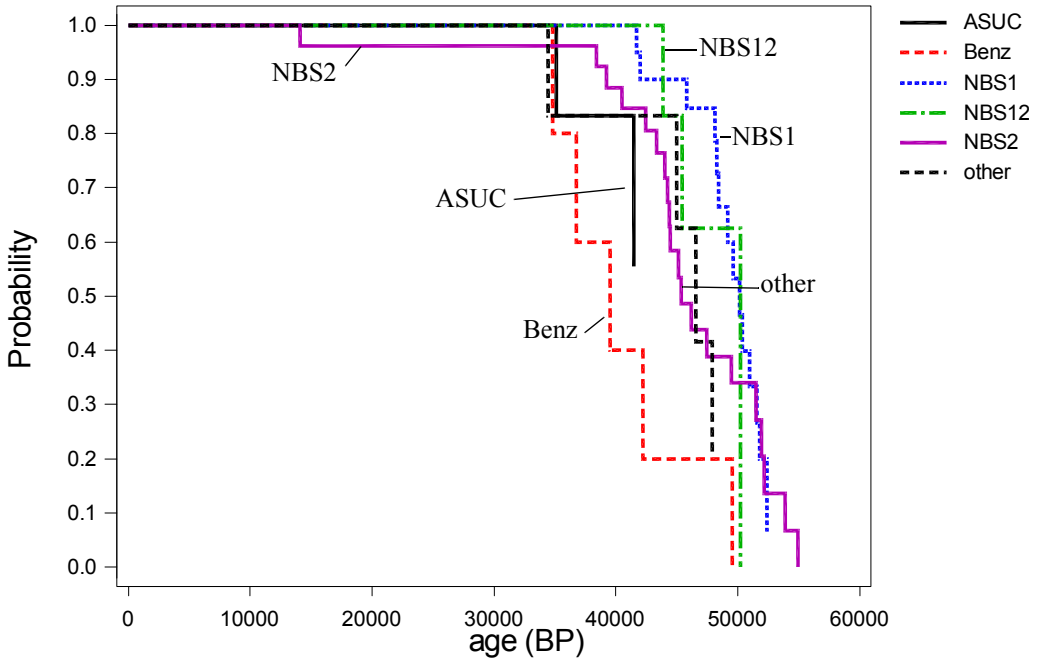

Figure 6.10 Cumulative age distribution by modern standard

6.6.4 Age Distribution by Background Material

Table 6.16 and Figure 6.11 repeat a similar analysis, but take into account the background material used.

Table 6.16 Age estimation by background material[a]

							95.0% normal CI	
	Mean	Median	Q1	Q3	IQR	Standard error	Lower	Upper
Other	47,051	48,305	44,400	50,380	5980	1260	44,581	49,522
Marble	41,988	*	—	—	—	—	*	—
Graphite	47,748	47,490	45,500	50,200	4700	1005	45,777	49,720
Coal	48,014	51,530	44,480	51,800	7320	1936	44,219	51,809
Charc	43,390	*	—	—	—	*	*	—
Anthracite	48,203	52,240	45,818	53,900	8082	2574	43,156	53,249

[a] * indicates that there were insufficient data to complete the calculation

The formal test again showed a significant difference in the age distribution between the different background materials (as evidenced in Figure 6.11). The results for laboratories using benzene as a background material lie below the curves for other background materials, so the reported ages tend to be younger for those laboratories using benzene as a background material (LSC laboratories) and this is confirmed in Table 6.16.

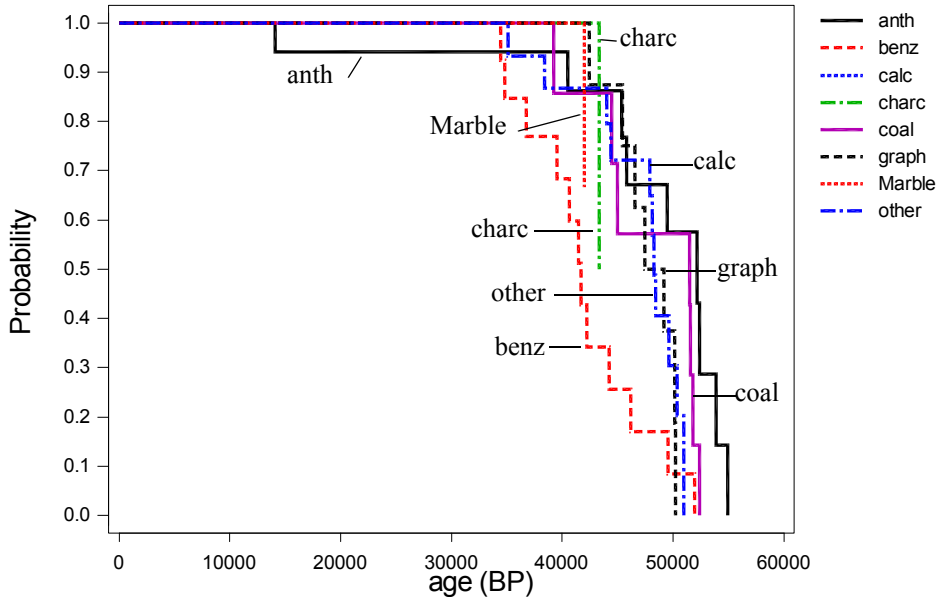

Figure 6.11 Cumulative age distribution by background material

6.7 ANALYSIS OF AGE, KAURI B

For Kauri B, a total of 83 measurements were reported, 51 of which were uncensored. The summary of the age distribution is given in the table below.

Table 6.17 Age estimation

						95.0% normal CI	
Mean	Median	Q1	Q3	IQR	Standard error	Lower	Upper
48,210	49,815	44,043	53,393	9350	730	46,779	49,641

The mean age is 48,210 BP, with the median age being approximately 1000 yr older, again suggesting that the distribution of ages has a long left tail (younger results). The IQR of just over 9300 yr again shows the considerable variation in the results reported.

6.7.1 Analysis by Laboratory Type

The formal test of equal cumulative distributions shows a significant difference in age for the different laboratory types (as shown in Table 6.18 and Figure 6.12). For LSC, 10 of 32 measurements were censored, GPC had 10 censored from 17 measurements, and AMS had 12 censored from 34 measurements. Again, we see in the figure that the LSC distribution lies clearly below the GPC and AMS distributions.

Table 6.18 Age distribution for laboratory type

							95.0% normal CI	
	Mean	Median	Q1	Q3	IQR	Standard error	Lower	Upper
LSC	44,423	44,900	39,200	49,900	10,700	999	42,643	46,382
AMS	50,612	51,000	46,660	54,500	7840	878	48,890	52,333
GPC	53,140	53,140	44,043	53,393	9350	1661	45,681	53,393

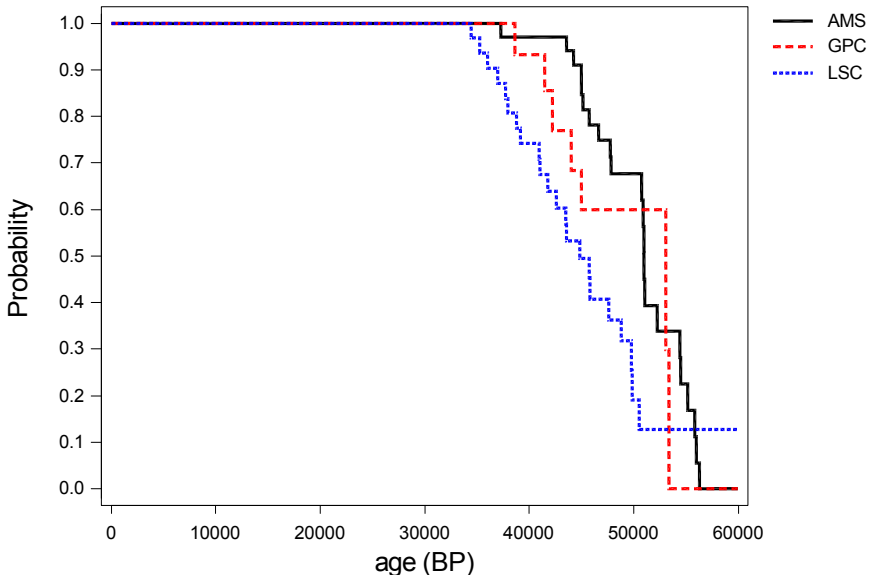

Figure 6.12 Cumulative age distribution by laboratory type

6.7.3 Age Distribution by Modern Standard

Figure 6.13 and Table 6.19 show the age distributions for the laboratories using different modern standards. The formal statistical test shows a significant difference in the age distribution, with those laboratories using benzene as the modern standard quoting results that are significantly younger on average.

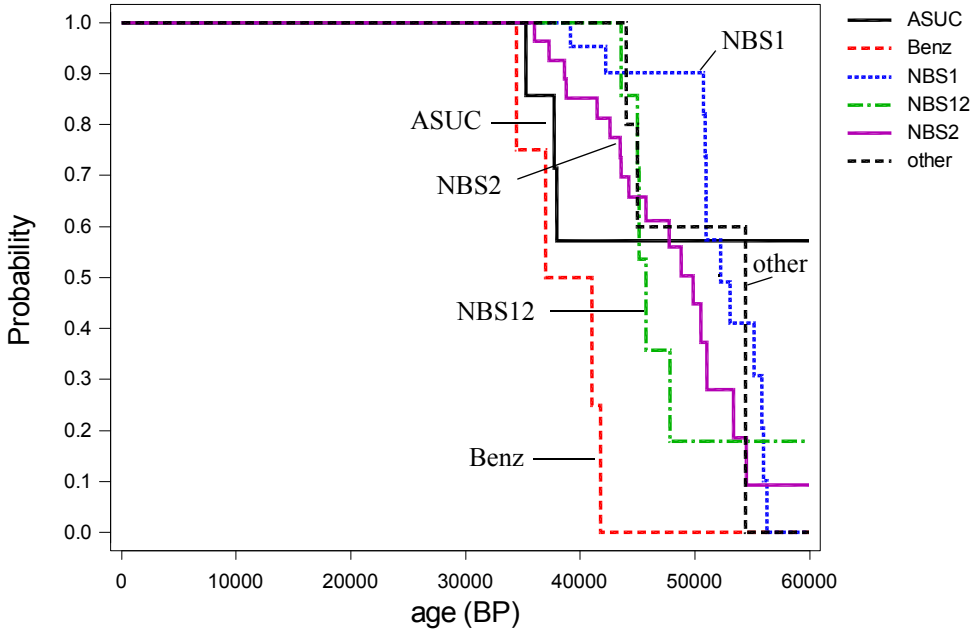

Figure 6.13 Cumulative age distribution by modern standard

Table 6.19 Age estimation for modern standard type[a]

	Mean	Median	Q1	Q3	IQR	Standard error	95.0% normal CI Lower	95.0% normal CI Upper
Other	50,492	54,473	45,000	54,473	9473	2675	45,248	55,736
NBS2	47,766	49,900	43,540	53,393	9853	1199	45,415	50,116
NBS12	46,014	45,800	45,000	47,900	2900	686	44,668	47,360
NBS1	52,250	52,300	50,900	55,900	5000	1092	50,109	54,390
Benz	38,549	37,000	34,420	41,013	6593	1728	35,161	41,937
Sucrose	37,584	*	—	—	*	431	36,739	38,429

[a] * indicates that there were insufficient data to complete the calculation

6.7.4 Effect of Background Material

The results (Table 6.20 and Figure 6.14) again show a clear difference in the age distributions due to the background material used. The formal test shows this result is statistically significant, with laboratories using benzene and marble as their background material quoting younger ages.

Table 6.20 Age estimation by background material[a]

	Mean	Median	Q1	Q3	IQR	Standard error	95.0% normal CI Upper	95.0% normal CI Lower
Other	49,533	50,800	44,300	50,900	6600	1812	45,982	53,085
Marble	41,473	42,231	*	*	*	928	39,654	43,292
Graph	49,605	47,900	45,800	52,300	6500	1270	47,115	52,095
Coal	49,496	51,090	45,000	56,000	11,000	2387	44,816	54,176
Benz	41,919	41,764	36,030	45,830	9800	1560	38,860	44,978
Anth	51,214	53,140	50,600	54,500	3900	1639	48,000	54,428

[a] * indicates that there were insufficient data to complete the calculation

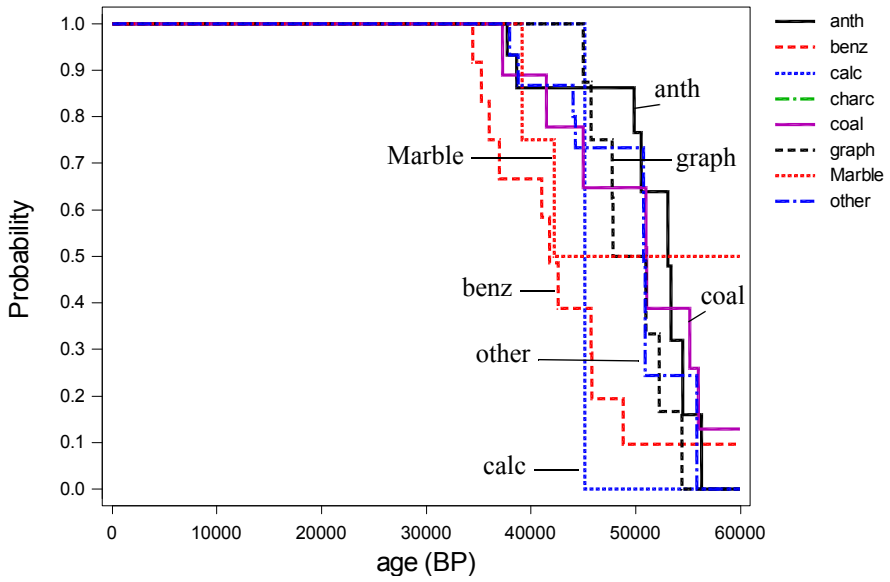

Figure 6.14 Cumulative age distribution by background material (charcoal not shown)

6.8 ANALYSIS OF AGE, COMBINED RESULTS

In total, of the 166 measurements on A and B combined, 57 were censored. The overall results are summarized below (Table 6.21) and show a median age of 48,305 BP, and the 50% range of the data as 43,900–51,800 BP.

Table 6.21 Age estimation (A and B combined)

						95.0% normal CI	
Mean	Median	Q1	Q3	IQR	Standard error	Upper	Lower
47,634	48,305	43,900	51,800	7900	555	46,545	48,723

6.8.1 Sources of Variation

The 3 main sources of variation—laboratory type, modern standard, and background material—are analyzed in the following sections.

6.8.1.1 Effect of Laboratory Type

Again, laboratory type is found to be highly significant. From Table 6.22, the mean and median age reported by AMS laboratories is approximately 2000 and 5000 yr greater than GPC and LSC laboratories, respectively. Figure 6.15 shows the cumulative age distribution curves, with the LSC curve lying below those for AMS and GPC.

Table 6.22 Age by laboratory type

							95.0% normal CI	
	Mean	Median	Q1	Q3	IQR	Standard error	Upper	Lower
AMS	50,007	50,800	47,490	52,300	4810	533	48,961	51,054
GPC	48,097	48,305	42,440	53,140	10,700	1090	45,960	50,234
LSC	45,039	44,300	40,190	49,580	9390	1130	42,824	47,254

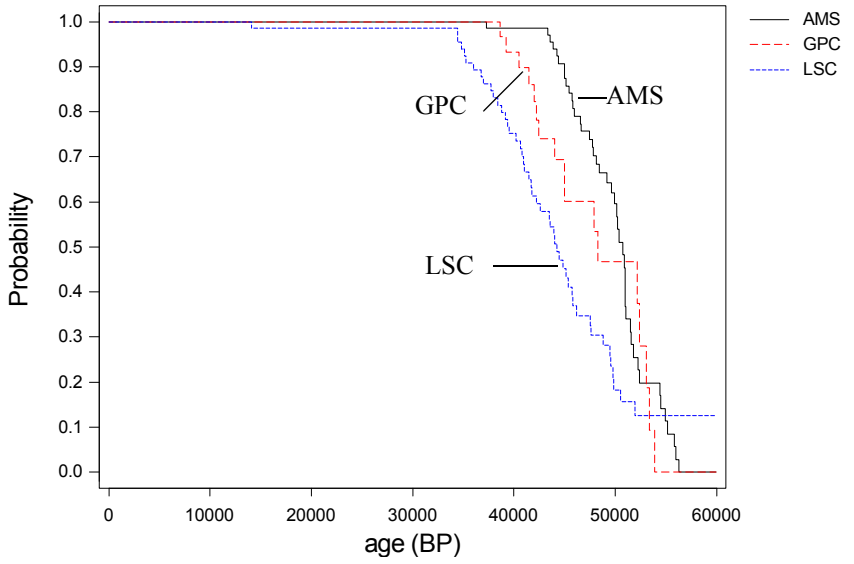

Figure 6.15 Cumulative age distribution by laboratory type

6.8.1.2 Effect of Modern Standard

A statistically significant difference in age distributions due to modern standard is found (Figure 6.16).

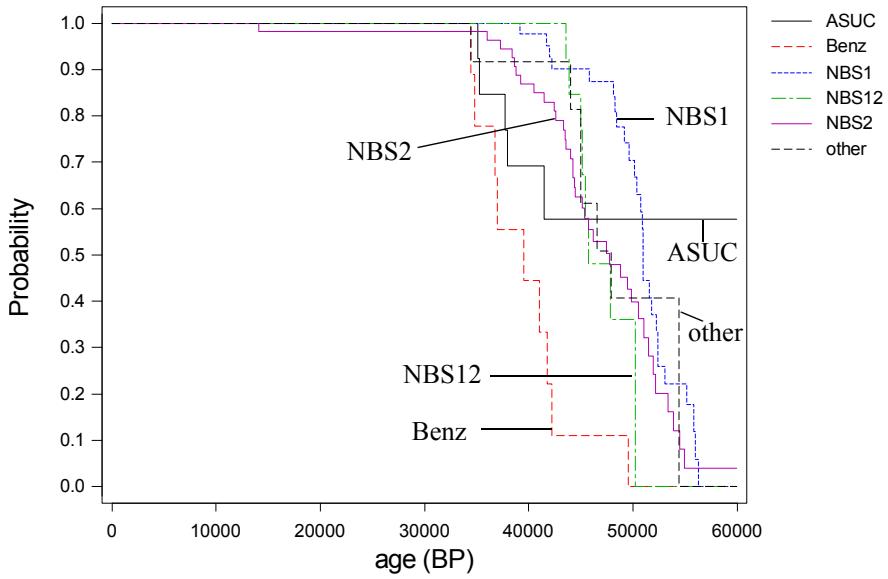

Figure 6.16 Cumulative age distribution by modern standard material

NIST OXI shows an age distribution that favors older ages, while laboratories using benzene as the modern standard quote overall younger ages, suggesting that the benzene activity is too high in comparison to the primary standards of NIST OXI and OXII.

6.8.1.3 Effect by Background Material

A statistically significant difference in the age distributions due to background material is found (Figure 6.17).

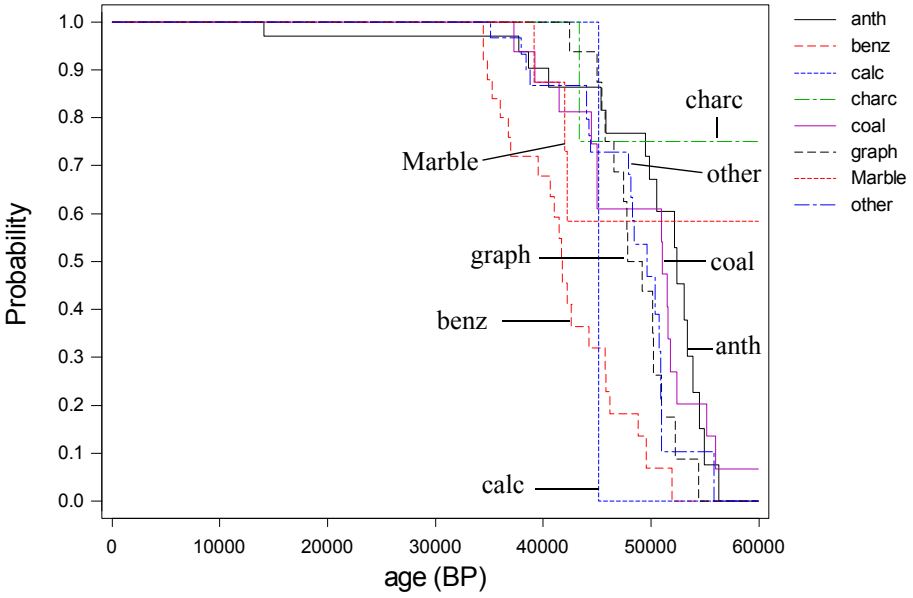

Figure 6.17 Cumulative age distribution by background material

There is a suggestion that laboratories using benzene as the background material are quoting younger ages on average.

6.9 CONCLUSIONS

Overall, this analysis has demonstrated significant differences between the laboratory types in the age distribution quoted for this near-background sample. At the same time, the effects of the modern standard and the background material have also been identified. This most sensitive sample to the laboratory parameters has shown significant differences due to laboratory type (LSC laboratories appear to be significantly different from AMS and GPC laboratories). This finding is further supported by the findings for the effects of modern standard and background material (where the use of benzene has been identified). Further, the intercomparison has also underlined the variation in the calculation and reporting formats for near-background samples.

SECTION 7: CHARACTERIZATION OF THE REFERENCE MATERIALS BY CONSENSUS VALUES

7.1 CALCULATION OF CONSENSUS VALUES

Each material needs to be characterized by estimating its activity, which creates a reference value for each material. This value then can be considered as the "known" activity of the material and future analyses can be compared to this to quantify the accuracy of the measurement. In this way, the materials remain useful for laboratory quality assurance.

The procedure used in the calculation of the consensus value comes from Rozanski et al. (1992) and is an iterative one. It is described below.

There are 3 stages.

- **Stage 1**: Outlying results are removed if they are greater than 3 interquartile ranges from the nearest of either the lower or upper quartiles. This occurs when a result is either greater than Q3 + 3(Q3–Q1) or less than Q1 – 3(Q3–Q1), where *Q1* and *Q3* are the lower and upper quartiles, respectively. Then, the preliminary consensus value is calculated as the median (*m*) of the remaining results.
- **Stage 2**: Remove results that are at least twice their quoted error (σ) from the preliminary consensus value. That is, only keep $|x–m| / \sigma < 2$, where *x* is the result, *m* the preliminary consensus value, and σ the quoted error.
- **Stage 3**: Calculate the final consensus as a weighted mean of the remaining results, using their σ^2 values as the weights.

7.1 Remarks on the Procedure

For FIRI Samples A and B (in yr), this approach is not very appropriate given that many laboratories did not quote finite ages. For these samples, an alternative approach was used based on the reliability analysis (see Section 6).

It should also be noted that averages are rather sensitive to extreme data values, which is why the outliers are removed in Step 1.

The approach has the advantage that an estimated error can be calculated for the consensus value (which will usually be very small since there are a large number of results).

7.2 INITIAL CONSENSUS VALUES

Consensus values are reported in Table 7.1, based on the Rozanski et al. procedure.

Table 7.1 Preliminary consensus values

FIRI sample	Weighted mean (1 σ)	AMS	GPC	LSC
C	18,173 (10.5)	18,183 (13)	18,229 (28)	18,140 (25)
DF	4508 (3)	4519 (4)	4484 (5)	4507 (6)
E	11,778 (7)	11,805 (9)	11,738 (19)	11,707 (17)
GJ	110.69 (0.04)	110.52 (0.05)	110.85 (0.07)	110.82 (0.08)
H	2232 (5)	2238 (6)	2198 (9)	2233 (9)
I	4485 (5)	4483 (7)	4456 (10)	4499 (11)

It should be noted that the results for Samples A and B are not included in this table, since this procedure only is possible using results where a quoted error is given. However, the results for Samples A and B will be returned to later in this section, when the analysis of the pMC is completed.

Figures 7.1 to 7.7 (Section 7 appendix, p 269–275) show the distribution of the laboratory results around these consensus values. They include the laboratory-quoted errors. In such figures, we can see how closely the results from the different laboratories agree (accounting for their quoted errors). The consensus values are also marked. In Step 2, laboratories quoting small errors will be excluded, unless they lie close to the consensus value, while laboratories quoting large errors will be included in Step 3. However, in Step 3, results with large errors will be down-weighted in the calculation and so will not have a large impact on the final result.

Therefore, there is an issue of how robust the initial consensus value is in Step 1, and how important its definition is on the final consensus value. Therefore, we consider variants of this original method, which at Step 1 exclude not simply extreme age/activity values, but also results with large quoted errors.

7.3 THE EFFECT OF SCREENING OUT RESULTS WITH LARGE QUOTED ERRORS IN CONSENSUS CALCULATIONS

7.3.1 σ Method 1

This method is the same as the original one, except that, between Stage 1 and Stage 2, results with a quoted uncertainty greater than a certain cut-off point are rejected.

7.3.2 σ Method 2

This method is the same as the σ Method 1, but this time, results with a quoted uncertainty greater than a certain cut-off point are rejected *before* Stage 1.

7.3.3 Choice of σ "Cut-off" Points

The choice of the cut-off points is subjective. However, from the histograms showing the distribution of σ and expert opinion, the cut-off points shown in Table 7.2 were used for both methods. Because of the subjectivity of the decisions, 2 (or, in AB's case, 3) different values for the cut-off points were chosen for each sample.

Table 7.2 Cut-off points used for the different samples

Sample	Cut-off points used			Units
Kauri wood, AB	0.3	0.15	0.1	pMC
Turbidite, C	200	150	—	yr BP
Belfast dendro-wood, DF	100	50	—	yr BP
Humic acid, E	150	100	—	yr BP
Barley mash, GJ	1	0.6	—	pMC
Hohenheim dendro-wood, H	100	50	—	yr BP
Belfast cellulose, I	100	50	—	yr BP

7.3.4 Results

From the results in Table 7.3, it can be seen that the various methods for calculating the consensus make very little difference to all but the AB sample (ranges of only 2.3 yr BP for Sample C, 1.4 yr BP for DF, 11.7 yr BP for Sample E, 0.06 pMC for GJ, 0.7 yr BP for H, and 10.6 yr BP for I).

For Sample AB, there is little difference within the σ Method 1 (a range of only 0.075 pMC), but there is for the σ Method 2, with the more restrictive cut-off points. When results with a σ greater than 0.1 pMC are screened, the consensus value becomes 0.2 pMC, less than two-thirds the value it is under the original method.

Table 7.3 Consensus values under the different methods

Sample		Original methods	Method 1			Method 2		
AB	σ cut-off	None	0.3	0.15	0.1	0.3	0.15	0.1
(pMC)	Consensus	0.330	0.330 (0.01)	0.327 (0.01)	0.325 (0.01)	0.324 (0.01)	0.251 (0.01)	0.203 (0.01)
C	σ cut-off	None	200	150	—	200	150	—
(yr BP)	Consensus	18,175.5 (10.5)	18,176.5 (9.7)	18,177.8 (9.3)	—	18,176.5 (9.7)	18,177.8 (9.3)	—
DF	σ cut-off	None	100	50	—	100	50	—
(yr BP)	Consensus	4508.3 (3)	4508.2 (3)	4506.8 (3)	—	4508.2 (3)	4506.8 (3)	—
E	σ cut-off	None	150	100	—	150	100.00	—
(yr BP)	Consensus	11,779.9 (7)	11,781.2 (8)	11,781.7 (7.6)	—	11,791.6 (7.8)	11,791.2 (8)	—
GJ	σ cut-off	None	1	0.6	—	1	0.6	—
(pMC)	Consensus	110.69 (0.04)	110.69 (0.04)	110.72 (0.04)	—	110.69 (0.04)	110.75 (0.04)	—
H	σ cut-off	None	100	50	—	100	50	—
(yr BP)	Consensus	2232.5 (5)	2232.3 (4.7)	2233.0 (4.7)	—	2232.3 (4.7)	2233.0 (4.7)	—
I	σ cut-off	None	100	50	—	100	50	—
(yr BP)	Consensus	4484.9 (5)	4485.1 (5)	4482.1 (5)	—	4485.1 (5)	4474.5 (5.3)	—

7.3.5 Discussion

The alternative methods for calculating the consensus only lead to very small differences, except in the case of the Kauri wood sample, AB. Here, screening out results with σs larger than a cut-off point before using the original method, shifted the consensus by large amounts when the cut-off was small (from 0.33–0.20 pMC, when the cut-off was 0.1 pMC). Possible reasons for this change could stem from AB being a sample at, or near, the limits of detection for ^{14}C dating.

Since the Kauri wood's activity is so low, some results are given as background or non-finite. This occurs when the σ is large with respect to its result. Obviously, those laboratories that have a lower σ can give finite results for older samples. Because background and non-finite results are excluded from the consensus calculation, this could bias the calculations.

Also, it is possible that laboratories have reported pMC results for samples that should be considered background or non-finite. At present, these results are not screened out. Such an approach could be valuable in providing a more reliable estimate of the activity in the Kauri wood sample.

7.3.6 Conclusion

The consensus calculations are robust in the initial screening stages for all but the Kauri wood samples. For this sample, the consensus age has been calculated by a different method and reported in Section 6. For the pMC results, the consensus calculation has been carried out, but with a number

of caveats. In AB's case, a better value for the consensus may be achieved if any results that are too small with respect to their quoted errors are screened out. If this does not help, then we may be left with a large range for the consensus value of the Kauri wood sample's activity. We recommend that the results in Table 7.1 and Table 6.20 be used as consensus values for the FIRI samples.

7.4 DEVIATIONS FROM CONSENSUS VALUES

We define the standardized deviation as the difference between the result and the consensus value, divided by the quoted uncertainty on the result. Using this summary, we can explore the distribution of laboratory performance. Ideally, we might expect a standardized deviation to lie between +2 and –2. Values greatly exceeding 2 or –2 indicate either a large absolute difference between the result and the consensus value or a "large" difference relative to the quoted error. This makes them sensitive indicators of general laboratory performance. The standardized deviations for each sample (except AB) can then be investigated for the effects of different laboratory factors.

7.4.1 Effect of Laboratory Type for Sample C: Turbidite

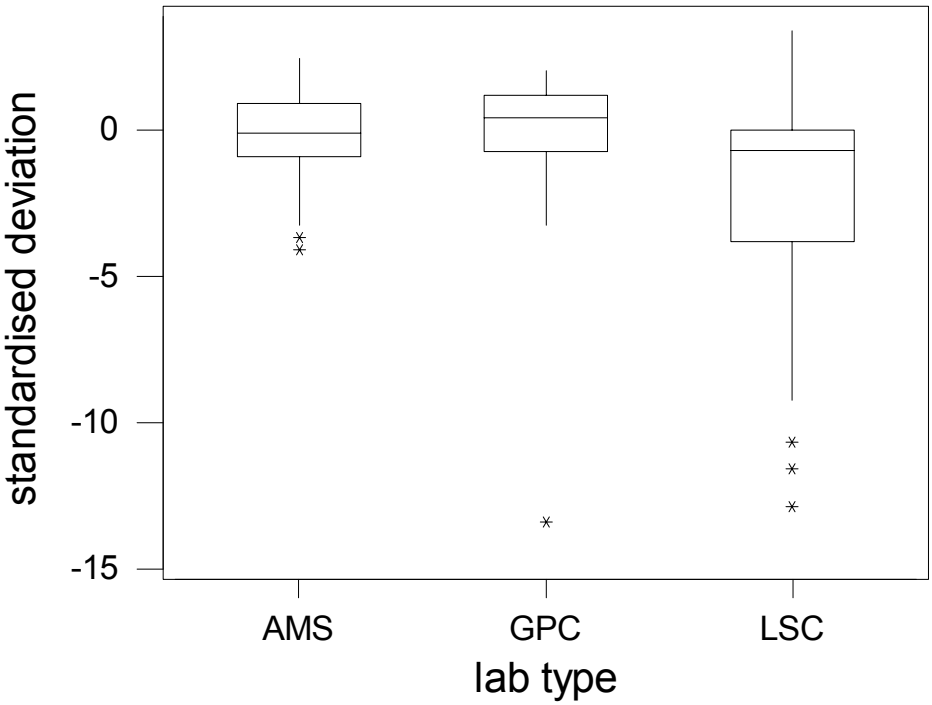

Figure 7.8 Distribution of standardized deviation for Sample C

We can see that AMS and GPC results appear to show a broadly similar distribution. For LSC results, the distribution is more widely scattered. Each laboratory type has a number of extreme values and this is more pronounced for the LSC set of results.

7.4.2 Effect of Laboratory Type for Sample D: Belfast Wood

A similar pattern is apparent; the median value lies close to 0, but there are a number of extreme values, typically reported by LSC laboratories.

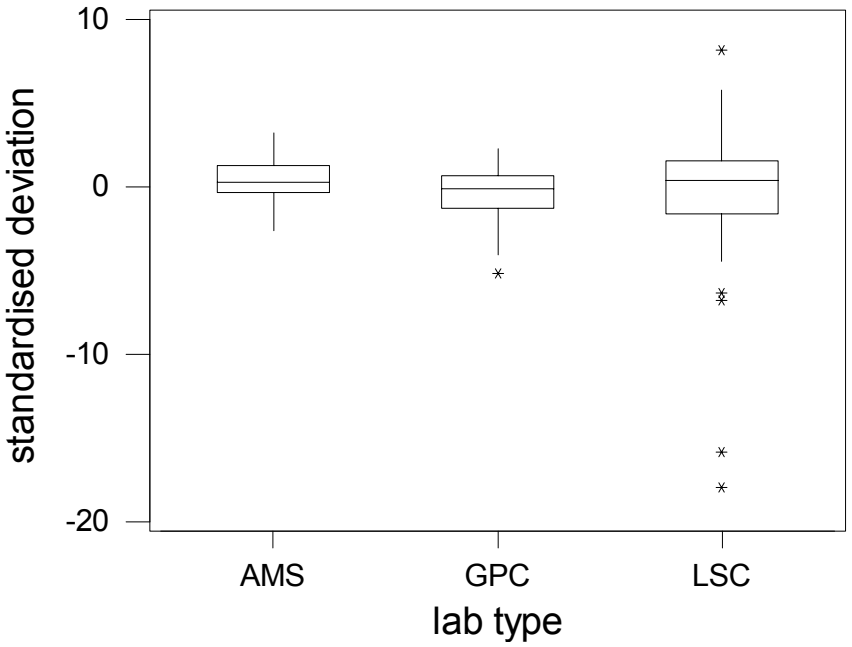

Figure 7.9 Distribution of standardized deviation for Sample D

7.4.3 Effect of Laboratory Type for Sample E: Humic Acid

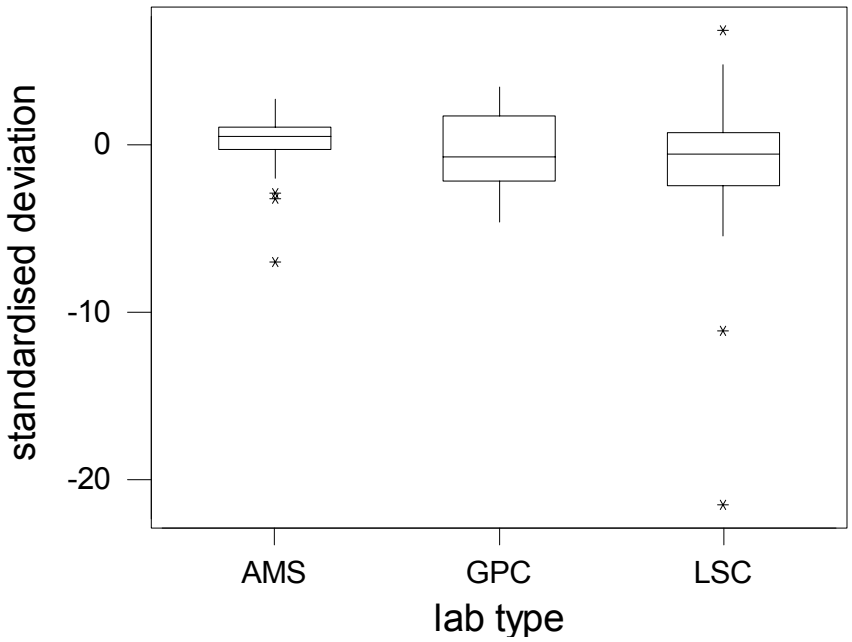

Figure 7.10 Distribution of standardized deviation for Sample E

A similar distributional pattern is apparent; the median value lies close to 0, but there are a small number of extreme values.

7.4.4 Effect of Laboratory Type for Sample F: Belfast Wood

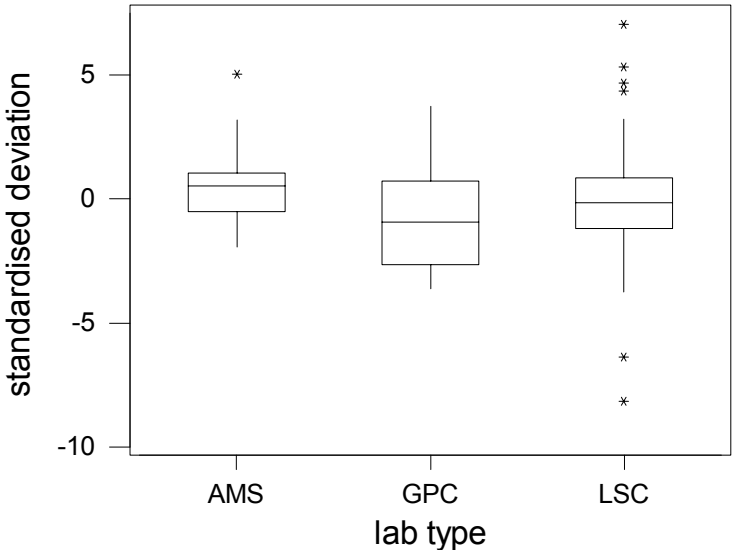

Figure 7.11 Distribution of standardized deviation for Sample F

The median value lies close to 0, but there are a number of extreme values, typically reported by LSC laboratories.

7.4.5 Effect of Laboratory Type for Sample G: Barley Mash

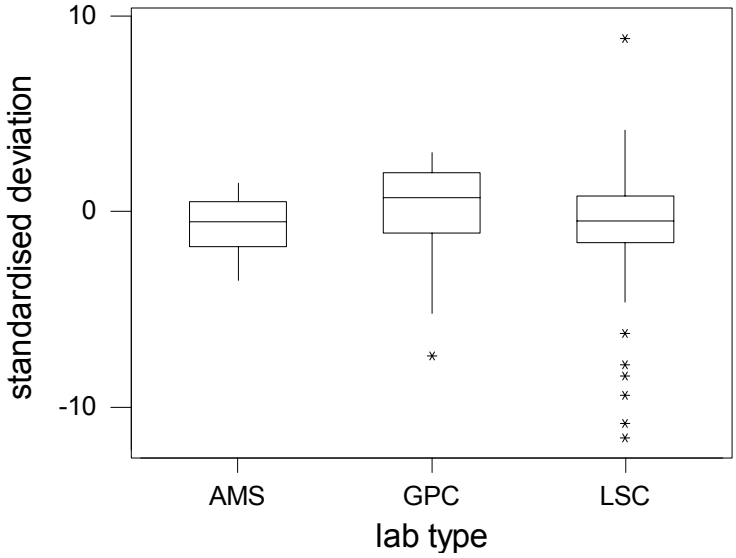

Figure 7.12 Distribution of standardized deviations for Sample G

The median value lies close to 0, but there are a number of extreme values, typically reported by LSC laboratories. Omitting these results would result in broadly similar distributions for the 3 laboratory types.

7.4.6 Effect of Laboratory Type for Sample H: Hohenheim Wood

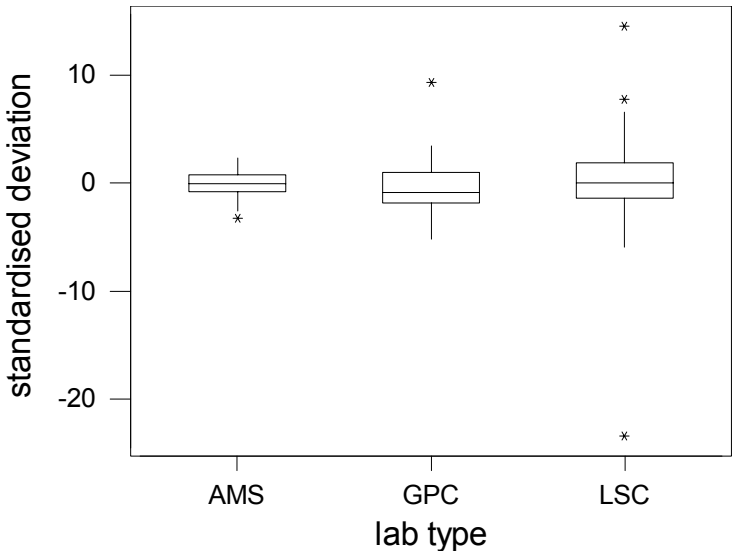

Figure 7.13 Distribution of standardized deviations for Sample H

A similar pattern is apparent. The median value lies close to 0, but there are a number of extreme values, typically reported by LSC laboratories.

7.4.7 Effect of Laboratory Type for Sample I: Belfast Cellulose

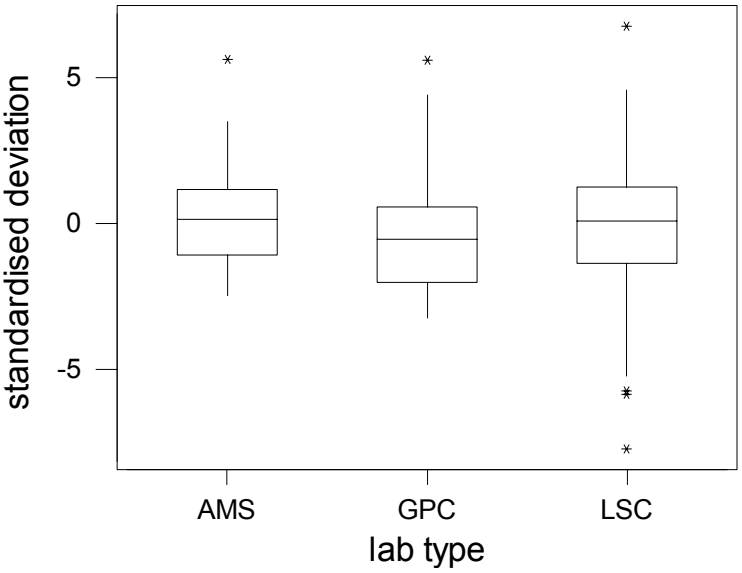

Figure 7.14 Distribution of standardized deviations for Sample I

The distribution of results is less wide for this sample. The median value lies close to 0, but there are a small number of extreme values, which are reported by LSC laboratories.

7.4.8 Effect of Laboratory Type for Sample J: Barley Mash

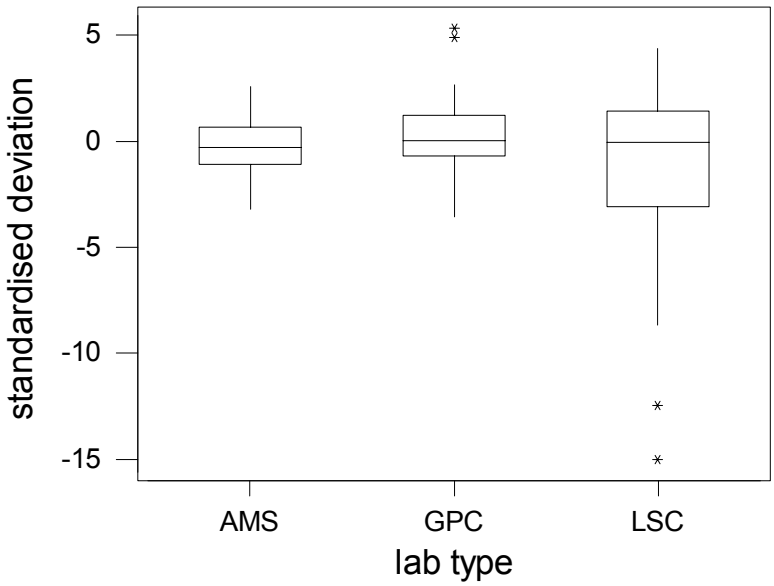

Figure 7.15 Distribution of standardized deviations for Sample J

A similar pattern is apparent, where the median value lies close to 0. The distribution of results is wider for LSC laboratories and there are several extreme values.

7.4.9 Effect of Laboratory Type for Sample A: Kauri Wood

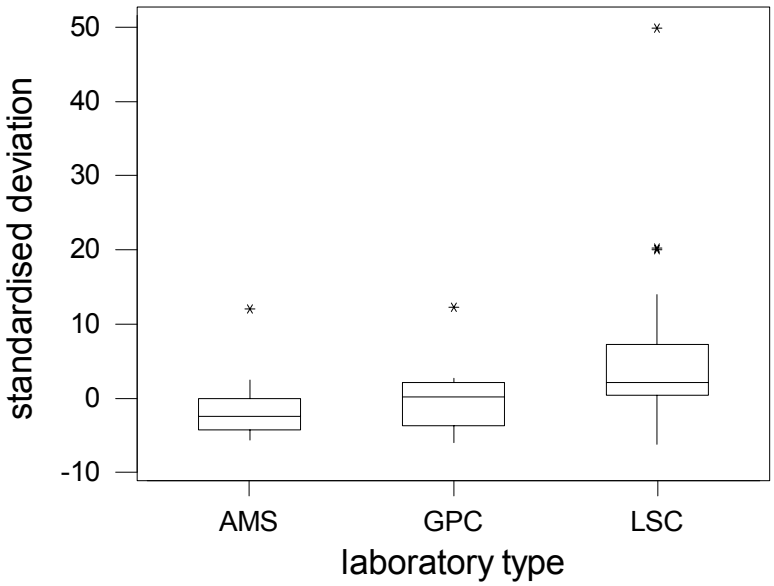

Figure 7.16 Distribution of standardized deviations for Sample A

7.4.10 Sample B: Kauri Wood

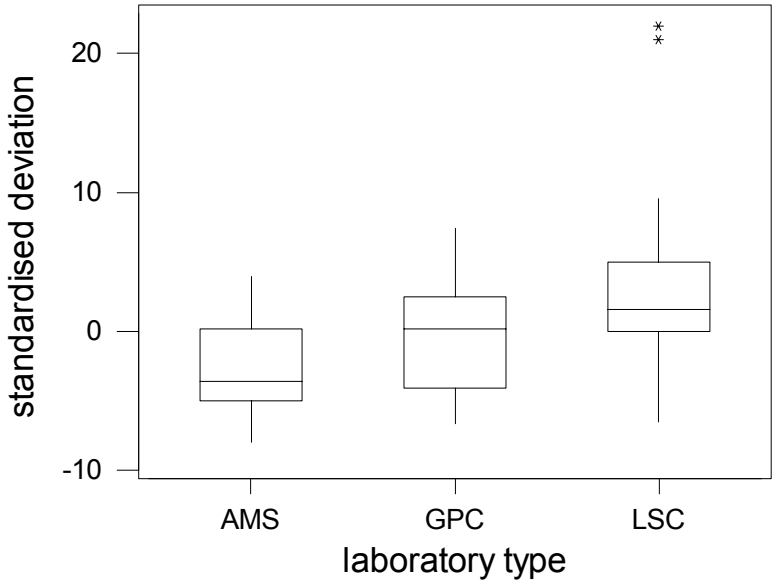

Figure 7.17 Distribution of standardized deviations for B

For Samples A and B, the calculations have been performed in pMC. We can see in Figures 7.16 and 7.17 that the distribution of results is skewed towards positive values, indicating that the laboratories reported results higher than the consensus value.

7.4.11 Effects of Other Laboratory Factors

It is of interest to explore the deviations from consensus values and to consider which factors, if any, can explain this variation. We have used the "initial" consensus values for this analysis and have not used Samples A and B. The consensus values were also all expressed in pMC to facilitate a global analysis over all the sample materials. We first consider the laboratory throughput.

The are 4 levels for the "number of analyses performed":

- *1* indicates <100 analyses done per yr by that laboratory;
- *2* indicates 100–200;
- *3* indicates 200–500;
- *4* indicates >500.

Table 7.4 Descriptive statistics for the standardized deviation by number of analyses

Nr of analyses	N	Mean	Median	StDev	Q1	Q3	Min	Max
1	109	–0.366	–0.163	4.044	–1.753	0.635	–18.15	20.25
2	266	0.753	0.380	5.156	–0.943	2.092	–15.01	49.94
3	118	–0.645	–0.040	3.770	–1.429	1.089	–19.75	11.00
4	384	–0.060	–0.202	2.537	–1.341	0.869	–8.00	12.30
Unknown	115	0.540	0.261	4.103	–0.967	1.556	–11.59	22.35

From the table, there are clearly some rather extreme values, but the IQR (Q1 to Q3) lies comfortably in the –2 to +2 range.

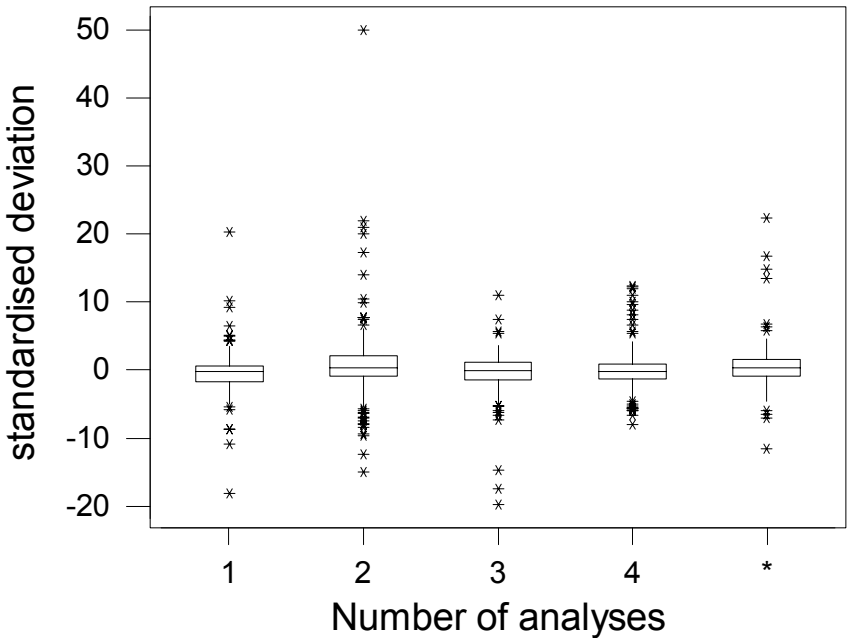

Figure 7.18 Distribution of standardized deviations by number of laboratory analyses

The results are highly skewed with many outliers. For further analysis, a statistical criteria can be used when an outlier in standardized deviation terms is greater than 4 or less than –4. The resultant numbers of values omitted are shown below in Table 7.5 by the laboratory type and by the modern standard.

Table 7.5a Number of results omitted by laboratory type

Laboratory type	Number omitted	% of results
AMS	40	19.4
GPC	42	20.3
LSC	124	60.2
All	**206**	**100**

Table 7.5b Number of results omitted by modern standard

Modern standard	Number omitted	% of results
ANU Sucr	20	10.3
Benzene	25	12.9
NBS OXI	52	26.9
NBS OXI/OXII	5	2.6
NBS OXII	66	34.2
Other	25	12.9
All	**193**	**100**

From the tables, it is clear that the majority of results omitted under this criterion are from LSC laboratories and that omission of results is more evenly distributed over the modern standard.

With the removal of the outliers, the distribution of results is more symmetrical.

Table 7.6 Descriptive statistics: outliers omitted

Analyses	Number of results	Mean	Median	Min	Max
1	87	–0.227	–0.101	–3.59	3.41
2	210	0.334	0.349	–3.93	3.86
3	103	–0.071	0.100	–3.94	3.61
4	350	–0.1073	–0.1626	–3.8	3.93
Unknown	99	0.116	0.231	–3.86	3.72

A formal analysis of the "laboratory throughput" is shown in Table 7.7 below.

Table 7.7 Effect of number of analyses

```
Source      DF         SS         MS         F         P
Analyses     3      32.38      10.79      4.18     0.006
Error      746    1925.88       2.58
Total      749    1958.26

                                    Individual 95% CIs For Mean
                                    Based on Pooled StDev
Level        N      Mean     StDev  -------+---------+---------+---------
1           87    -0.227     1.610    (---------*--------)
2          210     0.334     1.687                      (------*-----)
3          103    -0.071     1.680         (--------*--------)
4          350    -0.107     1.533            (----*----)
                                    -------+---------+---------+---------
Pooled StDev =     1.607               -0.35      0.00      0.35
```

Table 7.7 shows that there is a statistically significant difference in the average standardized deviation between the different categories of laboratory throughput.

However, we need to also consider that the number of analyses is very strongly related to laboratory type, in that AMS laboratories, in general, tend to have the highest throughput. Therefore, a further analysis, including both laboratory type and throughput, was carried out. The means of the standardized deviations are shown in Table 7.9, cross-classified by both laboratory type and throughput and the formal analysis is summarized in Table 7.8.

Table 7.8 Effect of laboratory type and number of analyses

```
Source      DF      Seq SS      Adj SS      Adj MS      F      P
Technique    2      68.831      69.824      34.912   13.99  0.000
Analyses     3      33.371      33.371      11.124    4.46  0.004
Error      744    1856.056    1856.056       2.495
Total      749    1958.258
```

The formal analysis showed that both the laboratory throughput and laboratory type are significant factors and affect the mean of the standardized deviations as shown in Table 7.9.

Table 7.9 Mean standardized deviation by type and number of analyses

	Nr of analyses				
Laboratory type	1	2	3	4	**All**
AMS	—	–0.7809	–0.3706	–0.3230	**–0.3438**
GPC	1.1584	–0.0591	0.4788	0.5738	**0.2695**
LSC	–0.3293	0.6259	–0.2347	0.5772	**0.2683**
All	–0.2267	0.3338	–0.0711	–0.1073	**0.0073**

For each sample and laboratory type, the average standardized deviation can be calculated for all samples. The results are shown in the table and figure below.

Table 7.10 Average standardized deviation for each sample by laboratory type

	AB	C	DF	E	GJ	H	I	**All**
AMS	–0.917	–0.048	–0.355	–0.293	–0.483	0.216	–0.115	**–0.311**
GPC	0.67	–0.131	0.442	0.073	0.301	0.318	0.482	**0.319**
LSC	0.567	0.551	–0.062	0.45	0.182	0.116	–0.034	**0.209**
All	0.065	0.184	–0.077	0.020	–0.078	0.196	0.038	**0.020**

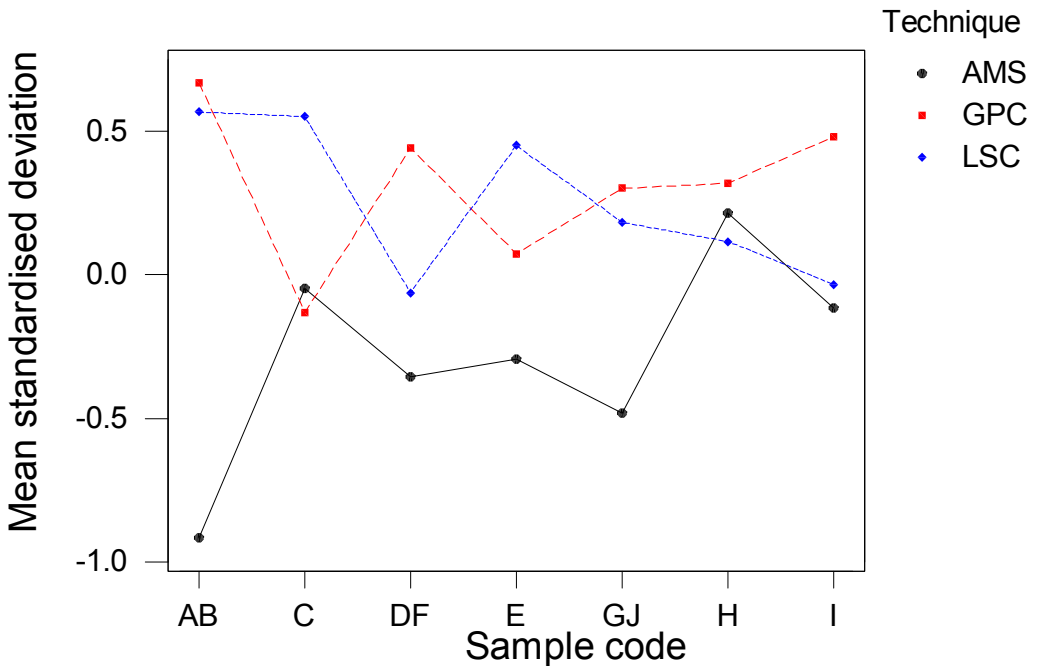

Figure 7.19 Mean standardized deviation by sample and laboratory type

The results for the 3 laboratory types are broadly similar (with the exception of AB, see Section 6) after the omission of outliers and all are generally acceptable (lying in a range of –1 to +1).

7.5 EVALUATION OF LABORATORY ACCURACY

Accepting the consensus values as, in some sense, the true age/activity for each material, we can evaluate the average laboratory difference from the consensus profile. The model used assumes that for a given laboratory there is a potential systematic offset from the consensus profile, which we can estimate, α, see Equation 1. These estimates are summarized in Table 7.11 and shown in Table 7.12.

$$\alpha = (\Sigma[x_i - \mu_i]^2 / s_i^2)\Sigma(1/s_i^2) \quad (1)$$

A summary of the results in Table 7.11. In the 2nd row outliers, offsets >2 or offsets <–2 are excluded.

Table 7.11 Summary of offset (pMC) for laboratories

Variable	N	Mean	Median	StDev	Min	Max	Q1	Q3
Offset	92	0.089	–0.010	1.403	–4.5	5.8	–0.3	0.3
Outliers excluded	85	–0.0005	–0.010	0.664	–1.3	1.8	–0.2	0.2

In summary, of the 90 labs for which an uncertainty estimate on the offset could be calculated, 59 were shown to have no offset. The distribution of offsets is shown in Figure 7.20.

Table 7.12 Laboratory offsets in pMC

Lab nr	Number of results	Lower limit on offset	Offset	Upper limit on offset
1	16	–0.24	–0.09	0.05
2	10	–0.19	–0.09	0.02
3	5	–0.71	–0.11	0.48
4	6	–0.77	–0.33	0.11
5	12	1.43	1.79	2.15
6	8	0.12	0.22	0.32
7	8	–0.02	0.29	0.61
8	8	–0.05	0.07	0.20
9	8	–0.21	0.22	0.64
10	6	1.97	2.91	3.86
11	12	–0.75	–0.02	0.72
12	8	–0.66	–0.27	0.12
13	8	–1.56	–0.87	–0.17
14	1	—	–0.50	—
15	11	–0.21	0.01	0.24
16	7	–3.45	–1.16	1.13
17	9	–1.49	–0.91	–0.34
18	8	–1.09	–0.37	0.36
19	8	–0.75	–0.11	0.53
20	9	–0.73	–0.35	0.04
21	8	–0.18	1.55	3.27
22	4	–1.08	–0.16	0.77
23	8	–1.58	–0.91	–0.25
24	7	–0.08	0.21	0.50
25	8	–0.18	0.03	0.23
26	6	1.50	5.81	10.13
27	8	–0.27	–0.11	0.05
28	6	–4.04	–1.27	1.50
29	8	–0.68	–0.40	–0.11
30	8	0.13	0.42	0.70
31	8	–0.21	–0.03	0.15
32	8	–1.17	–0.51	0.15
33	8	0.00	0.47	0.94
34	8	0.07	0.11	0.14
35	7	–1.76	–1.33	–0.91
36	10	–0.09	–0.01	0.07
37	13	–0.17	–0.07	0.04

Table 7.12 Laboratory offsets in pMC *(Continued)*

Lab nr	Number of results	Lower limit on offset	Offset	Upper limit on offset
38	9	0.02	0.12	0.22
39	8	–2.52	–1.33	–0.14
40	7	0.02	0.16	0.29
41	10	–0.08	0.07	0.21
42	6	0.07	1.69	3.31
43	8	–1.91	–1.06	–0.20
44	8	0.38	1.82	3.25
45	3	–3.14	–0.16	2.82
46	8	–0.28	–0.11	0.06
47	8	–0.65	–0.32	0.00
48	8	–0.20	0.07	0.35
49	22	–0.17	–0.07	0.03
50	16	–0.09	–0.02	0.05
51	28	–0.03	0.11	0.25
52	8	–0.28	–0.06	0.15
53	8	–11.05	–4.45	2.15
54	8	–0.05	0.30	0.65
55	8	–0.37	–0.12	0.12
56	6	–3.63	–2.56	–1.50
57	7	0.10	0.69	1.28
58	8	–0.40	–0.15	0.11
59	8	–2.26	–0.67	0.92
60	8	–0.29	–0.09	0.12
61	7	–0.55	–0.23	0.09
62	6	0.04	0.38	0.72
63	2	–8.15	–1.01	6.13
64	8	–0.09	0.08	0.26
65	8	0.05	0.13	0.22
66	8	0.30	0.48	0.65
67	5	–2.99	–1.05	0.88
68	8	–1.82	–0.94	–0.05
69	7	–7.31	–4.14	–0.97
70	16	1.49	4.85	8.22
71	7	0.12	0.97	1.81
72	10	–0.17	0.15	0.47
73	8	–0.15	0.01	0.17
74	11	–0.01	0.13	0.27
75	7	–0.25	0.36	0.98
76	8	–0.06	0.46	0.97
77	10	0.02	0.22	0.42
78	7	–3.68	0.10	3.89
79	8	–0.17	–0.05	0.06
80	1	—	5.80	—
81	8	–3.24	–1.01	1.22
82	8	–0.54	–0.28	–0.01

Table 7.12 Laboratory offsets in pMC *(Continued)*

Lab nr	Number of results	Lower limit on offset	Offset	Upper limit on offset
83	8	–0.10	0.01	0.12
84	20	–0.24	–0.09	0.06
85	8	0.34	0.43	0.51
86	8	–0.07	0.22	0.52
87	8	0.16	0.40	0.65
88	18	–0.17	–0.01	0.15
89	8	–0.53	0.45	1.42
90	12	0.94	1.55	2.17
91	8	–0.09	0.05	0.18
92	3	1.07	1.77	2.46

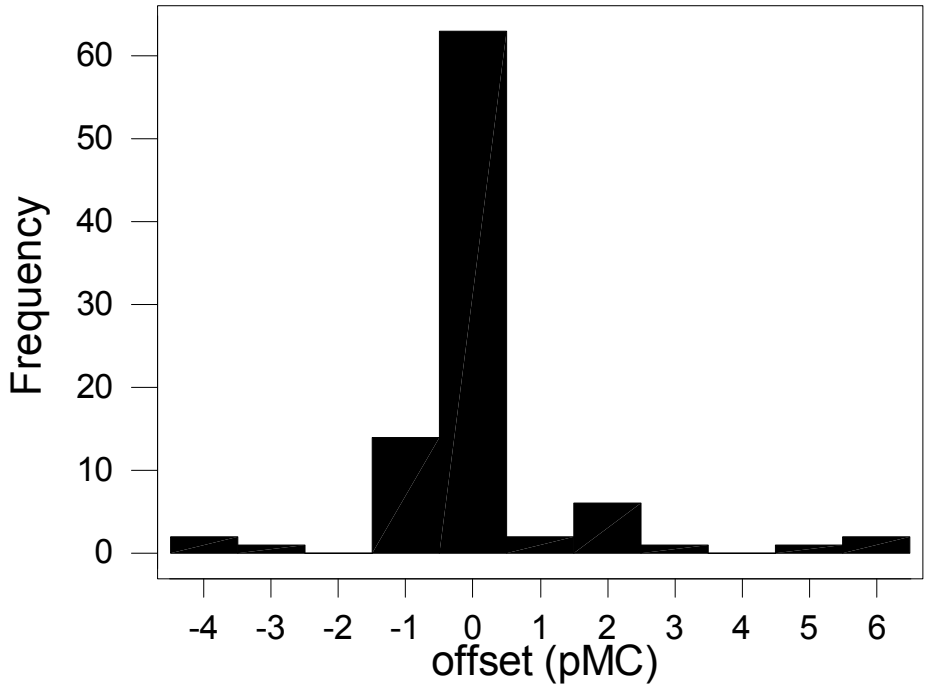

Figure 7.20 Distribution of laboratory offset relative to consensus values

Another possible calculation of offset can be based on the dendro-dated samples, of which 3 were included specifically for this purpose. Assuming a known age for these samples (based on the master chronology), an offset for each laboratory can then be estimated.

7.5.1 Offset Relative to the Dendro-Dated Wood Samples (yr BP)

A total of 4 dendro-dated wood samples were included in the list of core samples. They were Samples D and F (duplicates) from the Belfast master chronology, dendro-dated to 3200–3239 BC. Sample I (also from the Belfast master chronology), dendro-dated to 3299–3257 BC. Sample H was from the German oak chronology and was dendro-dated to 313–294 BC. A simple, exploratory summary of the findings and their comparison with the master calibration results is described in the following.

FIRI Samples D and F

Dendro-dated to 3239–3200 BC, this sample is linked to 4 samples on the master chronology. The average of the ^{14}C ages gives a "true" age of 4495 BP.

Table 7.13 Linked master calibration samples

Decadal midpoint	^{14}C age (1 σ)
3205	4528 ± 18
3215	4497 ± 11
3225	4495 ± 18
3235	4461 ± 18

FIRI Sample I

Dendro-dated to 3299–3257 BC, this sample is linked to 5 samples on the master chronology.

Table 7.14 Linked master calibration samples

Decadal midpoint	^{14}C age (1 σ)
3255	4455 ± 18
3265	4486 ± 18
3275	4480 ± 18
3285	4469 ± 18
3295	4468 ± 18

An average of the ^{14}C ages gives a "true" age of 4471 BP.

FIRI Sample H

Dendro-dated to 313–294 BC, this sample links to 3 samples on the master chronology.

Table 7.15 Linked master calibration samples

Decadal midpoint	^{14}C age (1 σ)
315	2210 ± 25
305	2211 ± 25
295	2225 ± 18

An average of the ^{14}C ages gives a "true" age of 2215 BP.

Similarly, using the master chronology ^{14}C ages as the "true" age for each laboratory, it is possible to estimate the systematic offset (if any) relative to these "true ages." However, it should be pointed out that, in fact, the consensus values for these samples are only slightly different from those extracted from the master calibration curve (4495 versus 4508 yr BP for DF, 4471 versus 4485 yr BP for I, and 2215 versus 2232 yr BP for Sample H).

Summarizing the offsets, we have:

Table 7.16 Offset in yr BP from the master ^{14}C ages

	N	Mean	Median	Min	Max	Q1	Q3	StDev
Offset	90	16.8	17.0	–642	414	–22	74	140.0
Outliers excluded	81	27	17	–218	209	–17	72	81

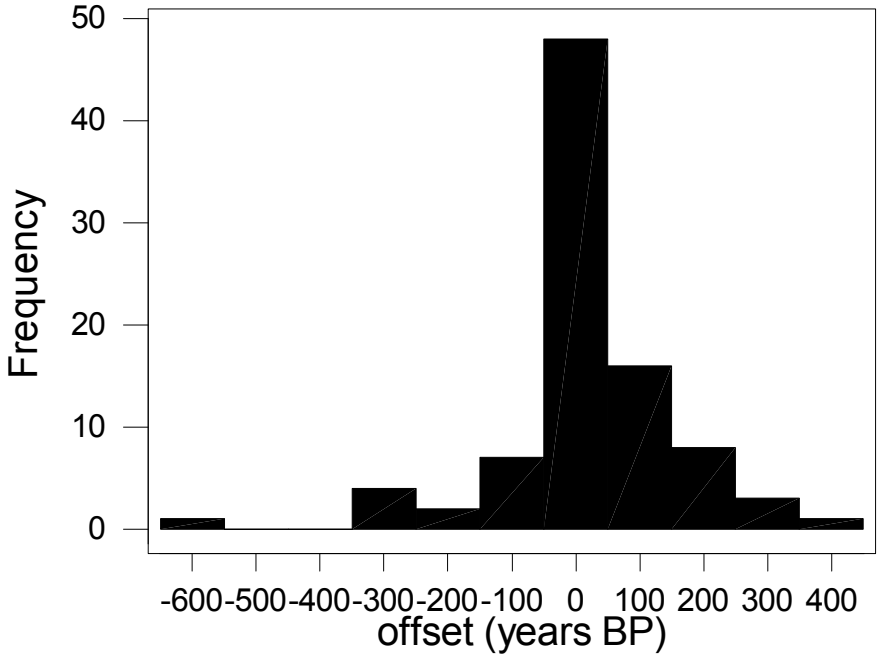

Figure 7.21 Distribution of offsets relative to the dendro-dated samples

Table 7.17 Lab offset (yr BP), based only on the dendro-dated samples (DF, I, H)

Lab nr	Number of results	Lower limit on offset	Offset	Upper limit on offset
1	8	48.83	80.86	112.89
2	6	6.53	37.24	67.96
3	2	–150.51	137.00	424.51
4	4	–31.83	89.87	211.56
5	7	–306.67	–218.46	–130.24
6	4	–46.34	–0.97	44.39
7	4	–135.38	–66.25	2.88
8	4	–46.65	–6.50	33.65
9	4	12.43	70.53	128.63
10	4	–420.29	–345.04	–269.79
11	7	–211.17	–62.76	85.65
12	4	–67.22	43.50	154.22
13	3	–84.91	199.67	484.24
15	7	–43.11	–6.04	31.03
16	3	94.17	203.95	313.73
17	5	–15.97	18.35	52.67
18	4	–140.97	12.64	166.25
19	4	1.11	62.13	123.14
20	5	73.72	109.46	145.21
21	4	–793.37	–327.44	138.50
22	3	–323.13	16.58	356.28

Table 7.17 Lab offset (yr BP), based only on the dendro-dated samples (DF, I, H) *(Continued)*

Lab nr	Number of results	Lower limit on offset	Offset	Upper limit on offset
23	4	75.46	209.49	343.52
24	4	–74.40	–14.28	45.84
25	4	–7.91	56.00	119.91
26	4	–1144.10	–642.19	–140.28
27	4	10.07	37.73	65.39
28	3	–626.91	–63.44	500.02
29	4	–39.33	75.73	190.79
30	4	–174.60	1.00	176.60
31	4	–70.59	8.44	87.46
32	4	–77.49	72.58	222.66
33	4	–147.00	–60.64	25.72
34	4	–94.50	–33.88	26.75
35	4	–62.96	104.21	271.38
36	6	–7.38	12.68	32.74
37	6	6.09	35.28	64.47
38	5	–41.22	–18.21	4.80
39	4	209.19	258.46	307.74
40	4	–51.43	–7.69	36.05
41	6	–20.89	10.32	41.54
42	4	–603.20	–321.25	–39.31
43	4	64.38	205.79	347.20
44	4	–257.58	–129.90	–2.21
45	2	–97.79	–35.86	26.06
46	4	–13.35	36.63	86.62
47	4	–31.27	125.28	281.82
48	4	–67.40	34.49	136.37
49	11	–11.40	17.41	46.23
50	8	–9.92	21.45	52.81
51	16	3.39	31.97	60.54
52	4	35.24	77.15	119.06
53	4	–1732.51	56.91	1846.32
54	3	–94.28	–20.99	52.30
55	4	–68.55	4.44	77.44
56	4	56.51	262.05	467.58
57	4	–226.99	–106.32	14.35
58	4	–29.62	42.03	113.68
59	4	–215.48	276.36	768.20
60	4	–36.92	42.28	121.49
61	4	–51.10	21.36	93.81
62	1	—	29.00	—
63	2	–873.23	160.00	1193.23
64	4	47.51	73.50	99.49
65	4	–39.40	–16.57	6.27
66	4	–50.75	–27.75	–4.75
67	4	–255.02	163.26	581.53

Table 7.17 Lab offset (yr BP), based only on the dendro-dated samples (DF, I, H) *(Continued)*

Lab nr	Number of results	Lower limit on offset	Offset	Upper limit on offset
68	4	169.60	192.55	215.50
69	4	26.45	414.43	802.42
70	8	–143.13	118.80	380.73
71	4	–245.85	–105.83	34.19
72	5	–87.53	20.01	127.55
73	4	–31.99	–1.17	29.65
74	7	–5.51	15.91	37.33
75	4	–189.71	–15.24	159.22
76	4	–36.24	–1.50	33.24
77	7	–60.72	–30.97	–1.22
78	4	–733.60	–27.86	677.87
79	4	–2.26	31.22	64.70
81	4	–116.91	182.93	482.76
82	4	28.77	102.39	176.01
83	4	–27.16	0.37	27.90
84	8	–37.68	–0.83	36.02
85	4	–55.00	–35.27	–15.53
86	4	–131.05	–26.97	77.12
87	4	–100.49	–49.26	1.98
88	9	–49.90	–16.60	16.70
89	4	–28.03	44.21	116.45
90	8	–239.84	–153.61	–67.37
91	4	–48.51	–7.57	33.36
92	1	—	–251.00	—

7.6 CONCLUSIONS

Consensus values (and their error) for the FIRI samples have been derived. Concerns remain over the consensus value for the Kauri wood sample due to the reporting difficulties for this sample. The sensitivity of the results to different calculation algorithms has been shown to be small (with the exception of the Kauri wood). Consensus values for the dendro-dated wood samples are very close to the values derived from the master calibration curve, which adds confidence in the results derived.

When considering laboratory performance, we have evaluated standardized deviations from the consensus values and have shown that these can be linked to the laboratory type. Calculation of the offsets has also shown that more than half the laboratories have no systematic offset, and that those laboratories that have a systematic offset, generally have small offsets (with only a few exceptions). Laboratories received this information for their consideration and, thus, were able to explore any causes, and then instigate any necessary corrective actions.

SECTION 7: CHARACTERIZATION OF THE REFERENCE MATERIALS BY CONSENSUS VALUES

7.1 CALCULATION OF CONSENSUS VALUES

Each material needs to be characterized by estimating its activity, which creates a reference value for each material. This value then can be considered as the "known" activity of the material and future analyses can be compared to this to quantify the accuracy of the measurement. In this way, the materials remain useful for laboratory quality assurance.

The procedure used in the calculation of the consensus value comes from Rozanski et al. (1992) and is an iterative one. It is described below.

There are 3 stages.

- **Stage 1**: Outlying results are removed if they are greater than 3 interquartile ranges from the nearest of either the lower or upper quartiles. This occurs when a result is either greater than Q3 + 3(Q3–Q1) or less than Q1 – 3(Q3–Q1), where *Q1* and *Q3* are the lower and upper quartiles, respectively. Then, the preliminary consensus value is calculated as the median (*m*) of the remaining results.
- **Stage 2**: Remove results that are at least twice their quoted error (σ) from the preliminary consensus value. That is, only keep $|x–m| / \sigma < 2$, where *x* is the result, *m* the preliminary consensus value, and σ the quoted error.
- **Stage 3**: Calculate the final consensus as a weighted mean of the remaining results, using their σ^2 values as the weights.

7.1 Remarks on the Procedure

For FIRI Samples A and B (in yr), this approach is not very appropriate given that many laboratories did not quote finite ages. For these samples, an alternative approach was used based on the reliability analysis (see Section 6).

It should also be noted that averages are rather sensitive to extreme data values, which is why the outliers are removed in Step 1.

The approach has the advantage that an estimated error can be calculated for the consensus value (which will usually be very small since there are a large number of results).

7.2 INITIAL CONSENSUS VALUES

Consensus values are reported in Table 7.1, based on the Rozanski et al. procedure.

Table 7.1 Preliminary consensus values

FIRI sample	Weighted mean (1 σ)	AMS	GPC	LSC
C	18,173 (10.5)	18,183 (13)	18,229 (28)	18,140 (25)
DF	4508 (3)	4519 (4)	4484 (5)	4507 (6)
E	11,778 (7)	11,805 (9)	11,738 (19)	11,707 (17)
GJ	110.69 (0.04)	110.52 (0.05)	110.85 (0.07)	110.82 (0.08)
H	2232 (5)	2238 (6)	2198 (9)	2233 (9)
I	4485 (5)	4483 (7)	4456 (10)	4499 (11)

It should be noted that the results for Samples A and B are not included in this table, since this procedure only is possible using results where a quoted error is given. However, the results for Samples A and B will be returned to later in this section, when the analysis of the pMC is completed.

Figures 7.1 to 7.7 (Section 7 appendix, p 269–275) show the distribution of the laboratory results around these consensus values. They include the laboratory-quoted errors. In such figures, we can see how closely the results from the different laboratories agree (accounting for their quoted errors). The consensus values are also marked. In Step 2, laboratories quoting small errors will be excluded, unless they lie close to the consensus value, while laboratories quoting large errors will be included in Step 3. However, in Step 3, results with large errors will be down-weighted in the calculation and so will not have a large impact on the final result.

Therefore, there is an issue of how robust the initial consensus value is in Step 1, and how important its definition is on the final consensus value. Therefore, we consider variants of this original method, which at Step 1 exclude not simply extreme age/activity values, but also results with large quoted errors.

7.3 THE EFFECT OF SCREENING OUT RESULTS WITH LARGE QUOTED ERRORS IN CONSENSUS CALCULATIONS

7.3.1 σ Method 1

This method is the same as the original one, except that, between Stage 1 and Stage 2, results with a quoted uncertainty greater than a certain cut-off point are rejected.

7.3.2 σ Method 2

This method is the same as the σ Method 1, but this time, results with a quoted uncertainty greater than a certain cut-off point are rejected *before* Stage 1.

7.3.3 Choice of σ "Cut-off" Points

The choice of the cut-off points is subjective. However, from the histograms showing the distribution of σ and expert opinion, the cut-off points shown in Table 7.2 were used for both methods. Because of the subjectivity of the decisions, 2 (or, in AB's case, 3) different values for the cut-off points were chosen for each sample.

Table 7.2 Cut-off points used for the different samples

Sample	Cut-off points used			Units
Kauri wood, AB	0.3	0.15	0.1	pMC
Turbidite, C	200	150	—	yr BP
Belfast dendro-wood, DF	100	50	—	yr BP
Humic acid, E	150	100	—	yr BP
Barley mash, GJ	1	0.6	—	pMC
Hohenheim dendro-wood, H	100	50	—	yr BP
Belfast cellulose, I	100	50	—	yr BP

7.3.4 Results

From the results in Table 7.3, it can be seen that the various methods for calculating the consensus make very little difference to all but the AB sample (ranges of only 2.3 yr BP for Sample C, 1.4 yr BP for DF, 11.7 yr BP for Sample E, 0.06 pMC for GJ, 0.7 yr BP for H, and 10.6 yr BP for I).

For Sample AB, there is little difference within the σ Method 1 (a range of only 0.075 pMC), but there is for the σ Method 2, with the more restrictive cut-off points. When results with a σ greater than 0.1 pMC are screened, the consensus value becomes 0.2 pMC, less than two-thirds the value it is under the original method.

Table 7.3 Consensus values under the different methods

Sample		Original methods	Method 1			Method 2		
AB	σ cut-off	None	0.3	0.15	0.1	0.3	0.15	0.1
(pMC)	Consensus	0.330	0.330 (0.01)	0.327 (0.01)	0.325 (0.01)	0.324 (0.01)	0.251 (0.01)	0.203 (0.01)
C	σ cut-off	None	200	150	—	200	150	—
(yr BP)	Consensus	18,175.5 (10.5)	18,176.5 (9.7)	18,177.8 (9.3)	—	18,176.5 (9.7)	18,177.8 (9.3)	—
DF	σ cut-off	None	100	50	—	100	50	—
(yr BP)	Consensus	4508.3 (3)	4508.2 (3)	4506.8 (3)	—	4508.2 (3)	4506.8 (3)	—
E	σ cut-off	None	150	100	—	150	100.00	—
(yr BP)	Consensus	11,779.9 (7)	11,781.2 (8)	11,781.7 (7.6)	—	11,791.6 (7.8)	11,791.2 (8)	—
GJ	σ cut-off	None	1	0.6	—	1	0.6	—
(pMC)	Consensus	110.69 (0.04)	110.69 (0.04)	110.72 (0.04)	—	110.69 (0.04)	110.75 (0.04)	—
H	σ cut-off	None	100	50	—	100	50	—
(yr BP)	Consensus	2232.5 (5)	2232.3 (4.7)	2233.0 (4.7)	—	2232.3 (4.7)	2233.0 (4.7)	—
I	σ cut-off	None	100	50	—	100	50	—
(yr BP)	Consensus	4484.9 (5)	4485.1 (5)	4482.1 (5)	—	4485.1 (5)	4474.5 (5.3)	—

7.3.5 Discussion

The alternative methods for calculating the consensus only lead to very small differences, except in the case of the Kauri wood sample, AB. Here, screening out results with σs larger than a cut-off point before using the original method, shifted the consensus by large amounts when the cut-off was small (from 0.33–0.20 pMC, when the cut-off was 0.1 pMC). Possible reasons for this change could stem from AB being a sample at, or near, the limits of detection for ^{14}C dating.

Since the Kauri wood's activity is so low, some results are given as background or non-finite. This occurs when the σ is large with respect to its result. Obviously, those laboratories that have a lower σ can give finite results for older samples. Because background and non-finite results are excluded from the consensus calculation, this could bias the calculations.

Also, it is possible that laboratories have reported pMC results for samples that should be considered background or non-finite. At present, these results are not screened out. Such an approach could be valuable in providing a more reliable estimate of the activity in the Kauri wood sample.

7.3.6 Conclusion

The consensus calculations are robust in the initial screening stages for all but the Kauri wood samples. For this sample, the consensus age has been calculated by a different method and reported in Section 6. For the pMC results, the consensus calculation has been carried out, but with a number

of caveats. In AB's case, a better value for the consensus may be achieved if any results that are too small with respect to their quoted errors are screened out. If this does not help, then we may be left with a large range for the consensus value of the Kauri wood sample's activity. We recommend that the results in Table 7.1 and Table 6.20 be used as consensus values for the FIRI samples.

7.4 DEVIATIONS FROM CONSENSUS VALUES

We define the standardized deviation as the difference between the result and the consensus value, divided by the quoted uncertainty on the result. Using this summary, we can explore the distribution of laboratory performance. Ideally, we might expect a standardized deviation to lie between +2 and –2. Values greatly exceeding 2 or –2 indicate either a large absolute difference between the result and the consensus value or a "large" difference relative to the quoted error. This makes them sensitive indicators of general laboratory performance. The standardized deviations for each sample (except AB) can then be investigated for the effects of different laboratory factors.

7.4.1 Effect of Laboratory Type for Sample C: Turbidite

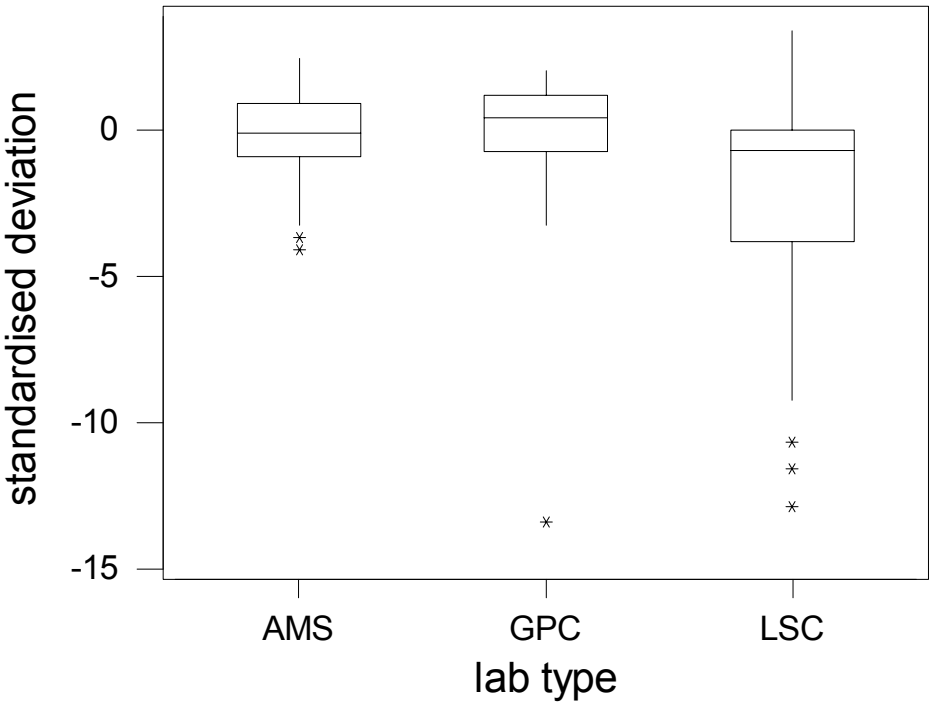

Figure 7.8 Distribution of standardized deviation for Sample C

We can see that AMS and GPC results appear to show a broadly similar distribution. For LSC results, the distribution is more widely scattered. Each laboratory type has a number of extreme values and this is more pronounced for the LSC set of results.

7.4.2 Effect of Laboratory Type for Sample D: Belfast Wood

A similar pattern is apparent; the median value lies close to 0, but there are a number of extreme values, typically reported by LSC laboratories.

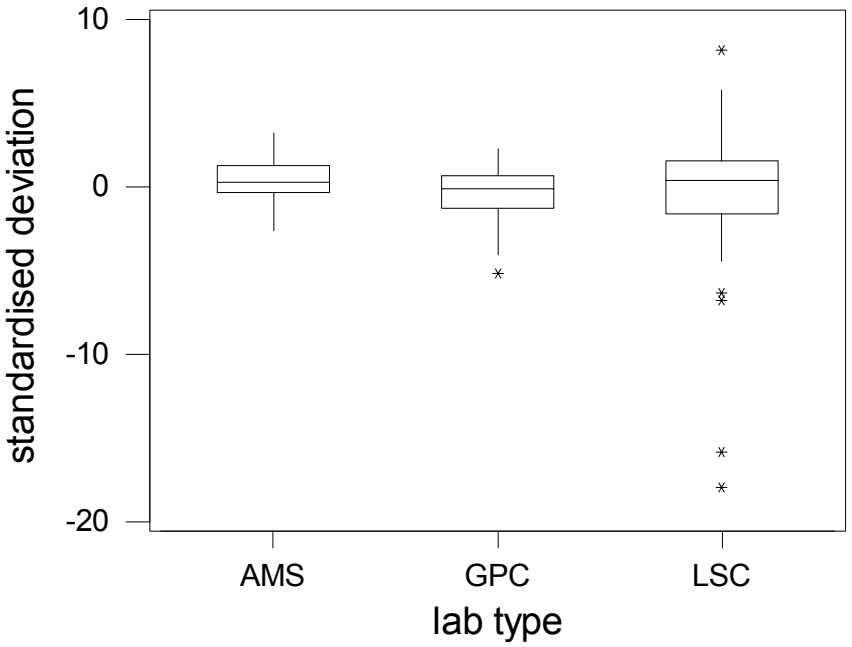

Figure 7.9 Distribution of standardized deviation for Sample D

7.4.3 Effect of Laboratory Type for Sample E: Humic Acid

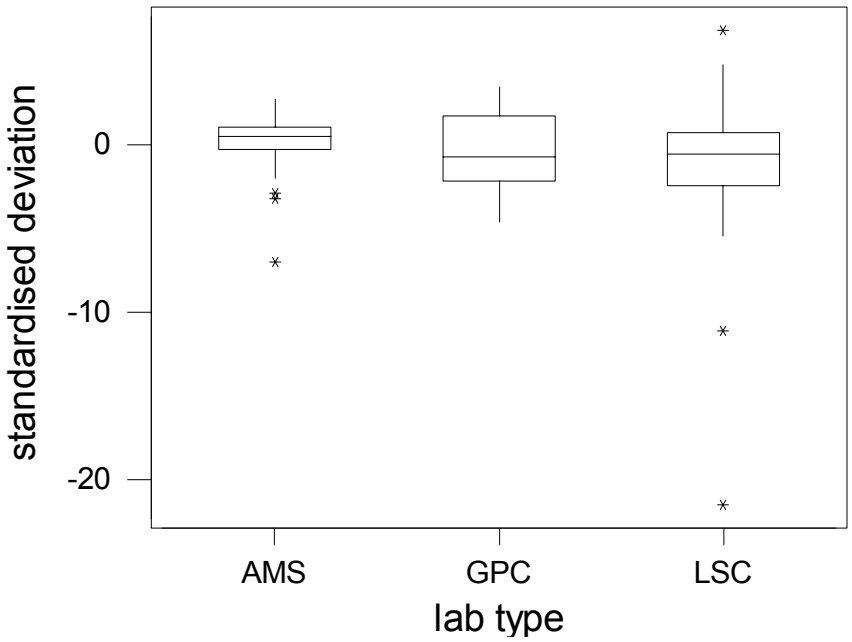

Figure 7.10 Distribution of standardized deviation for Sample E

A similar distributional pattern is apparent; the median value lies close to 0, but there are a small number of extreme values.

7.4.4 Effect of Laboratory Type for Sample F: Belfast Wood

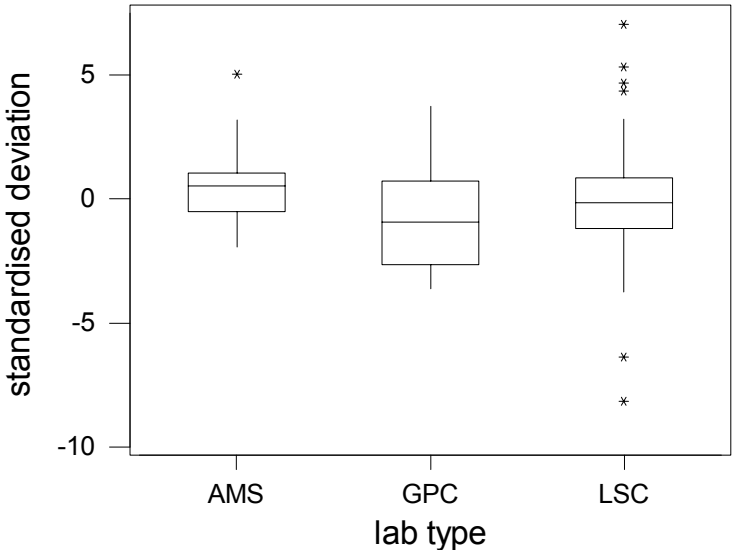

Figure 7.11 Distribution of standardized deviation for Sample F

The median value lies close to 0, but there are a number of extreme values, typically reported by LSC laboratories.

7.4.5 Effect of Laboratory Type for Sample G: Barley Mash

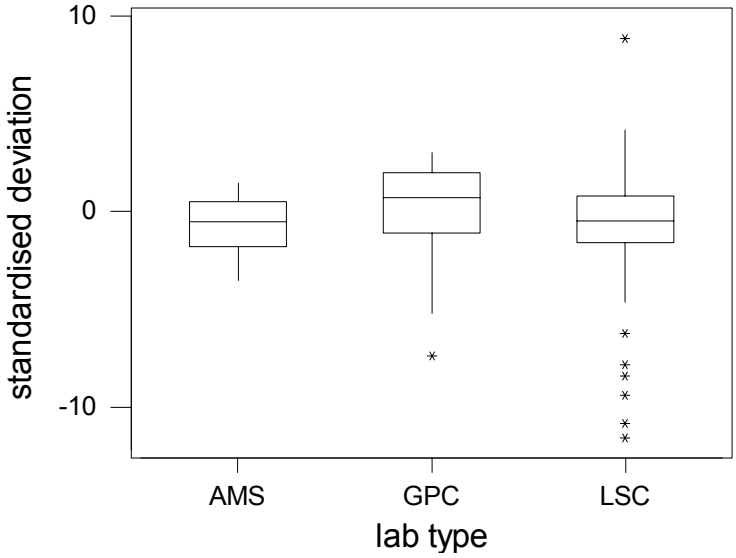

Figure 7.12 Distribution of standardized deviations for Sample G

The median value lies close to 0, but there are a number of extreme values, typically reported by LSC laboratories. Omitting these results would result in broadly similar distributions for the 3 laboratory types.

7.4.6 Effect of Laboratory Type for Sample H: Hohenheim Wood

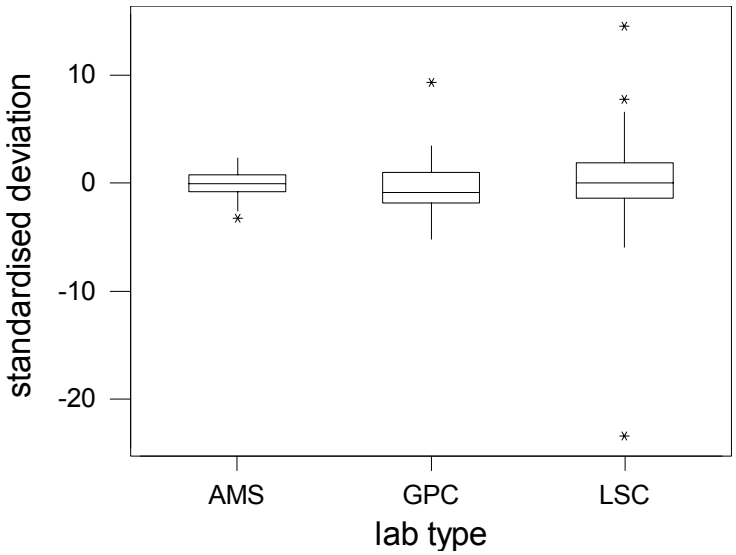

Figure 7.13 Distribution of standardized deviations for Sample H

A similar pattern is apparent. The median value lies close to 0, but there are a number of extreme values, typically reported by LSC laboratories.

7.4.7 Effect of Laboratory Type for Sample I: Belfast Cellulose

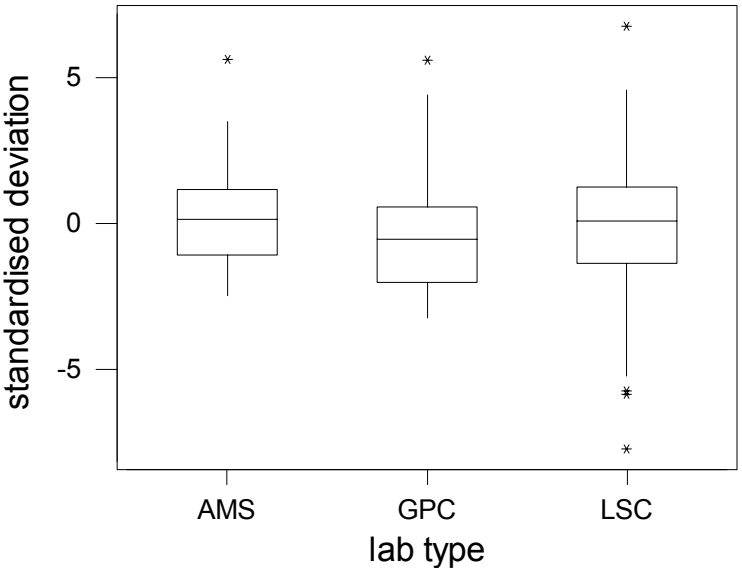

Figure 7.14 Distribution of standardized deviations for Sample I

The distribution of results is less wide for this sample. The median value lies close to 0, but there are a small number of extreme values, which are reported by LSC laboratories.

7.4.8 Effect of Laboratory Type for Sample J: Barley Mash

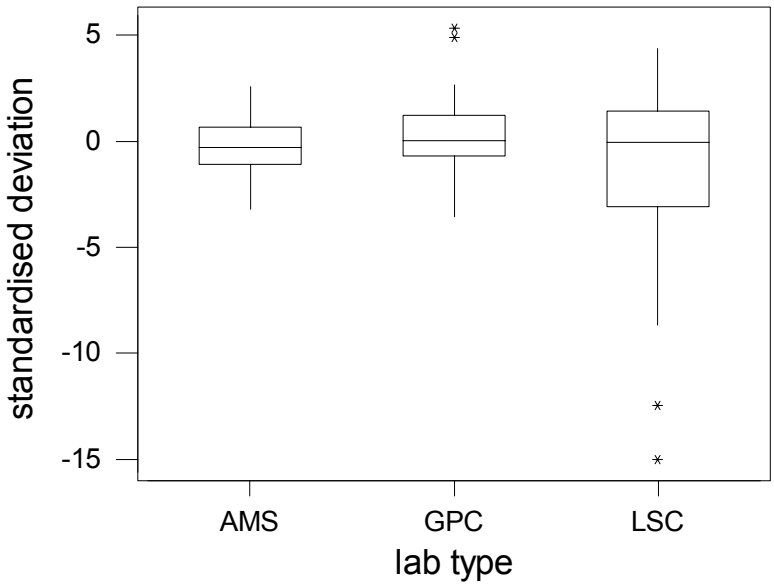

Figure 7.15 Distribution of standardized deviations for Sample J

A similar pattern is apparent, where the median value lies close to 0. The distribution of results is wider for LSC laboratories and there are several extreme values.

7.4.9 Effect of Laboratory Type for Sample A: Kauri Wood

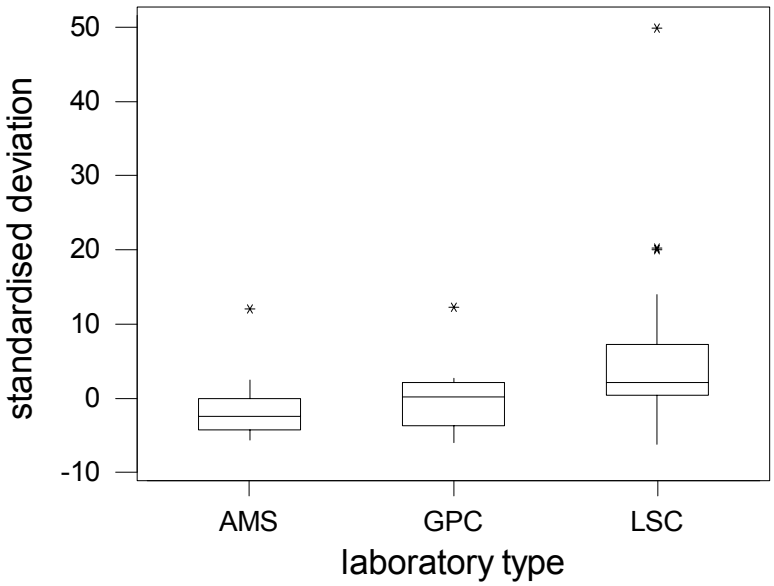

Figure 7.16 Distribution of standardized deviations for Sample A

7.4.10 Sample B: Kauri Wood

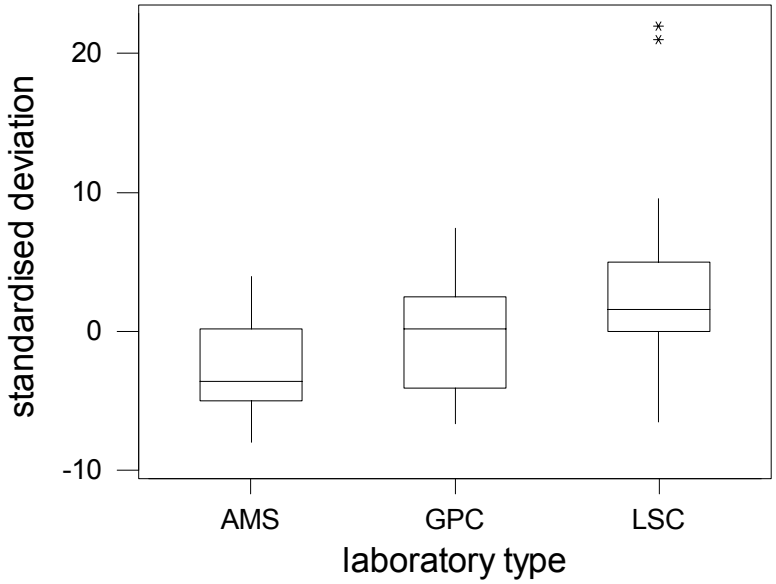

Figure 7.17 Distribution of standardized deviations for B

For Samples A and B, the calculations have been performed in pMC. We can see in Figures 7.16 and 7.17 that the distribution of results is skewed towards positive values, indicating that the laboratories reported results higher than the consensus value.

7.4.11 Effects of Other Laboratory Factors

It is of interest to explore the deviations from consensus values and to consider which factors, if any, can explain this variation. We have used the "initial" consensus values for this analysis and have not used Samples A and B. The consensus values were also all expressed in pMC to facilitate a global analysis over all the sample materials. We first consider the laboratory throughput.

The are 4 levels for the "number of analyses performed":

- *1* indicates <100 analyses done per yr by that laboratory;
- *2* indicates 100–200;
- *3* indicates 200–500;
- *4* indicates >500.

Table 7.4 Descriptive statistics for the standardized deviation by number of analyses

Nr of analyses	N	Mean	Median	StDev	Q1	Q3	Min	Max
1	109	–0.366	–0.163	4.044	–1.753	0.635	–18.15	20.25
2	266	0.753	0.380	5.156	–0.943	2.092	–15.01	49.94
3	118	–0.645	–0.040	3.770	–1.429	1.089	–19.75	11.00
4	384	–0.060	–0.202	2.537	–1.341	0.869	–8.00	12.30
Unknown	115	0.540	0.261	4.103	–0.967	1.556	–11.59	22.35

From the table, there are clearly some rather extreme values, but the IQR (Q1 to Q3) lies comfortably in the –2 to +2 range.

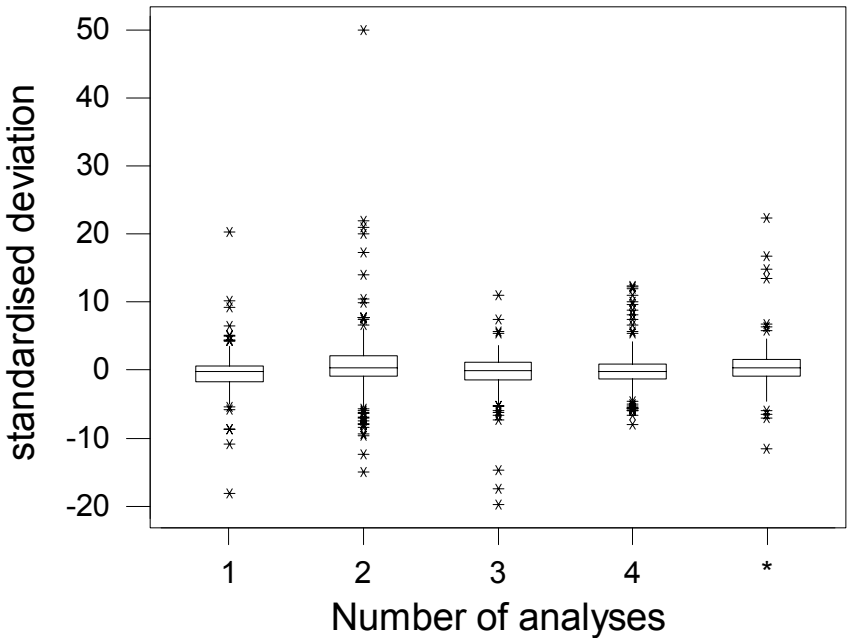

Figure 7.18 Distribution of standardized deviations by number of laboratory analyses

The results are highly skewed with many outliers. For further analysis, a statistical criteria can be used when an outlier in standardized deviation terms is greater than 4 or less than –4. The resultant numbers of values omitted are shown below in Table 7.5 by the laboratory type and by the modern standard.

Table 7.5a Number of results omitted by laboratory type

Laboratory type	Number omitted	% of results
AMS	40	19.4
GPC	42	20.3
LSC	124	60.2
All	**206**	**100**

Table 7.5b Number of results omitted by modern standard

Modern standard	Number omitted	% of results
ANU Sucr	20	10.3
Benzene	25	12.9
NBS OXI	52	26.9
NBS OXI/OXII	5	2.6
NBS OXII	66	34.2
Other	25	12.9
All	**193**	**100**

From the tables, it is clear that the majority of results omitted under this criterion are from LSC laboratories and that omission of results is more evenly distributed over the modern standard.

With the removal of the outliers, the distribution of results is more symmetrical.

Table 7.6 Descriptive statistics: outliers omitted

Analyses	Number of results	Mean	Median	Min	Max
1	87	–0.227	–0.101	–3.59	3.41
2	210	0.334	0.349	–3.93	3.86
3	103	–0.071	0.100	–3.94	3.61
4	350	–0.1073	–0.1626	–3.8	3.93
Unknown	99	0.116	0.231	–3.86	3.72

A formal analysis of the "laboratory throughput" is shown in Table 7.7 below.

Table 7.7 Effect of number of analyses

```
Source      DF         SS         MS         F         P
Analyses     3      32.38      10.79      4.18     0.006
Error      746    1925.88       2.58
Total      749    1958.26

                                    Individual 95% CIs For Mean
                                    Based on Pooled StDev
Level        N      Mean     StDev  -------+---------+---------+---------
1           87    -0.227     1.610    (---------*--------)
2          210     0.334     1.687                      (------*-----)
3          103    -0.071     1.680         (--------*--------)
4          350    -0.107     1.533            (----*----)
                                    -------+---------+---------+---------
Pooled StDev =     1.607               -0.35      0.00      0.35
```

Table 7.7 shows that there is a statistically significant difference in the average standardized deviation between the different categories of laboratory throughput.

However, we need to also consider that the number of analyses is very strongly related to laboratory type, in that AMS laboratories, in general, tend to have the highest throughput. Therefore, a further analysis, including both laboratory type and throughput, was carried out. The means of the standardized deviations are shown in Table 7.9, cross-classified by both laboratory type and throughput and the formal analysis is summarized in Table 7.8.

Table 7.8 Effect of laboratory type and number of analyses

```
Source      DF      Seq SS      Adj SS      Adj MS      F      P
Technique    2      68.831      69.824      34.912   13.99  0.000
Analyses     3      33.371      33.371      11.124    4.46  0.004
Error      744    1856.056    1856.056       2.495
Total      749    1958.258
```

The formal analysis showed that both the laboratory throughput and laboratory type are significant factors and affect the mean of the standardized deviations as shown in Table 7.9.

Table 7.9 Mean standardized deviation by type and number of analyses

	Nr of analyses				
Laboratory type	1	2	3	4	**All**
AMS	—	–0.7809	–0.3706	–0.3230	**–0.3438**
GPC	1.1584	–0.0591	0.4788	0.5738	**0.2695**
LSC	–0.3293	0.6259	–0.2347	0.5772	**0.2683**
All	–0.2267	0.3338	–0.0711	–0.1073	**0.0073**

For each sample and laboratory type, the average standardized deviation can be calculated for all samples. The results are shown in the table and figure below.

Table 7.10 Average standardized deviation for each sample by laboratory type

	AB	C	DF	E	GJ	H	I	**All**
AMS	–0.917	–0.048	–0.355	–0.293	–0.483	0.216	–0.115	**–0.311**
GPC	0.67	–0.131	0.442	0.073	0.301	0.318	0.482	**0.319**
LSC	0.567	0.551	–0.062	0.45	0.182	0.116	–0.034	**0.209**
All	0.065	0.184	–0.077	0.020	–0.078	0.196	0.038	**0.020**

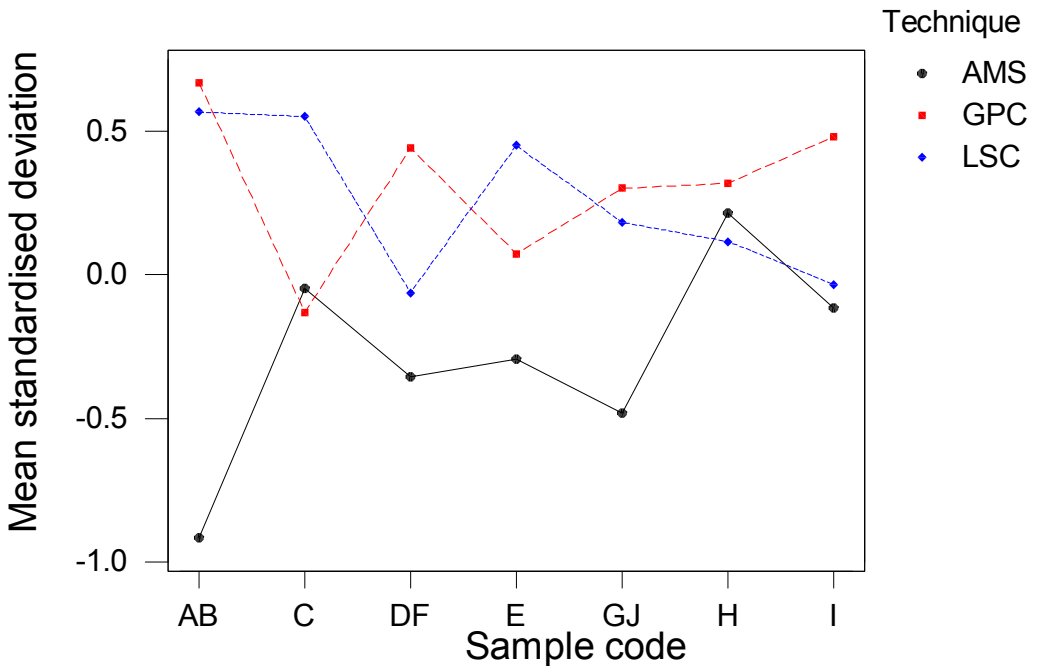

Figure 7.19 Mean standardized deviation by sample and laboratory type

The results for the 3 laboratory types are broadly similar (with the exception of AB, see Section 6) after the omission of outliers and all are generally acceptable (lying in a range of –1 to +1).

7.5 EVALUATION OF LABORATORY ACCURACY

Accepting the consensus values as, in some sense, the true age/activity for each material, we can evaluate the average laboratory difference from the consensus profile. The model used assumes that for a given laboratory there is a potential systematic offset from the consensus profile, which we can estimate, α, see Equation 1. These estimates are summarized in Table 7.11 and shown in Table 7.12.

$$\alpha = (\Sigma[x_i - \mu_i]^2 / s_i^2)\Sigma(1/s_i^2) \quad (1)$$

A summary of the results in Table 7.11. In the 2nd row outliers, offsets >2 or offsets <–2 are excluded.

Table 7.11 Summary of offset (pMC) for laboratories

Variable	N	Mean	Median	StDev	Min	Max	Q1	Q3
Offset	92	0.089	–0.010	1.403	–4.5	5.8	–0.3	0.3
Outliers excluded	85	–0.0005	–0.010	0.664	–1.3	1.8	–0.2	0.2

In summary, of the 90 labs for which an uncertainty estimate on the offset could be calculated, 59 were shown to have no offset. The distribution of offsets is shown in Figure 7.20.

Table 7.12 Laboratory offsets in pMC

Lab nr	Number of results	Lower limit on offset	Offset	Upper limit on offset
1	16	–0.24	–0.09	0.05
2	10	–0.19	–0.09	0.02
3	5	–0.71	–0.11	0.48
4	6	–0.77	–0.33	0.11
5	12	1.43	1.79	2.15
6	8	0.12	0.22	0.32
7	8	–0.02	0.29	0.61
8	8	–0.05	0.07	0.20
9	8	–0.21	0.22	0.64
10	6	1.97	2.91	3.86
11	12	–0.75	–0.02	0.72
12	8	–0.66	–0.27	0.12
13	8	–1.56	–0.87	–0.17
14	1	—	–0.50	—
15	11	–0.21	0.01	0.24
16	7	–3.45	–1.16	1.13
17	9	–1.49	–0.91	–0.34
18	8	–1.09	–0.37	0.36
19	8	–0.75	–0.11	0.53
20	9	–0.73	–0.35	0.04
21	8	–0.18	1.55	3.27
22	4	–1.08	–0.16	0.77
23	8	–1.58	–0.91	–0.25
24	7	–0.08	0.21	0.50
25	8	–0.18	0.03	0.23
26	6	1.50	5.81	10.13
27	8	–0.27	–0.11	0.05
28	6	–4.04	–1.27	1.50
29	8	–0.68	–0.40	–0.11
30	8	0.13	0.42	0.70
31	8	–0.21	–0.03	0.15
32	8	–1.17	–0.51	0.15
33	8	0.00	0.47	0.94
34	8	0.07	0.11	0.14
35	7	–1.76	–1.33	–0.91
36	10	–0.09	–0.01	0.07
37	13	–0.17	–0.07	0.04

Table 7.12 Laboratory offsets in pMC *(Continued)*

Lab nr	Number of results	Lower limit on offset	Offset	Upper limit on offset
38	9	0.02	0.12	0.22
39	8	–2.52	–1.33	–0.14
40	7	0.02	0.16	0.29
41	10	–0.08	0.07	0.21
42	6	0.07	1.69	3.31
43	8	–1.91	–1.06	–0.20
44	8	0.38	1.82	3.25
45	3	–3.14	–0.16	2.82
46	8	–0.28	–0.11	0.06
47	8	–0.65	–0.32	0.00
48	8	–0.20	0.07	0.35
49	22	–0.17	–0.07	0.03
50	16	–0.09	–0.02	0.05
51	28	–0.03	0.11	0.25
52	8	–0.28	–0.06	0.15
53	8	–11.05	–4.45	2.15
54	8	–0.05	0.30	0.65
55	8	–0.37	–0.12	0.12
56	6	–3.63	–2.56	–1.50
57	7	0.10	0.69	1.28
58	8	–0.40	–0.15	0.11
59	8	–2.26	–0.67	0.92
60	8	–0.29	–0.09	0.12
61	7	–0.55	–0.23	0.09
62	6	0.04	0.38	0.72
63	2	–8.15	–1.01	6.13
64	8	–0.09	0.08	0.26
65	8	0.05	0.13	0.22
66	8	0.30	0.48	0.65
67	5	–2.99	–1.05	0.88
68	8	–1.82	–0.94	–0.05
69	7	–7.31	–4.14	–0.97
70	16	1.49	4.85	8.22
71	7	0.12	0.97	1.81
72	10	–0.17	0.15	0.47
73	8	–0.15	0.01	0.17
74	11	–0.01	0.13	0.27
75	7	–0.25	0.36	0.98
76	8	–0.06	0.46	0.97
77	10	0.02	0.22	0.42
78	7	–3.68	0.10	3.89
79	8	–0.17	–0.05	0.06
80	1	—	5.80	—
81	8	–3.24	–1.01	1.22
82	8	–0.54	–0.28	–0.01

Table 7.12 Laboratory offsets in pMC *(Continued)*

Lab nr	Number of results	Lower limit on offset	Offset	Upper limit on offset
83	8	–0.10	0.01	0.12
84	20	–0.24	–0.09	0.06
85	8	0.34	0.43	0.51
86	8	–0.07	0.22	0.52
87	8	0.16	0.40	0.65
88	18	–0.17	–0.01	0.15
89	8	–0.53	0.45	1.42
90	12	0.94	1.55	2.17
91	8	–0.09	0.05	0.18
92	3	1.07	1.77	2.46

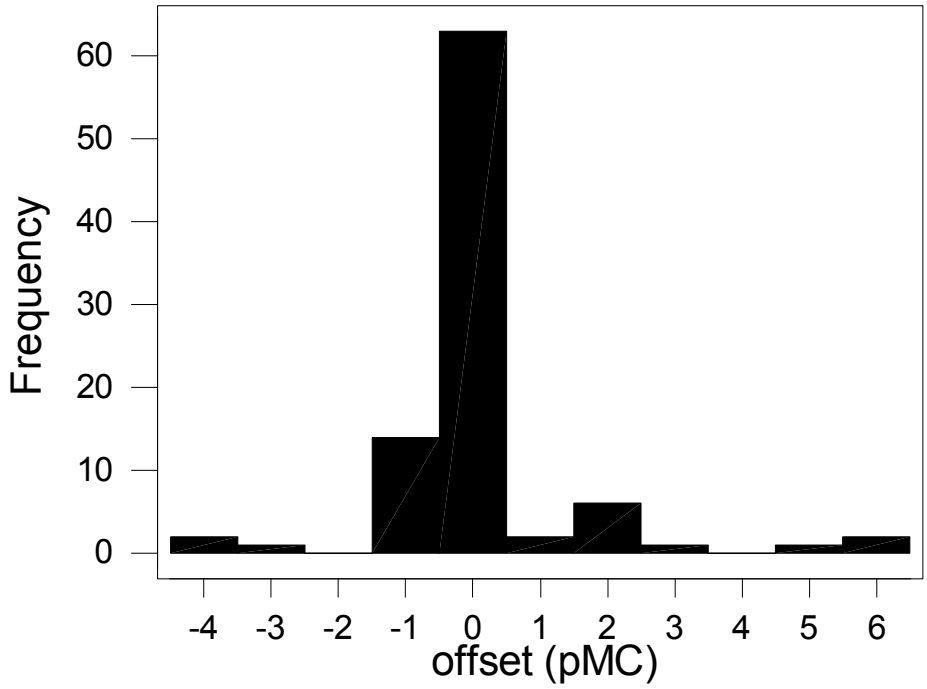

Figure 7.20 Distribution of laboratory offset relative to consensus values

Another possible calculation of offset can be based on the dendro-dated samples, of which 3 were included specifically for this purpose. Assuming a known age for these samples (based on the master chronology), an offset for each laboratory can then be estimated.

7.5.1 Offset Relative to the Dendro-Dated Wood Samples (yr BP)

A total of 4 dendro-dated wood samples were included in the list of core samples. They were Samples D and F (duplicates) from the Belfast master chronology, dendro-dated to 3200–3239 BC. Sample I (also from the Belfast master chronology), dendro-dated to 3299–3257 BC. Sample H was from the German oak chronology and was dendro-dated to 313–294 BC. A simple, exploratory summary of the findings and their comparison with the master calibration results is described in the following.

FIRI Samples D and F

Dendro-dated to 3239–3200 BC, this sample is linked to 4 samples on the master chronology. The average of the ^{14}C ages gives a "true" age of 4495 BP.

Table 7.13 Linked master calibration samples

Decadal midpoint	^{14}C age (1 σ)
3205	4528 ± 18
3215	4497 ± 11
3225	4495 ± 18
3235	4461 ± 18

FIRI Sample I

Dendro-dated to 3299–3257 BC, this sample is linked to 5 samples on the master chronology.

Table 7.14 Linked master calibration samples

Decadal midpoint	^{14}C age (1 σ)
3255	4455 ± 18
3265	4486 ± 18
3275	4480 ± 18
3285	4469 ± 18
3295	4468 ± 18

An average of the ^{14}C ages gives a "true" age of 4471 BP.

FIRI Sample H

Dendro-dated to 313–294 BC, this sample links to 3 samples on the master chronology.

Table 7.15 Linked master calibration samples

Decadal midpoint	^{14}C age (1 σ)
315	2210 ± 25
305	2211 ± 25
295	2225 ± 18

An average of the ^{14}C ages gives a "true" age of 2215 BP.

Similarly, using the master chronology ^{14}C ages as the "true" age for each laboratory, it is possible to estimate the systematic offset (if any) relative to these "true ages." However, it should be pointed out that, in fact, the consensus values for these samples are only slightly different from those extracted from the master calibration curve (4495 versus 4508 yr BP for DF, 4471 versus 4485 yr BP for I, and 2215 versus 2232 yr BP for Sample H).

Summarizing the offsets, we have:

Table 7.16 Offset in yr BP from the master ^{14}C ages

	N	Mean	Median	Min	Max	Q1	Q3	StDev
Offset	90	16.8	17.0	–642	414	–22	74	140.0
Outliers excluded	81	27	17	–218	209	–17	72	81

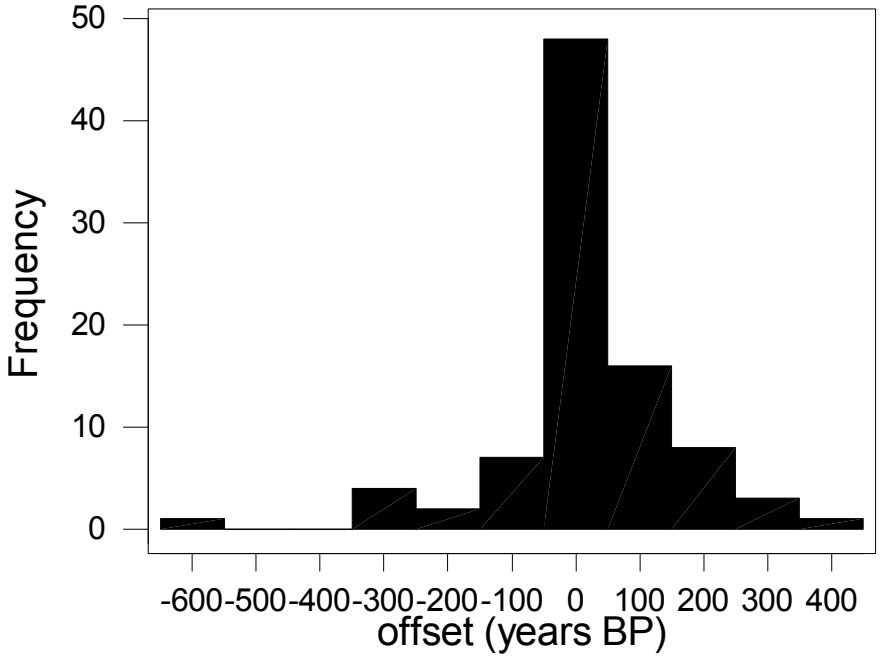

Figure 7.21 Distribution of offsets relative to the dendro-dated samples

Table 7.17 Lab offset (yr BP), based only on the dendro-dated samples (DF, I, H)

Lab nr	Number of results	Lower limit on offset	Offset	Upper limit on offset
1	8	48.83	80.86	112.89
2	6	6.53	37.24	67.96
3	2	–150.51	137.00	424.51
4	4	–31.83	89.87	211.56
5	7	–306.67	–218.46	–130.24
6	4	–46.34	–0.97	44.39
7	4	–135.38	–66.25	2.88
8	4	–46.65	–6.50	33.65
9	4	12.43	70.53	128.63
10	4	–420.29	–345.04	–269.79
11	7	–211.17	–62.76	85.65
12	4	–67.22	43.50	154.22
13	3	–84.91	199.67	484.24
15	7	–43.11	–6.04	31.03
16	3	94.17	203.95	313.73
17	5	–15.97	18.35	52.67
18	4	–140.97	12.64	166.25
19	4	1.11	62.13	123.14
20	5	73.72	109.46	145.21
21	4	–793.37	–327.44	138.50
22	3	–323.13	16.58	356.28

Table 7.17 Lab offset (yr BP), based only on the dendro-dated samples (DF, I, H) *(Continued)*

Lab nr	Number of results	Lower limit on offset	Offset	Upper limit on offset
23	4	75.46	209.49	343.52
24	4	–74.40	–14.28	45.84
25	4	–7.91	56.00	119.91
26	4	–1144.10	–642.19	–140.28
27	4	10.07	37.73	65.39
28	3	–626.91	–63.44	500.02
29	4	–39.33	75.73	190.79
30	4	–174.60	1.00	176.60
31	4	–70.59	8.44	87.46
32	4	–77.49	72.58	222.66
33	4	–147.00	–60.64	25.72
34	4	–94.50	–33.88	26.75
35	4	–62.96	104.21	271.38
36	6	–7.38	12.68	32.74
37	6	6.09	35.28	64.47
38	5	–41.22	–18.21	4.80
39	4	209.19	258.46	307.74
40	4	–51.43	–7.69	36.05
41	6	–20.89	10.32	41.54
42	4	–603.20	–321.25	–39.31
43	4	64.38	205.79	347.20
44	4	–257.58	–129.90	–2.21
45	2	–97.79	–35.86	26.06
46	4	–13.35	36.63	86.62
47	4	–31.27	125.28	281.82
48	4	–67.40	34.49	136.37
49	11	–11.40	17.41	46.23
50	8	–9.92	21.45	52.81
51	16	3.39	31.97	60.54
52	4	35.24	77.15	119.06
53	4	–1732.51	56.91	1846.32
54	3	–94.28	–20.99	52.30
55	4	–68.55	4.44	77.44
56	4	56.51	262.05	467.58
57	4	–226.99	–106.32	14.35
58	4	–29.62	42.03	113.68
59	4	–215.48	276.36	768.20
60	4	–36.92	42.28	121.49
61	4	–51.10	21.36	93.81
62	1	—	29.00	—
63	2	–873.23	160.00	1193.23
64	4	47.51	73.50	99.49
65	4	–39.40	–16.57	6.27
66	4	–50.75	–27.75	–4.75
67	4	–255.02	163.26	581.53

Table 7.17 Lab offset (yr BP), based only on the dendro-dated samples (DF, I, H) *(Continued)*

Lab nr	Number of results	Lower limit on offset	Offset	Upper limit on offset
68	4	169.60	192.55	215.50
69	4	26.45	414.43	802.42
70	8	–143.13	118.80	380.73
71	4	–245.85	–105.83	34.19
72	5	–87.53	20.01	127.55
73	4	–31.99	–1.17	29.65
74	7	–5.51	15.91	37.33
75	4	–189.71	–15.24	159.22
76	4	–36.24	–1.50	33.24
77	7	–60.72	–30.97	–1.22
78	4	–733.60	–27.86	677.87
79	4	–2.26	31.22	64.70
81	4	–116.91	182.93	482.76
82	4	28.77	102.39	176.01
83	4	–27.16	0.37	27.90
84	8	–37.68	–0.83	36.02
85	4	–55.00	–35.27	–15.53
86	4	–131.05	–26.97	77.12
87	4	–100.49	–49.26	1.98
88	9	–49.90	–16.60	16.70
89	4	–28.03	44.21	116.45
90	8	–239.84	–153.61	–67.37
91	4	–48.51	–7.57	33.36
92	1	—	–251.00	—

7.6 CONCLUSIONS

Consensus values (and their error) for the FIRI samples have been derived. Concerns remain over the consensus value for the Kauri wood sample due to the reporting difficulties for this sample. The sensitivity of the results to different calculation algorithms has been shown to be small (with the exception of the Kauri wood). Consensus values for the dendro-dated wood samples are very close to the values derived from the master calibration curve, which adds confidence in the results derived.

When considering laboratory performance, we have evaluated standardized deviations from the consensus values and have shown that these can be linked to the laboratory type. Calculation of the offsets has also shown that more than half the laboratories have no systematic offset, and that those laboratories that have a systematic offset, generally have small offsets (with only a few exceptions). Laboratories received this information for their consideration and, thus, were able to explore any causes, and then instigate any necessary corrective actions.

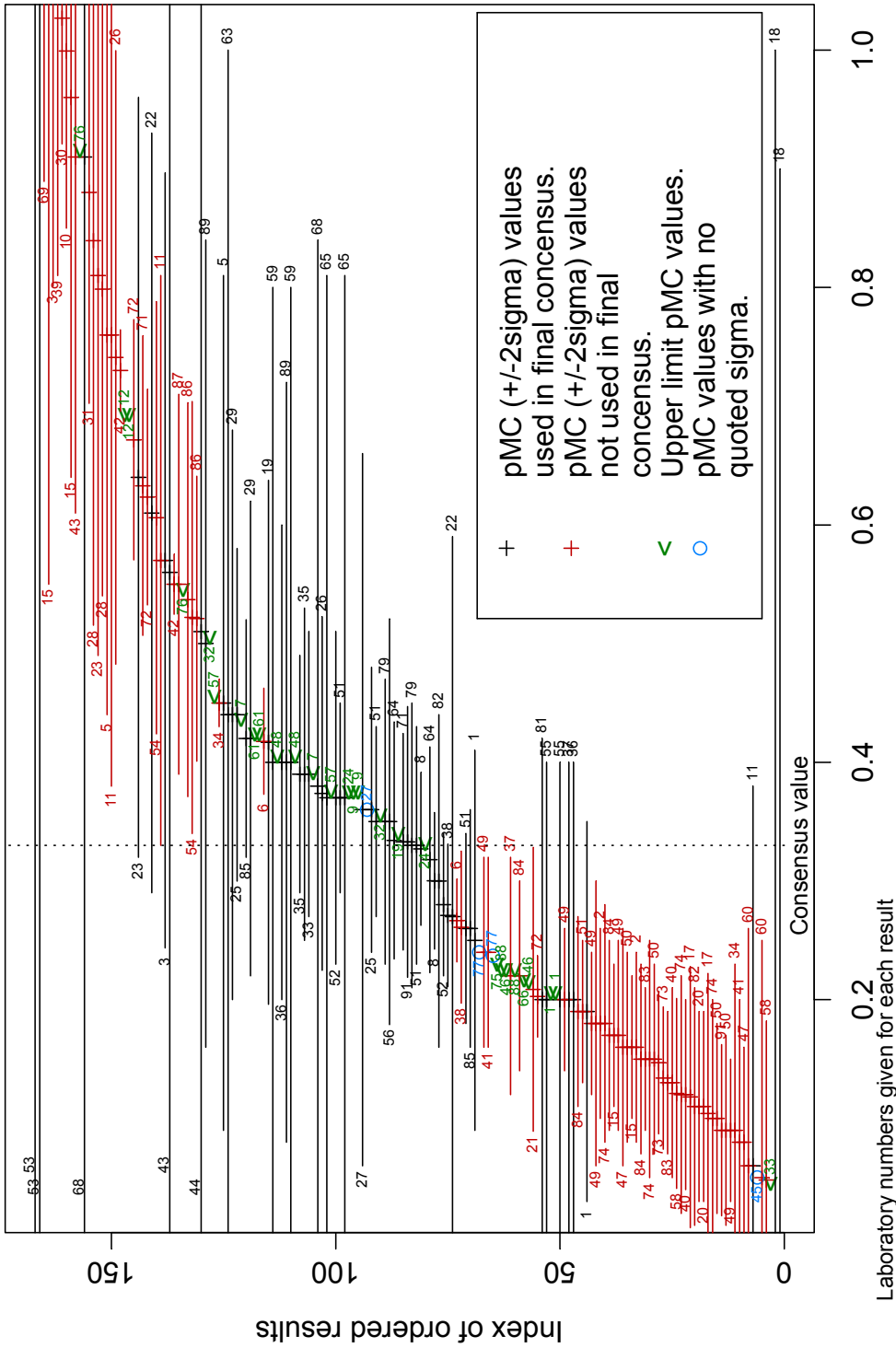

Figure 7.1 Distribution plot of Kauri wood, A & B, pMC with consensus value for all laboratories

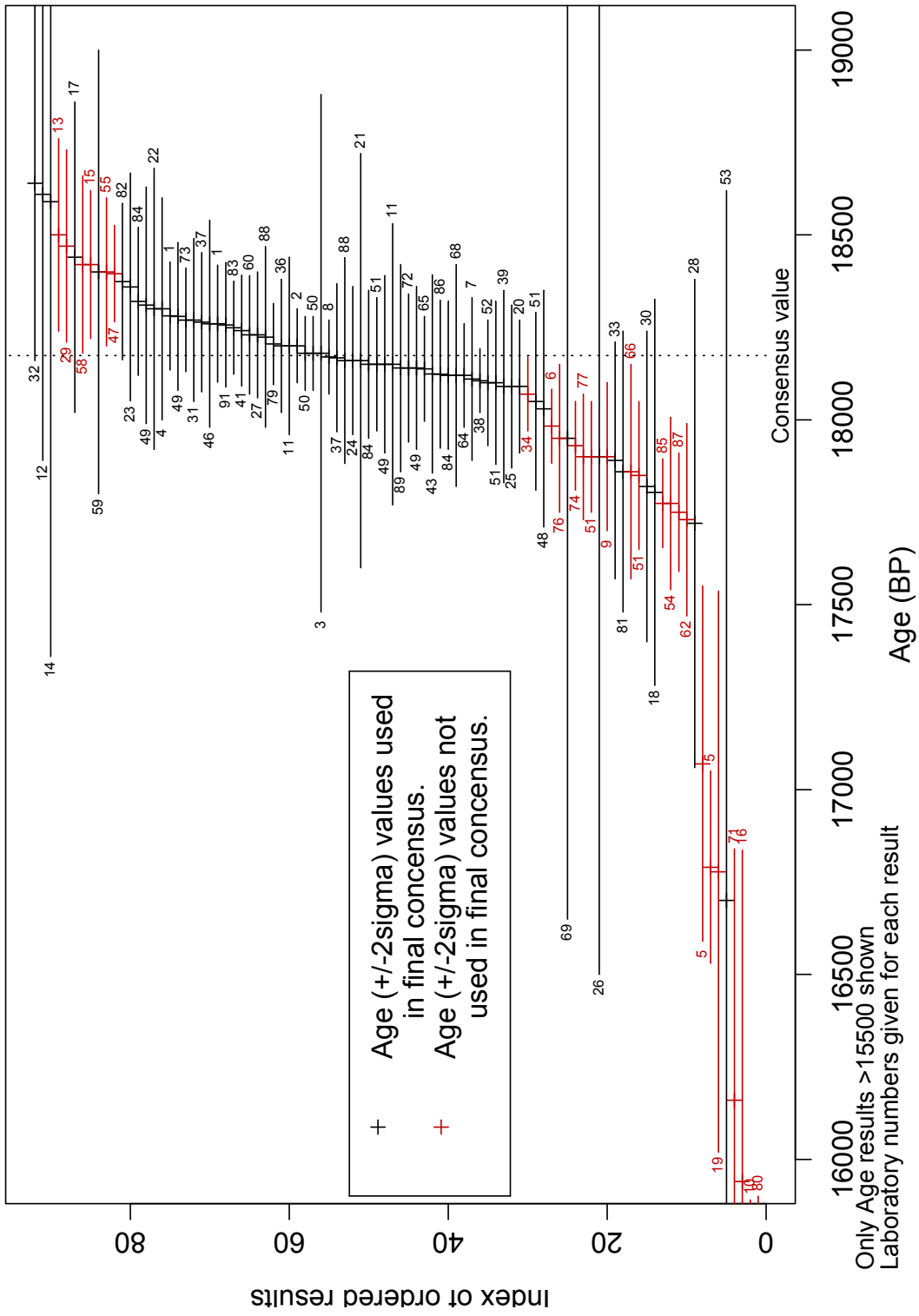

Figure 7.2 Distribution plot of turbidite, C, age with consensus value for all laboratories

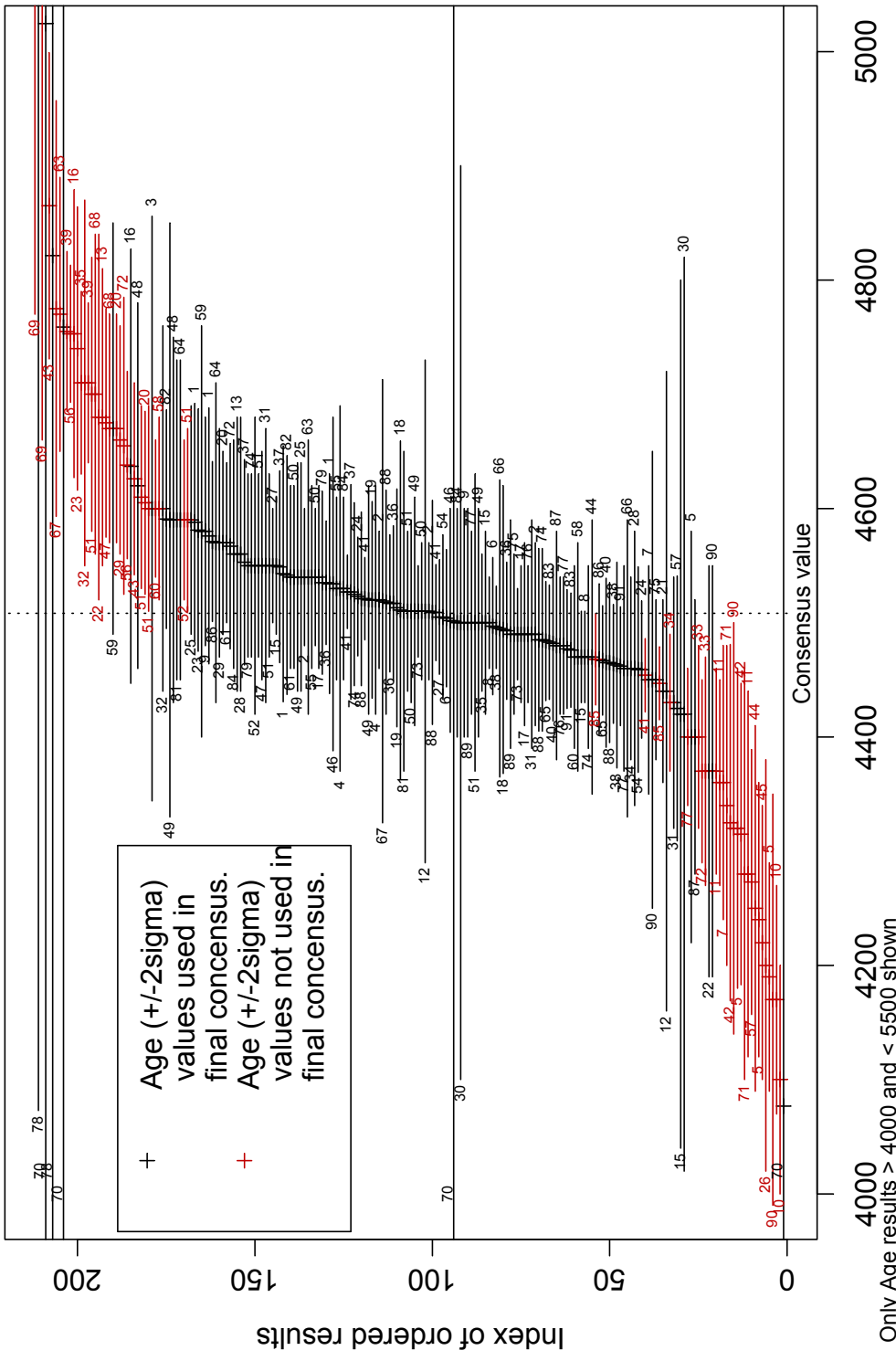

Figure 7.3 Distribution plot of Belfast dendro-dated wood, D & F, age with consensus value for all laboratories

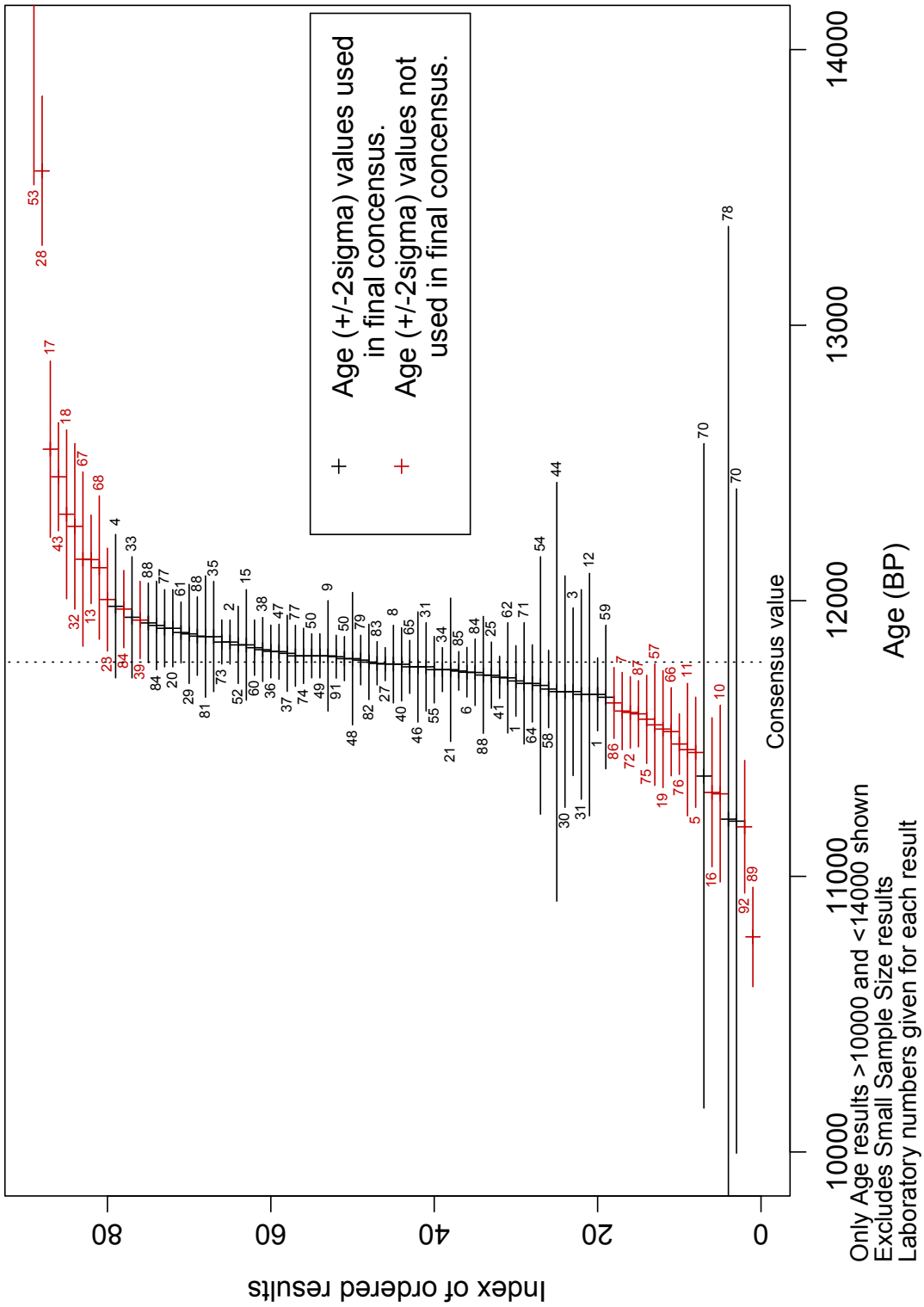

Figure 7.4 Distribution plot of humic acid, E, age with consensus value for all laboratories

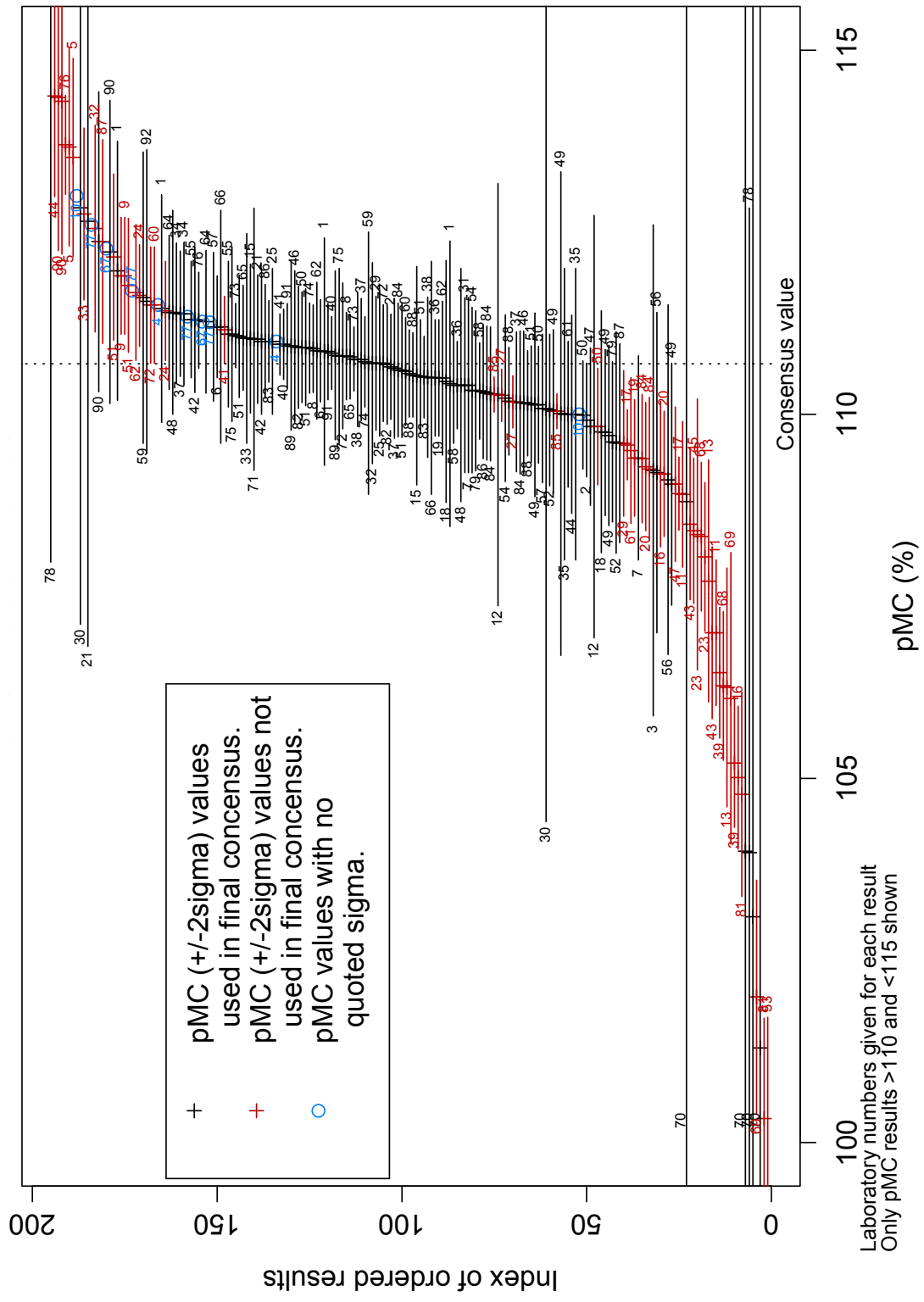

Figure 7.5 Distribution plot of barley mash, G & J, pMC with consensus value for all laboratories

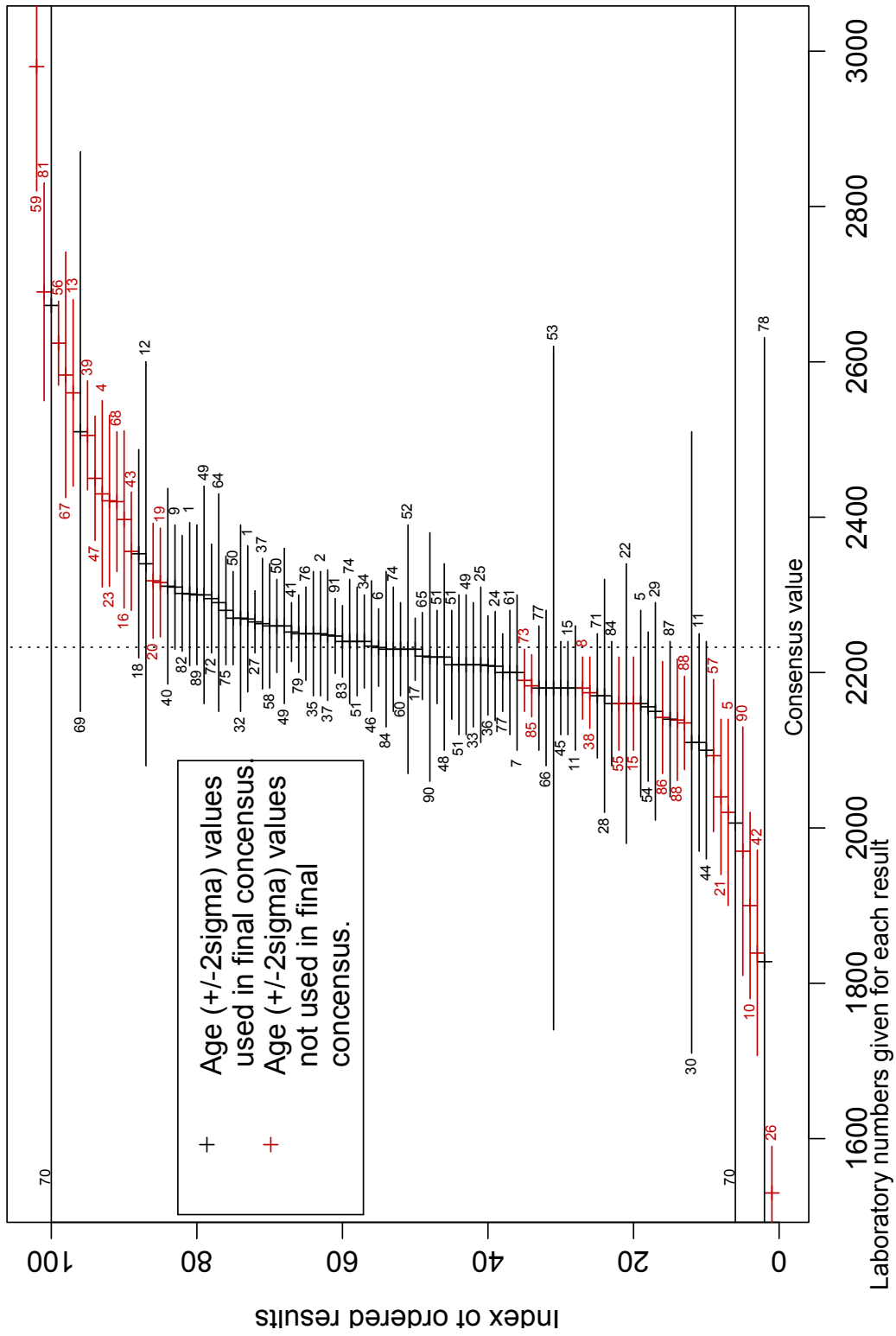

Figure 7.6 Distribution plot of Hohenheim dendro-dated wood, H, age with consensus value for all laboratories

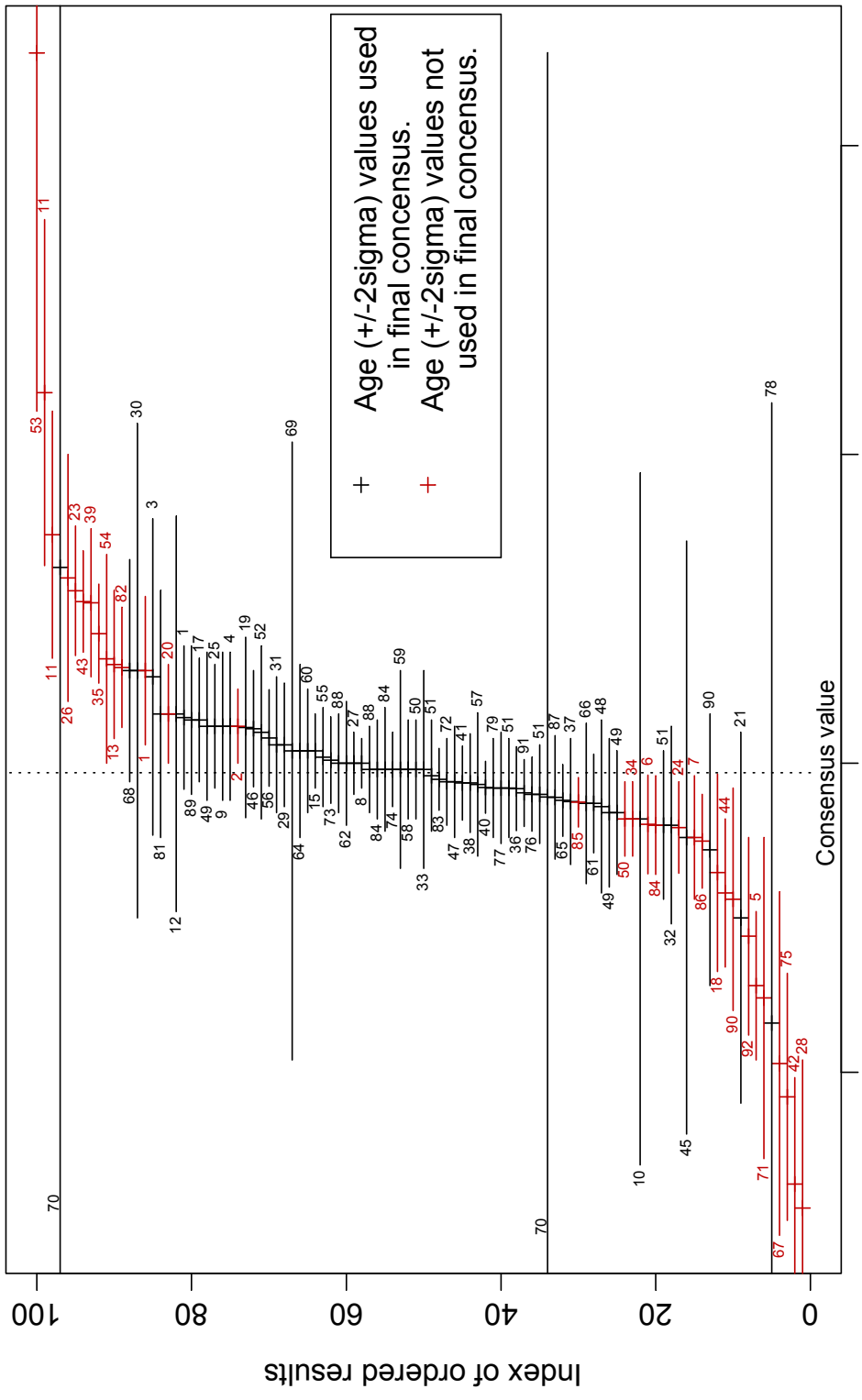

Figure 7.7 Distribution plot of Belfast cellulose, I, age with consensus value for all laboratories

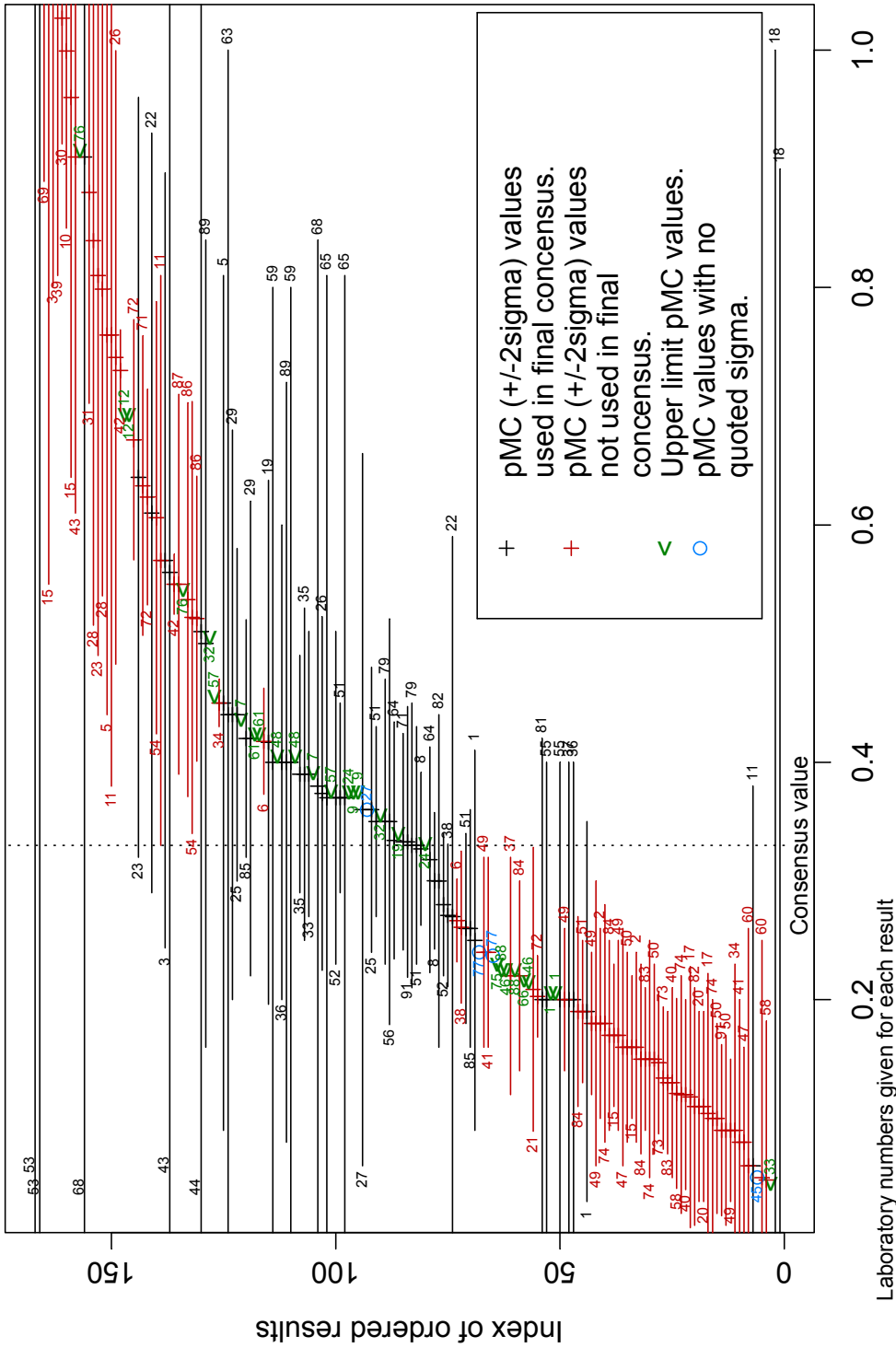

Figure 7.1 Distribution plot of Kauri wood, A & B, pMC with consensus value for all laboratories

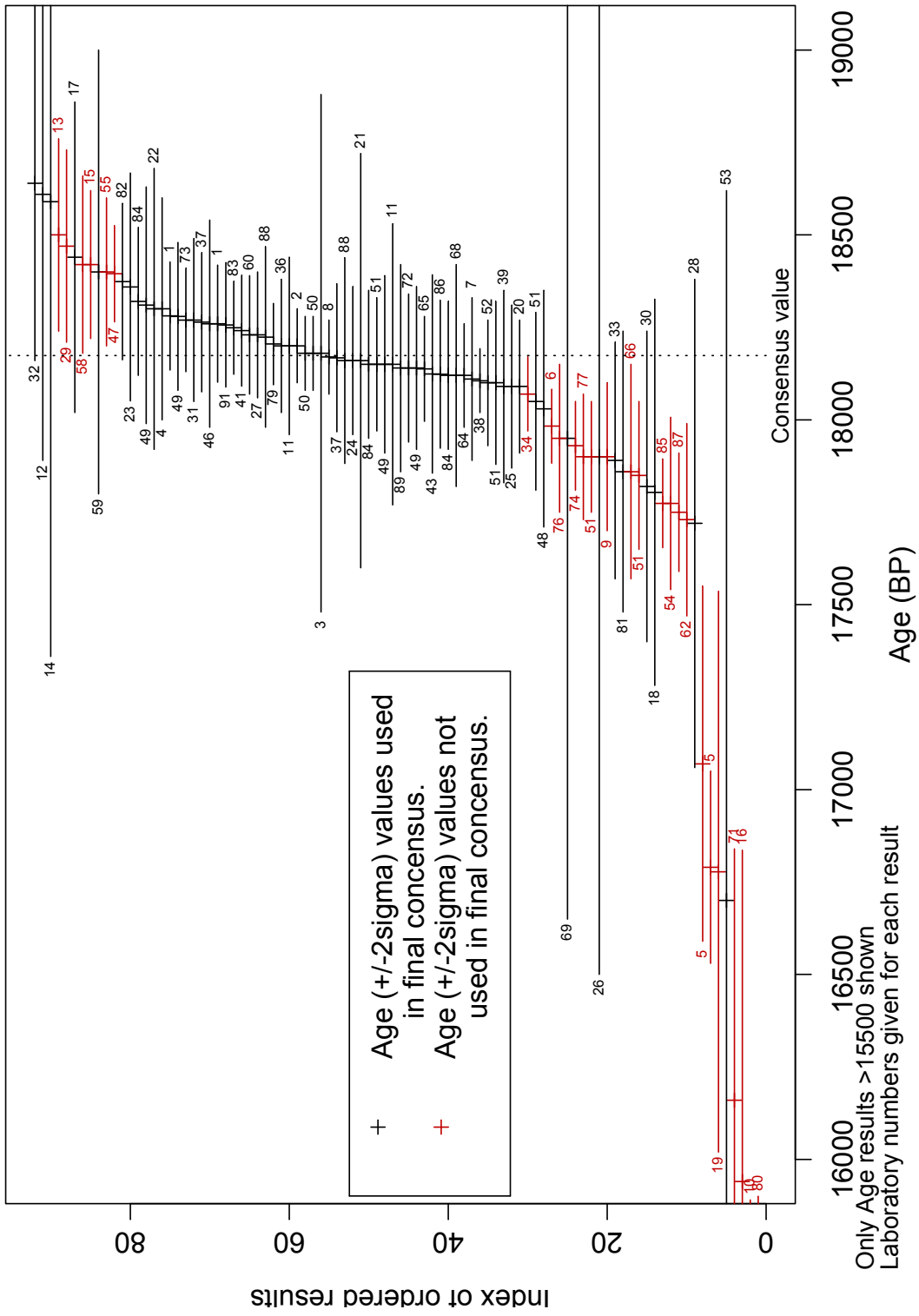

Figure 7.2 Distribution plot of turbidite, C, age with consensus value for all laboratories

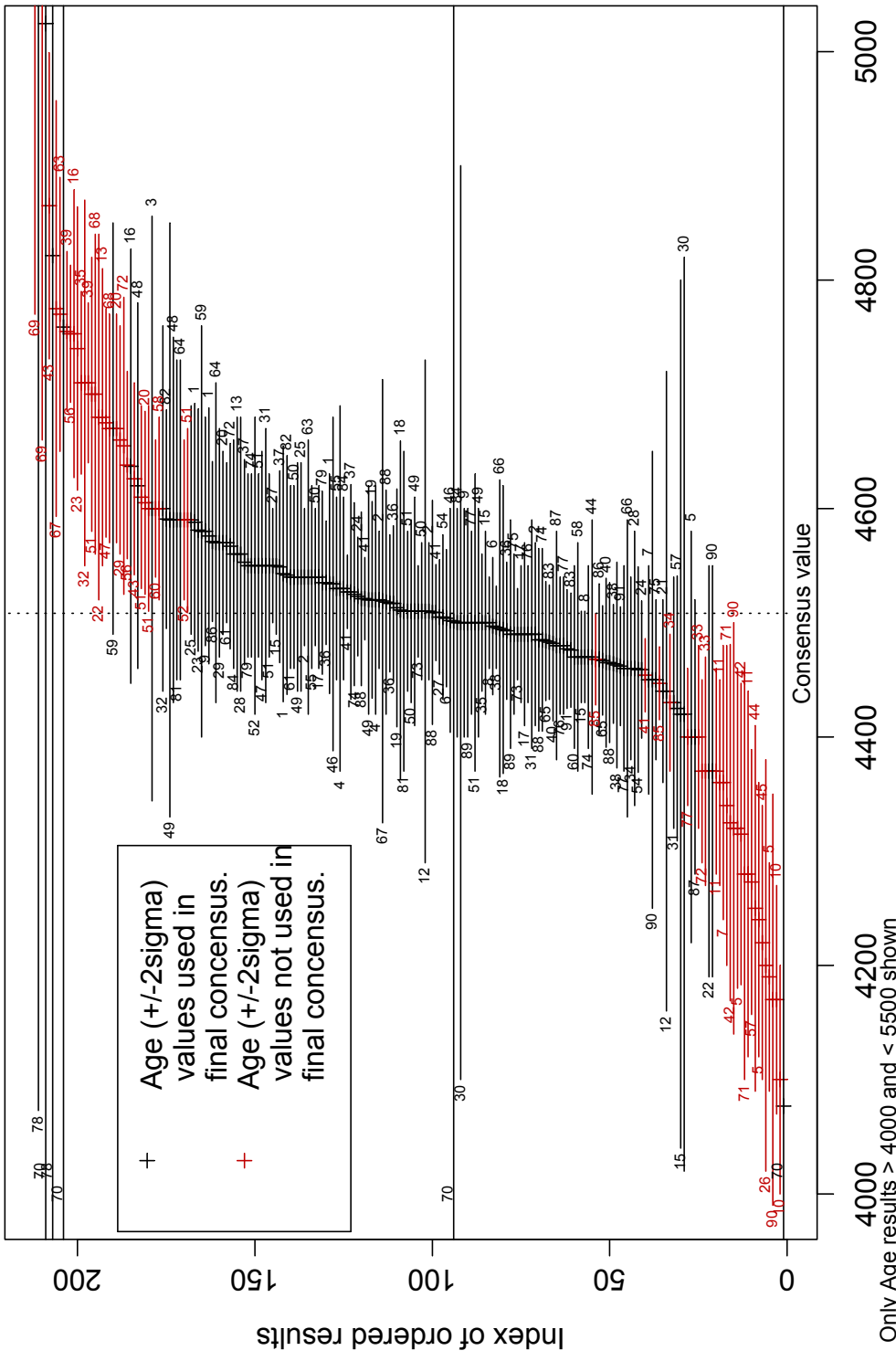

Figure 7.3 Distribution plot of Belfast dendro-dated wood, D & F, age with consensus value for all laboratories

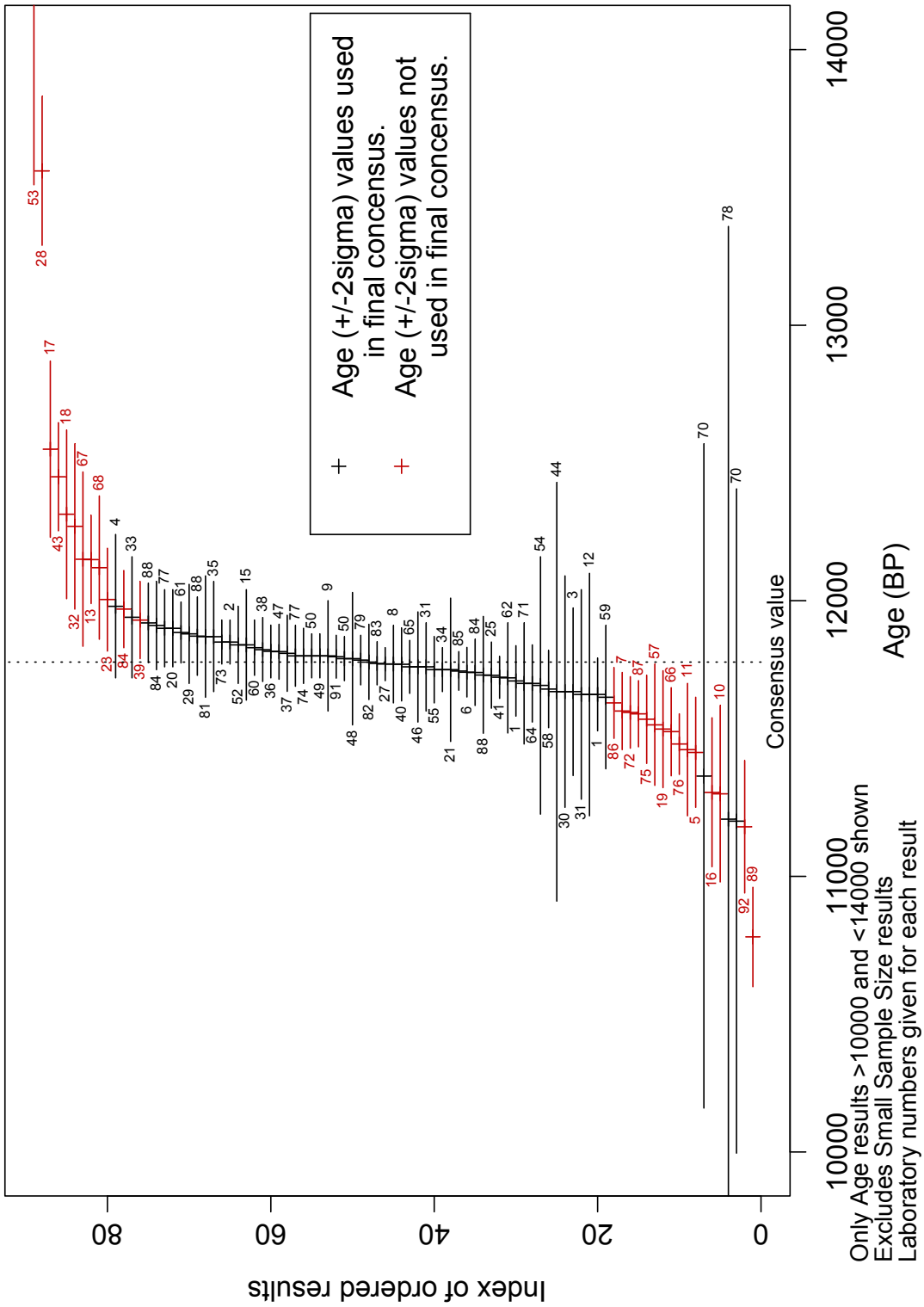

Figure 7.4 Distribution plot of humic acid, E, age with consensus value for all laboratories

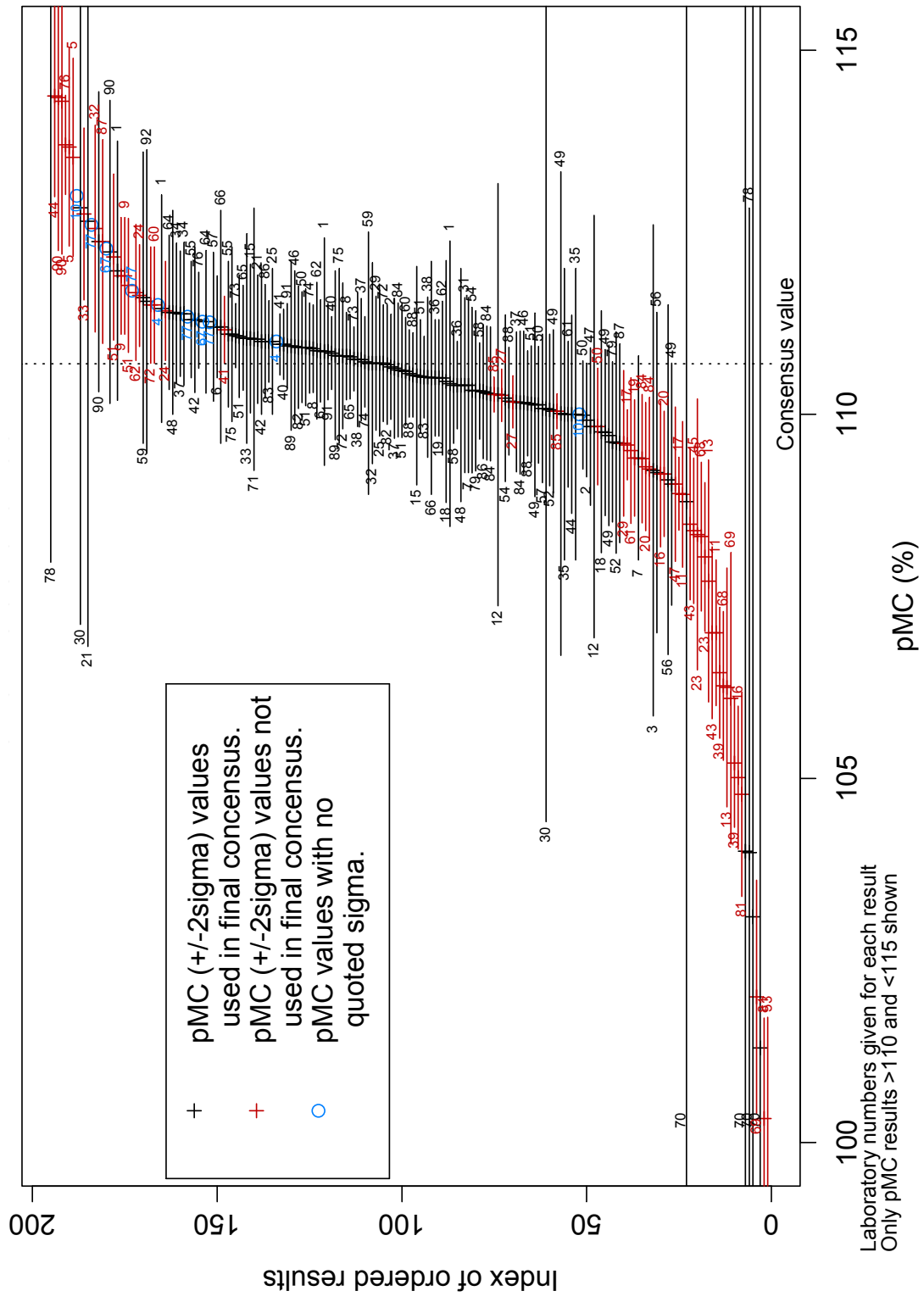

Figure 7.5 Distribution plot of barley mash, G & J, pMC with consensus value for all laboratories

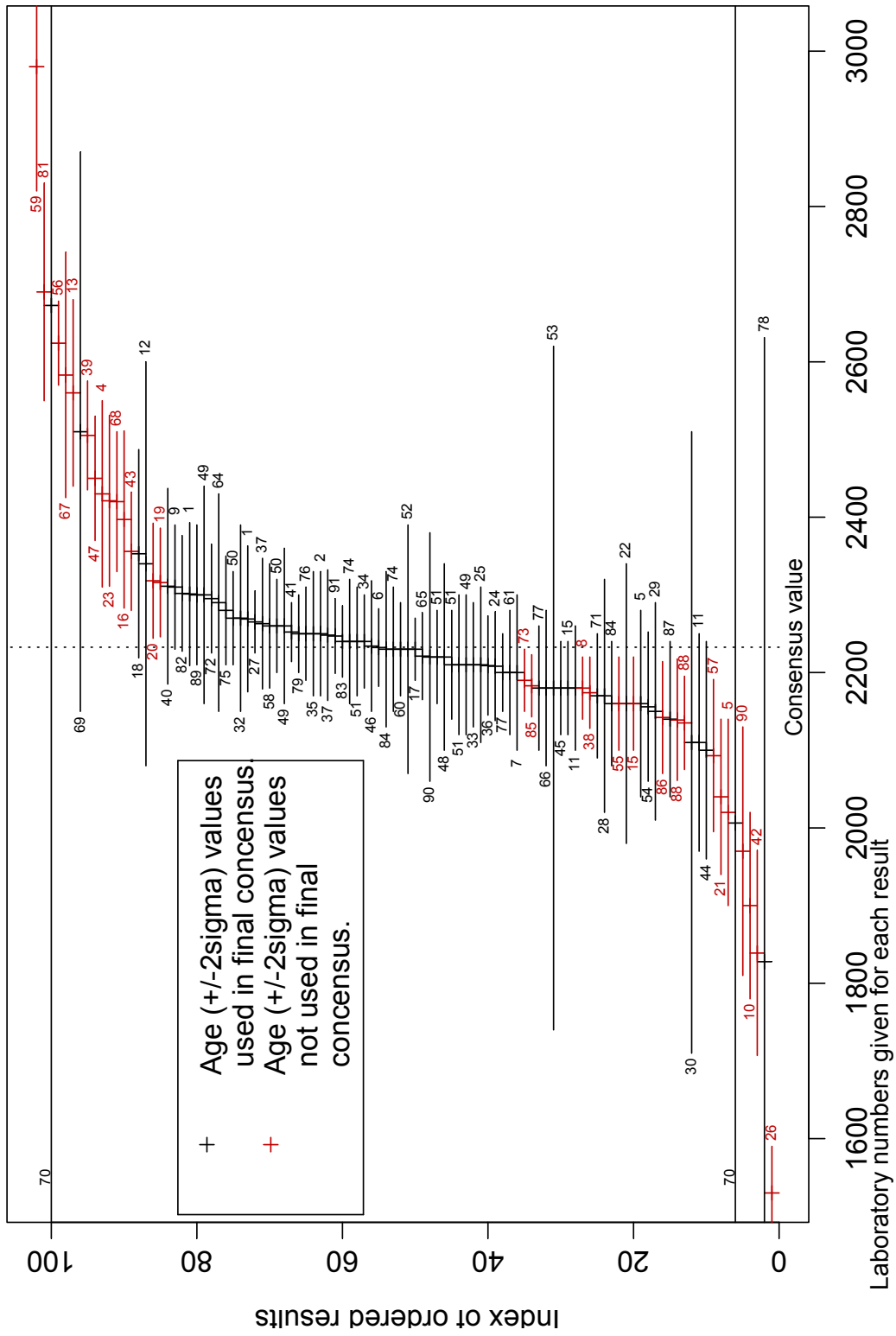

Figure 7.6 Distribution plot of Hohenheim dendro-dated wood, H, age with consensus value for all laboratories

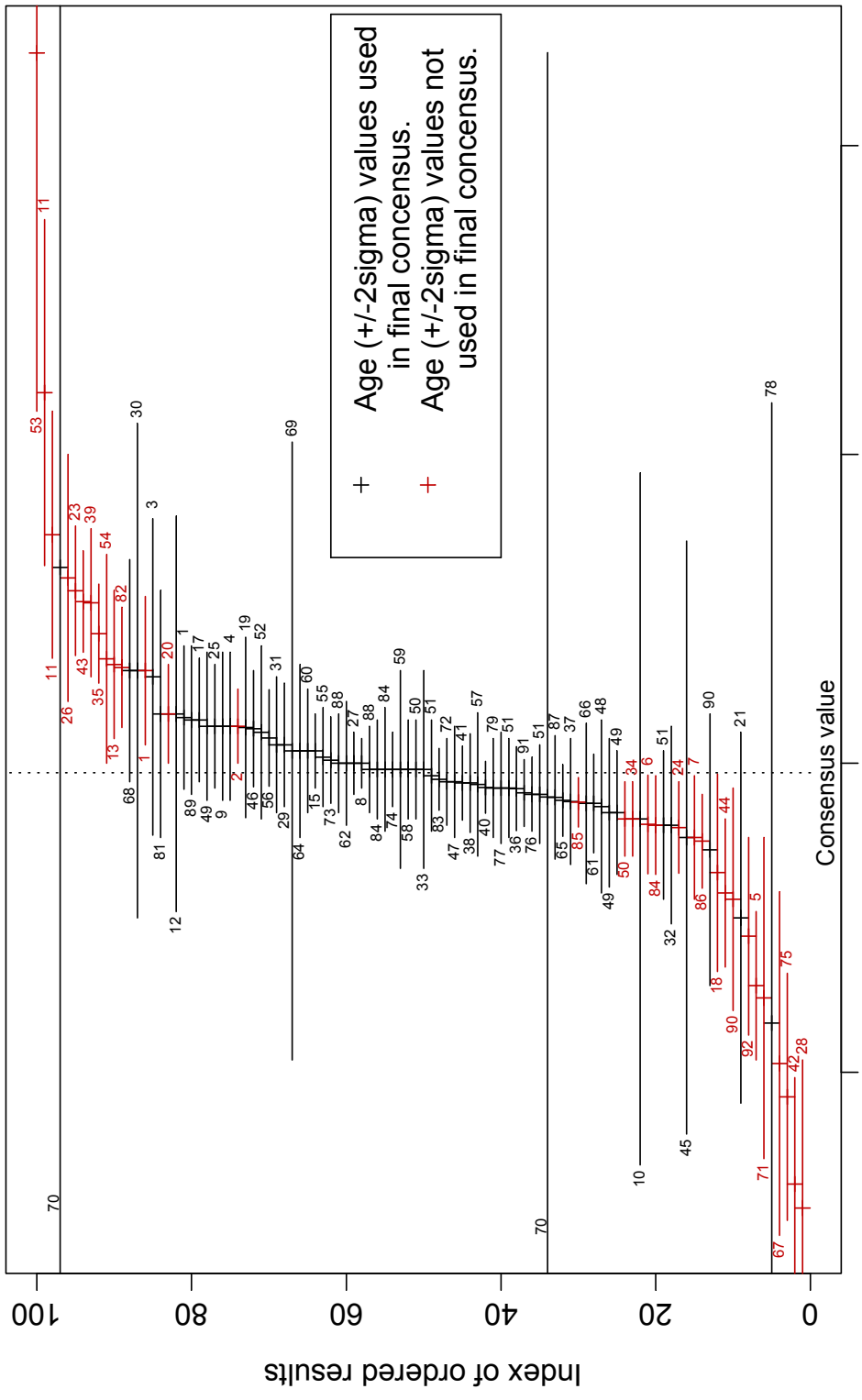

Figure 7.7 Distribution plot of Belfast cellulose, I, age with consensus value for all laboratories

SECTION 8: OPTIONAL FURTHER STUDIES

8.1 INTRODUCTION

As part of the FIRI program, it was recognized that providing samples in sufficient quantity for laboratory procedures is close to ideal and does not represent "typical" conditions. Therefore, laboratories were asked to consider 2 optional studies: investigating the effects of sample size on results and achieving high precision. The sample size study was focused on Sample E, humic acid, which had been chosen because of the rigorous pretreatment it had undergone in the solution stage, and which would ensure sample homogeneity. The precision study was focused on Sample D, the Belfast dendro-dated wood sample, given its importance in the master calibration.

8.1.1 Small Sample Size Results

Laboratories were asked to provide results at the smallest sample size they would consider. This resulted in an additional 52 results from 27 laboratories. A summary of these is given in Table 8.1.

Table 8.1 Summary of small sample size age (yr BP) results for Sample E

N	Mean	Median	Minimum	Maximum	Q1	Q3
52	11,776	11,796	313	10,370	13,000	11,880

A few laboratories provided more than 1 result. One (laboratory 15) provided 44 separate results for Sample E. These results are not included in the summary of the 52 results since this would bias the analysis. Laboratory 15 (AMS) provided results across a wide range of sample sizes, from their optimal size to the smallest size they would analyze.

8.1.2 High-Precision Samples

Laboratories were asked to provide high-precision results for the Belfast dendro-dated wood, Sample D. Two laboratories (15 and 25) indicated that their results were of high-precision—the ages of these were 4510 (10) BP and 4586 (28) BP, respectively. Given there were so few results identified as being reported with "high precision," no further analysis was completed on this part of the study.

8.2 THE EFFECT OF SAMPLE SIZE

In this section, the focus is on modelling the relationship between sample size and the absolute deviations from the consensus value for this sample. The effect of the quoted sigma on this relationship will also be explored. The AMS laboratories were best able to contribute to this study since they were able to report multiple results at a variety of sample sizes.

A total of 11 AMS laboratories gave 90 results, with almost half of these coming from a single laboratory (laboratory 15) and 6 laboratories giving 3 or fewer results. A scatterplot of the absolute deviations against the sample size (carbon mass) of the results is given in Figure 8.1. From the figure, we can see that results above 1 mg tend to have much smaller deviations than results from smaller carbon mass samples.

Log transformation of both the sample sizes and the deviations were used to examine the relationship and to control the skew. A plot of the log transformed data is given in Figure 8.2.

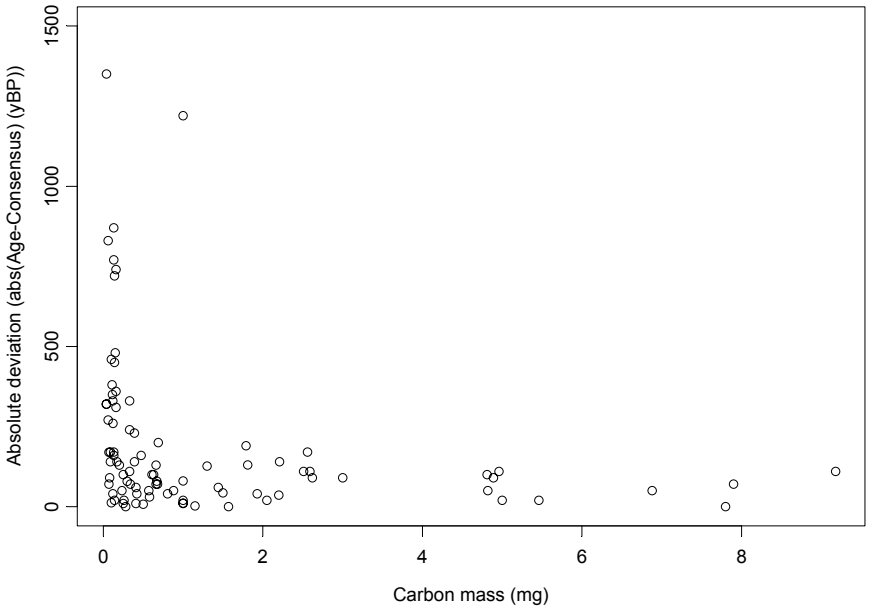

Figure 8.1 Scatterplot of sample size versus absolute deviation from consensus of Sample E results from AMS laboratories with at least 1 small sample size result

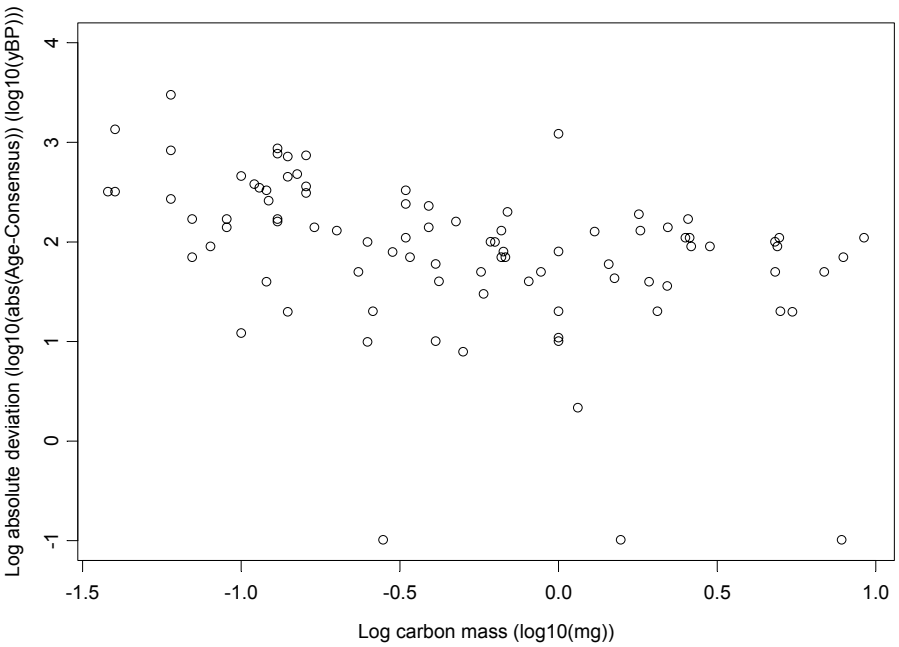

Figure 8.2 Scatterplot of $\log_{10}$ sample size versus $\log_{10}$ absolute deviation from consensus of Sample E results from AMS laboratories with at least 1 small sample size result

Figure 8.2 indicates that the log transformations of the data show a more linear relationship, which can then be modelled formally. A linear relationship was estimated and the resulting equation is:

$$\text{Log (absolute deviation)} = 1.7559 - 0.4996 \times \log \text{(carbon mass)}$$

The R^2 value is 16.4% (a measure of the variation in deviation explained by sample size), which is extremely low. Thus, sample size, although statistically significant, does not provide a good explanation of the scatter in the absolute deviation.

8.2.1 Comments

From this output, it can be seen that there is very strong evidence that the size of the sample does influence the average absolute standard deviation of the results via the $\log_{10}$ transformations of both the predictor and the response variable.

The analysis indicates that, although the regression is significant, it does not account for that much of the variation in the deviations. This is evident also from the low R^2 value (only 16%) for the model.

8.3 THE EFFECT OF QUOTED ERROR AND SAMPLE SIZE

So far, we have not considered the effect of the quoted sigma on this relationship. Two approaches to investigate this question are considered:

1. Scale the absolute deviations by the quoted sigma and re-analyze using the scaled values as the response;
2. Include quoted errors as another covariate in the regression modelling.

8.3.1 Scaling the Deviation Using the Quoted Sigma

Figure 8.4 indicates that by scaling the response by quoted sigma, there is much less of an evident pattern in the relationship with sample size; this is made more obvious when carbon mass is logged in Figure 8.5.

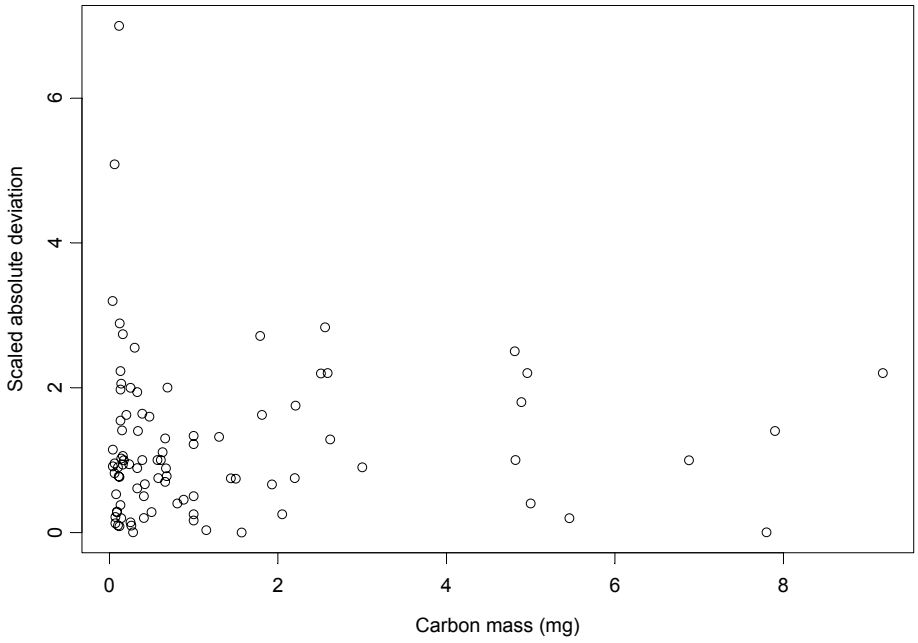

Figure 8.4 Scatterplot of scaled absolute deviation from the consensus versus the sample size of Sample E, results from AMS labs with at least 1 small sample size result

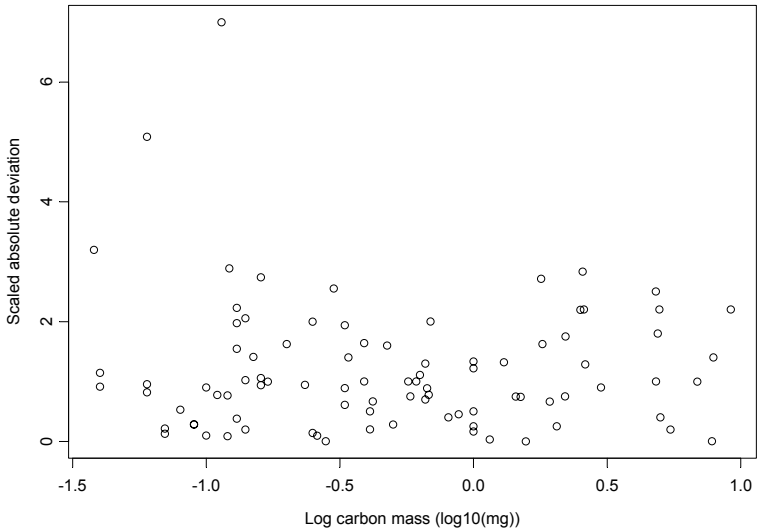

Figure 8.5 Scatterplot of scaled absolute deviation from the consensus versus the $\log_{10}$ sample size of Sample E, results from AMS labs with at least 1 small sample size result

This apparent lack of relationship is confirmed by the regression analysis, which gave a p-value for log (carbon mass) of 0.56; thus, we could conclude that there was no statistically significant relationship between the scaled deviation and the carbon mass. These results indicate that after scaling the absolute deviations by their associated quoted sigmas, there is no longer any dependence on sample size.

8.3.3 Including Quoted Sigma as a Second Covariate in the Regression

Figure 8.6 indicates that there does appear to be a linear relationship between the $\log_{10}$ quoted sigma and the $\log_{10}$ absolute deviation from the consensus. The model with the $\log_{10}$ quoted sigma added as a covariate showed that there was not a statistically significant relationship with the carbon mass ($p = 0.65$), but that there was one with the quoted sigma ($p < 0.05$).

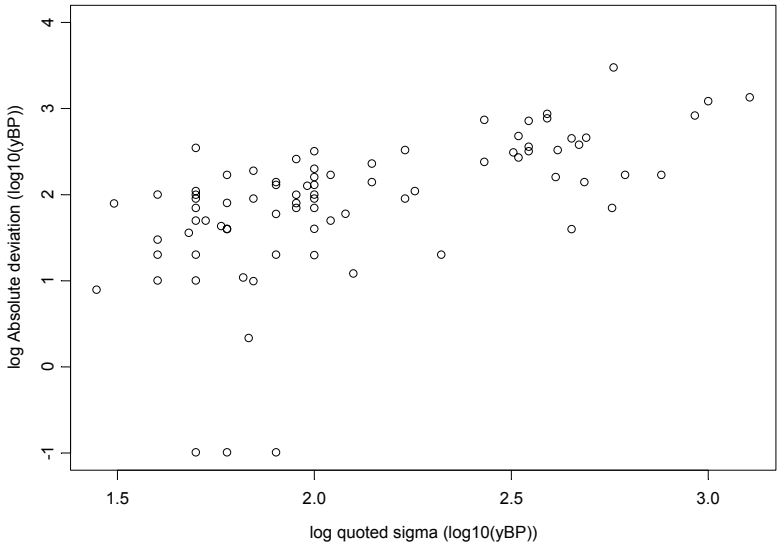

Figure 8.6 Scatterplot of $\log_{10}$ absolute deviation from consensus versus $\log_{10}$ quoted sigma of Sample E, results from AMS labs with at least 1 small sample size result

The $\log_{10}$ sample size is no longer a significant predictor, the but $\log_{10}$ quoted sigma is highly significant, indicating that the quoted sigma is better than sample size as a predictor, and that both of these 2 predictors are correlated. Therefore, it makes sense to fit the model with only $\log_{10}$ quoted sigma.

Fitting such a model, results in an R^2 value of 30%, with the coefficient on the $\log_{10}$ (quoted sigma) estimated as 1.055

This model explains over 30% of the variation in the response, which is almost twice as much as the model with the $\log_{10}$ sample size as a response. This is still rather poor, indicating that there are other factors which explain the variation in deviations.

Though not completely satisfying all the assumptions of simple linear regression, the model performs better than the model involving sample size. The presence of outliers is apparent and these may also impact any analysis.

8.4 CONCLUSIONS

Quoted sigmas determine the average size of the absolute deviations better than the sample size, though sample size is a significant predictor, if quoted sigmas are not used in the model. One reason that sample size may not be such a useful predictor in regression terms is that there seems (from Figure 8.1) to be a threshold, above which, increases in sample size have little or no impact on the average absolute deviations, but below which, changes in sample size seem to have a much greater effect on the deviations. This threshold could be said to be at about 1 mg. One possible further analysis could be to look at results obtained below the sample size threshold. This may show a stronger relationship between the sample size and the deviations from the consensus.

SECTION 9: OPTIONAL SAMPLES

9.1 INTRODUCTION

After the first and main phase of FIRI, which focused on routinely measured materials, an optional series of samples were also made available to participating laboratories. This second list included archaeological samples, mammoth tusks, and modern cellulose. Not all samples were available in sufficient quantity for radiometric measurement (in particular, the mammoth tusks). The samples are briefly described in Table 9.1 below.

Table 9.1 Description of optional samples

Sample	Description
K	Cambridge cellulose
L	Dogee Barrow wood
M	whole peat
N	mammoth tusk
O	mammoth tusk
P	mammoth tusk

9.2 SAMPLE DESCRIPTION

For Sample K, the dendro-age is known. Sample M had been previously pretested and came from the same site as Sample E. The Dogee Barrow site had been extensively dated, as had the mammoth tusks.

Sample K came from a tree that was planted around AD 1722 and material corresponding to the period AD 1820–1880 (a relatively flat area on the calibration curve) provided the sample. It has been homogeneity tested (approximately 98 pMC).

The 3 mammoth tusks had been previously dated with results for Sample N (T-13440, 28,075 ± 255 and LU-3983, 29,170 ± 340), Sample O (Lu-4170, age 39,320 ± 960), and Sample P (Lu-1967, age 12,820 ± 60).

Sample L came from the burial mound of Dogee Barrow, grave 8 (the Tuva king barrows). The approximate age was 2300–2400 BP.

Only a limited number of laboratories measured the optional samples and the summary statistics are shown below (the full table of results is given in Appendix 2).

Table 9.2 Descriptive statistics for the optional samples (in yr BP)

Sample	N	Mean	Median	StDev	Minimum	Maximum	Q1	Q3
K	6	126.2	76.5	104	40	310	58	220
L	10	2505	2500	123	2386	2790	2406	2548
M	15	11,139	11,120	191	10,710	11,413	11,070	11,300
N	5	28,100	28,574	1177	26,000	28,746	27,265	28,698
O	5	37,815	37,980	2143	34,700	40,504	35,910	39,639
P	5	12,558	12,600	151	12,300	12,696	12,443	12,653

Table 9.3 Descriptive statistics for the optional samples (in pMC)

Sample	N	Mean	Median	StDev	Minimum	Maximum	Q1	Q3
K	7	98.737	99.1	1.407	96.2	100.482	97.7	99.727
L	8	73.514	73.644	0.752	72.333	74.29	72.918	74.203
M	11	24.849	24.79	0.603	24.14	26.3	24.4	25.16
N	5	3.048	2.85	0.477	2.79	3.9	2.81	3.385
O	5	0.922	0.88	0.243	0.65	1.3	0.725	1.14
P	5	20.94	20.84	0.385	20.59	21.6	20.695	21.235

9.2.1 Comments

We can see that the results are in general agreement with the previous dating results and the known-age dendro date. No further analysis of the results for these materials has been undertaken. Given the small number of results, consensus values have not been calculated, but there still remains a sufficient archive to allow laboratories to measure these materials as part of their in-house QA procedures.

SECTION 10: SUMMARY AND CONCLUSIONS

A significant and substantial effort has been made by the ^{14}C community in quality assurance (QA) procedures, of which participation in FIRI is only one part but one that provides an independent and blind check on laboratory performance. The overwhelming willingness to participate is a testament to the importance which laboratories place on quality.

FIRI provides a spot-check of operational performance at the time it was carried out. It is not intended to be a means to create a league chart of laboratories. FIRI does not measure consistent performance over a period of time and this is one reason why the FIRI results are published without attribution to laboratories. Feedback is provided to laboratories, which they may choose to act upon, and it has been the case that a number of laboratories have identified and corrected problems as a result of participation in FIRI.

Derived reference values for the FIRI materials have been obtained and are given in Table 10.1.

Table 10.1 Consensus values and estimated standard error

Sample	Known age	Consensus value (estimated 1 σ precision)
AB (pMC)	—	0.24 pMC[a] (95% CI [0.23 – 0.30])
C (yr BP)	—	18,176 (10.5) yr BP[b]
DF (yr BP)	3200–3239 BC (^{14}C age 4495 BP)	4508 (3) yr BP
E (yr BP)	—	11,780 (7) yr BP
GJ (pMC)	—	110.7 (0.04) pMC
H (yr BP)	313–294 BC (^{14}C age 2215 BP)	2232 (5) yr BP
I (yr BP)	3299–3257 BC (^{14}C age 4471 BP)	4485 (5) yr BP

[a]pMC = percent modern carbon
[b]^{14}C years before present (yr BP) is 1950

The findings of FIRI are best considered in terms of some of the overall design aims for FIRI. These can be expressed in the form of some general questions posed and answered in the following.

10.1 HOW COMPARABLE ARE THE LABORATORIES?

Comparability can be considered in terms of the average result and also in terms of the variation in results.

On Average

We find overall, and on average, no evidence of significant differences between AMS, GPC, and LSC laboratories, with the exception of the near-background Kauri sample, where, on average, the age reported by AMS laboratories is highly likely to be older.

Variation

In terms of the variation reported, we find that more LSC laboratories reported results identified as extreme or outliers. Outliers were also identified for GPC laboratories, but in less number. In addition, there is some evidence as a group that the overall variation in the results is less for AMS laboratories; however, there are several factors which may, in part, explain this result, namely: a) feeder labs may have used a common AMS facility, and b) given the sample size requirements,

AMS facilities are able to prepare multiple targets and quote average results, which would be expected to show less variation.

10.2 HOW VARIED ARE THE RESULTS AND WHAT FACTORS EXPLAIN THE VARIATION?

Components of Variation

The components of variation can be considered in two ways: random and systematic.

Random

Random components of variation would be apparent from the amount of scatter in the results, which shows no specific pattern but perhaps manifests itself through either outliers or anomalous values. The chances of outliers occurring, assuming they occur by chance, is roughly 1 in 20. For an individual laboratory, random variation might also manifest itself in that the difference between duplicates or the difference between the measurement and the known age tend to be larger than would be expected, given the laboratory quoted error.

Thus, in 10 results (as in FIRI), the presence of 1 outlier in the set is not unlikely and, therefore, does not indicate a problem. However, more than 1 outlier in a set of 10 is increasingly unlikely, assuming that such observations occur by chance.

Systematic

Systematic components of variation are apparent as a shift or offset in the results, i.e., results are always too high or always too low relative to a known age (or a reference value). Possible reasons could be incorrect estimation of the background, calibration of the modern standard, or a source (constant) of modern or very old carbon within the laboratory.

In the analysis of the FIRI results, we see evidence of both random and systematic sources of variation.

- Roughly 10% of the total results are identified as outliers (which is around twice as frequent as would be expected). Yet, it should be noted that the distribution of outliers is not uniform across the laboratories, with the majority of outliers coming from around only 14% of the laboratories. The distribution of outliers across samples is uniform, so no one sample material is more varied than any other.
- Comparing laboratory results to both dendro-dated samples and the derived reference values for the materials, we find evidence for small laboratory offsets relative to the derived reference values for some laboratories.

10.3 CAN WE IDENTIFY ANY REASONS FOR THE VARIATION IN RESULTS?

We have studied the effect of the modern standard and background material used and found no evidence that these factors make a significant contribution to the overall variation observed. For some samples, we did see evidence of an effect of pretreatment (FIRI C). Issues of outlier identification showed that they were often associated with the modern standard used by the laboratories and this is a recurring theme in much of the analysis.

10.4 ACCURACY AND PRECISION ISSUES

Precision

Within the measurement process, the quoted error is a measure of precision on the measurement. Ideally, it quantifies the variation to be expected in the measurement were it to be repeated many times. For the radiometric laboratories, its basis is the Poisson counting/decay process, although other sources of random variation are also typically included in its calculation. For the AMS laboratories, the quoted error is not based on the Poisson decay process, but its interpretation remains similar to that for the radiometric laboratories.

The duplicate samples included in FIRI allow estimation of precision (without issues of true age being considered).

From the 3 sets of duplicate results, we see that, on average, the difference in duplicates is zero (for all laboratories and also for individual laboratories), but the magnitude of difference is frequently large relative to the quoted errors (and larger than expected given the interpretation of the quoted error). The implication is that a source of variation is not completely accounted for in the quoted error in these cases. In a number of cases, we also see evidence of agreement between the duplicates, which is, in fact, better than would be expected given the quoted errors.

Accuracy

Accuracy is concerned with the "correctness of the result." Ideally, with exactly known-age samples, this could be independently estimated (for our dendro-dated samples, the true ^{14}C age is not known exactly, but only within a range, due to that fact that it is measured). The master measurements are based on decadal samples, which do not correspond exactly to the samples provided in FIRI. This range could be as much as 100 yr, which corresponds to twice a commonly-quoted error value.

For our materials, we must assume that we can define (through calculation) what the "true" ^{14}C age will be (the consensus value), and then, we can estimate for each laboratory whether there is a constant offset from this consensus (hence, a measure of accuracy).

This is not an ideal situation since the issue of precision of the estimate of the consensus value should also be considered. However, the consensus value is based on a large number of results and so its precision is high, relative to the individual measurements.

We found evidence that a number of laboratories had small, but significant, offsets relative to the consensus profile. One possible explanation is that of mis-estimation of the background or modern standard activity, but other reasons are possible. Results from FIRI do not allow further examination of this.

Overall, the evidence supports the fact that ^{14}C laboratories are generally accurate and precise, but that notwithstanding internal QA procedures, some problems still occur that can best be detected by participation in intercomparisons such as FIRI. The results from FIRI are significant in that they show a broad measure of agreement between measurements made in different laboratories on a wide range of materials. They also demonstrate no statistically significant difference between measurements made by radiometric or AMS techniques.

Finally, some of the same features identified in FIRI were also observed in the previous exercise ("Part 2" of this issue). This reinforces the idea that an extra, independent check on laboratory per-

formance is required, and suggests that internal QA procedures, while essential, do not address all QA issues. When advised of the analysis, laboratories are able to instigate a number of corrective measures and we would anticipate that FIRI would result in similar activity.

There is a clearly demonstrated need for standards and reference materials to which laboratories have ready access to allow checking and correction. As a result of FIRI (and previous Glasgow-led programs, especially TIRI, see Part 2), a small archive of natural materials has been created for use by the ^{14}C community. Some of the materials are extremely limited and sufficient remains exist for AMS measurement only, while for some others, a substantial store exists. However, information concerning its existence has been, and is being, disseminated to laboratories, with the purpose that such samples could be used to check laboratory performance.

Table 10.2 Archived material from TIRI

Sample identifier	Description	Activity range
H	Ellanmore peat	Less than 3 half-lives
A	Barley mash	Modern
J	Buiston Crannog wood	Less than one half-life
G	Fuglaness wood	More than 5 half-lives
I	Travertine	—
K	Turbidite	More than 3 half-lives
F	Doublespar	Background
L	Whalebone	More than 2 half-lives

Table 10.3 Archived material from FIRI

Sample identifier	Description	Activity range
A,B	Kauri wood	More than 5 half-lives
C	Turbidite	More than 3 half-lives
E	Humic acid	Less than 3 half-lives
G, J	Barley mash	Modern
H	Dendro-dated wood	Less than 1 half-life
I	Dendro-dated cellulose	Less than 1 half-life
K	Dendro-dated cellulose	Before bomb

10.5 FURTHER INTERCOMPARISONS

At the end of FIRI, a small follow-up questionnaire was circulated to all ^{14}C laboratories, seeking their views on the intercomparison just completed and any comments they might have on future requirements and organization. Of those who responded, around 80% thought the FIRI workload had been sufficient, that the timescale was sufficient, that there was sufficient sample material, and that the feedback had been timely and in sufficient detail.

The view on the frequency of intercomparisons was roughly split, with 50% thinking that an interval of 4–5 yr was optimal and 37.5% preferring an interval of 3 yr.

A total of 33% thought that there should be fewer than 10 samples, while 59% thought 10 samples was reasonable.

With respect to the anonymity of the laboratories, 44% thought laboratories should be anonymous, 41% thought it should be up to the individual laboratory, and 16% thought laboratories should not be anonymous.

An overwhelming majority said they would participate in a future intercomparison (94%).

From the experience gathered from both TIRI and FIRI and also the response to the questionnaire, it would appear that the ^{14}C community is fully supportive of intercomparisons and see them as benefiting them greatly. Thus, further intercomparisons are seen as an essential part of the community's QA.

Design Issues in Future Intercomparisons

There are a number of design issues relating to the organization of a laboratory intercomparison. Many relate to the sample material, but also there are issues concerning the conduct of the trial, which are briefly discussed below.

Sample Material

There are 2 options in the selection of material: first, all samples are of a single class of material (e.g., only shell or peat or wood), this of course limits the ability to generalize the results, so more commonly for ^{14}C dating at least, the materials used have been representative of routinely-dated material.

Activity Range

The activity or age of the test samples should cover the applied ^{14}C timescale.

Sample Size and Homogeneity

A key question, especially when using natural samples, is the homogeneity of the material, which should be tested. Obviously, as sample requirements in terms of weight may vary quite widely (through differences in pretreatment procedure, counting, and technique), it is necessary that the sample should be demonstrably homogeneous at the finest level required. This is an important issue as there is ever growing requests for dates from smaller and smaller samples.

Number of Samples

The number of samples is balanced between the needs of the statistical analysis of the data and, of course, the practical commitments of the participating laboratories. Preferably, numbers of test samples should be greater than 4 and there should be replication (with the identity of duplicate pairs withheld from the participating laboratories). The presence of duplicate samples allows a direct assessment of a laboratory's repeatability or the within-lab variation.

Perceived Needs

All of the previous ^{14}C intercomparisons have provided valuable information to laboratories, and hence, to users. As a result, it is clear that such checks as FIRI and others are, and will continue to be, necessary and that they must operate in addition to any within-laboratory procedures. Nor is it clear in these previous studies that the increased availability of an extensive range of reference materials has presented an immediate solution to the problem of laboratory comparability, as might have been hoped. Increasing the scope of reference materials and standards is important, since by their inclusion, the dating determinations can be better constrained, but only if laboratories make regular use of them in routine operation. There is increasing pressure to date smaller (even to the molecular level) and older samples. More conventional laboratories are forming close collaborations with accelerator laboratories, which has meant developing in-house techniques for

target preparation. Thus, an accelerator laboratory may have a number of target preparation laboratories providing it with targets presenting new issues of comparability. However, perhaps the most significant factor is that as we strive to measure smaller and smaller samples, the issue of sample homogeneity becomes more and more important; indeed, the definition of a sample becomes critical. In some of the studies already completed in which AMS laboratories have participated, some evidence of sample in-homogeneity has been reported, which the conventional laboratories were not able to detect. There are difficulties in taking a representative sub-sample from the bulk of material; indeed, how do we know it is representative? We do not fully know the potential scale of natural ^{14}C variation in sample matrices.

10.6 FUTURE PROPOSALS

Continuation in this work is important. The linkage to previous work provides an invaluable continuity (e.g., IAEA and other reference materials are still available and should be used), but further, new materials should be sought, including known-age material, and certainly a "background" organic sample is essential. For the conventional laboratory, the typical sample requirement might be 5gC, with sample age ranges from 1 to 4 half-lives. However, for the AMS laboratories and for those conventional laboratories where small samples are dated, we need to explore the natural variation in reportedly single-event samples (deposits of charcoal, grain from a single growing season, single insects from a well-defined stratum). This information is not just important for the laboratory, but is also of fundamental importance for the sample submitter who must select samples referring to the event of interest. There are new challenges for ^{14}C dating in continuing to ensure the quality of results.

Discussion with laboratories and results from the questionnaire responses have indicated a general desire for further intercomparisons of this more classical nature such as FIRI. Yet, in consultation, an additional proposal has been drawn up such that there should be a rolling 4-yr program. A major intercomparison such as FIRI would be organized every 4 yr, but in each of the 3 preceding years, a small number of samples (e.g., 3) would be sent to laboratories to be analyzed in a short time and feedback given within a short follow-up period. In this way, the "spot-check" nature of FIRI and the lack of continuous monitoring of performance would be remedied. This would be of more use to the participating laboratories, but would also provide a better guarantee of quality assurance to the user communities. Plans are currently being drawn-up for the next intercomparison (VIRI) and will be presented at the 2003 Radiocarbon International Conference in New Zealand.

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PART 2: THE THIRD INTERNATIONAL RADIOCARBON INTERCOMPARISON (TIRI)

1. THE THIRD INTERNATIONAL RADIOCARBON INTERCOMPARISON (TIRI)

TIRI was officially launched at the 14th International Radiocarbon Conference in Arizona in 1991. Prior to the conference, 150 laboratories received a letter describing the general intention to organize an intercomparison and over 90 laboratories from around the world responded positively to the invitation to participate. Simply stated, the aims of this intercomparison were:

1. To function as the third arm of the quality assurance (QA) procedure.
2. To provide an objective measure of the maintenance and improvement in analytical quality.
3. To assist in the development of a "self-help" scheme for participating laboratories.

TIRI followed through on previous intercomparisons, including that organized by IAEA for the launch of 6 new reference materials, C1–C6 (Rozanksi et al. 1992), and the International Collaborative Study (Scott et al. 1990).

1.1 Structure of TIRI

A total of 13 different samples were collected and prepared for TIRI. They were classified as either *core* or *optional*. Every laboratory received 6 core samples. The remaining 7 samples were of a more specialized nature; therefore, laboratories were allowed to choose the samples they wished to receive.

Core samples were dispatched to laboratories in March 1992 with results expected by March 1993. The optional samples were dispatched in May 1993 at the same time as a preliminary report on the core results.

1.2 Sample Selection and Preparation

The samples used in TIRI were natural, and generally required full laboratory processing, including pretreatment. They were also selected with the following criteria in mind:

- There should be sufficient quantity of material available to meet requirements.
- They should be of archaeological and geological interest.
- They should cover the broad spectrum of laboratory experience.
- They should satisfy rigorous homogeneity conditions.

In some instances, the sample had undergone some preparation before dispatch, and where necessary, had been homogeneity tested.

The range of ^{14}C activities of the samples spanned from "modern" to "close to background," although the majority of samples were clustered in the range of 1000 to 15,000 yr.

2. SAMPLES AVAILABLE IN TIRI

2.1 Core Samples

Each sample was identified by a name and a code. Detailed information was provided concerning each sample. The approximate sample ages were broadly categorized as a rough guide for laboratories in the following way:

Table 2.1 Sample age classification used

Modern
Less than 1 half-life
Between 1 and 2 half-lives
Between 2 and 3 half-lives
Greater than 3 half-lives

2.1.1 Glengoyne Barley Mash, Sample A

Age: Modern

Composition/Provenance: This core sample in the series of TIRI standards comprises a barley grain by-product from the manufacture of malt whiskey.

As a first stage in malt whiskey distillation, barley grains are allowed to sprout to catalyze the conversion of the constituent starch to sugars. This "malted" barley is mixed with water to produce a "mash", which is allowed to ferment. The alcoholic liquor is then separated for multiple distillation, leaving the solid "mash" residue.

A bulk sample of "mash" residue was obtained from Glengoyne Distillery during October 1991 by G T Cook and D D Harkness.

Pretreatment/Preparation: The bulk sample was taken from a single fermentation vat, and therefore, was already very well mixed in the industrial process. The material was immediately force-dried to avoid the possible development of mold growths and was finally subjected to physical mixing.

2.1.2 Belfast Pine, Sample B

Approximate age: 1 half-life

Composition/Provenance: This core sample comprises Scots pine (*Pinus sylvestris*), collected by Prof M Baillie in December 1991. It grew on the western side of Garry Bog, Co. Antrim, and is designated Q7780.

Each sample is a block of 40 rings, representing growth rings 74–113 of the 347-yr-old tree. The samples conform exactly to 2 of the bidecadal samples of oak used in the original high-precision calibration (Pearson and Stuiver 1986). This sample was dendro-dated to 3239–3200 BC.

Pretreatment/Preparation: The material was provided dried and split radially; no further processing was undertaken.

2.1.3 IAEA-Cellulose, Sample C

Activity: 129.41 pMC

Composition/Provenance: A batch of cellulose produced in 1989 from one season's harvest of about 40-yr-old trees was supplied by a paper factory in Bergum, the Netherlands.

This material is Sample C-3 in the IAEA ^{14}C quality assurance materials. The consensus value was 129.41 pMC (with an estimated standard error of 0.06).

Pretreatment/Preparation: The material was provided already packaged and had undergone no further processing.

2.1.4 Hekla Peat, Sample D

Approximate age: less than 1 half-life

Composition/Provenance: Peat was sampled at Svinavatn, North Iceland, in August 1991 with the help of Dr A Dugmore and Mr A Newton. It is associated with a tephra layer corresponding to one of the largest eruptions of the Hekla volcano.

The tephra layer corresponding to the eruption was exposed over a length of 2 m and a depth of approximately 1 m below the overlying vegetation. The tephra layer was then removed and a 1-cm-thick layer of peat lying beneath the tephra was extracted.

Pretreatment/Preparation: The bulk peat was dried at room temperature, ground to a fine powder, and thoroughly mixed.

This material, as provided, contains about 30% by weight of carbon.

2.1.5 Ellanmore Humic Acid, Sample E

Approximate age: between 2 and 3 half-lives

Composition/Provenance: Details for this core sample in the series of TIRI standards are identical to those describing the optional "whole peat" standard (Sample H).

Pretreatment/Preparation: Approximately 5 kg of the dried bulk peat was digested in 0.25M KOH at 80 °C. The alkali extracts were filtered and combined into one volume. The bulk aqueous solution was thoroughly mixed and the humic acids then precipitated by adjusting the solution pH to <3 by the stirred addition of 2M HCl. The solid precipitate was recovered by filtration and given a preliminary wash with cold distilled water. After drying to constant weight, the crystalline humic acid was washed free of chloride inclusions with hot distilled water.

The final product contains about 45% by weight of carbon.

2.1.6 Icelandic Doublespar, Sample F

Approximate age: 0% activity

Composition/Provenance: Iceland spar is a variety of crystalline calcite, its chemical composition is calcium carbonate. It occurs as pure, large, and single crystals concentrated between sheets of basic volcanic lava.

All the material used for TIRI came from the spar-mine at Helgustadir, Iceland, and was provided from the Museum of Natural History, Reykjavik, by Dr S Jakobssen.

Pretreatment/Preparation: Larger crystals provided were broken into smaller pieces and packaged in sealed bags for dispatch.

Samples from the spar-mine had been measured previously by the Radiological Dating Laboratory, NTNU, Trondheim. After removal of the outer 10%, measurements showed no excess activity compared to freshly-cut marble and CO_2 from natural gas. Thus, it is obvious that the crystalline structure provides excellent preservation from contamination during storage (Gulliksen and Thomsen 1992).

2.2 OPTIONAL SAMPLES

2.2.1 Fugla Ness Wood Fragments, Sample G

Approximate age: greater than 4 half-lives

Composition/Provenance: This optional TIRI standard comprises fragments of wood (*Pinus* sp.) recovered from a well-documented bed of *in-situ* peat within glacial deposits.

The Fugla Ness section is exposed on the extreme northwest coast of Mainland Shetland, Scotland (60°30′N, 1°25′W, Natl Grid Ref HU 311 913). The stratigraphy was first described by Chapelhow (1965) as a 1.5-m band of amorphous peat buried beneath 2 tills. On the basis of its pollen and rich macrofossil content, Birks and Ransom (1969) concluded that the peat layer was of interglacial age and with strong Gortian (cf. Hoxnian) affinities. A critical re-evaluation of the pollen-stratigraphic evidence is provided by Lowe (1984).

A bulk sample of wood fragments was collected by fresh excavation of the section during August 1991 by A M Hall, D D Harkness, G Whittington, and N J Alexander.

Pretreatment/Preparation: The wood fragments had been subjected to a preliminary cleaning to discard adhering peat and other soluble organic residues.

The raw sample was soaked in distilled water for several days, digested in 0.5M KOH at 80 °C, and then re-soaked in fresh distilled water. Individual fragments were then scrubbed using a wire brush and digested overnight in hot 2M HCl. The wood was again soaked in several washes of distilled water to remove excess acid, and then dried to constant weight in a vacuum oven.

Further decontamination by either acid/alkali/acid digestion and/or extraction of the component cellulose is strongly recommended prior to any attempt to date this natural material.

2.2.2 Ellanmore Whole Peat, Sample H

Approximate age: between 2 and 3 half-lives

Composition/Provenance: This optional TIRI standard is finely-ground peat from a well-defined stratigraphic section. The Ellanmore peat occurs as an approximately 50-cm-thick horizon intercalated with glacial diamicts and is exposed in a stream bank section of the Reisgill Burn, Ellanmore, Caithness, Scotland (58°18′N, 3°17′W, Natl Grid Ref ND 237 370). The stratigraphical section is described and discussed in detail by Hall and Whittington (1981).

During September 1991, a bulk sample comprising about 10 kg of peat was cut from a freshly cleaned exposure by A M Hall and D D Harkness.

Pretreatment/Preparation: The bulk peat was air-dried at room temperature. Approximately half of the available material was ground to a fine powder and thoroughly mixed to produce an age homogeneous standard.

This material, as provided, contains about 40% by weight of carbon.

2.2.3 Caerwys Quarry Travertine, Sample I

Approximate age: within 1 and 2 half-lives

Composition/Provenance: This optional TIRI standard was available for distribution to those laboratories that had an interest in dating freshwater travertines (tufas) of postglacial origin.

A bulk sample of fresh material (98% Ca CO_3) was collected from a well-documented exposure at Caerwys Quarry, North Wales (Natl Grid Ref 33 129 719), during April/May 1992.

2.2.4 Buiston Crannog Wood, Sample J

Approximate age: less than 1 half-life

Composition/Provenance: This timber, available as an optional TIRI standard, was in the form of a large morticed baulk, lying just behind the outer palisade of Buiston Crannog, near Kilmaurs, Ayrshire, Scotland (55°40′N, 4°18′W, NGR 4154 4351). Although no longer *in situ*, it resembled the morticed planks used to secure the stakes of the outer palisade and is interpreted here as having formed part of the latter. The sample was supplied by Dr B A Crone, Archaeological Operations and Conservation, Fleming House, Newcraighall, Edinburgh.

Pretreatment/Preparation: The samples were cut from a single timber. No chemical treatment had been undertaken.

2.2.5 Turbidite Carbonate (Mainly Coccolith Calcite), Sample K

Approximate age: 3 half-lives

Composition/Provenance: This optional TIRI standard is from a single, distal turbidite emplaced on the Madeira Abyssal Plain, east of Great Meteor Seamount, a few hundred years ago. A remarkable feature of these turbidites is their homogeneity. The basal layers are graded and inhomogeneous, but are overlain by relatively thick, ungraded deposits. These are further overlain by surficial (approximately a 10-cm layer) material which is, again, non-homogeneous. The material used in this study is derived from the middle ungraded deposit. The sample was supplied by Dr J Thomson, Institute of Oceanographic Sciences, Deacon Laboratory, Wormley, England.

Pretreatment/Preparation: On receipt, the sample was immediately oven-dried (~50 °C), ground, and fully homogenized.

2.2.6 Whalebone, Sample L

Approximate age: between 2 and 3 half-lives

Composition/Provenance: This optional TIRI standard comprises sections of whalebone recovered from a complete whale skeleton discovered in Flatanger, Norway. The skeleton has been buried under approximately 2 m of Quaternary till and beach gravel.

The whole skeleton was freshly excavated in March 1992 by Sigmund Alsaker in collaboration with the Geological Survey of Norway (NGU) and the Radiological Dating Laboratory in Trondheim.

The further financial support of the municipality of Flatanger is gratefully acknowledged.

2.2.7 Icelandic Peat, Sample M

Approximate age: less than 1 half-life

Composition/Provenance: This optional TIRI standard comprises peat sampled in August 1991 from Solheimajokull, South Iceland, with the help of Dr A Dugmore and Mr A Newton.

The peat sample was taken from a thin section between 2 tephra layers, at approximately 1 m below the underlying vegetation layer.

Pretreatment/Preparation: The whole peat was dried and ground to a fine powder, then thoroughly mixed. This material, as provided, contains approximately 10% by weight of carbon.

3. RESULTS FOR STAGE 1: CORE SAMPLES

3.1 PARTICIPATING LABORATORIES

A total of 93 sets of samples were dispatched and 67 sets of results were received. A number of laboratories submitted more than 1 set of results, the additional sets of results typically having been produced as a result of collaboration with an accelerator laboratory (target preparation in 1 laboratory, measurement in another). In total, 42 sets of results were produced using liquid scintillation technology (LSC), including 1 by direct CO_2 absorption (CARB), 18 by gas proportional counting (GPC), and 11 by accelerator mass spectrometry (AMS). The list of participating laboratories is shown in Table 3.1 and the full results are given in Appendix 4.

Table 3.1 Laboratories participating in TIRI

Laboratory name	Country	Laboratory type
INRA, Science du Sol	France	LSC
RAWS, Heidelberg	Germany	GPC
Datación por Carbono-14	Spain	LSC
Svedberg Lab, Uppsala University	Sweden	AMS
Rafter Lab, Nuclear Science	New Zealand	AMS
Physical Research Lab	India	GPC
Physical Research Lab	India	LSC
NLB, Radiocarbon Lab	Germany	GPC
LOYDC, Paris	France	LSC
Dating Lab, University of Helsinki	Finland	GPC
Radiocarbon Dating	England	LSC
INAN, University of Louvain	Belgium	GPC
National Museum	Denmark	GPC
Weizmann Institute	Israel	GPC
Institute of Material Culture	Russia	LSC
Institute of Geography	China	LSC
MWG MacIntosh Centre	Australia	LSC
University of California	USA	GPC
University of Texas	USA	LSC
SUERC	Scotland	LSC
Geologie du Quaternaire	France	LSC
ATOMKI	Hungary	GPC
University of Rome	Italy	GPC
AMS lab, Aarhus	Denmark	AMS
CAMS/LLNL	USA	AMS
Techniques Nucleaire	Algeria	LSC
Van de Graaf Lab	Netherlands	AMS
Institute of Zoology and Botany	Estonia	LSC
Saskatchewan Research Council	Canada	LSC
Research Lab for Archaeology	England	AMS
Centre de Datation	France	LSC
Belfast	Ireland	LSC
Kyoto Sangyo University	Japan	LSC
Tallinn ^{14}C Lab	Estonia	LSC
Kraków	Poland	LSC
Illinois Geological Survey	USA	LSC
Ruđer Bošković Institute	Croatia	GPC

Table 3.1 Laboratories participating in TIRI *(Continued)*

Laboratory name	Country	Laboratory type
ICEN/LNETI	Portugal	LSC
National Taiwan University	Taiwan	LSC
LATYR	Argentina	LSC
Bhabha Atomic Research	India	LSC
CRAD	Italy	LSC
UFZ	Germany	LSC
Institut für Radiumforschung	Austria	GPC
Department of Geography	Wales	GPC
Japan Radiosiotope	Japan	GPC
Geographical Institute	Russia	LSC
Atomic Energy for peace	Thailand	LSC
Palaeoclimatologie im WIP	Germany	LSC
CSIRO	Australia	CARB
Department of Geosciences	USA	LSC
Scienze della Terra	Italy	LSC
Institut für Kernphysik	Germany	GPC
Bergakademie	Germany	LSC
WHOI	USA	AMS
DAI	Germany	GPC
University of Rome	Italy	LSC
NERC ^{14}C Lab	Scotland	LSC
Radiologisk Datering	Norway	GPC
Beta Analytic	USA	LSC/AMS
WHOI	USA	GPC
British Museum	England	LSC
SMU	USA	LSC
Radiologisk Datering	Norway	AMS
University of Barcelona	Spain	LSC
NSF-Arizona AMS	USA	AMS
University of Waikato	New Zealand	LSC
Geological Survey of Canada	Canada	GPC

3.2 SUMMARY STATISTICS

The summary statistics for each sample are presented below and follow a common pattern:

a) Boxplots for $\delta^{14}C$, $\Delta^{14}C$, and %Modern (pMC) or age, are shown. Such diagrams show the overall distribution of results, indicating (by the central box) the middle 50% of the data (the interquartile range, IQR), the extremes (minimum and maximum), and any outlying observations (indicated by * and 0 on the diagrams).

b) A numerical summary of the results is given in an accompanying table. *N* indicates the number of observations. The mean and median give estimates of the central value (used as the consensus) and the quartiles Q1 and Q3 give the range within which the mid-50% of the data lie.

c) The presentation of the results by laboratories was quite varied; sometimes only age was reported, on other occasions $\delta^{14}C$, $\Delta^{14}C$, and age were given. In the summary tables, we have based the calculations on all the results for a particular quantity, including results on different sub-samples of the same sample. Thus, in some of the tables, the number of results being summarized exceed the number of laboratories that participated.

For Sample F, we have provided several tables since there was an additional complication that results were often censored (reported in the form of a "greater than" age).

3.2.1 Statistical Summary for TIRI-A: Glengoyne Barley Mash

Figure 3.1 Distribution of results for Barley mash TIRI-A

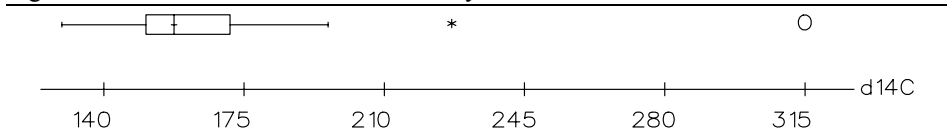

Figure 3.1a Boxplot for $d^{14}C$

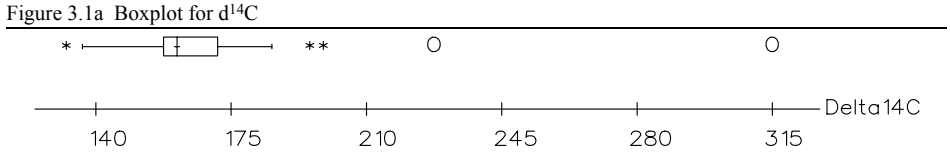

Figure 3.1b Boxplot for $D^{14}C$

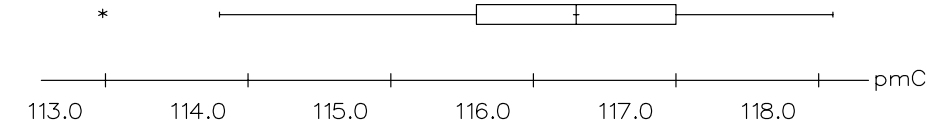

Figure 3.1c Boxplot for pMC

Table 3.2 Numerical summary for TIRI-A

	N	Mean	Median	Q1	Q3
$d^{14}C$	50	165.06	160.18	151.80	171.57
$D^{14}C$	62	167.91	164.14	157.88	172.20
pMC	25	116.12	116.35	115.30	117.08

Comments: The mean activity is 116.12 pMC, with an interquartile range of 1.78.

3.2.2 Statistical Summary of Results for TIRI-B: Belfast Pine

Figure 3.2 Distribution of results for Belfast pine TIRI-B

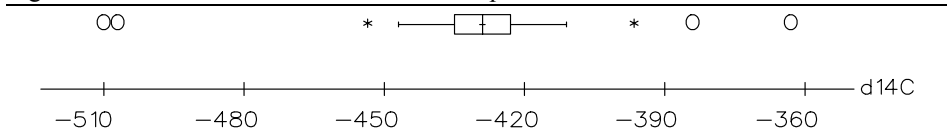

Figure 3.2a Boxplot for $d^{14}C$

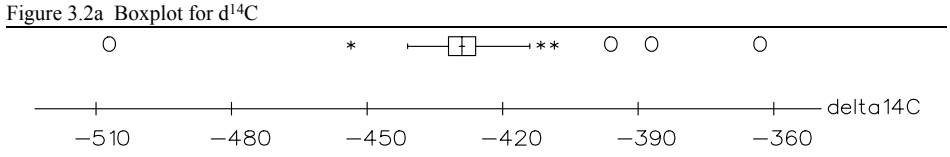

Figure 3.2b Boxplot for $D^{14}C$

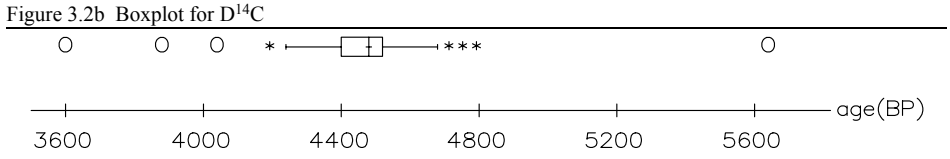

Figure 3.2c Boxplot for age (yr BP)

Table 3.3 Numerical summary for TIRI-B

	N	Mean	Median	Q1	Q3
$d^{14}C$	47	–428.52	–427.30	–434.17	–421.79
$D^{14}C$	63	–426.93	–428.96	–434.40	–423.10
Age	78	4485	4500	4420	4540

Comments: The mean ^{14}C age is 4485 BP (the "expected" age from the dendro-dates is approximately 4495 BP). The IQR is 140 yr.

3.2.3 Statistical Summary for TIRI-C: IAEA-Cellulose

Figure 3.3 Boxplots for TIRI-C: IAEA-cellulose

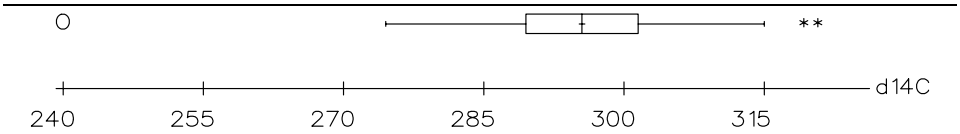

Figure 3.3a Boxplot for $d^{14}C$

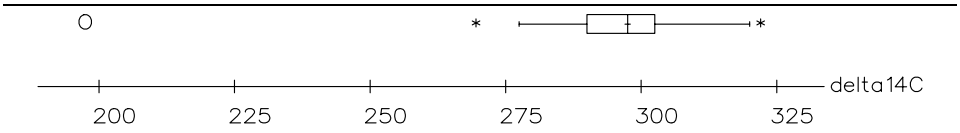

Figure 3.3b Boxplot for $D^{14}C$

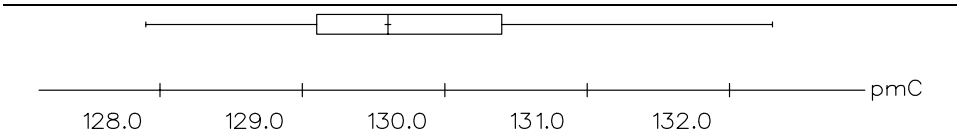

Figure 3.3c Boxplot for pMC

Table 3.4 Numerical summary for TIRI-C

	N	Mean	Median	Q1	Q3
$d^{14}C$	45	295.42	295.50	290.2	302.1
$D^{14}C$	58	295.98	297.35	291.3	303.6
pMC	25	129.81	129.60	129.1	130.5

Comments: The mean activity is 129.81 pMC, compared with the IAEA reference value of 129.41. The IQR is 1.4.

3.2.4 Statistical Summary of TIRI D: Hekla Peat

Figure 3.4 Distribution of results TIRI D: Hekla peat

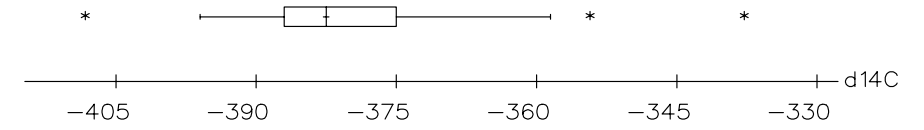

Figure 3.4a Boxplot for $d^{14}C$

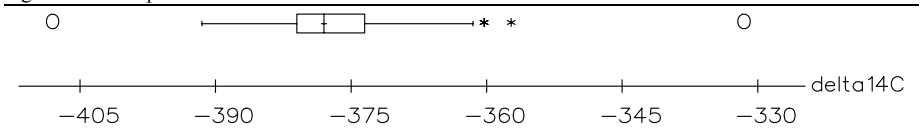

Figure 3.4b Boxplot for $D^{14}C$

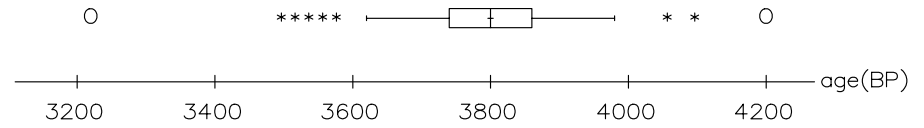

Figure 3.4c Boxplot for age (yr BP)

Table 3.5 Numerical summary for TIRI-D

	N	Mean	Median	Q1	Q3
$d^{14}C$	46	–379.25	–381.25	–386.4	–373.6
$D^{14}C$	60	–376.25	–377.25	–380.9	–372.6
Age (BP)	72	3799	3805	3752	3865

Comments: The mean is 3799 yr BP, with an IQR of 113 yr BP. A number of outliers are identified.

3.2.5 Statistical Summary for TIRI E: Ellanmore Humic

Figure 3.5 Statistical summary for TIRI E: Ellanmore humic

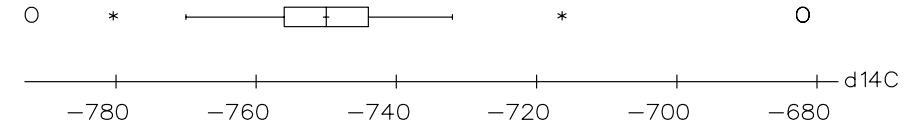

Figure 3.5a Boxplot for $d^{14}C$

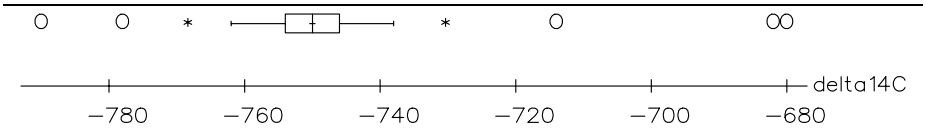

Figure 3.5b Boxplot for $D^{14}C$

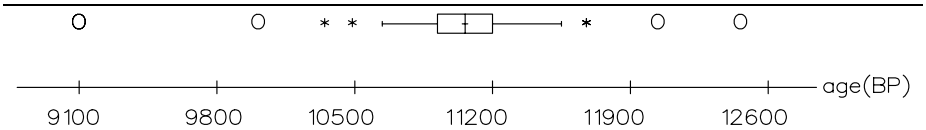

Figure 3.5c Boxplot for age (yr BP)

Table 3.6 Numerical summary for TIRI-E

	N	Mean	Median	Q1	Q3
$d^{14}C$	43	–747.7	–749.5	–755.5	–743.0
$D^{14}C$	56	–747.0	–748.6	–752.2	–745.2
Age (BP)	68	11,066	11,105	10,965	11,240

Comments: The mean age is 11,066 yr BP, with an IQR of 175 yr. A number of outliers are apparent.

3.2.6 Statistical Summary for TIRI F: Icelandic Doublespar

Figure 3.6 Distribution of results for TIRI F: Icelandic doublespar

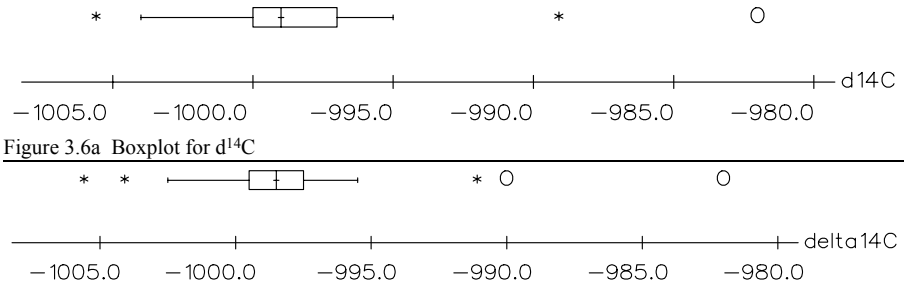

Figure 3.6a Boxplot for $d^{14}C$

Figure 3.6b Boxplot for $D^{14}C$

Table 3.7 Numerical summary for TIRI-F

	N	Mean	Median	Q1	Q3
$d^{14}C$	33	–997.9	–998.6	–1000.0	–996.6
$D^{14}C$	51	–997.9	–998.2	–999.5	–997.0

Comments: For this sample, 21 results were simply classified as "background" and 19 results were given in the form of a finite age. These results are summarized below:

	Mean	Median	Q1	Q3
Age (BP)	48,198	49,030	44,160	52,106

Twenty-two results were given in the form of >age (BP). The ages are summarized below:

	Mean	Median	Q1	Q3
> Age (BP)	46,076	46,150	39,450	53,400

3.3 PRELIMINARY CONCLUSIONS

1. For the 2 modern samples, A and C, we found that a number of laboratories calculated a slightly different form for pMC (by incorporating an allowance for decay). To ensure that all results are directly comparable, we asked labs to confirm their results without decay correction.
2. For Sample A, it is clear that there are a number of outlying observations. The preliminary consensus value is 116.35 pMC.
3. For Samples B, D, and E, there are no obvious computational problems. A few outlying observations are apparent. Consensus values are 4500 BP, 3805 BP, and 11,105 BP,

respectively. For Sample B, the known-age sample, the known ^{14}C age is 4495 BP, so the results are in good agreement.

4. For Sample C, a few outlying observations are apparent. The consensus value is 129.6, which is slightly higher than the IAEA value of 129.4.
5. For Sample F, the results have proved particularly interesting. This material was selected to function as a background sample. A relatively large number of laboratories reported a finite age for this sample, indicating a statistically significant ^{14}C count rate relative to their accepted background.
   Other laboratories simply stated that the sample was background, while others gave their result in the form of >age (BP). Generally speaking, the consensus value would indicate an age greater than 46,150 yr BP.
6. Given the diversity of form of results for this sample, perhaps there is a need for careful consideration of the limiting age calculations. For almost all samples, outliers or extreme values have been graphically identified.

3.4 CONSENSUS VALUES

Consensus values for each sample were evaluated using the same method used in the characterization of the IAEA reference samples (Rozanski et al. 1992). Briefly, a preliminary robust consensus value (rcv) was evaluated (the median of all the results with identified outliers removed) for each of the samples. To evaluate the final consensus value, the standardized difference (sd) between the robust consensus value (rcv) and each result is calculated (sd = [result–rcv] / quoted error). If the standardized difference exceeds 2, then that result is not used in any subsequent calculation. In this way, results that do not lie within ±2 quoted errors of the robust consensus value are removed. The final consensus value is calculated as a weighted average of the remaining results.

The following tables show the consensus values for the core samples, evaluated using the criterion stated above.

Table 3.8 Consensus values for core samples

Sample	Consensus value	Estimated precision (1 σ)
A: barley mash	116.35 pMC	0.0084
B: Belfast pine	4503 BP	6
C: IAEA cellulose	129.7 pMC	0.08
D: Hekla peat	3810 BP	7
E: Ellanmore humic	11,129 BP	12
F: Icelandic doublespar (BP)	46,750 BP	208
F: Icelandic doublespar (pMC)	0.18 pMC	0.006

For each sample, a number of outliers were removed (up to a maximum of 10, but more typically less than 5). When the consensus value was calculated, results were also omitted due to the ±2 σ criterion not being satisfied.

The results are presented graphically in Figures 3.7–3.13, where plots for each core sample show the results for the individual laboratories and their differences from the consensus values. The vertical bars represent ±2 quoted uncertainties. Where a laboratory has not quoted an uncertainty (e.g., for Sample F), the result is shown without bars. We would expect that the results should be scattered around zero and this is the case. The figures also show the variation in the quoted errors among laboratories.

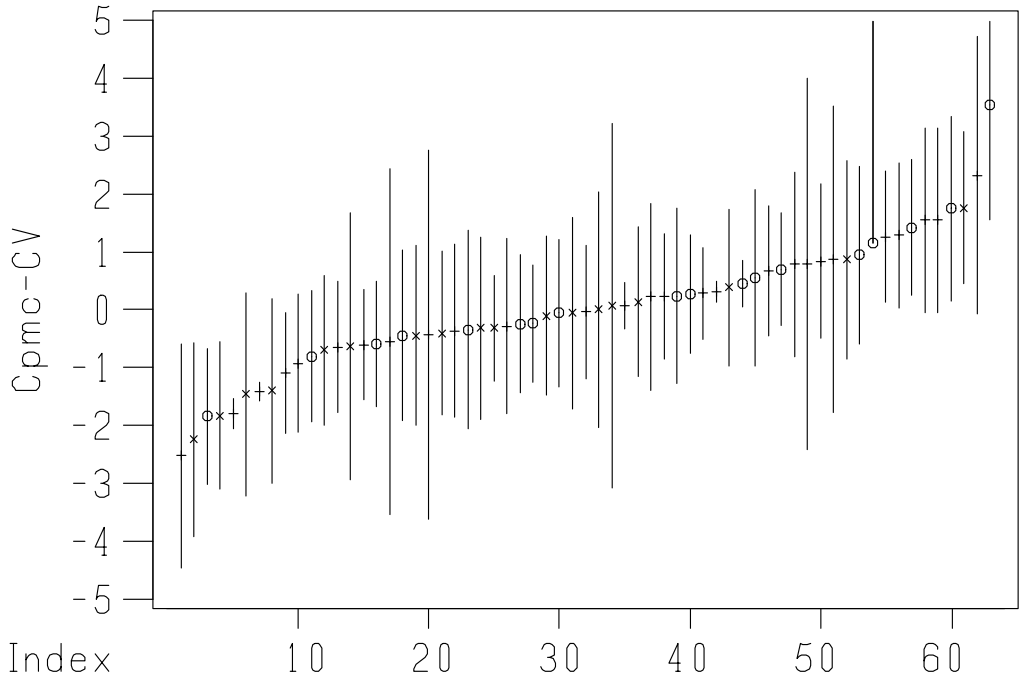

Figure 3.7 Sample A: Glengoyne barley mash

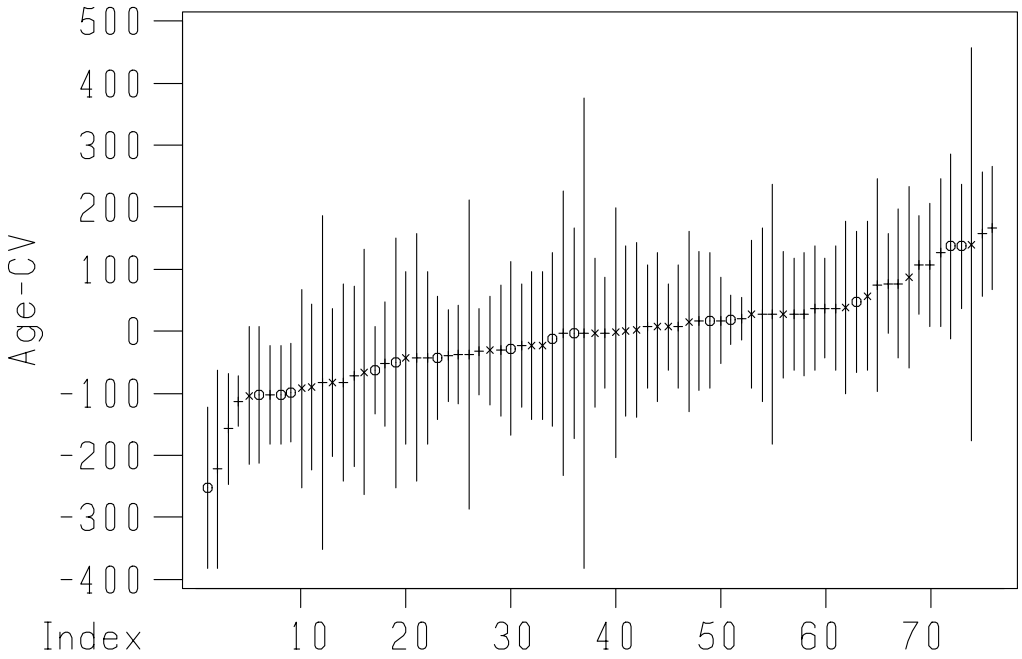

Figure 3.8 Sample B: Belfast wood

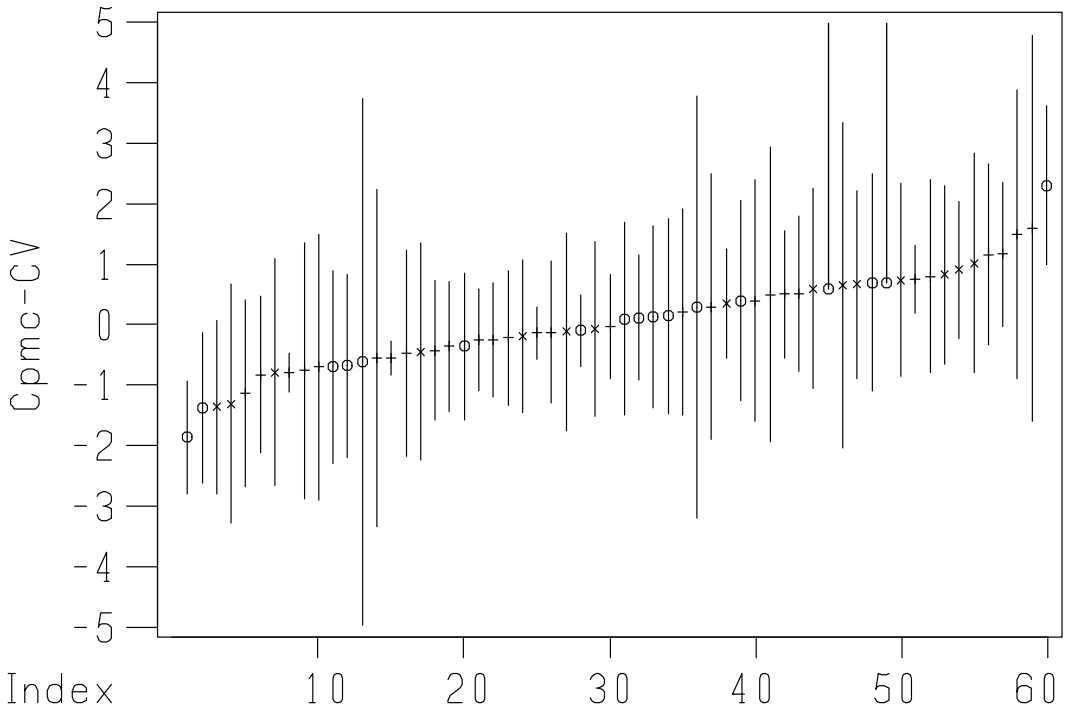

Figure 3.9 Sample C: IAEA cellulose

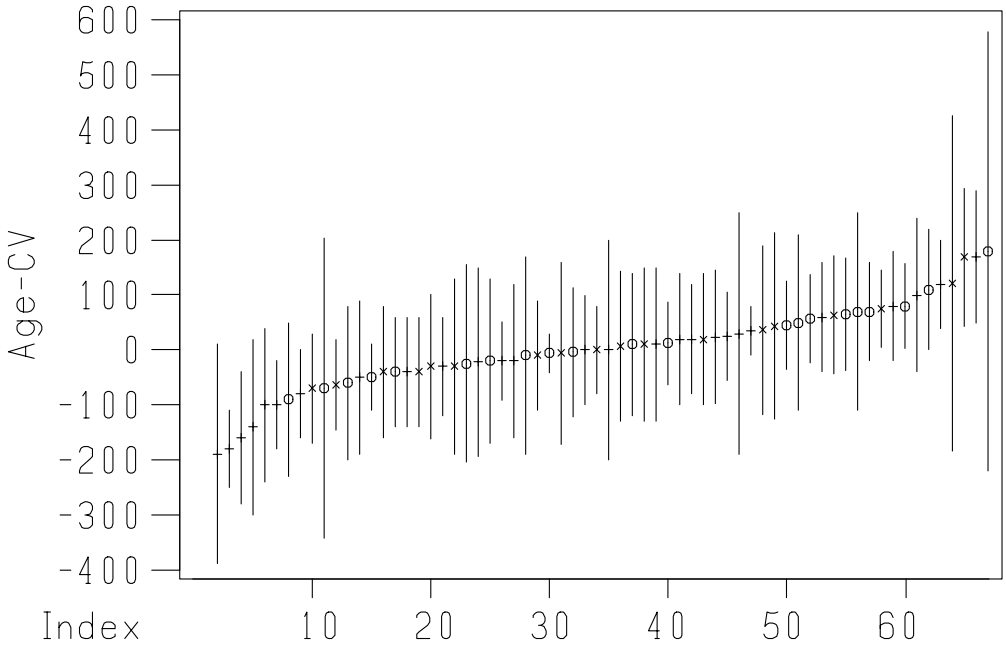

Figure 3.10 Sample D: Hekla peat

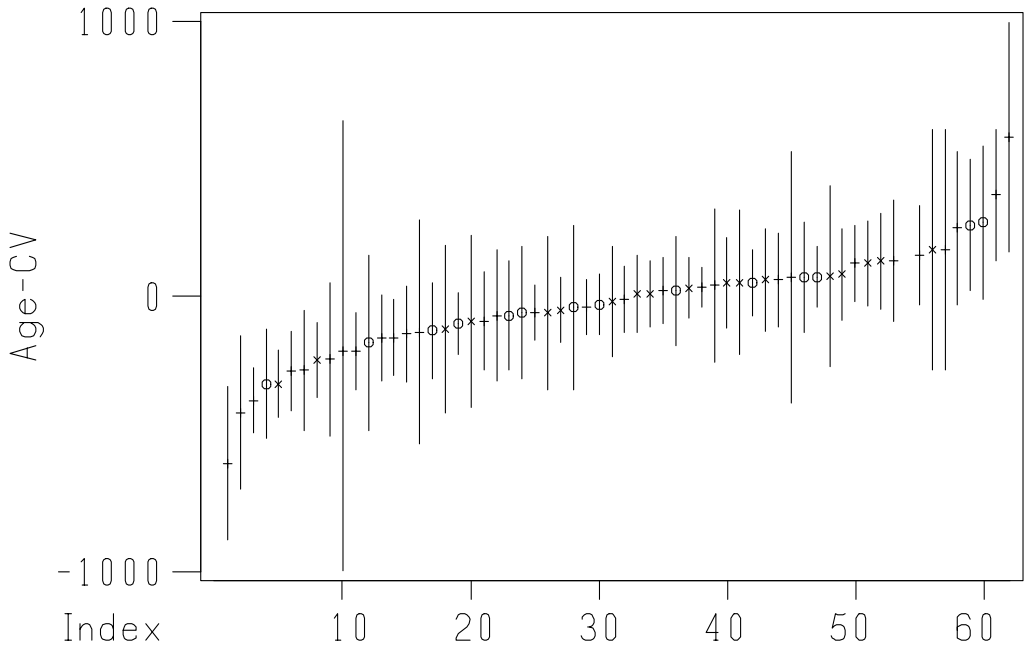

Figure 3.11 Sample E: Ellanmore humic

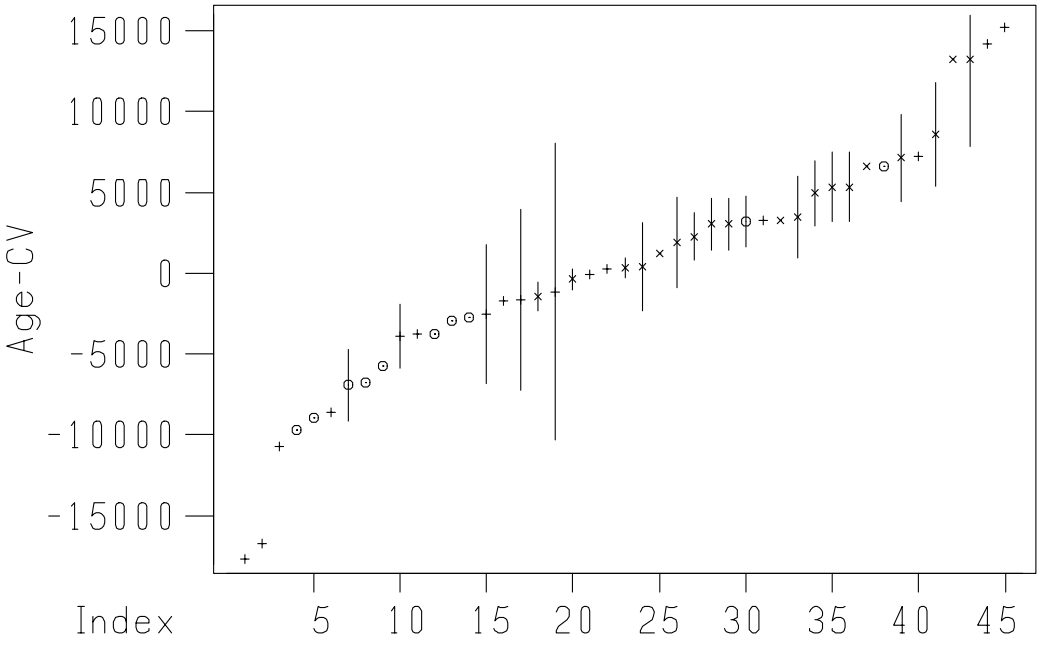

Figure 3.12 Sample F: Icelandic doublespar (yr BP)

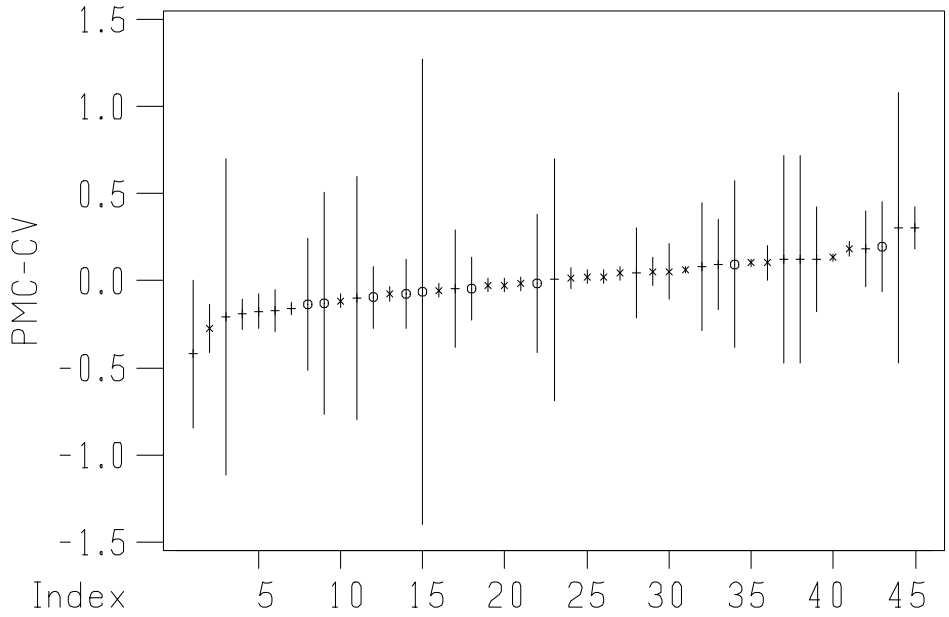

Figure 3.13 Sample F: Icelandic doublespar (pMC)

Further, to allow a comparison of the scatter of results for the different samples, deviations have been calculated, where the deviation is defined as:

Deviation = (laboratory result – consensus value) / quoted uncertainty

We would anticipate that deviations should generally lie between ±2, (normal counting statistics). Figures 3.14–3.19 show the deviations for the 6 core samples for LSC, GPC, and AMS labs. In the main, the results are very tight, but we do see some evidence of wider scatter in Sample F for LSC and AMS labs.

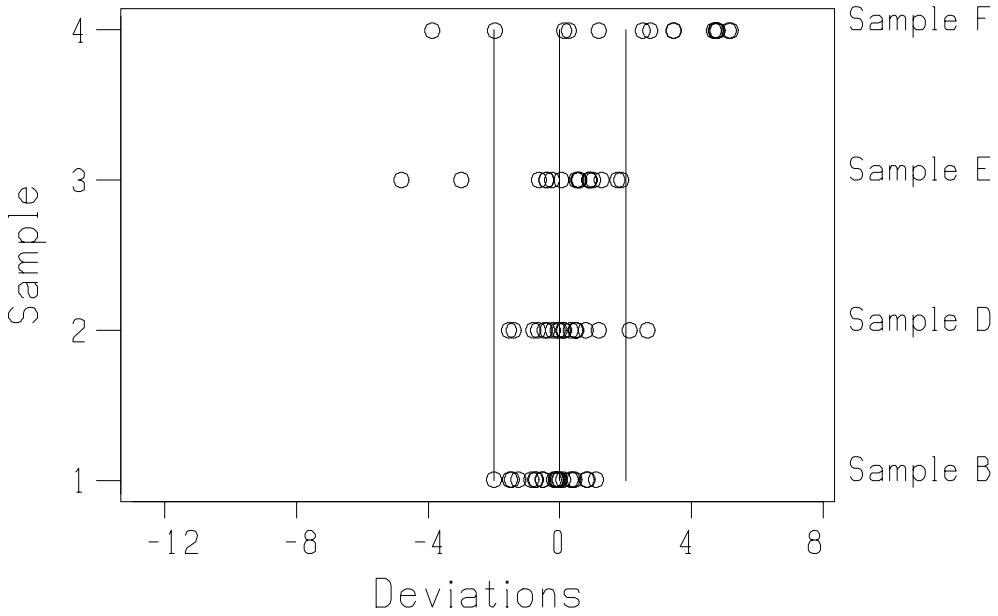

Figure 3.14 Age deviations for AMS Laboratories by sample

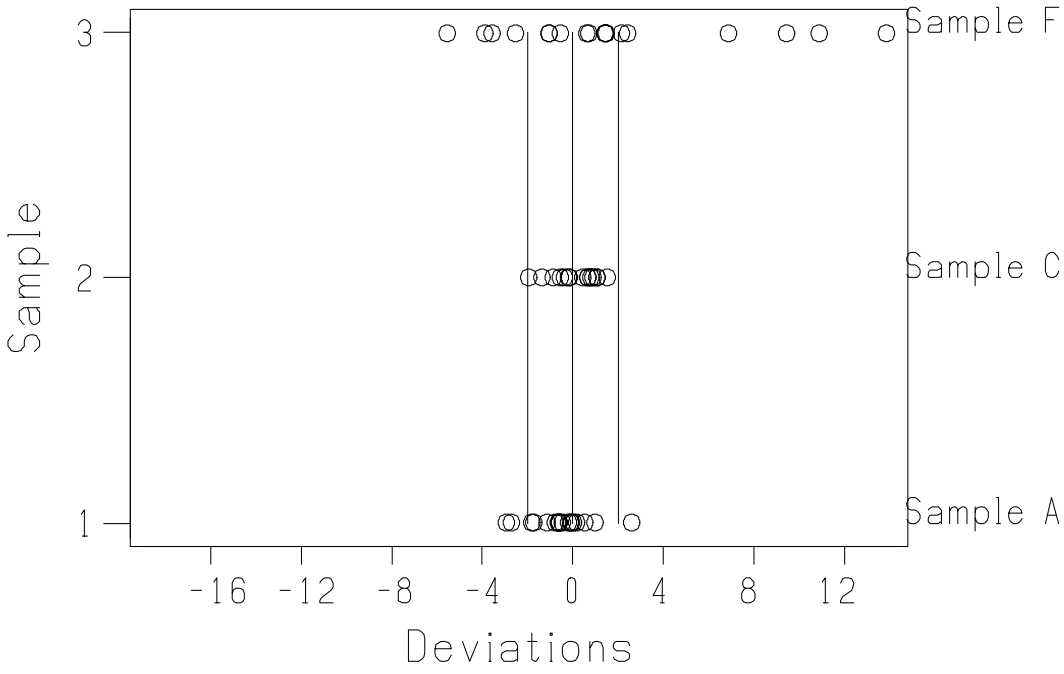

Figure 3.15 pMC deviations for AMS Laboratories by sample

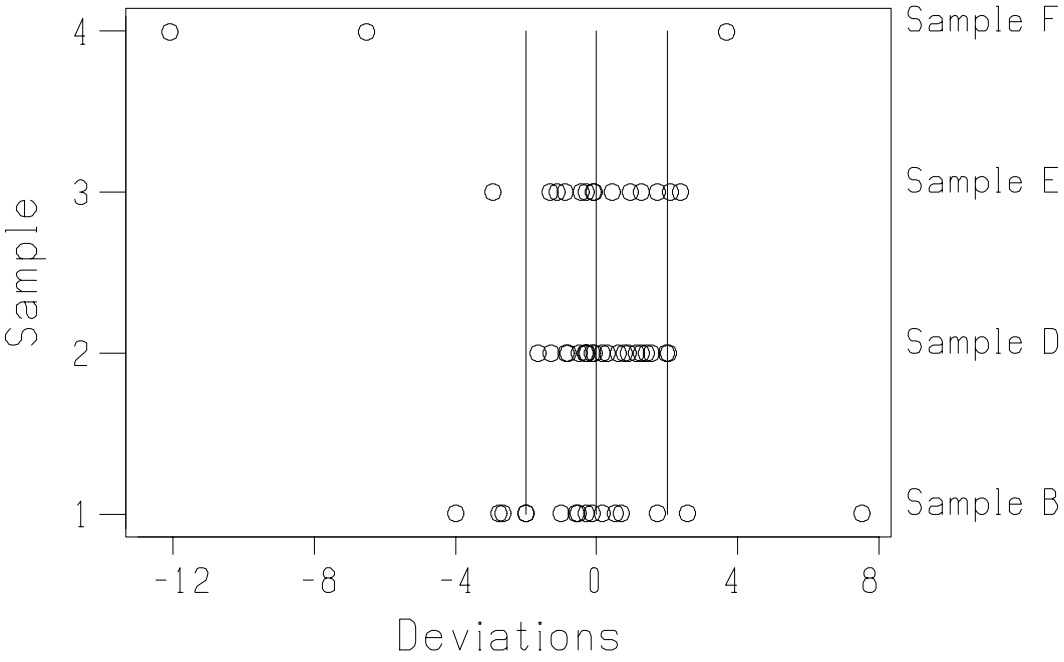

Figure 3.16 Age deviations for GPC laboratories by sample

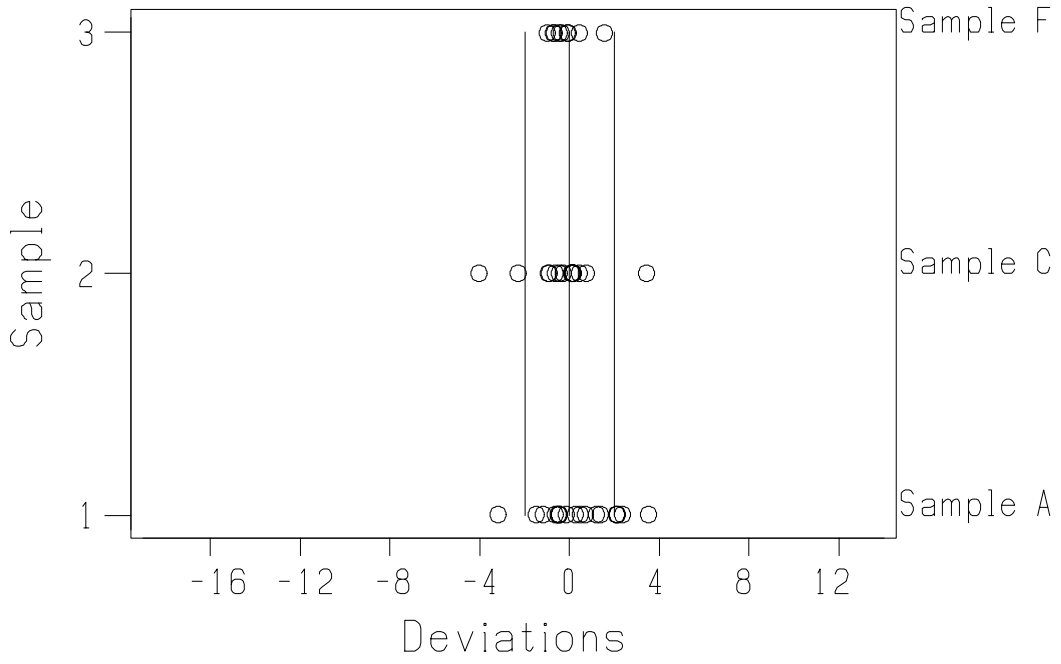

Figure 3.17 pMC deviations for GPC laboratories by sample

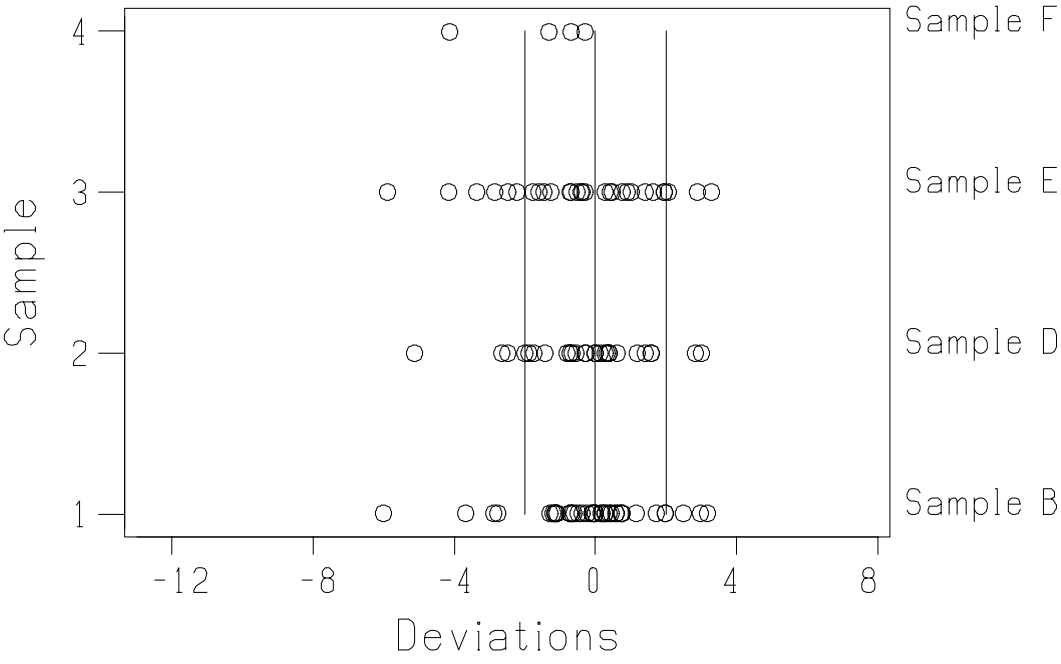

Figure 3.18 Age deviations for LSC laboratories by sample

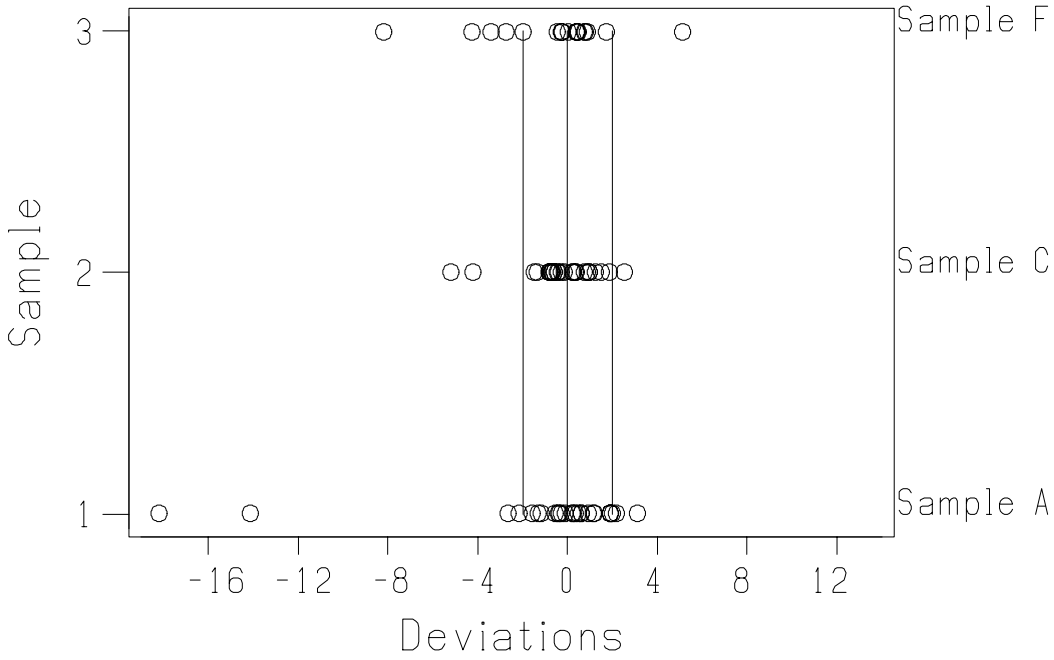

Figure 3.19 pMC deviations for LSC laboratories by sample

4. RESULTS FOR STAGE 2: OPTIONAL SAMPLES

4.1 PARTICIPATING LABORATORIES

In the second stage of TIRI, a number of optional samples were available and participating labs selected those most appropriate to their dating practices. Seven samples were available and are listed below. Results from a total of 40 laboratories were received for TIRI Stage 2 samples (11 GPC, 25 LSC, 3 AMS, and 1 lab using CO_2 absorption). The full results are available in Appendix 4.

Table 4.1 Laboratories participating in Stage 2 of TIRI

Laboratory name	Country	Laboratory type
Datación por Carbono-14	Spain	LSC
Physical Research Lab	India	LSC
NLB, Radiocarbon Lab	Germany	GPC
Radiocarbon Dating	England	LSC
National Museum	Denmark	GPC
Weizmann Institute	Israel	GPC
Institute of Material Culture	Russia	LSC
University of California	USA	GPC
University of Texas	USA	LSC
SUERC	Scotland	LSC
ATOMKI	Hungary	GPC
University of Rome	Italy	GPC
Institute of Zoology and Botany	Estonia	LSC
Saskatchewan Research Council	Canada	LSC
Research Lab for Archaeology	England	AMS

Table 4.1 Laboratories participating in Stage 2 of TIRI *(Continued)*

Laboratory name	Country	Laboratory type
Centre de Datation	France	LSC
Kyoto Sangyo University	Japan	LSC
Tallinn ^{14}C lab	Estonia	LSC
Illinois Geological Survey	USA	LSC
Ruđer Bošković Institute	Croatia	GPC
ICEN/LNETI	Portugal	LSC
National Taiwan University	Taiwan	LSC
LATYR	Argentina	LSC
Bhabha Atomic Research	India	LSC
CRAD	Italy	LSC
UFZ	Germany	LSC
Department of Geology	Wales	GPC
Geographical Institute	Russia	LSC
Palaeoclimatologie im WIP	Germany	LSC
CSIRO	Australia	CARB
Department of Geosciences	USA	LSC
Scienze della Terra	Italy	LSC
Institut für Kernphysik	Germany	GPC
DAI	Germany	GPC
University of Rome	Italy	LSC
NERC ^{14}C lab	Scotland	LSC
University of Barcelona	Spain	LSC
NSF, Arizona AMS	USA	AMS
Geological Survey of Canada	Canada	GPC
University of Waikato	New Zealand	LSC

Table 4.2 Optional samples

Sample description	Expected age
G: Fuglaness wood	greater than 4 half-lives
H: Ellanmore whole peat	between 2 and 3 half-lives
I: travertine	between 1 and 2 half-lives
J: Crannog wood	less than 1 half-life
K: turbidite carbonate	approximately 3 half-lives
L: whalebone	between 2 and 3 half-lives
M: Icelandic peat	less than 1 half-life

4.3 SUMMARY STATISTICS

The individual statistical summaries of the results for each sample are given in the following. The summaries used are the mean and median (the average value); the standard deviation (a measure of the scatter in the results), denoted *Stdev*; the standard error of the mean (the precision of the average), denoted *Semean*; the minimum and maximum results; and the lower and upper quartiles (the middle-50% range of the data), denoted *Q1* and *Q3*. The results for the age have also been summarized graphically using a boxplot (described in Section 3 on the core samples). A number of "outlying" observations are also indicated (marked by *); although at this stage, these results have not been further investigated nor removed from the calculations.

4.3.1 Sample G: Fugla Ness Wood Fragments

The expected age of this sample was greater than 4 half-lives, the sample having been recovered from a peat bed within glacial deposits.

Thirteen laboratories reported a finite age for the sample, 18 laboratories quoted results in the form of "greater than," and 7 simply gave their result as "background."

The results are summarized in Table 4.3.

Table 4.3a Summary of finite ages

Age	N	Mean	Median	StDev	Semean	Min	Max	Q1	Q3
	13	41,372	42,710	5273	1463	31,800	50,510	37,460	45,450

Table 4.3b Summary for censored values

Age	N	Mean	Median	StDev	Semean	Min	Max	Q1	Q3
	18	42,962	40,918	5826	1373	35,000	54,025	39,500	47,750

Table 4.3c Summary of other measurement information

	N	N*	Mean	Median	StDev	Semean	Min	Max	Q1	Q3
$\delta^{13}C$	35	3	–26.518	–26.680	1.122	0.190	–28.060	–23.500	–27.520	–25.900
$\delta^{14}C$	28	10	–996.20	–996.75	3.86	0.73	–1000.50	–981.70	–998.50	–995.16
$\Delta^{14}C$	28	10	–995.39	–996.52	4.72	0.89	–1000.50	–980.80	–998.47	–993.57

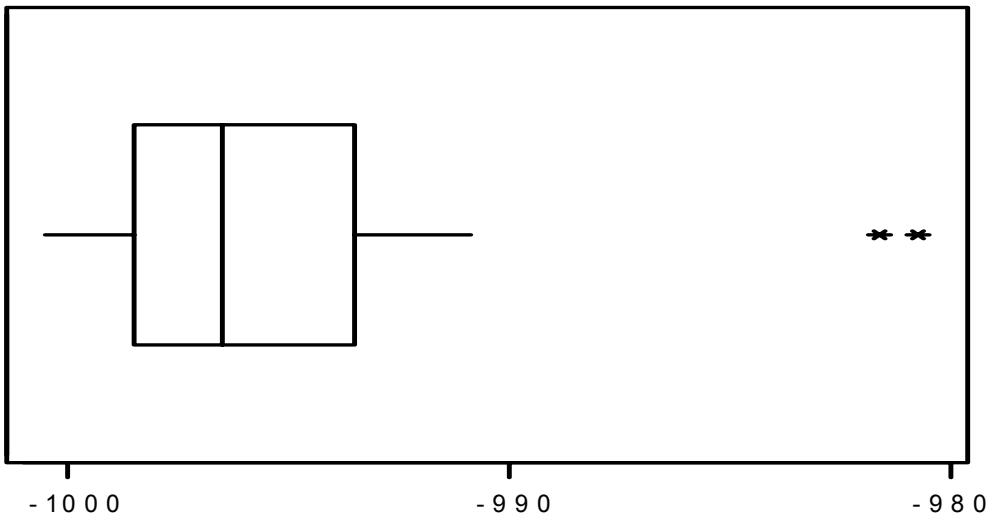

Figure 4.1 Distribution of $\Delta^{14}C$ for Sample G

4.3.2 Sample H: Ellanmore Whole Peat (Raw Material of Sample E in Stage 1)

The expected age for this sample is between 2 and 3 half-lives. In the earlier stage, the humic acid extract from the bulk was supplied as Sample E. The previous mean result was 11,066 yr BP.

Table 4.4 Summary statistics for Sample H

	N	N*	Mean	Median	StDev
$\delta^{13}C$	33	2	–28.392	–28.600	0.679
$\delta^{14}C$	24	11	–749.25	–749.53	9.95
$\Delta^{14}C$	32	3	–749.41	–749.99	9.45
Age	35	0	11,115	11,130	311
Error	35	0	115.5	100.0	90.9

	Semean	Min	Max	Q1	Q3
$\delta^{13}C$	0.118	–29.200	–26.200	–28.800	–28.050
$\delta^{14}C$	2.03	–772.90	–723.90	–756.00	–745.04
$\Delta^{14}C$	1.67	–771.50	–722.10	–754.65	–744.27
Age	53	10,280	11,860	10,915	11,300
Error	15.4	25.0	580.0	70.0	140.0

From the table, it can be seen that the range of results is approximately 1000 yr and that the mean age is 11,115 yr. The middle 50% of the data lie between 10,915 and 11,300 yr, a span of approximately 380 yr. A 95% confidence interval for the "true" age is 11,008–11,221 yr BP.

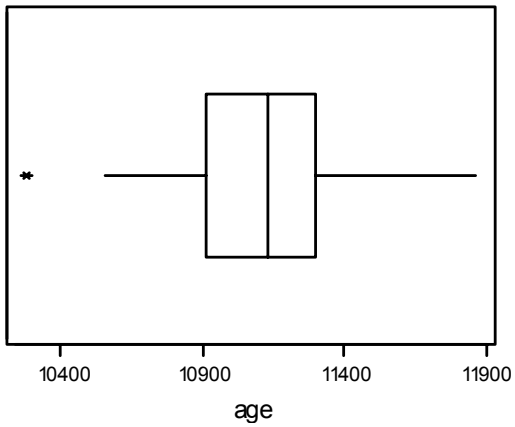

Figure 4.2 Distribution of age for Sample H

4.3.3 Sample I: Travertine

This sample had an expected age between 1 and 2 half-lives

Table 4.5 Summary statistics for Sample I

	N	N*	Mean	Median	StDev
$\delta^{13}C$	32	2	–9.556	–9.900	1.224
$\delta^{14}C$	24	10	–740.42	–740.06	5.82
$\Delta^{14}C$	31	3	–747.09	–748.00	8.51
Age	34	0	11,034	11,073	276
Error	34	0	126.9	100.0	114.1

	Semean	Min	Max	Q1	Q3
$\delta^{13}C$	0.216	–10.700	–4.100	–9.958	–9.690
$\delta^{14}C$	1.19	–755.14	–730.40	–741.82	–736.30
$\Delta^{14}C$	1.53	–762.45	–711.92	–750.40	–743.90
Age	47	9990	11,550	10,931	11,144
Error	19.6	35.0	570.0	70.8	132.5

The average age is 11,034 yr BP, with a range of 1500 yr based on 34 results. The middle 50% of the data lie in a range 10,931–11,144, a span of approximately 250 yr. A 95% confidence interval for the true age is 10,937–11,130.

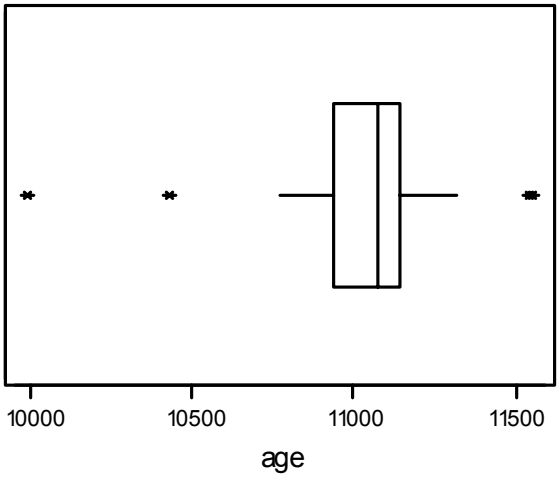

Figure 4.3 Distribution of age for TIRI-I

Three observations are highlighted as extreme, but it is clear that the middle 50% range is relatively tight.

4.3.4 Sample J: Wood, Expected Age Less Than 1 Half-Life

Table 4.6 Summary statistics

	N	N*	Mean	Median	StDev
$\delta^{13}C$	33	3	–26.579	–26.800	1.147
$\delta^{14}C$	26	10	–184.03	–185.50	12.88
$\Delta^{14}C$	31	5	–178.89	–179.80	12.66
Age	36	0	1593.0	1597.5	119.1
Error	36	0	49.97	45.00	18.57

	Semean	Min	Max	Q1	Q3
$\delta^{13}C$	0.200	–28.200	–22.490	–27.400	–25.975
$\delta^{14}C$	2.53	–211.88	–149.70	–189.93	–175.82
$\Delta^{14}C$	2.27	–209.91	–147.10	–186.60	–172.00
Age	19.9	1315.0	1890.0	1522.5	1660.0
Error	3.09	10.00	82.00	37.75	65.75

The average age is 1593 yr, with the range of results approximately 500 yr. The mid-50% span is 1522–1660, a spread of 140 yr. A 95% confidence interval for the true age is 1553–1633.

The boxplot shows a highly symmetrical distribution with 2 extreme observations (1 low, 1 high).

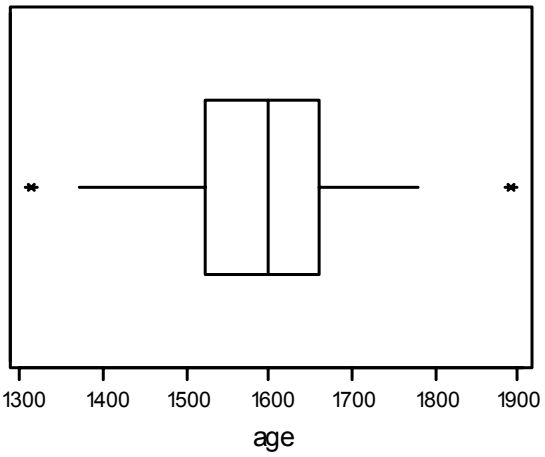

Figure 4.4 Distribution of age for TIRI-J

4.3.4 Sample K: Turbidite Carbonate, Expected Age of 3 Half-Lives

Table 4.7 Summary statistics for Sample K

	N	N*	Mean	Median	StDev
$\delta^{13}C$	28	2	1.321	1.100	1.260
$\delta^{14}C$	22	8	–890.57	–890.35	5.21
$\Delta^{14}C$	27	32	–895.8	–895.3	10.8
Age	30	0	18,166	18,147	928
Error	30	0	237.8	150.0	360.7

	Semean	Min	Max	Q1	Q3
$\delta^{13}C$	0.238	0.000	7.300	0.863	1.475
$\delta^{14}C$	1.11	–898.90	876.30	–894.78	–887.85
$\Delta^{14}C$	2.1	–933.1	–863.3	–899.9	–892.9
Age	169	15,980	21,700	17,986	18,522
Error	65.9	80.0	2100.0	110.0	216.2

The average age is 18,166 yr BP, with an observed range of approximately 5000 yr in the results. The mid-50% lies in the range of 17,986–18,522, a span of just over 500 yr. A 95% confidence interval for the true age is 17,820–18,513 BP.

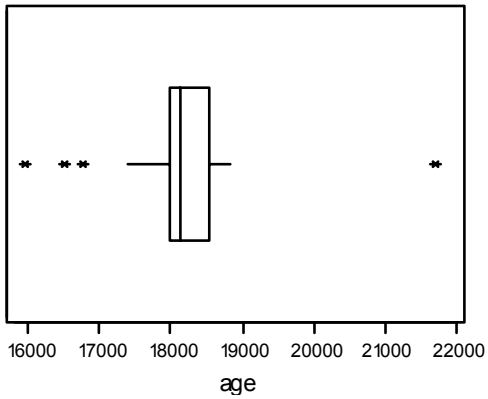

Figure 4.5 Distribution of age for TIRI-K

The boxplot identifies several extreme observations.

4.3.5 Sample L: Whalebone, Expected Age Between 2 and 3 Half-Lives

Table 4.8 Summary statistics for Sample L

	N	N*	Mean	Median	StDev
$\delta^{13}C$	21	2	–15.06	–14.770	1.602
$\delta^{14}C$	17	6	–789.04	–789.10	9.18
$\Delta^{14}C$	18	5	–792.61	–793.25	8.48
Age	23	0	12,605	12,680	449
Error	23	0	127.5	110.0	72.1

	Semean	Min	Max	Q1	Q3
$\delta^{13}C$	0.35	–19.400	–13.200	–15.305	–14.15
$\delta^{14}C$	2.23	–800.00	–762.00	–795.68	–786.15
$\Delta^{14}C$	2.00	–804.00	–767.00	–799.02	–789.90
Age	94	11,050	13,091	12,580	12,900
Error	15.0	40.0	310.0	70.0	154.0

The average age is 12,600 yr BP and the full spread of results is 2000 yr. The mid-50% of the data lie in the range of 12,580–12,900, a span of 320 yr. A 95% confidence interval for the true age is 12,410–12,799 BP.

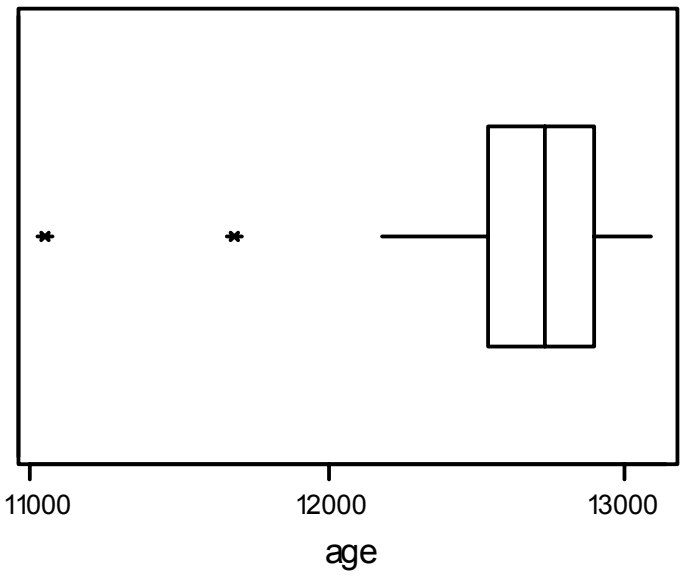

Figure 4.6 Distribution of age for TIRI-L

Two low values are identified under 12,000 yr BP.

4.3.6 Sample M: Peat, Expected Age Less Than 1 Half-Life

Table 4.9 Summary statistics for Sample M

	N	N*	Mean	Median	StDev
$\delta^{13}C$	28	2	–28.178	–28.150	0.841
$\delta^{14}C$	22	8	–212.07	–196.95	41.05
$\Delta^{14}C$	27	3	–203.45	–189.00	39.04
Age	29	1	1842.8	1710.0	408.9
Error	29	1	83.6	60.0	63.7

	Semean	Min	Max	Q1	Q3
$\delta^{13}C$	0.159	–29.800	–26.600	–28.792	–27.800
$\delta^{14}C$	8.75	–361.38	–169.40	–219.94	–189.50
$\Delta^{14}C$	7.51	–358.79	–165.20	–212.43	–184.50
Age	75.9	1448.0	3570.0	1642.5	1920.0
Error	11.8	30.0	250.0	44.0	95.0

The average age is 1842 yr BP, the spread of results is 2000 yr, with the mid-50% lying between 1642–1920, a span of 300 yr. The 95% confidence interval for the true age is 1687–1998 BP.

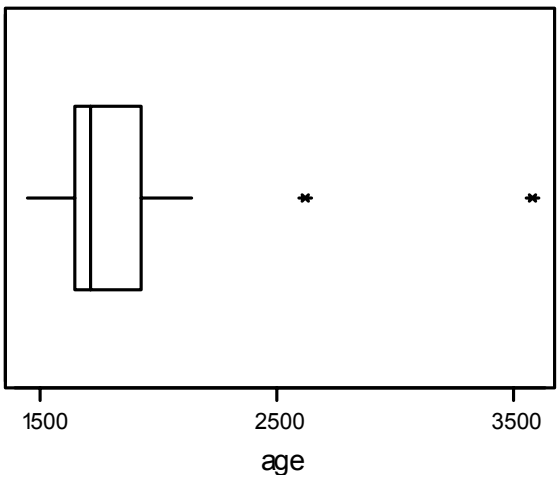

Figure 4.7 Distribution of age for TIRI-M

Two extreme observations over 2500 yr are identified and contribute to the very large range observed.

4.4 ANALYSIS AND CONCLUSIONS

If we calculate the coefficient of variation (defined as StDev / Mean × 100), then we gain an impression of the variability in the results relative to the mean. In this way, we can also compare more directly the results for the different samples.

Table 4.10 Material coefficient of variations

Sample	Material	Mean (BP)	Span of results	Mid-50% span	CV(%)
G	wood	41,372	18,710	7990	12.7
H	whole peat	11,115	1580	385	2.8
I	travertine	11,034	1560	213	2.5
J	wood	1590	575	138	7.4
K	turbidite carbonate	18,166	5720	536	5.1
L	whalebone	12,601	2041	320	3.6
M	Icelandic peat	1842	2122	278	22.2

Comparing the results, we see that the 2 most variable samples (relative to their average age) are G (Fugla Ness wood) and M (Icelandic peat), followed by J (Crannog wood), and then K (turbidite carbonate). These differences will reflect in part the natural sample variability, and so are wholly realistic.

The span of results for Sample G is large, reflecting the fact that this sample is close to background for many laboratories and, again, emphasizes the fact that at this level of activity, differences between laboratories are emphasized. For Sample J, we see a large span relative to the age, but that the mid-50% span is pleasingly tight. The large span of results is perhaps surprising given that the sample was cut from a single timber (roughly 50-yr growth). For Sample M, (of a roughly equivalent age to J), the span of all results is considerably larger, though the mid-50% span is approximately the same as J. The overall span can, of course, be heavily influenced by small numbers of extreme observations. For the rest—Samples H, I, and L—they are virtually identical in terms of range of results. Thus, it seems unlikely that there have been any particular problems linked to the dating of bone. Sample K shows a wider range of results, though the mid-50% span is just over 500 yr relative to an age of approximately 18,000 yr.

4.5 CONSENSUS VALUES

Consensus values for each sample were evaluated using the same method used in the characterization of the IAEA reference samples (Rozanski et al. 1992) and for the core samples.

Table 4.11 Consensus values for optional samples

Sample	Consensus value (BP)	Estimated precision (1 σ)
G: Fuglaness wood	39,784	620
H: Ellanmore whole peat	11,152	23
I: travertine	11,060	17
J: Crannog wood	1605	8
K: turbidite carbonate	18,155	34
L: whalebone	12,788	30
M: Icelandic peat	1682	15

For each sample, a number of outliers were removed (up to a maximum of 10, but more typically less than 5). When the consensus value was calculated, results were also omitted due to the $\pm 2\sigma$ criterion not being satisfied.

Similar to the presentation for core samples, Figures 4.8–4.14 show individual laboratory differences from the consensus value. Figures 4.15–4.16 show the deviations for LSC and GPC laboratories. There is no such figure for AMS laboratories, since too few participated in the optional

program. These latter plots again show up to 1 or 2 large deviations for a number of the samples, but there is no evidence of any significant difference in performance overall for the 2 laboratory types. The figures again demonstrate that Sample G (at close to background) was the most scattered.

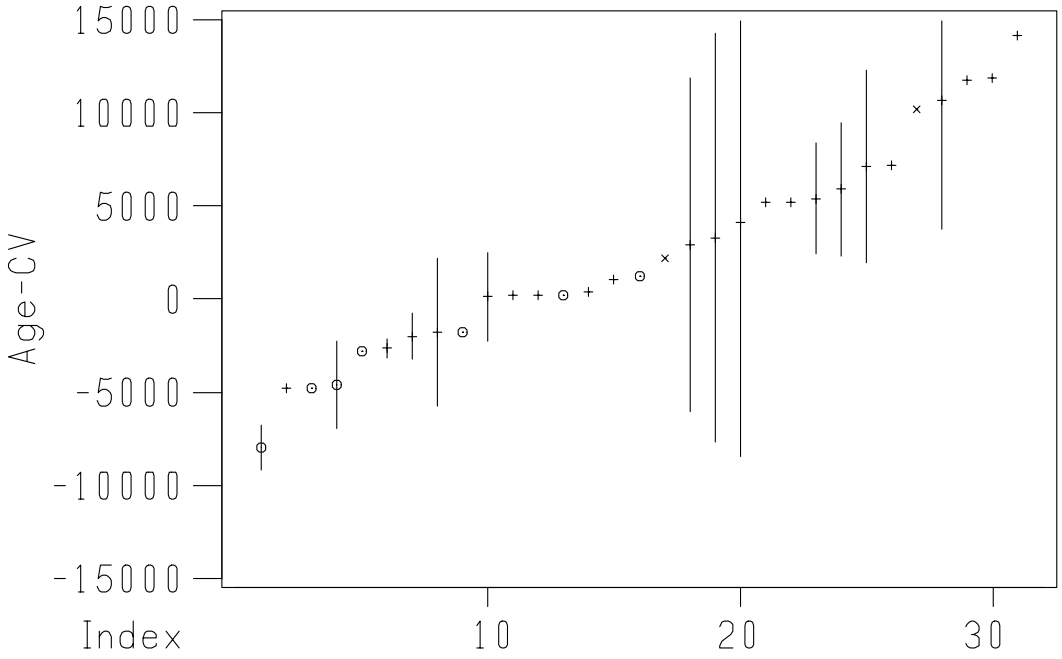

Figure 4.8 Sample G: Fuglaness wood

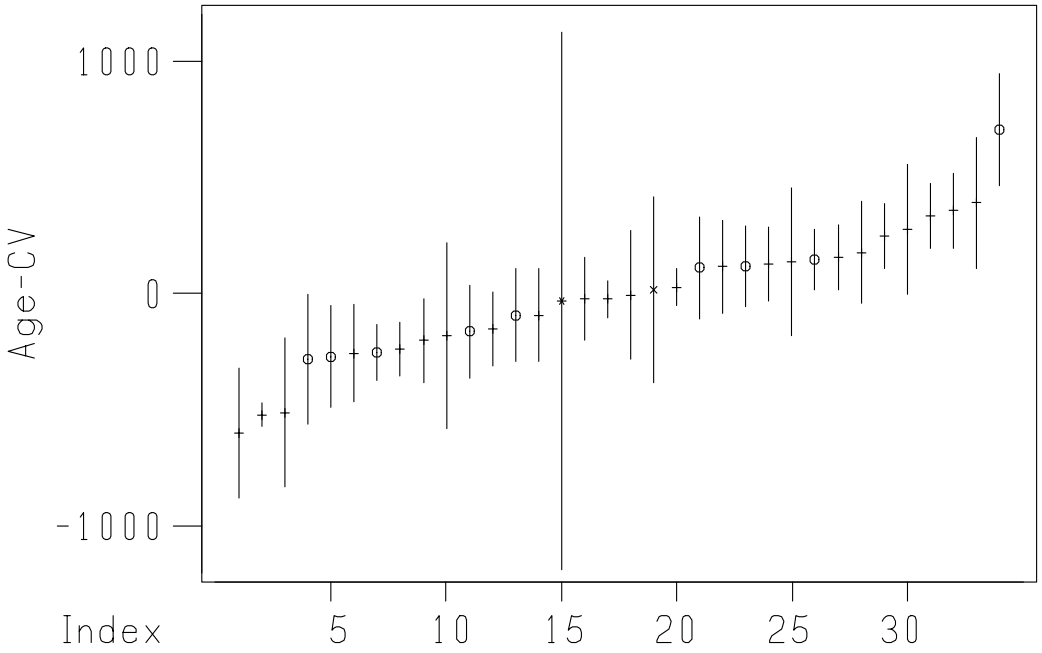

Figure 4.9 Sample H: Ellanmore peat

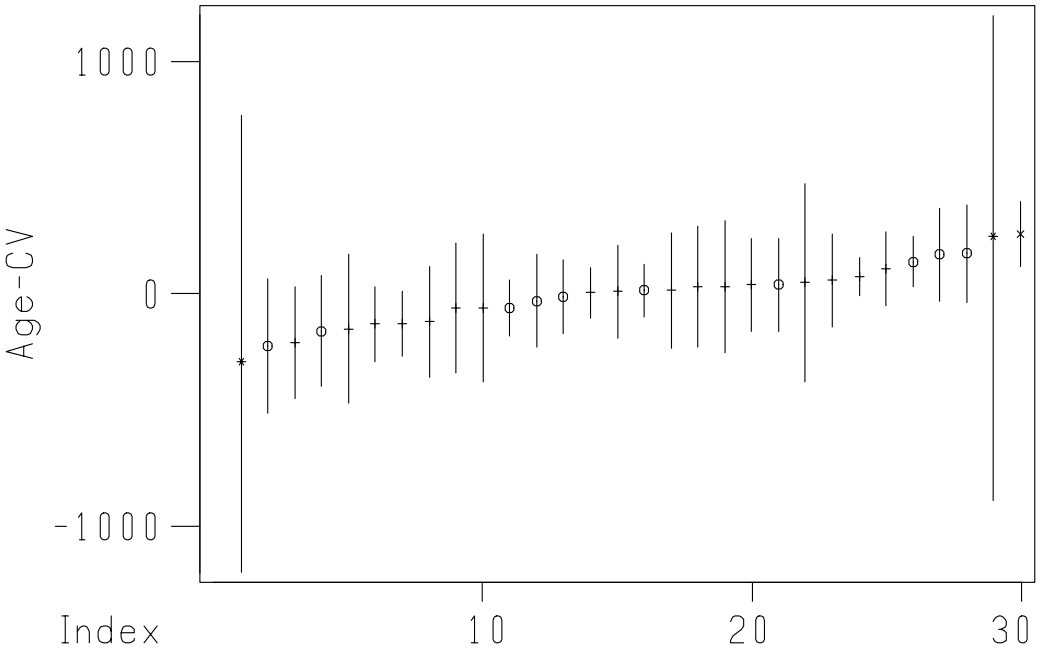

Figure 4.10 Sample I: travertine

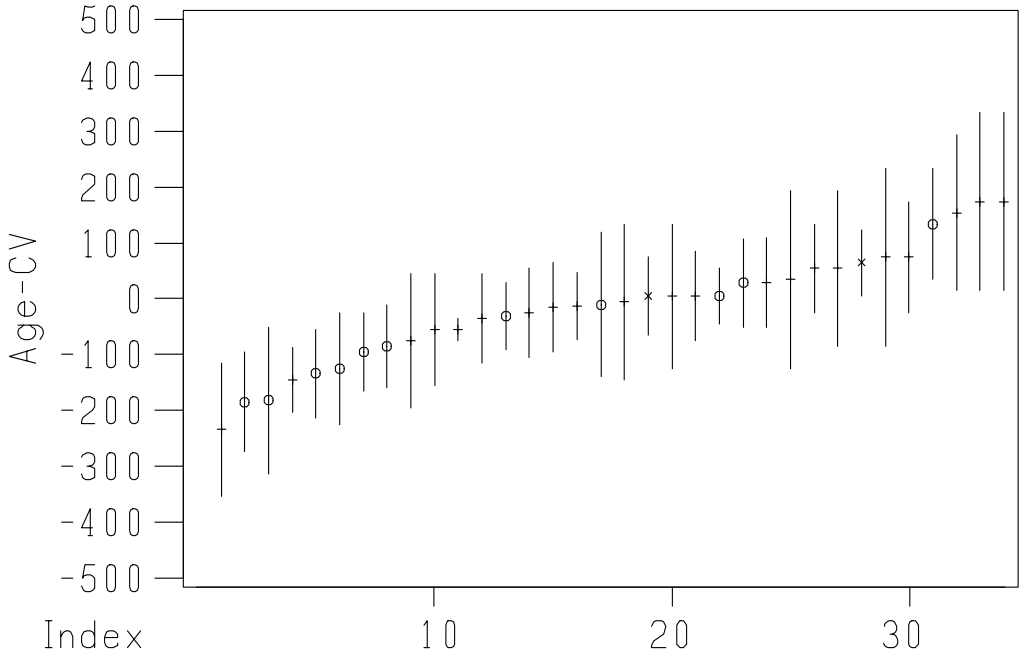

Figure 4.11 Sample J: wood (Buiston Crannog)

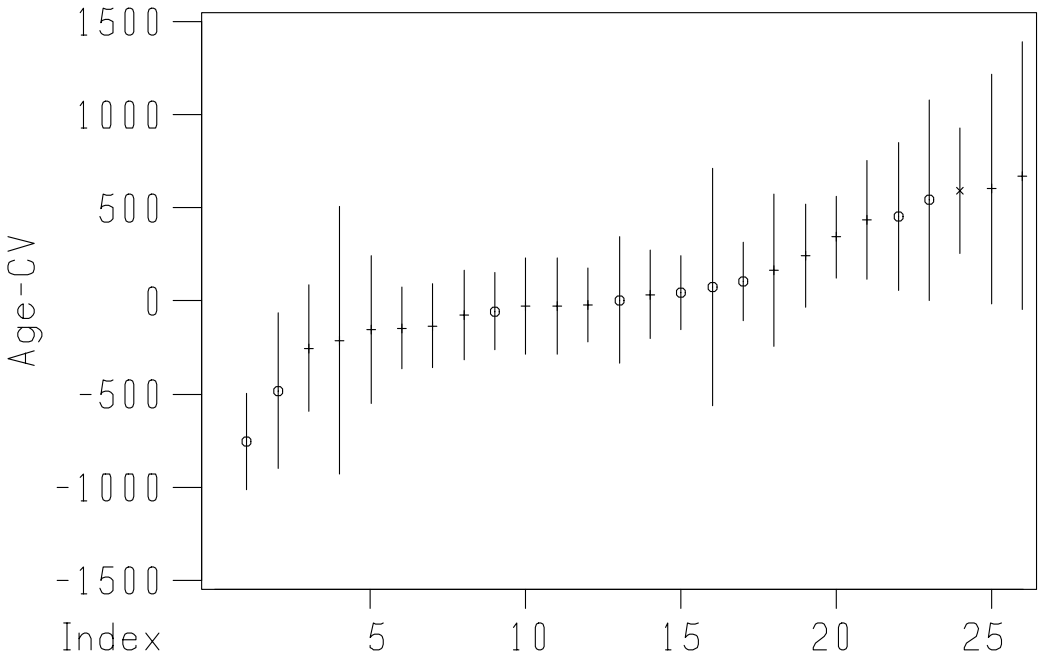

Figure 4.12 Sample K: turbidite carbonate

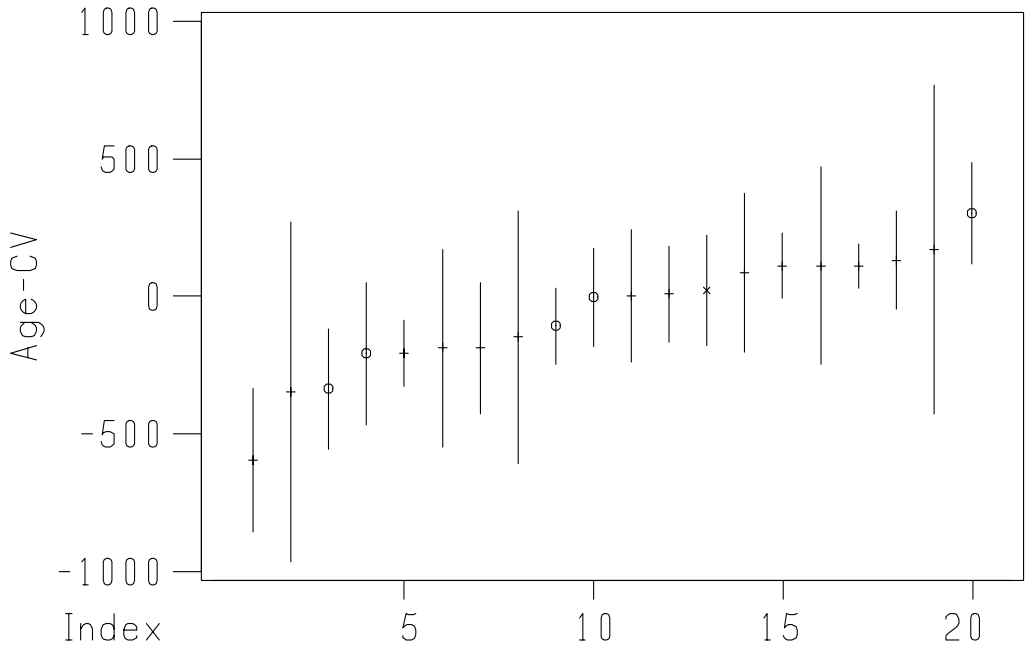

Figure 4.13 Sample L: whalebone

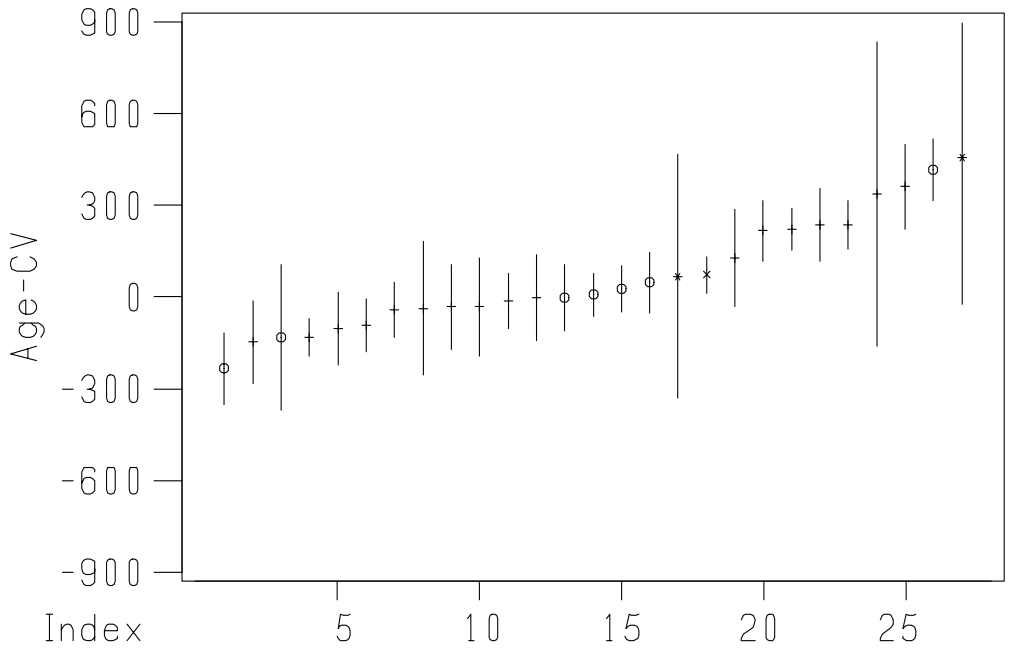

Figure 4.14 Sample M: Icelandic peat

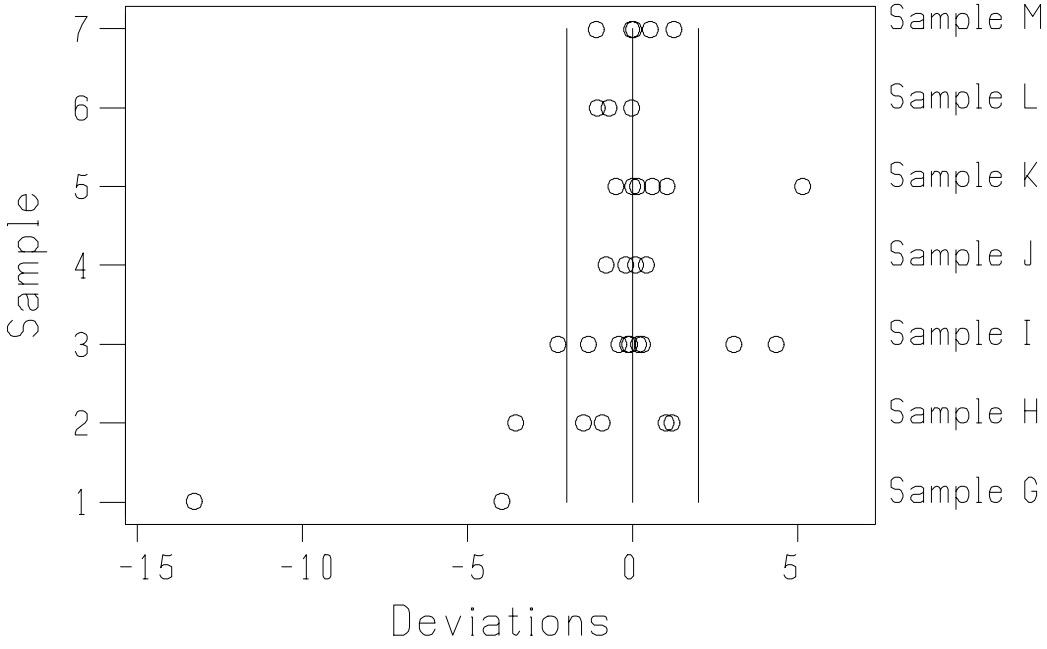

Figure 4.15 Deviations for GPC laboratories by sample

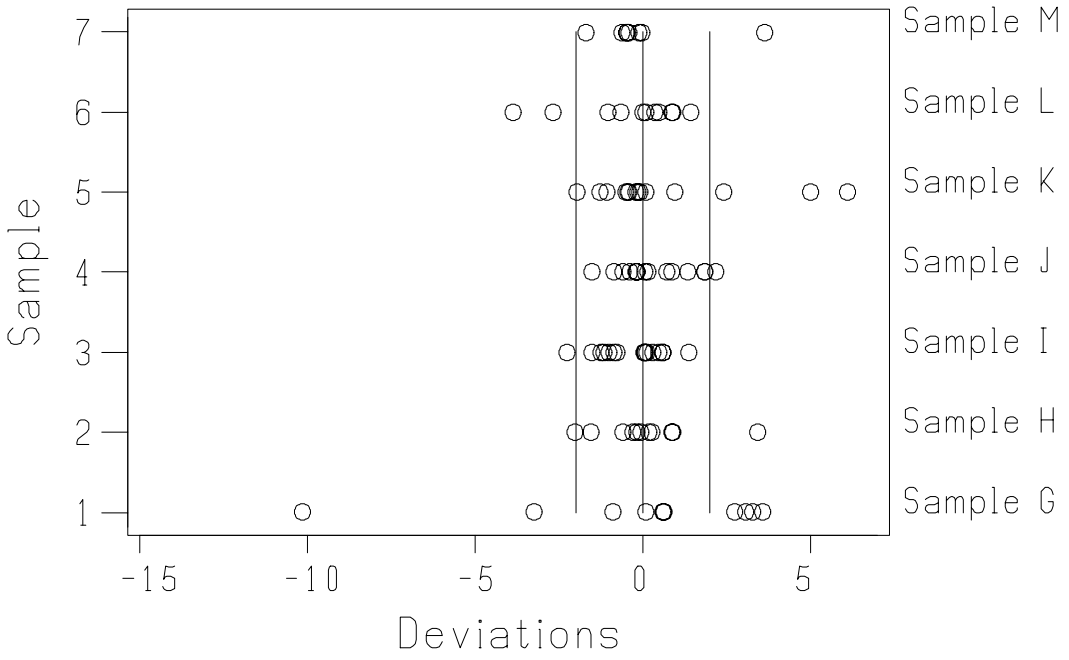

Figure 4.16 Deviations for LSC laboratories by sample

5. LABORATORY PERFORMANCE

5.1 BIAS AND ERROR MULTIPLIERS

Finally, as a summary of the individual laboratory performance, the relative bias (relative to the consensus values) and the error multiplier have been calculated based on the deviations as calculated for each lab and using results from both core and optional samples.

Measurement Model Used:

X_{ij} = Consensus value$_i$ + ε_{ij} for I = 1,..,N (number of labs) and j = 1,…, J (number of samples)

where X_{ij} is the ^{14}C age for sample *i* given by lab *j* and Consensus value$_i$ is the consensus value for sample *i*.

We further assume that ε_{ij} is normally distributed with mean 0 and variance = $S_{ij}^2\sigma_{ij}^2$

where S_{ij} is the quoted uncertainty and σ_{ij} is the error multiplier.

For each laboratory, we first carry out a formal test of a non-zero offset (relative bias) from the consensus values. This corresponds to a simple t-test of the deviations, with the null hypothesis that the mean value is 0. Eleven laboratories were found (at 5% level) to have a bias significantly different from zero. An additional 4 laboratories had a bias significantly different from zero (at a 10% level). For those laboratories for which there is no evidence of a relative bias, the error multiplier is evaluated and formally tested. This formal test simply evaluates whether the error multiplier is equal to 1. A value of 1 would indicate that the size of the deviations from the consensus value are in agreement with the size of the quoted uncertainties. A 95% confidence interval for σ_{ij} is calculated based on a χ^2 distributional result.

For those labs without relative bias, the error multiplier, σ_i, has been calculated under the model, assuming no bias, shown below as:

$$\sigma_{ij}^2 = 1 / J \, \Sigma d_{ij}^2$$

where d_{ij} is the deviation for lab *j* for sample *i*, and *J* is the total number of samples reported by lab *j*.

Further, a 95% confidence interval for the error multiplier has also been calculated (the value 1 should lie within this range for laboratories whose deviations from the consensus values agree within their quoted uncertainties).

The results are shown in the Table 5.1: a * value in the table indicates a laboratory with an error multiplier of plausibly 1.

Table 5.1 Interval estimates for error multiplier for those labs with no relative bias

Laboratory	Type	Lower limit	Upper limit
1	LSC	0.39314	1.5448*
2	GPC	0.75462	3.6203*
3	LSC	1.72561	4.5800
4	AMS	1.36298	5.3555
6	GPC	3.26318	12.8220
7	LSC	3.85350	9.2430
8	GPC	2.98044	6.6297
9	LSC	1.82828	7.1838
10	GPC	2.55963	16.8388
11	LSC	1.78315	4.4783
12	GPC	1.12415	7.3953
15	LSC	3.84395	10.2024
16	LSC	0.62025	2.9757*
17	LSC	2.30556	7.0962
18	GPC	1.39678	4.7739
19	LSC	0.92575	1.6943*
20	LSC	0.29319	1.0021*
21	LSC	1.09995	3.7594
22	GPC	0.77278	1.5439*
23	GPC	2.55769	6.4235
24	AMS	0.67305	1.2318*
25	AMS	3.22388	5.7437
26	LSC	0.60067	3.9516*
27	AMS	0.32694	1.1174*
30	AMS	0.62148	2.4420*
31	LSC	1.79200	4.7562
32	LSC	2.30850	7.8900
34	LSC	1.12385	2.9829
35	LSC	1.90813	6.5216
36	LSC	3.71198	7.9961
41	LSC	0.20635	1.3575*
42	LSC	1.62698	4.0861
43	LSC	1.16020	2.1234
45	GPC	1.86341	7.3219
47	LSC	1.24230	2.9774
48	LSC	1.16715	3.9891

Table 5.1 Interval estimates for error multiplier for those labs with no relative bias *(Continued)*

Laboratory	Type	Lower limit	Upper limit
49	LSC	1.06416	2.5505
50	other	0.71129	2.1893*
51	LSC	0.78706	2.0890*
52	LSC	1.30224	4.0081
53	GPC	1.53774	2.6762
54	LSC	4.17980	20.0528
55	GPC	0.95746	6.2988*
56	GPC	1.40190	4.7914
57	LSC	1.76777	5.4410
58	LSC	1.80718	4.1622
59	GPC	0.46164	1.8139*
60	LSC	1.70803	5.8377
61	AMS	1.81663	3.7125
62	LSC	0.51584	2.0269*
64	LSC	0.59069	2.3210*
66	AMS	1.60930	6.3234
67	AMS	1.71646	5.8666
68	AMS	0.36383	1.4296*
69	LSC	1.79715	4.7699
72	AMS	1.23534	2.5883
74	LSC	1.27743	3.2080
75	GPC	1.08569	3.7107

A histogram (Figure 5.1) of the error multipliers is given, as well as a boxplot (Figure 5.2) showing error multipliers by laboratory type. It is clear that the median error multiplier is around 2, suggesting that the quoted uncertainties are, in general, too small. However, it is also the case, that although for each of the laboratories, we have found no statistical evidence of a relative bias, the mean offset may still be non-zero. By ignoring this fact, the error multipliers are, in fact, slightly inflated as a result.

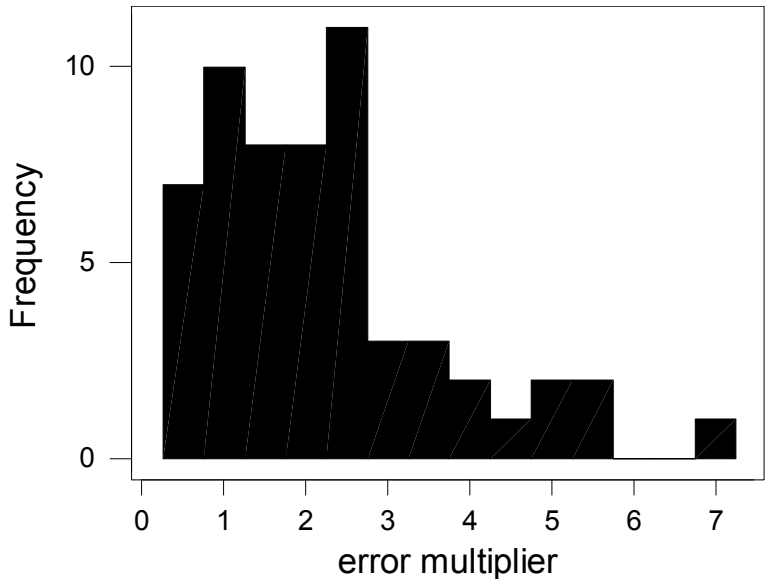

Figure 5.1 Histogram of error multipliers

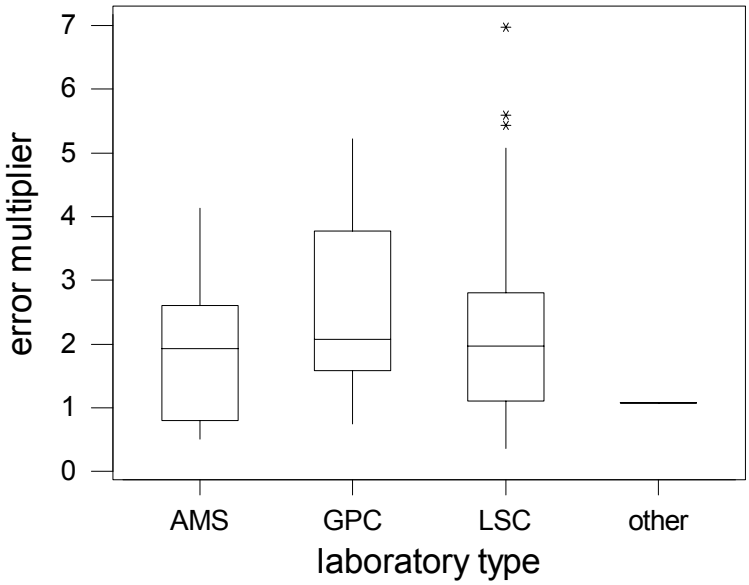

Figure 5.2 Distribution of error multipliers by laboratory type

6. CONCLUSIONS

TIRI provided valuable information to laboratories (both well-established and new) and hence, to users. It demonstrated the existence of additional variation (through the error multiplier) in the results, part of which must be due to the natural variation of the samples. Anomalous observations were found, although there is no evidence that they occurred on a frequent basis. There is evidence of significant between-laboratory variation, but no indications of differences in performance amongst the different laboratory types.

In the analysis the error multiplier was used. This is a rather simple tool, which has advantages and disadvantages in its use. Its main advantage is that it is very simple to use, and relates the observed variation in a direct way to the quoted uncertainties. However, it is difficult to meaningfully interpret, at least from the analyst's perspective and it is highly sensitive to anomalous observations. It refers to the results as reported and, thus, may not be generalizable beyond the study to which it refers.

Nevertheless, in TIRI as in the other studies, it points to variation in the results beyond that described by the quoted uncertainties. TIRI was not intended to explore the sources of the variation in the results, but it should be noted that at the TIRI workshop (Gulliksen and Scott 1995), there had been discussion concerning the homogeneity of the test samples, the issues of selection of small samples for AMS dating and the question of differing measured ^{14}C contents depending on the chemical fraction dated. It is clear, that in any study using natural samples some part of the extra variation must be due to the sampling of the bulk material. These issues are ones that will become increasingly important in future dating exercises (see discussion in FIRI on sample homogeneity testing).

Fourteen laboratories were found to have a significant bias, and for 55 laboratories, no such systematic bias was found. For these 55 laboratories, an error multiplier was then evaluated. Of the 55 laboratories, 28 had an error multiplier less than 2, and a significant number of these had an error multiplier less than 1.

Consensus values for the materials were derived and are shown in Table 6.1. Some of these materials were archived and re-used in FIRI. A store of material still remains for use by the ^{14}C dating community.

Table 6.1a Consensus values for Stage 1 TIRI samples

Sample	Consensus value	Estimated precision (1 σ)
A: barley mash	116.35 pMC	0.0084
B: Belfast pine	4503	6
C: IAEA cellulose	129.7 pMC	0.08
D: Hekla peat	3810	7
E: Ellanmore humic	11,129	12
F: Icelandic doublespar	46,750	208
F: Icelandic doublespar	0.18 pMC	0.006

Table 6.1b Consensus values for Stage 2 TIRI samples

Sample	Consensus value	Estimated precision (1 σ)
G: Fuglaness wood	39,784	620
H: Ellanmore whole peat	11,152	23
I: travertine	11,060	17
J: Crannog wood	1605	8
K: turbidite carbonate	18,155	34
L: whalebone	12,788	30
M: Icelandic peat	1682	15

It is also of interest to compare the 2 samples that are common in both TIRI and FIRI. These are the TIRI-B and FIRI-D and FIRI-F (Belfast pine), and TIRI-K and FIRI-C (marine turbidite from the same source).

Table 6.2 TIRI and FIRI samples in common

Sample description	Consensus value	True age	Estimated precision (1σ)
TIRI-B: Belfast dendro-dated wood	4503 yr BP	3200–3239 BC (^{14}C age 4495 BP)	6
FIRI-D, F: Belfast dendro-dated wood	4508 yr BP	3200–3239 BC (^{14}C age 4495 BP)	3
TIRI-K: turbidite	18,155 yr BP	—	34
FIRI-C: turbidite	18,176 yr BP	—	10.5

The consensus values, as estimated from the 2 different studies, are virtually identical. The estimated precisions are different. This is likely due to 3 reasons: a) the larger number of laboratories that participated in FIRI compared to TIRI; b) the tighter screening criteria used in FIRI; and c) the reduced scatter in the set of measurements once outliers have been removed.

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Scott EM, Aitchison TC, Harkness DD, Cook GT, Baxter MS. 1990. An overview of all three stages of the international radiocarbon intercomparison. *Radiocarbon* 32(3):309–19.

APPENDIX 1: RESULTS FOR ALL SAMPLES (FIRI)

Column Labelling and Data Entry

In the following tables:

- M/E is coded for measured (M) or estimated (E) $\delta^{13}C$
- "Limit" is used when a sample is non-finite and the coded entries are:
    - •B: indistinguishable from background;
    - •M: modern;
    - •>: greater than age.
- σ_L and σ_U are used if the error is asymmetrical
- pMClim: limit for pMC

Table A1.1 Full results for Sample A

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMClim	pMCσ
1	–23.5	—	M	48,180	—	—	—	2480	0.25	—	0.08
1	–23.7	—	M	50,380	—	—	—	3300	0.19	—	0.08
2	–23	—	M	51,640	—	—	—	2100	0.16	—	0.04
3	–24.2	—	M	41,490	—	—	—	2300	0.57	—	—
4	–22.98	0.45	M	—	B	—	—	—	—	B	—
5	–25	2	E	40,000	>	—	—	—	0.45	—	0.18
6	–22.975	0.004	M	44,006	—	420	440	—	—	—	—
7	–22.7	—	M	43,760	>	—	—	—	—	—	—
8	–20.9	—	M	45,970	—	—	—	790	—	—	—
9	–24.5	—	M	45,000	>	—	—	—	—	—	—
10	—	—	—	34,800	—	—	—	500	—	—	—
11	–30.7	0.1	M	39,250	—	—	—	1800	0.76	—	0.19
12	–22.8	—	M	40,000	>	—	—	—	—	—	—
13	–24.65	—	M	—	B	—	—	—	—	B	—
15	–23.33	0.75	M	51,530	—	1260	1490	—	0.16	—	0.03
15	–25.5	0.69	M	33,790	—	2190	3020	—	1.49	—	0.47
16	—	—	—	35,086	—	—	—	480	1.27	—	0.07
17	–25	—	E	55,200	>	—	—	—	0.104	—	0.059
18	–24.85	0.21	M	37,000	>	—	—	—	0	—	0.5
19	–25.2	—	M	44,024	—	—	—	2127	—	—	—
20	–22.1	—	M	55,000	—	—	—	2800	0.11	—	0.04
21	—	—	E	49,580	—	—	—	2300	—	—	—
22	–25.56	0.15	—	47,600	—	3700	7070	—	0.27	—	0.16
23	–23.02	0.2	M	40,545	—	—	—	1966	0.64	—	0.16
24	–23.68	—	—	46,000	>	—	—	—	—	—	—
25	–21.6	—	M	45,200	—	—	—	1400	0.36	—	0.06
26	—	—	—	39,400	—	—	—	1400	—	—	—
27	–26.2	—	M	45,000	>	—	—	—	0.36	—	—
28	—	—	—	38,400	—	1400	1700	—	—	—	—

Table A1.1 Full results for Sample A *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMClim	pMCσ
29	–21.4	—	M	43,900	—	—	—	2000	0.42	—	0.1
30	–24.17	—	M	36,780	—	410	430	—	1.027	—	0.053
31	–24.3	—	M	35,100	—	540	580	—	1.27	—	—
32	–23.5	—	M	42,810	>	—	—	—	0.5	<	—
33	–23.5	—	M	44,480	—	—	—	1280	0.39	—	0.06
34	–23.4	—	M	43,390	—	—	—	180	0.45	—	0.01
35	–25	—	M	44,400	—	—	—	1600	0.39	—	0.07
36	–25.6	—	M	50,000	—	2800	4000	—	0.2	—	0.1
37	–21.9	0.2	M	51,971	—	2418	3475	—	0.2	—	0.1
38	–23.8	—	M	47,490	—	—	—	890	0.271	—	0.03
39	–23.87	0.01	M	32,990	—	—	—	820	1.65	—	0.17
40	–23.6	0.6	M	49,922	>	—	—	—	0.12	—	0.04
41	–24.2	—	M	48,305	—	—	—	1592	0.24	—	0.04
42	–24.5	—	E	39,556	—	—	—	189	0.73	—	—
43	–24.234	0.004	M	40,000	>	—	—	—	0.56	—	0.66
44	–24.1	—	M	42,440	—	3880	7790	—	0.51	—	0.31
45	—	—	—	61,000	—	—	—	—	—	—	—
46	–22.1	—	M	49,000	>	—	—	—	0.21	<	—
47	–21.9	0.1	M	50,860	>	—	—	—	0.08	—	0.04
48	–24.84	—	M	44,000	>	—	—	—	0.4	<	—
49	—	—	—	48,500	—	—	—	1200	0.24	—	0.04
49	—	—	—	51,000	—	—	—	1700	0.17	—	0.04
49	—	—	—	49,700	—	—	—	1300	0.2	—	0.03
50	–23.4	—	M	52,400	—	—	—	2300	0.15	—	0.04
50	–23.4	—	M	51,800	—	—	—	2100	0.16	—	0.04
51	–23.5	0.1	M	45,500	—	—	—	1000	0.35	—	0.04
51	–23.9	0.1	M	50,300	—	—	—	1500	0.19	—	0.03
52	–23.2	0.3	M	47,250	>	—	—	—	0.28	—	0.03

Table A1.1 Full results for Sample A *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMClim	pMCσ
53	—	—	E	20,880	—	—	—	8010	7.43	—	4.69
54	–24.5	0.5	M	42,211	—	1295	1545	—	0.522	—	0.091
55	–24.4	—	M	49,500	—	2100	2850	—	0.2	—	0.1
56	–24.1	0.2	M	45,398	—	1724	2199	—	0.35	—	—
57	–25	—	E	43,400	>	—	—	—	0.45	<	—
58	–22.78	—	M	53,900	—	2300	3300	—	0.121	—	0.04
59	–22.1	—	M	42,000	>	—	—	—	0.4	—	0.2
60	–24.09	—	M	48,100	>	—	—	—	0.05	—	0.1
61	–24.9	—	M	44,000	>	—	—	—	—	—	—
63	–25	—	E	28,420	—	—	—	750	2.91	—	0.28
64	–24.8	—	M	46,200	—	—	—	1200	—	—	—
65	–24.61	—	M	45,000	—	3800	7300	—	0.37	—	0.22
66	–23.4	—	M	33,410	—	—	—	500	—	—	—
67	–25	—	E	23,250	>	—	—	—	—	—	—
68	–22.7	—	M	—	—	—	—	—	0.91	—	0.7
69	–23.8	0.2	M	32,400	B	—	—	2000	—	—	—
70	—	—	—	—	—	—	—	—	—	B	—
71	—	—	—	40,660	—	—	—	800	0.633	—	—
72	–21.9	—	M	40,790	—	—	—	585	—	—	—
72	–21.9	—	M	40,190	—	—	—	605	—	—	—
73	–23.84	—	M	52,400	—	1500	1830	—	0.147	—	0.03
74	–21.6	—	M	50,000	>	—	—	—	0.18	—	0.05
74	–20.1	—	M	50,000	>	—	—	—	0.15	—	0.05
75	—	—	—	34,450	—	—	—	300	1.14	—	—
76	–24.06	—	M	37,500	>	—	—	—	0.91	<	—

Table A1.1 Full results for Sample A *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMClim	pMCσ
77	–23.98	—	M	38,430	>	—	—	—	0.24	—	—
77	–23.98	—	M	39,760	>	—	—	—	–0.043	—	—
78	—	—	—	—	—	—	—	—	10.62	—	3.097
79	–23.59	—	M	—	—	—	—	—	0.35	—	0.06
81	–25	3	E	14,090	—	—	—	160	17.31	—	0.34
82	–24.08	—	M	46,610.96	—	—	—	2090.35	0.3	—	0.07
83	–24.1	—	M	52,240	—	—	—	1698	0.15	—	0.03
84	–22.4	—	M	49,200	—	—	—	1800	0.22	—	0.04
84	–24.8	—	M	50,200	—	—	—	2000	0.19	—	0.04
85	–24.2	—	E	47,948	—	—	—	1907	0.26	—	0.05
86	–24.4	—	M	41,988	—	1148	1340	—	0.537	—	0.083
87	–22.9	—	M	41,700	—	—	—	1200	0.55	—	0.08
88	–23.3	—	M	49,000	>	—	—	—	0.22	<	—
89	–23.85	—	M	44,300	—	—	—	3300	0.4	—	0.16
90	–25.18	—	M	—	B	—	—	—	—	—	—
90	–24.79	—	M	—	B	—	—	—	—	—	—
91	–24.4	1	M	45,818	—	—	—	1374	0.333	—	0.057

Table A1.2 Full results for Sample B

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMClim	pMCσ
1	–25.4	—	M	47,000	>	—	—	—	0.2	<	—
1	–23.2	—	M	47,000	>	—	—	—	0.2	<	—
2	–22.3	—	M	50,980	—	—	—	1930	0.18	—	0.04
3	–24.03	—	M	35,310	—	—	—	1400	1.23	—	—
4	–24.08	0.48	M	—	B	—	—	—	—	B	—
5	–25	2	E	39,200	—	—	—	1700	0.76	—	0.16
6	–23.142	0.012	M	47,610	—	510	540	—	—	—	—
7	–22.4	—	M	44,650	>	—	—	—	—	—	—
8	–21.4	—	M	46,660	—	—	—	770	—	—	—
9	–25.7	—	M	45,000	>	—	—	—	—	—	—
10	—	—	—	37,000	—	—	—	600	—	—	—
11	–31	0.1	M	41,500	—	—	—	1800	0.57	—	0.12
11	–31	0.1	M	42,000	>	—	—	—	0.06	—	0.16
12	–22.4	—	M	40,000	>	—	—	—	—	—	—
13	–24.09	—	M	—	B	—	—	—	—	B	—
15	–23.69	0.86	M	51,090	—	1230	1460	—	0.17	—	0.03
15	–26.82	0.72	M	37,320	—	1210	1420	—	0.96	—	0.16
16	—	—	—	33,521	—	—	—	960	1.54	—	0.19
17	–25	—	E	54,200	>	—	—	—	0.118	—	0.055
18	–24.4	0.17	M	37,000	>	—	—	—	–0.3	—	0.60
19	–25.2	—	E	45,800	>	—	—	—	—	—	—
20	–22.3	—	M	54,500	—	—	—	2600	0.11	—	0.04
21	—	—	E	32,640	—	—	—	1180	—	—	—
22	–24.9	0.15	—	41,000	—	1870	2450	—	0.61	—	0.16
23	–23.39	0.2	M	38,663	—	—	—	1609	0.81	—	0.16
24	–23.58	—	—	45,000	>	—	—	—	—	—	—
25	–21.9	—	M	43,600	—	—	—	1300	0.44	—	0.07
26	—	—	—	44,900	—	—	—	1600	—	—	—

Table A1.2 Full results for Sample B *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMClim	pMCσ
27	–24.5	—	M	45,200	—	—	—	3400	0.36	—	0.15
28	—	—	—	38,800	—	1200	1400	—	—	—	—
29	–21.5	—	M	43,600	—	—	—	2200	0.44	—	0.12
30	–23.83	—	M	34,420	—	310	320	—	1.378	—	0.054
31	–24.6	—	M	38,000	—	770	850	—	0.88	—	—
32	–23.97	—	M	45,480	>	—	—	—	0.35	<	—
33	–24	—	M	56,840	>	—	—	—	0.04	<	—
34	–24	—	M	48,800	>	—	—	—	0.09	—	0.07
35	–24	—	M	44,300	—	—	—	1300	0.39	—	0.05
36	–25.6	—	M	45,000	—	1600	2000	—	0.4	—	0.1
37	–21.5	0.2	M	48,829	—	1745	2232	—	0.22	—	0.05
38	–23.6	—	M	47,780	—	—	—	970	0.261	—	0.032
39	–23.92	0.01	M	36,030	—	—	—	1130	1.13	—	0.16
40	–23.6	0.6	M	49,478	>	—	—	—	0.13	—	0.04
41	–24.5	—	M	—	B	—	—	—	0.08	—	0.06
42	–24.5	—	E	41,764	—	—	—	184	0.55	—	—
43	–23.464	0.003	M	37,782	—	—	—	1314	0.91	—	0.15
44	–25.2	—	M	32,340	—	810	900	—	1.74	—	0.19
46	–21.9	—	M	49,000	>	—	—	—	0.22	<	—
47	–21.8	0.1	M	49,760	>	—	—	—	0.16	—	0.05
48	–25.89	—	M	44,000	>	—	—	—	0.4	<	—
49	—	—	—	55,900	—	—	—	2900	0.09	—	0.03
49	—	—	—	50,800	—	—	—	2600	0.18	—	0.06
49	—	—	—	50,900	—	—	—	1500	0.18	—	0.03
50	–24	—	M	56,000	—	—	—	3600	0.09	—	0.04
50	–24	—	M	55,200	—	—	—	3300	0.1	—	0.04
51	–23.4	0.1	M	45,800	—	—	—	1200	0.33	—	0.05
51	–23.2	0.1	M	45,000	—	—	—	950	0.37	—	0.04
51	–22.7	0.1	M	47,900	—	—	—	1300	0.26	—	0.04

Table A1.2 Full results for Sample B *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMClim	pMCσ
52	–23.3	0.3	M	45,010	>	—	—	—	0.37	—	0.07
53	—	—	E	24,290	—	—	—	5360	4.86	—	4.61
54	–24.1	0.5	M	41,013	—	1128	1313	—	0.606	—	0.091
55	–24	—	M	50,600	—	2050	2750	—	0.2	—	0.1
56	–23.9	0.2	M	—	B	—	—	—	—	—	—
57	–25	—	E	45,000	>	—	—	—	0.37	<	—
58	–22.86	—	M	50,600	>	—	—	—	0.048	—	0.067
59	–21.8	—	M	41,000	>	—	—	—	0.4	—	0.2
60	–23.2	—	M	47,800	>	—	—	—	0.06	—	0.1
61	–25	—	M	44,000	>	—	—	—	—	—	—
63	–25	—	E	43,540	—	—	—	3970	0.44	—	0.28
64	–23.9	—	M	45,800	—	—	—	1200	—	—	—
65	–24.85	—	M	45,000	—	3800	7300	—	0.37	—	0.22
66	–24.1	—	M	49,500	>	—	—	—	—	—	—
67	–25	—	E	24,800	>	—	—	—	—	—	—
68	–23.5	—	M	—	—	—	—	—	0.38	—	0.23
69	–25.2	0.2	M	32,500	B	—	—	1000	—	—	—
70	—	—	—	—	—	—	—	—	—	B	—
71	—	—	—	45,830	—	—	—	1100	0.333	—	—
72	–21.7	—	M	49,815	—	—	—	679	—	—	—
73	–24.37	—	M	53,140	—	1620	2030	—	0.134	—	0.03
74	–23.5	—	M	50,000	>	—	—	—	0.1	—	0.05
74	–21.4	—	M	50,000	>	—	—	—	0.12	—	0.05
75	—	—	—	49,000	>	—	—	—	—	—	—
76	–23.7	—	M	41,200	>	—	—	—	0.54	<	—
77	–24.44	—	M	38,600	>	—	—	—	0.237	—	—
77	–24.44	—	M	39,830	>	—	—	—	–0.004	—	—
78	—	—	—	—	—	—	—	—	8.41	—	2.876106

Table A1.2 Full results for Sample B *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMClim	pMCσ
79	–23.69	—	M	—	—	—	—	—	0.33	—	0.06
81	–25	3	E	49,900	—	3490	6310	—	0.2	—	0.11
82	–24.38	—	M	54,473.53	—	—	—	4296.82	0.11	—	0.05
83	–24.7	—	M	53,393	—	—	—	1951	0.13	—	0.03
84	–24.1	—	M	52,300	—	—	—	2700	0.15	—	0.04
84	–25.3	—	M	51,000	—	—	—	2300	0.17	—	0.04
85	–24.5	—	E	44,051	—	—	—	1062	0.42	—	0.05
86	–24.3	—	M	42,231	—	870	976	—	0.521	—	0.06
87	–23.5	—	M	—	B	—	—	—	—	—	—
88	–24	—	M	49,000	>	—	—	—	0.22	<	—
89	–24.35	—	M	42,600	—	—	—	2700	0.5	—	0.17
90	–25.37	—	M	—	B	—	—	—	—	—	—
90	—	—	—	—	B	—	—	—	—	—	—
91	–24.9	1	M	56,366	—	—	—	3126	0.09	—	0.036

Table A1.3 Full results for Sample C

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
1	0.9	—	M	18,260	—	—	79	10.30	0.10
1	0.9	—	M	18,281	—	—	73	10.27	0.09
2	0.9	—	M	18,200	—	—	50	10.38	0.05
3	1.107	—	M	18,180	—	—	350	10.40	—
4	1.17	0.02	M	18,300	—	—	150	10.25	—
5	0	2	E	16,790	—	—	130	12.37	0.20
5	0	2	E	17,070	—	—	240	11.95	0.36
6	1.2	0.01	M	17,983	—	—	50	—	—
7	1	—	M	18,110	—	—	110	—	—
8	1.4	—	M	18,170	—	—	50	—	—
9	1.1	—	M	17,900	—	—	100	—	—
10	—	—	—	15,470	—	—	210	—	—
11	–2.4	0.1	M	18,200	—	—	120	10.38	0.16
11	–2.4	0.1	M	18,150	—	—	190	10.44	0.28
12	1.2	—	M	18,610	—	—	360	9.86	0.44
13	3.864	—	M	18,500	—	—	130	10.02	0.16
14	1.5	—	M	18,590	590	640	—	9.90	0.80
15	–0.37	0.67	M	18,420	—	—	100	10.1	0.12
16	—	—	—	15,940	—	—	448	13.75	0.77
17	0	—	E	18,440	—	—	210	10.07	0.497
18	0	—	E	17,804	—	—	261	10.89	0.35
19	0	—	E	16,778	—	—	379	—	—
20	0.4	—	M	18,090	—	—	90	10.52	0.12
21	—	—	E	18,160	—	—	280	—	—
22	1.07	0.15	—	18,300	—	—	190	10.22	0.24
23	1.17	0.2	M	18,359	—	—	154	10.17	0.20
24	1.38	—	—	18,160	—	—	100	—	—
25	1.2	—	M	18,090	—	—	110	10.52	0.14
26	—	—	—	17,900	—	—	700	—	—

Table A1.3 Full results for Sample C *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
27	1.3	—	M	18,230	—	—	85	10.34	0.11
28	—	—	—	17,720	320	340	—	—	—
29	1.6	—	M	18,470	—	—	130	10.03	0.16
30	1.12	—	M	17,820	—	—	210	10.882	0.282
31	0.8	—	M	18,270	—	—	110	—	—
32	0.83	—	M	18,640	—	—	240	9.71	0.3
33	0.8	—	M	17,890	—	—	160	10.79	0.21
34	0.8	—	M	18,070	—	—	50	10.55	0.06
36	0.8	—	M	18,200	—	—	90	10.4	0.1
37	1.1	0.2	M	18,168	—	—	100	10.41	0.12
37	1.1	0.2	M	18,264	—	—	94	10.29	0.12
38	1.1	—	M	18,106	—	—	43	10.5	0.06
39	0.65	0.01	M	18,090	—	—	130	10.52	0.18
41	1.1	—	M	18,241	—	—	75	10.32	0.1
43	1.17	0.05	M	18,124	—	—	134	10.47	0.17
44	–0.3	—	M	15,230	—	—	220	15.02	0.42
46	1.1	—	M	18,260	—	—	140	10.3	0.18
47	0.9	0.1	M	18,395	—	—	65	10.13	0.08
48	–0.72	—	M	18,030	—	—	160	10.6	0.2
49	—	—	—	18,280	—	—	100	10.27	0.12
49	—	—	—	18,150	—	—	120	10.44	0.16
49	—	—	—	18,140	—	—	110	10.45	0.15
49	—	—	—	18,310	—	—	160	10.23	0.21
50	1.5	—	M	18,180	—	—	50	10.4	0.05
50	1.5	—	M	18,180	—	—	50	10.41	0.06
51	1.1	0.1	M	18,100	—	—	110	10.53	0.15
51	1	0.1	M	17,850	—	—	100	10.81	0.14
51	1.1	0.1	M	17,900	—	—	75	10.8	0.1
51	1.2	0.1	M	18,150	—	—	90	10.43	0.11

Table A1.3 Full results for Sample C *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
51	1.1	0.1	M	18,050	—	—	120	10.56	0.16
52	1.6	0.3	M	18,100	—	—	85	10.5	0.1
53	—	—	E	16,700	—	—	960	12.51	1.41
54	1.15	0.5	M	17,774	116	117	—	10.941	0.15
55	1.1	—	M	18,400	—	—	100	10.1	0.1
58	1.36	—	M	18,420	—	—	120	10.094	0.146
59	1.4	—	M	18,400	—	—	300	10.1	0.4
60	0.69	—	M	18,230	—	—	80	10.34	0.1
62	1.47	0.05	M	17,730	—	—	130	11.0	0.175
64	1	—	M	18,120	—	—	70	—	—
65	1.13	—	M	18,138	—	—	71	10.46	0.09
66	1.3	—	M	17,860	—	—	145	—	—
68	1.3	—	M	18,120	—	—	150	10.48	0.19
69	1	0.2	M	17,950	—	—	650	—	—
70	—	—	—	—	—	—	—	20.8	1.56
70	—	—	—	—	—	—	—	24.8	1.86
71	—	—	—	16,160	—	—	340	13.38	—
72	–22.6	—	M	18,140	—	—	100	—	—
73	1	—	M	18,270	—	—	70	10.28	0.09
74	3.1	—	M	17,930	—	—	60	10.73	0.08
76	–0.9	—	M	17,950	—	—	100	10.71	0.13
77	1.9	—	M	17,900	—	—	85	10.746	—
79	2.522	—	M	18,205	—	—	55	—	—
80	1.44	0.06	M	14,600	—	—	650	16.2	1.3
81	–5	5	E	17,860	—	—	190	10.83	0.25
82	1.06	—	M	18,373.98	—	—	105.64	10.15	0.13
83	1	—	M	18,249	—	—	63	10.31	0.08

Table A1.3 Full results for Sample C *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
84	0	—	E	18,150	—	—	100	10.44	0.13
84	0	—	E	18,320	—	—	100	10.22	0.13
84	0	—	E	18,120	—	—	100	10.48	0.13
85	1.1	—	E	18,205	—	—	48	10.94	0.07
86	1.3	—	M	18,123	—	—	100	10.48	0.13
87	–3.4	—	M	17,750	—	—	80	10.9	—
88	1.1	—	M	18,224	—	—	122	10.35	0.16
88	0.9	—	M	18,160	—	—	139	10.43	0.18
89	1.19	—	M	18,140	—	—	140	10.45	0.19
91	1.5	1	M	18,257	—	—	84	—	—

Table A1.4 Full results for Sample D

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
1	–24.2	—	M	4543	—	—	56	56.8	0.4
1	–24.2	—	M	4576	—	—	56	56.57	0.4
2	–22.8	—	M	4510	—	—	30	57.07	0.18
2	–22.8	—	M	4490	—	—	40	57.21	0.23
2	–22.8	—	M	4520	—	—	30	56.94	0.19
4	–23.81	0.47	M	4520	—	—	50	57	—
5	–25	2	E	4320	—	—	70	58.43	0.49
5	–25	2	E	4240	—	—	60	59.01	0.44
6	–24.328	0.024	M	4497	—	—	30	—	—
7	–23.3	—	M	4360	—	—	60	—	—
8	–23.5	—	M	4500	—	—	20	—	—
9	–25.3	—	M	4580	—	—	50	—	—
10	—	—	—	4170	—	—	50	—	—
11	–31.4	0.1	M	4370	—	—	45	58.04	0.33
12	–23.6	—	M	4510	—	—	110	57.05	1.1
13	–26.37	—	M	4680	60	70	—	55.89	0.45
15	–25.19	0.63	M	4510	—	—	10	57.06	0.1
15	–23.43	0.82	M	4500	—	—	40	57.13	0.31
15	–26.55	1.36	M	4550	—	—	30	56.74	0.22
16	—	—	—	4753	—	—	63	55.34	0.44
17	–25	—	E	4540	—	—	40	56.849	0.441
18	–25.2	0.19	M	4511	—	—	74	56.68	0.53
19	–26.2	—	M	4520	—	—	43	—	—
20	–23.7	—	M	4605	—	—	40	56.4	0.3
20	–23.9	—	M	4670	—	—	50	55.9	0.3
21	—	—	E	3790	—	—	40	—	—
22	–26.48	0.15	—	4680	—	—	80	55.81	0.54
23	–24.64	0.2	M	4581	—	—	53	56.54	0.37
24	–24.8	—	—	4523	—	—	26	—	—

Table A1.4 Full results for Sample D *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
25	–23.3	—	M	4590	—	—	50	56.47	0.35
25	—	—	M	4586	—	—	28	56.5	0.2
26	—	—	—	3400	—	—	70	—	—
27	–25.2	—	M	4550	—	—	25	56.76	0.18
28	—	—	—	4560	—	—	60	—	—
29	–22.9	—	M	4660	—	—	50	56	0.32
30	–25.01	—	M	4420	—	—	200	57.646	1.456
31	–25.2	—	M	4490	—	—	50	—	—
31	–25.4	—	M	4430	—	—	55	—	—
32	–25.54	—	M	4600	—	—	80	56.48	0.52
33	–25.54	—	M	4400	—	—	40	57.97	0.28
34	–25.5	—	M	4430	—	—	30	57.64	0.21
35	–25	—	M	4500	—	—	30	57.11	0.22
36	–23.4	—	M	4517	—	—	34	57	0.2
36	–23.4	—	M	4517	—	—	30	57	0.2
36	–26.1	—	M	4535	—	—	27	56.9	0.2
37	–23.4	0.2	M	4553	—	—	45	56.73	0.31
37	–23.6	0.2	M	4549	—	—	42	56.76	0.29
38	–25	—	M	4464	—	—	26	57.37	0.19
38	–22.8	—	M	4496	—	—	18	57.14	0.13
39	–25.09	0.02	M	4710	—	—	35	55.63	0.26
40	–24.8	0.6	M	4482	—	—	34	57.24	0.24
41	–25.5	—	M	4509	—	—	21	57.05	0.15
41	–25.1	—	M	4521	—	—	18	56.96	0.13
42	–24.5	—	E	4325	—	—	78	58.4	—
43	–24.988	0.002	M	4626	—	—	42	56.22	0.29
44	–23.1	—	M	4470	—	—	60	57.34	0.4
46	–22.8	—	M	4534	—	—	73	56.87	0.52
47	—	—	M	4675	—	—	50	55.87	0.35

Table A1.4 Full results for Sample D *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
48	–25.5	—	M	4620	—	—	80	56.3	0.6
49	—	—	—	4520	—	—	50	56.95	0.38
49	—	—	—	4500	—	—	50	57.13	0.34
50	–24.1	—	M	4510	—	—	30	57.02	0.2
50	–24.1	—	M	4540	—	—	30	56.84	0.2
51	–24	0.1	M	4500	—	—	65	57.08	0.46
51	–23.5	0.1	M	4590	—	—	40	56.44	0.28
51	–24.4	0.1	M	4550	—	—	40	56.73	0.3
52	–24.3	0.3	M	4590	—	—	35	56.51	0.25
53	—	—	E	2990	—	—	240	68.88	1.99
54	–25.8	0.5	M	4505	—	—	36	57.074	0.255
55	–23.9	—	M	4540	—	—	40	56.9	0.3
56	–26.8	0.2	M	4753	—	—	30	55.3	0.2
57	–25	—	E	4273	—	—	58	58.75	0.4
58	–24.33	—	M	4600	—	—	40	56.402	0.3
59	–23.4	—	M	4580	—	—	90	56.5	0.7
60	–24.26	—	M	4600	—	—	30	56.43	0.2
61	–25.6	—	M	4570	—	—	35	—	—
63	–25	—	E	4540	—	—	60	56.83	0.36
64	–24.9	—	M	4590	—	—	70	—	—
65	–25.98	—	M	4467	—	—	24	—	—
66	–26	—	M	4495	—	—	65	—	—
67	–25	—	E	4775	—	—	91	—	—
68	–23.9	—	M	4700	—	—	70	55.72	0.47
69	–25.2	0.2	M	5060	—	—	200	—	—
70	—	—	—	—	—	—	—	55.3	4.1475
70	—	—	—	—	—	—	—	57.1	4.2825
71	—	—	—	4340	—	—	70	58.23	—
72	–22.8	—	M	4567	—	—	45	—	—

Table A1.4 Full results for Sample D *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
72	–22.8	—	M	4655	—	—	65	—	—
73	–25.2	—	M	4510	—	—	20	57.04	0.12
74	–25.5	—	M	4485	—	—	40	57.22	0.27
74	–21.6	—	M	4470	—	—	40	57.33	0.22
75	—	—	—	4450	—	—	35	—	—
76	–25	—	M	4490	—	—	30	57.18	0.21
77	–24.89	—	M	4480	—	—	30	57.221	—
77	–24.89	—	M	4500	—	—	40	57.096	—
78	—	—	—	—	—	—	—	54.87	3.539823
79	–22.91	—	M	4535	—	—	40	—	—
81	–25	3	E	4590	—	—	70	56.49	0.52
82	–25.12	—	M	4541.69	—	—	52.2	56.81	0.37
83	–24.8	—	M	4476	—	—	25	57.28	0.18
84	–23.9	—	M	4530	—	—	40	56.89	0.24
85	–25.1	—	E	4446	—	—	12	57.48	0.09
86	–23.2	—	M	4468	—	—	33	57.34	0.23
87	–25.9	—	M	4400	—	—	60	57.44	—
88	–24.7	—	M	4465	—	—	35	57.37	0.25
88	–24.6	—	M	4518	—	—	49	56.98	0.35
88	–24.7	—	M	4521	—	—	38	56.96	0.27
89	–25.69	—	M	4500	—	—	50	57.08	0.37
90	–25.92	—	M	4450	—	—	100	57.46	0.69
90	–26.03	—	M	4370	—	—	90	58.05	0.66
91	–25.1	1	M	4462	—	—	26	—	—

Table A1.5 Full results for Sample E

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
1	–28.9	—	M	11,660	—	—	66	23.42	0.19
1	–28.9	—	M	11,709	—	—	64	23.28	0.18
2	–28.7	—	M	11,850	—	—	40	22.87	0.09
3	–29.3	—	M	11,670	—	—	152	23.3	—
4	–27.59	0.55	M	11,980	—	—	130	22.5	—
4	–28.05	0.56	M	12,000	—	—	110	22.4	—
5	–25	2	E	11,450	—	—	100	24.05	0.31
5	–25	2	E	11,760	—	—	130	23.14	0.4
6	–29.502	0.012	M	11,741	—	—	45	—	—
7	–27.7	—	M	11,600	—	—	70	—	—
8	–28.9	—	M	11,770	—	—	70	—	—
9	–29.5	—	M	11,800	—	—	100	—	—
10	—	—	—	11,300	—	—	160	—	—
11	–32.9	0.1	M	11,530	—	—	170	23.8	0.5
11	–32.9	0.1	M	11,460	—	—	120	24.01	0.45
12	–23.2	—	M	11,660	—	—	220	23.41	0.62
13	–29.4	—	M	12,150	—	—	80	22.06	0.23
15	–29.58	1.8	M	11,840	—	—	100	22.9	0.28
16	—	—	—	11,305	—	—	135	24.48	0.4
17	–27	—	E	12,550	—	—	160	20.963	0.585
18	–29.02	0.16	M	12,314	—	—	153	21.45	0.41
19	–27	—	E	11,534	—	—	106	—	—
20	–28.2	—	M	11,900	—	—	70	22.73	0.2
21	—	—	E	11,750	—	—	130	—	—
23	–28.68	0.2	M	12,004	—	—	93	22.44	0.26
24	—	—	—	—	—	—	—	—	—
25	–29.1	—	M	11,730	—	—	60	23.22	0.17
25	–29.1	—	M	11,920	—	—	130	22.68	0.36
26	—	—	—	7700	—	—	190	—	—

Table A1.5 Full results for Sample E *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
27	–28.8	—	M	11,770	—	—	30	23.11	0.08
28	—	—	—	13,560	130	140	—	—	—
29	–28.8	—	M	11,880	—	—	90	22.8	0.24
30	–29.27	—	M	11,670	—	—	210	23.394	0.609
30	–29.3	—	M	11,610	—	—	220	23.561	0.636
31	–29.6	—	M	11,760	—	—	80	—	—
31	–29.5	—	M	11,660	—	—	190	—	—
32	–29.87	—	M	12,270	—	—	150	21.84	0.42
33	–29.87	—	M	11,940	—	—	110	22.63	0.32
34	–29.9	—	M	11,750	—	—	40	23.16	0.01
35	–27	—	M	11,870	—	—	100	22.8	0.3
35	–27	—	M	13,000	—	—	1000	—	—
36	–30.7	—	M	11,820	—	—	60	23	0.2
36	–31.1	—	M	11,640	—	—	140	23.5	0.4
36	–29.9	—	M	11,816	—	—	48	23	0.1
37	–29.6	0.2	M	11,809	—	—	69	22.99	0.19
38	–28.9	—	M	11,823	—	—	58	22.95	0.17
38	–29	—	M	11,830	—	—	53	22.93	0.15
39	–29.2	0.01	M	11,930	—	—	70	22.44	0.21
40	–29.8	0.6	M	11,769	—	—	66	23.11	0.19
40	–29	0.5	M	11,772	—	—	28	23.1	0.13
40	–30.5	0.6	M	11,859	—	—	31	22.85	0.09
41	–29.5	—	M	11,722	—	—	38	23.24	0.11
43	–29.056	0.003	M	12,450	—	—	98	21.23	0.26
44	–29.2	—	M	11,670	—	—	380	23.4	1.1
46	–29.1	—	M	11,760	—	—	100	23.12	0.29
46	–29	—	M	11,653	—	—	96	23.44	0.28
47	–29.6	0.1	M	11,815	—	—	50	22.97	0.14
47	–31.1	0.1	M	11,880	—	—	95	22.79	0.27

Table A1.5 Full results for Sample E *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
48	–29.5	—	M	11,790	—	—	120	23	0.4
49	—	—	—	11,800	—	—	40	23.03	0.1
49	—	—	—	11,910	—	—	70	22.7	0.21
49	—	—	—	12,020	—	—	270	22.39	0.77
49	—	—	—	11,840	—	—	120	22.89	0.34
49	—	—	—	11,940	—	—	100	22.61	0.28
49	—	—	—	11,820	—	—	100	22.96	0.28
49	—	—	—	11,730	—	—	110	23.23	0.31
49	—	—	—	11,720	—	—	80	23.25	0.23
49	—	—	—	11,780	—	—	80	23.06	0.23
49	—	—	—	11,800	—	—	80	23.01	0.24
49	—	—	—	11,920	—	—	80	22.69	0.23
50	–28.4	—	M	11,790	—	—	40	23.05	0.1
50	–28.4	—	M	11,800	—	—	40	23.02	0.1
50	–28.4	—	M	11,810	—	—	40	23	0.1
50	–28.4	—	M	11,830	—	—	50	22.92	0.13
50	–28.4	—	M	11,850	—	—	50	22.89	0.14
50	–28.4	—	M	11,790	—	—	50	23.05	0.12
50	–28.4	—	M	11,780	—	—	60	23.08	0.16
50	–28.4	—	M	11,770	—	—	70	23.09	0.17
50	–28.4	—	M	11,950	—	—	110	22.59	0.29
50	–28.4	—	M	11,760	—	—	100	23.14	0.27
50	–28.4	—	M	11,870	—	—	170	22.82	0.46
50	–28.4	—	M	12,100	—	—	350	22.17	0.95
52	–28.7	0.3	M	11,840	—	—	70	22.9	0.2
53	—	—	E	15,150	—	—	820	15.17	1.47
54	–29.5	0.5	M	11,693	230	237	—	23.327	0.678
55	–29.6	—	M	11,750	—	—	60	23.1	0.2
55	–29.2	—	M	12,100	290	300	—	22.2	0.8

Table A1.5 Full results for Sample E *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
57	–25	—	E	11,550	—	—	110	23.75	0.31
58	–30.11	—	M	11,680	—	—	70	23.37	0.2
58	–30.36	—	M	11,880	—	—	110	22.8	0.3
59	–29.4	—	M	11,650	—	—	130	23.5	0.4
60	–29.8	—	M	11,830	—	—	50	22.94	0.12
61	–28.3	—	M	11,885	—	—	55	—	—
62	–29.08	0.05	M	11,720	—	—	100	23.245	0.281
64	–29.5	—	M	11,510	—	—	230	—	—
64	–29.5	—	M	11,700	—	—	70	—	—
65	–29.53	—	M	11,760	—	—	48	23.13	0.14
66	–29.3	—	M	11,525	—	—	80	—	—
67	–25	—	E	12,151	—	—	158	—	—
67	–25	—	E	11,721	—	—	230	—	—
68	–27	—	E	12,120	—	—	130	22.14	0.22
70	—	—	—	—	—	—	—	24.3	1.8225
70	—	—	—	—	—	—	—	24.8	1.86
70	—	—	—	—	—	—	—	18.6	1.395
71	—	—	—	11,700	—	—	110	23.31	—
72	–23	—	M	11,595	—	—	65	—	—
73	–29.28	—	M	11,850	—	—	40	22.88	0.13
74	–27.7	—	M	11,800	—	—	50	23.03	0.13
74	–34.3	—	M	11,860	—	—	60	22.85	0.17
74	–12.3	—	M	11,650	—	—	80	23.45	0.24
74	–28.6	—	M	12,050	—	—	330	22.3	0.96
75	—	—	—	11,570	—	—	80	—	—
76	–28.79	—	M	11,480	—	—	55	23.96	0.16
77	–30.2	—	M	11,800	—	—	55	22.962	—
77	–30.2	—	M	11,900	—	—	70	22.832	—
77	–31.33	—	M	11,300	—	—	105	24.415	—

Table A1.5 Full results for Sample E *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
78	—	—	—	—	—	—	—	24.78	3.318
79	—	—	M	11,785	—	—	45	—	—
81	–25	3	E	11,870	—	—	110	22.82	0.31
82	–28.59	—	M	11,792.04	—	—	125.61	23.04	0.36
82	–28.73	—	M	11,777.74	—	—	68.17	23.08	0.2
83	–29.1	—	M	11,773	—	—	39	23.09	0.11
83	–30.4	—	M	12,098	—	—	134	22.18	0.37
83	–30.5	—	M	11,652	—	—	125	23.45	0.36
84	–29.1	—	M	11,740	—	—	60	23.2	0.16
84	–29.1	—	M	11,910	—	—	80	22.71	0.21
84	–29.1	—	M	11,970	—	—	70	22.55	0.18
84	–29.1	—	M	11,680	—	—	50	23.37	0.12
84	–29.1	—	M	11,520	—	—	90	23.84	0.24
84	–29.1	—	M	11,430	—	—	50	24.11	0.14
84	–29.1	—	M	11,460	—	—	100	24.01	0.28
85	–29.5	—	E	11,673	—	—	26	23.38	0.08
85	–29.5	—	E	11,539	—	—	82	23.56	0.27
86	–29.9	—	M	11,629	—	—	64	23.51	0.19
86	–29.8	—	M	11,619	—	—	114	23.54	0.34
87	–29.5	—	M	11,590	—	—	60	23.48	—
88	–29	—	M	11,920	—	—	72	22.68	0.2
88	–28.9	—	M	11,872	—	—	71	22.81	0.2
88	–29	—	M	11,731	—	—	106	23.22	0.31
89	–29.42	—	M	10,780	—	—	90	26.13	0.3
89	–29.42	—	M	11,870	—	—	550	22.8	1.6
91	–28.1	1	M	11,797	—	—	39	—	—
92	–29	—	M	11,180	—	—	120	24.88	0.37
92	–29	—	M	10,370	—	—	470	27.48	1.6

Table A1.6 Full results for Sample F

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
1	–23.1	—	M	4588	—	—	52	56.48	0.37
1	–23.7	—	M	4534	—	—	48	56.87	0.34
2	–21.7	—	M	4540	—	—	30	56.84	0.18
3	–24.92	—	M	4600	—	—	128	56.4	—
4	–24.01	0.48	M	4530	—	—	80	56.9	—
5	–25	2	E	4190	—	—	50	59.36	0.4
5	–25	2	E	4400	—	—	90	57.83	0.7
6	–24.068	0.014	M	4504	—	—	30	—	—
7	–23.8	—	M	4450	—	—	50	—	—
8	–22.9	—	M	4470	—	—	20	—	—
9	–26.2	—	M	4500	—	—	50	—	—
10	—	—	—	4100	—	—	50	—	—
11	–32.2	0.1	M	4360	—	—	45	58.11	0.33
11	–32.2	0.1	M	4280	—	—	80	58.7	0.9
12	–24	—	M	4440	—	—	140	57.52	1.01
13	–25.48	—	M	4560	—	—	60	56.74	0.42
15	–24.94	0.42	M	4470	—	—	20	57.34	0.13
15	–27.04	0.01	M	4420	—	—	190	57.71	1.38
16	—	—	—	4637	—	—	95	56.14	0.69
17	–25	—	E	4490	—	—	40	57.162	0.464
17	–25	—	E	4490	—	—	30	57.176	0.436
18	–25.05	0.21	M	4494	—	—	63	56.8	0.44
19	–26.2	—	E	4513	—	—	52	—	—
20	–22.9	—	M	4570	—	—	40	56.6	0.3
21	—	—	E	4440	—	—	40	—	—
22	–27.15	0.15	—	4370	—	—	90	58.02	0.63
23	–24.2	0.2	M	4740	—	—	62	55.43	0.43
24	–25.05	—	—	4459	—	—	30	—	—
25	–23.8	—	M	4540	—	—	50	56.83	0.35

Table A1.6 Full results for Sample F *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
26	—	—	—	4200	—	—	90	—	—
27	–25	—	M	4505	—	—	25	57.08	0.17
28	—	—	—	4460	—	—	60	—	—
29	–23.1	—	M	4570	—	—	50	56.6	0.32
30	–24.6	—	M	4500	—	—	200	57.101	1.444
31	–25.2	—	M	4550	—	—	60	—	—
32	–25	—	M	4710	—	—	80	55.64	0.52
33	–25	—	M	4370	—	—	50	58.04	0.37
34	–25	—	M	4460	—	—	40	57.42	0.22
35	–24	—	M	4710	—	—	40	55.64	0.08
36	–26	—	M	4493	—	—	30	57.2	0.2
37	–23.6	0.2	M	4527	—	—	47	56.92	0.33
38	–24.7	—	M	4463	—	—	45	57.38	0.32
39	–25.46	0.01	M	4755	—	—	35	55.26	0.24
40	–24.8	0.5	M	4465	—	—	37	57.36	0.26
41	–25.3	—	M	4454	—	—	16	57.44	0.11
41	–24.9	—	M	4527	—	—	16	56.92	0.11
42	–24.5	—	E	4315	—	—	66	58.4	—
43	–25.145	0.005	M	4865	—	—	67	54.58	0.46
44	–26.1	—	M	4250	—	—	80	58.93	0.61
46	–23.6	—	M	4502	—	—	49	57.09	0.35
47	–23.3	0.1	M	4550	—	—	50	56.77	0.34
48	–25	—	M	4590	—	—	80	56.4	0.6
49	—	—	—	4540	—	—	50	56.82	0.37
49	—	—	—	4510	—	—	50	57.02	0.37
49	—	—	—	4590	—	—	130	56.46	0.93
50	–25	—	E	4540	—	—	40	56.85	0.25
50	–25	—	E	4510	—	—	40	57.07	0.23
51	–24	0.1	M	4610	—	—	40	56.33	0.27

Table A1.6 Full results for Sample F *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
51	–24.2	0.1	M	4550	—	—	40	56.73	0.27
51	–23.8	0.1	M	4510	—	—	35	57.01	0.26
51	–23.9	0.1	M	4700	—	—	60	55.73	0.41
51	–24	0.1	M	4600	—	—	45	56.42	0.3
52	–24.2	0.3	M	4550	—	—	65	56.76	0.45
53	—	—	E	5870	—	—	290	48.15	1.72
54	–25.8	0.5	M	4459	—	—	45	57.401	0.324
55	–26	—	M	4530	—	—	40	56.9	0.3
56	–25.2	0.2	M	4638	—	—	41	56.1	0.9
57	–25	—	E	4425	—	—	58	57.65	0.4
58	–23.25	—	M	4470	—	—	50	57.31	0.31
59	–24.1	—	M	4670	—	—	90	55.9	0.6
60	–24.97	—	M	4470	—	—	40	57.31	0.22
61	–25.4	—	M	4540	—	—	40	—	—
63	–25	—	E	4770	—	—	60	55.24	0.36
64	–24.9	—	M	4570	—	—	70	—	—
65	–26.62	—	M	4484	—	—	26	57.22	0.19
66	–25.5	—	M	4460	—	—	65	—	—
67	–25	—	E	4519	—	—	97	—	—
68	–25.4	—	M	4670	—	—	50	55.9	0.32
69	–24.9	0.2	M	5270	—	—	250	—	—
70	—	—	—	—	—	—	—	53.5	4.0125
70	—	—	—	—	—	—	—	60.2	4.515
71	—	—	—	4280	—	—	90	58.71	—
72	–23	—	M	4370	—	—	40	—	—
73	–25.25	—	M	4490	—	—	20	57.19	0.15
74	–24.9	—	M	4525	—	—	40	56.93	0.21
74	–25	—	M	4550	—	—	40	56.76	0.2
75	—	—	—	4490	—	—	35	—	—

Table A1.6 Full results for Sample F *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
76	–25.41	—	M	4480	—	—	30	57.24	0.23
77	–25.33	—	M	4400	—	—	30	57.847	—
77	–25.33	—	M	4460	—	—	45	57.405	—
78	—	—	—	—	—	—	—	52.65	3.539823
79	–24.51	—	M	4550	—	—	40	—	—
81	–25	3	E	4510	—	—	70	57.02	0.52
82	–24.88	—	M	4590.68	—	—	47.84	56.47	0.34
83	–24.8	—	M	4483	—	—	25	57.23	0.18
84	–23.5	—	M	4500	—	—	50	57.08	0.3
84	–24.5	—	M	4560	—	—	50	56.69	0.31
85	–24.9	—	E	4469	—	—	16	57.34	0.12
86	–25.5	—	M	4571	—	—	35	56.61	0.25
87	–23.7	—	M	4480	—	—	50	56.92	—
88	–25.3	—	M	4485	—	—	40	57.22	0.27
88	–25	—	M	4509	—	—	49	57.04	0.35
89	–25.33	—	M	4490	—	—	50	57.2	0.37
90	–26.48	—	M	4320	—	—	90	58.45	0.66
90	–26.24	—	M	4170	—	—	90	59.54	0.67
91	–24.1	1	M	4477	—	—	26	—	—

Table A1.7 Full results for Sample G

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMCσ
1	–28.4	—	M	–872	—	—	—	57	111.45	0.78
1	–28.5	—	M	–909	—	—	—	64	111.97	0.89
2	–28.9	—	M	—	M	—	—	—	109.92	0.39
3	–29.33	—	M	–709	—	—	—	124	109.23	—
4	–29.04	0.58	M	—	M	—	—	—	111	—
5	–25	2	E	—	M	—	—	—	113.53	0.68
6	–29.106	0.004	M	—	—	—	—	—	110.87	0.35
7	–29.3	—	M	—	—	—	—	—	110.4	0.6
8	–29.4	—	M	—	—	—	—	—	110.8	0.3
9	–30.5	—	M	—	—	—	—	—	111.9	0.4
10	—	—	—	—	M	—	—	—	110	—
11	–33.1	0.1	M	—	M	—	—	—	107	0.5
12	–29.6	—	M	—	M	—	—	—	110.27	1.45
13	–29.93	—	M	—	M	—	—	—	107.71	0.83
15	–28.79	1.25	M	–800	—	—	—	50	110.53	0.75
16	—	—	—	—	—	—	—	—	105.01	0.49
17	–29.1	—	E	—	—	—	—	—	109.58	0.24
18	–29.04	0.59	M	—	M	—	—	—	109.76	0.83
19	–29.2	—	M	—	—	—	—	—	110.5	0.4
20	–28.5	—	M	—	—	—	—	—	109.18	0.43
21	—	—	E	—	—	—	—	—	111.03	0.44
23	–28.94	0.2	M	—	M	—	—	—	108.04	0.51
24	–28.97	—	—	–869	—	—	—	27	111.42	0.34
25	–29.3	—	M	—	M	—	—	—	111	0.5
26	—	—	—	—	—	—	—	—	121	—
27	–29.8	—	M	—	M	—	—	—	110.26	0.18
28	—	—	—	—	M	—	—	—	—	—
29	–28.9	—	M	—	M	—	—	—	109.6	0.5
30	–28.98	—	M	—	M	—	—	—	112.835	2.861

Table A1.7 Full results for Sample G *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMCσ
31	–29.4	—	M	–795	—	—	—	—	110.4	0.6
32	–29.3	—	M	—	M	—	—	—	112.55	0.71
33	–29.3	—	M	—	M	—	—	—	112.75	0.59
34	–29.3	—	M	—	M	—	—	—	111.39	0.48
35	–26	—	M	—	M	—	—	—	110	1
36	–29.9	—	M	–804	—	—	—	31	110.5	0.4
37	–29.2	0.2	M	—	M	—	—	—	110.75	0.42
37	–29.2	0.2	M	—	M	—	—	—	110.17	0.48
38	–28.7	—	M	–820	—	—	—	33	110.75	0.46
39	–28.83	0.01	M	—	M	—	—	—	106.45	0.45
40	–29.3	0.6	M	—	M	—	—	—	110.84	0.25
41	–29.6	—	M	–850	—	—	—	17	111.16	0.23
42	–24.5	—	E	–858	M	—	—	59	111.3	0.5
43	–28.653	0.005	M	—	M	—	—	—	107	0.59
44	–28.9	—	M	–770	—	—	—	50	110	0.68
46	–29.2	—	M	—	M	—	—	—	110.93	0.52
47	–28.9	0.1	M	—	M	—	—	—	109.04	0.53
48	–29.3	—	M	—	M	—	—	—	110.4	0.8
49	—	—	—	–745	—	—	—	40	109.75	0.57
49	—	—	—	–700	—	—	—	60	109.04	0.83
49	—	—	—	–745	—	—	—	45	109.71	0.62
50	–28.3	—	M	—	M	—	—	—	109.83	0.4
50	–28.3	—	M	—	M	—	—	—	110.13	0.4
51	–29.8	0.1	M	—	M	—	—	—	110.92	0.4
51	–29.3	0.1	M	—	M	—	—	—	110.14	0.4
51	–28.9	0.1	M	—	M	—	—	—	111.07	0.42
52	–28.7	0.3	M	—	M	—	—	—	109.61	0.76
53	—	—	E	480	—	—	—	180	94.17	2.11
54	–29.5	0.5	M	—	M	—	—	—	110.33	0.568

Table A1.7 Full results for Sample G *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMCσ
55	–29.7	—	M	—	M	—	—	—	111.3	0.4
56	–28.9	0.2	M	—	M	—	—	—	109.1	1.2
57	–25	—	E	–772	—	—	—	41	110.08	0.51
58	–29.65	—	M	—	M	—	—	—	110.32	0.33
59	–29.2	—	M	–880	—	—	—	70	111.6	1
60	–28.81	—	M	—	M	—	—	—	110.59	0.37
61	–29.8	—	M	—	—	—	—	—	110	0.5
62	–29.68	0.05	M	–800	—	—	—	40	110.497	0.528
64	–29.3	—	M	—	—	—	—	—	111.27	0.49
65	–29.77	—	M	–823	—	—	—	21	110.79	0.29
66	–29.5	—	M	—	—	—	—	—	111.2	0.8
67	–25	—	E	—	—	—	—	—	111.271	—
68	–29	—	M	—	M	—	—	—	108.32	0.51
69	–25.9	0.2	M	—	—	—	—	—	102	0.8
70	—	—	—	—	—	—	—	—	104	7.8
70	—	—	—	—	—	—	—	—	103.1	7.7325
72	–28.6	—	M	—	—	—	—	—	110.7	0.4
72	–28.6	—	M	—	—	—	—	—	110.8	0.5
73	–30.27	—	M	—	M	—	—	—	111.08	0.22
74	–31.1	—	M	—	M	—	—	—	110.91	0.31
75	—	—	—	—	—	—	—	—	111.1	0.6
76	–28.61	—	M	—	M	—	—	—	113.7	0.34
77	–29.49	—	M	—	M	—	—	—	111.698	—
77	–29.49	—	M	—	M	—	—	—	112.6	—
78	—	—	—	—	—	—	—	—	103.98	4.424779
79	–28.65	—	M	—	M	—	—	—	110.32	0.55
81	–25	3	E	—	M	—	—	—	104.78	0.7
82	–28.87	—	M	–810.01	—	—	—	30.15	110.68	0.41
83	–29.2	—	M	–838	—	—	—	20	111	0.28

Table A1.7 Full results for Sample G *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMCσ
84	–28.2	—	M	—	M	—	—	—	109.25	0.49
84	–28.2	—	M	—	M	—	—	—	109.39	0.44
84	–28.2	—	M	—	M	—	—	—	110.29	0.46
85	–29.6	—	E	—	M	—	—	—	111.05	0.12
86	–29.6	—	M	–838	—	—	—	29	111	0.39
87	–28.2	—	M	–785	—	—	—	50	109.6	—
88	–28.7	—	M	–805	—	—	—	21	110.54	0.29
88	–28.7	—	M	–776	—	—	—	26	110.15	0.36
89	–29.05	—	M	–835	—	—	—	40	110.94	0.58
90	–31.04	—	M	–1070	—	—	—	70	114.3	1.05
90	–30.02	—	M	–1080	—	—	—	70	114.35	1.05
91	–28.5	1	M	–828	—	—	—	24	110.86	0.33
92	–28.85	—	M	—	M	—	—	—	111.55	1.04

Table A1.8 Full results for Sample H

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
1	–23.9	—	M	2269	—	—	47	75.39	0.44
1	–24.5	—	M	2301	—	—	46	75.09	0.43
2	–25.1	—	M	2250	—	—	40	75.57	0.36
4	–23.8	0.47	M	2430	—	—	60	73.9	—
5	–25	2	E	2160	—	—	60	76.4	0.56
5	–25	2	E	2020	—	—	60	77.79	0.59
6	–24.124	0.006	M	2232	—	—	25	—	—
7	–24	—	M	2200	—	—	50	—	—
8	–23.1	—	M	2180	—	—	20	—	—
9	–21.1	—	M	2310	—	—	40	—	—
10	—	—	—	1900	—	—	60	—	—
11	–31.1	0.1	M	2180	—	—	40	76.23	0.38
11	–31.1	0.1	M	2110	—	—	70	76.9	1.2
12	–24.7	—	M	2340	—	—	130	74.8	1.2
13	–25.19	—	M	2560	—	—	60	72.67	0.52
15	–25.65	0.79	M	2180	—	—	30	76.26	0.27
15	–28.08	0.07	M	2160	—	—	30	76.39	0.27
16	—	—	—	2397	—	—	57	74.2	0.54
17	–25	—	E	2230	—	—	20	75.777	0.566
18	–25.86	0.15	M	2353	—	—	67	74.15	0.62
19	–25.8	—	M	2316	—	—	35	—	—
20	–23.9	—	M	2318	—	—	37	74.93	0.34
21	—	—	E	2040	—	—	50	—	—
22	–27.09	0.15	—	2160	—	—	90	76.44	0.83
23	–24.36	0.02	M	2421	—	—	55	73.98	0.51
24	–25.12	—	—	2208	—	—	35	—	—
25	–24.5	—	M	2210	—	—	50	75.95	0.47
26	—	—	—	1530	—	—	30	—	—
27	–25.5	—	M	2265	—	—	20	75.42	0.2

Table A1.8 Full results for Sample H *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
28	—	—	—	2170	70	80	—	—	—
29	–23	—	M	2150	—	—	70	76.51	0.58
30	–24.84	—	M	2110	—	—	200	76.888	1.951
32	–24.8	—	M	2270	—	—	60	75.41	0.57
33	–24.8	—	M	2210	—	—	40	75.92	0.39
34	–24.8	—	M	2240	—	—	30	75.67	0.28
35	–26	—	M	2250	—	—	40	75.57	0.38
36	–26.5	—	M	2209	—	—	32	76	0.3
37	–23.7	0.2	M	2263	—	—	42	75.44	0.39
37	–23.6	0.2	M	2248	—	—	42	75.58	0.39
38	–24.8	—	M	2174	—	—	23	76.29	0.22
39	–25.33	0.02	M	2505	—	—	35	73.18	0.32
40	–25.4	0.5	M	2311	—	—	63	75	0.59
41	–25.5	—	M	2252	—	—	19	75.56	0.18
42	–24.5	—	E	1839	—	—	66	79.5	—
43	–25.23	0.002	M	2356	—	—	38	74.58	0.35
44	–24.2	—	M	2100	—	—	70	77.02	0.62
45	—	—	—	2180	—	—	30	—	—
46	–23.6	—	M	2234	—	—	42	75.72	0.39
47	–24.3	0.1	M	2450	—	—	40	73.69	0.39
48	–25.8	—	M	2220	—	—	60	75.8	0.6
49	—	—	—	2210	—	—	45	75.93	0.45
49	—	—	—	2300	—	—	70	75.15	0.69
49	—	—	—	2260	—	—	50	75.51	0.48
50	–24.5	—	M	2260	—	—	30	75.45	0.27
50	–24.5	—	M	2270	—	—	30	75.42	0.27
51	–25.5	0.1	M	2210	—	—	45	75.97	0.44
51	–24.9	0.1	M	2220	—	—	30	75.81	0.31
51	–25.2	0.1	M	2240	—	—	35	75.66	0.31

Table A1.8 Full results for Sample H *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
51	–25	0.1	M	2210	—	—	35	75.95	0.34
52	–24.3	0.3	M	2230	—	—	80	75.76	0.75
53	—	—	E	2180	—	—	220	76.24	2.03
54	–25.3	0.5	M	2156	—	—	48	76.464	0.456
55	–25.5	—	M	2160	—	—	30	76.4	0.3
56	–24.7	0.2	M	2624	—	—	27	72.1	0.2
57	–25	—	E	2093	—	—	49	77.07	0.44
58	–25.01	—	M	2260	—	—	40	75.49	0.4
59	–24.1	—	M	2980	—	—	80	69	0.7
60	–24.2	—	M	2230	—	—	30	75.78	0.26
61	–25.8	—	M	2200	—	—	40	—	—
64	–24.9	—	M	2290	—	—	70	—	—
65	–25.82	—	M	2221	—	—	28	75.84	0.27
66	–24.8	—	M	2180	—	—	50	—	—
67	–25	—	E	2583	—	—	79	—	—
68	–24.7	—	M	2420	—	—	45	73.95	0.41
69	–27	0.2	M	2510	—	—	180	—	—
70	—	—	—	—	—	—	—	71.7	5.3775
70	—	—	—	—	—	—	—	77.9	5.8425
71	—	—	—	2170	—	—	40	76.3	—
72	–23.7	—	M	2295	—	—	35	—	—
73	–25.1	—	M	2190	—	—	20	76.12	0.15
74	–25.8	—	M	2240	—	—	40	75.66	0.25
74	–24.4	—	M	2230	—	—	40	75.74	0.25
75	—	—	—	2280	—	—	35	—	—
76	–24.92	—	M	2250	—	—	30	75.59	0.2
77	–24.83	—	M	2200	—	—	25	76.086	—
77	–24.83	—	M	2180	—	—	40	76.187	—
78	—	—	—	—	—	—	—	79.65	3.982301

Table A1.8 Full results for Sample H *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
79	–24.75	—	M	2250	—	—	25	—	—
81	–25	3	E	2690	—	—	70	71.52	0.66
82	–24.82	—	M	2301.72	—	—	37.2	75.09	0.35
83	–25.1	—	M	2240	—	—	23	75.66	0.21
84	–23.9	—	M	2160	—	—	40	76.39	0.36
84	–22.3	—	M	2230	—	—	50	75.75	0.41
85	–25.5	—	E	2175	—	—	11	76.2	0.11
86	–25.5	—	M	2142	—	—	36	76.59	0.34
87	–24	—	M	2140	—	—	50	76.11	—
88	–25.1	—	M	2135	—	—	30	76.64	0.27
88	–25.1	—	M	2139	—	—	39	76.62	0.37
89	–25.21	—	M	2300	—	—	45	75.1	0.44
90	–25.47	—	M	2220	—	—	80	75.89	0.79
90	–25.56	—	M	1970	—	—	80	78.23	0.81
91	–24.6	1	M	2247	—	—	24	—	—

Table A1.9 Full results for Sample I

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
1	–23.5	—	M	4650	—	—	60	56.06	0.42
1	–23.7	—	M	4574	—	—	58	56.59	0.41
2	–23.2	—	M	4560	—	—	30	56.67	0.18
3	–23.47	—	M	4640	—	—	128	56.12	—
4	–22.05	0.57	M	4560	—	—	60	56.7	—
5	–25	2	E	4140	—	—	60	59.7	0.45
6	–24.043	0.01	M	4401	—	—	40	—	—
7	–23.9	—	M	4380	—	—	50	—	—
8	–23.5	—	M	4500	—	—	20	—	—
9	–25	—	M	4560	—	—	60	—	—
10	—	—	—	4410	—	—	280	—	—
11	–21.7	0.1	M	4870	—	—	100	54.54	0.68
11	–21.7	0.1	M	5100	—	—	140	53.03	1.1
12	–24.1	—	M	4580	—	—	160	56.54	1.13
13	–23.9	—	M	4660	—	—	60	56	0.43
15	–22.81	0.66	M	4520	—	—	30	56.99	0.24
17	–25	—	E	4570	—	—	50	56.636	0.464
18	–25	—	E	4323	—	—	80	58.02	0.58
19	–20	—	E	4558	—	—	73	—	—
20	–22.3	—	M	4580	—	—	40	56.56	0.29
21	—	—	E	4250	—	—	150	—	—
23	–24.36	0.2	M	—	—	—	—	55.16	0.36
24	–23.55	—	—	4396	—	—	37	—	—
25	–23.6	—	M	4560	—	—	50	56.69	0.35
26	—	—	—	4800	—	—	100	—	—
27	–25.1	—	M	4500	—	—	25	57.12	0.17
28	—	—	—	3780	—	—	120	—	—
29	–23.2	—	M	4530	—	—	50	56.91	0.33
30	–23.73	—	M	4650	—	—	200	56.068	1.422

Table A1.9 Full results for Sample I *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
31	–23.9	—	M	4530	—	—	55	—	—
32	–23.73	—	M	4400	—	—	80	57.77	0.58
33	–23.7	—	M	4490	—	—	80	57.15	0.59
34	–23.7	—	M	4410	—	—	30	57.78	0.21
35	–24	—	M	4710	—	—	40	55.64	0.08
36	–24	—	M	4459	—	—	34	57.4	0.2
37	–23.8	0.2	M	4438	—	—	51	57.55	0.36
38	–23.5	—	M	4468	—	—	40	57.34	0.28
39	–23.74	0.02	M	4760	—	—	60	55.31	0.38
40	–23.4	0.5	M	4461	—	—	21	57.39	0.15
41	–23.9	—	M	4468	—	—	30	57.34	0.21
42	–24.5	—	E	3819	—	—	86	62.2	—
43	–23.432	0.007	M	4762	—	—	41	55.28	0.28
44	–25.5	—	M	4290	—	—	60	58.63	0.41
45	—	—	—	4380	—	—	240	—	—
46	–23.6	—	M	4556	—	—	47	56.72	0.34
47	–24.2	0.1	M	4470	—	—	45	57.31	0.3
48	–24.2	—	M	4430	—	—	70	57.6	0.5
49	—	—	—	4420	—	—	50	57.67	0.39
49	—	—	—	4420	—	—	60	57.71	0.43
49	—	—	—	4560	—	—	60	56.69	0.39
50	–23	—	M	4410	—	—	30	57.72	0.21
50	–23	—	M	4490	—	—	40	57.18	0.26
51	–23.8	0.1	M	4460	—	—	40	57.41	0.3
51	–23.5	0.1	M	4480	—	—	45	57.28	0.33
51	–23.5	0.1	M	4400	—	—	60	57.8	0.42
51	–23.6	0.1	M	4450	—	—	40	57.48	0.28
52	–23.1	0.3	M	4550	—	—	70	56.77	0.49
53	—	—	E	5650	—	—	290	49.53	1.73

Table A1.9 Full results for Sample I *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
54	–24.8	0.5	M	4669	84	85	—	55.921	0.587
55	–23.7	—	M	4510	—	—	40	57	0.3
56	–23.4	0.2	M	4541	—	—	39	56.8	0.9
57	–25	—	E	4466	—	—	58	57.36	0.4
58	–24.6	—	M	4490	—	—	40	57.15	0.27
59	–23.7	—	M	4490	—	—	80	57.2	0.6
60	–23.23	—	M	4520	—	—	50	56.97	0.3
61	–24.3	—	M	4435	—	—	40	—	—
62	–23.89	0.05	M	4500	—	—	50	57.128	0.327
64	–23.6	—	M	4520	—	—	70	—	—
65	–24.5	—	M	4440	—	—	29	57.54	0.21
66	–23.9	—	M	4435	—	—	65	—	—
67	–25	—	E	4014	—	—	139	—	—
68	–23.9	—	M	4650	—	—	90	56.03	0.59
69	–23.9	0.2	M	4520	—	—	250	—	—
70	—	—	—	—	—	—	—	57.5	4.3125
70	—	—	—	—	—	—	—	54.9	4.1175
71	—	—	—	4120	—	—	130	59.89	—
72	–23.2	—	M	4470	—	—	35	—	—
73	–23.94	—	M	4505	—	—	35	57.08	0.23
74	–22.8	—	M	4490	—	—	30	57.15	0.2
75	—	—	—	3960	—	—	100	—	—
76	–23.54	—	M	4450	—	—	30	57.45	0.21
77	–24.4	—	M	4460	—	—	45	57.38	—
78	—	—	—	—	—	—	—	60.18	3.761062
79	–24.85	—	M	4460	—	—	40	—	—
81	–25	3	E	4580	—	—	100	56.55	0.72
82	–23.31	—	M	4655.23	—	—	48.57	56.02	0.34
83	–23.8	—	M	4474	—	—	25	57.3	0.18

Table A1.9 Full results for Sample I *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
84	–23.1	—	M	4490	—	—	50	57.2	0.29
84	–23.1	—	M	4400	—	—	40	57.81	0.27
84	–23.1	—	M	4490	—	—	40	57.2	0.26
85	–23.9	—	E	4455	—	—	14	57.56	0.1
86	–24.5	—	M	4374	—	—	38	58.01	0.27
87	–24.25	—	M	4445	—	—	50	57.16	—
88	–23.3	—	M	4500	—	—	40	57.13	0.29
88	–23.3	—	M	4490	—	—	35	57.17	0.23
89	–23.76	—	M	4570	—	—	60	56.62	0.42
90	–24.96	—	M	4360	—	—	110	58.1	0.81
90	–24.43	—	M	4280	—	—	90	58.71	0.67
91	–24.9	1	M	4452	—	—	27	—	—
92	–23.73	—	M	4220	—	—	80	59.15	0.62

Table A1.10 Full results for Sample J

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMCσ
1	–28	—	M	–797	—	—	—	72	110.42	0.98
1	–28.2	—	M	–829	—	—	—	57	110.86	0.78
2	–28.7	—	M	—	M	—	—	—	110.65	0.36
4	–28.96	0.57	M	—	M	—	—	—	111.5	—
5	–25	2	E	—	M	—	—	—	113.67	0.68
6	–29.317	0.006	M	—	—	—	—	—	111.2	0.35
7	–29.2	—	M	—	—	—	—	—	109.4	0.7
8	–29.5	—	M	—	—	—	—	—	110.9	0.3
9	–30.8	—	M	—	—	—	—	—	111.9	0.4
10	—	—	—	—	M	—	—	—	113	—
11	–34.1	0.1	M	—	M	—	—	—	108.9	0.5
12	–29.6	—	M	—	M	—	—	—	109.83	1.45
13	–29.66	—	M	—	M	—	—	—	106.25	0.82
15	–28.51	1.06	M	–840	—	—	—	40	111.04	0.51
16	—	—	—	—	—	—	—	—	109.18	0.5
17	–29.1	—	E	—	—	—	—	—	108.91	0.25
18	–29.28	0.19	M	—	M	—	—	—	110.45	0.83
19	–29.2	—	E	—	—	—	—	—	109.4	0.4
20	–27.6	—	M	—	—	—	—	—	109.28	0.44
21	—	—	E	—	—	—	—	—	112.65	2.92
23	–29.68	0.2	M	—	M	—	—	—	108.35	0.93
24	–29.05	—	—	–884	—	—	—	28	111.63	0.35
25	–29.2	—	M	—	M	—	—	—	110.7	0.5
26	—	—	—	—	—	—	—	—	122	—
27	–29.7	—	M	—	M	—	—	—	110.17	0.18
28	—	—	—	—	M	—	—	—	—	—
29	–28.8	—	M	—	M	—	—	—	110.7	0.46
30	–28.93	—	M	—	M	—	—	—	110.078	2.836
32	–29.7	—	M	—	M	—	—	—	110.7	0.69

Table A1.10 Full results for Sample J *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMCσ
33	–29.7	—	M	—	M	—	—	—	111.04	0.72
34	–29.7	—	M	—	M	—	—	—	111.38	0.49
35	–26	—	M	—	M	—	—	—	110	1
36	–30.1	—	M	–796	—	—	—	22	110.4	0.3
37	–29.4	0.2	M	—	M	—	—	—	111.38	0.43
37	–29.4	0.2	M	—	M	—	—	—	110.63	0.48
38	–28.9	—	M	–803	—	—	—	40	110.51	0.55
39	–29.36	0.02	M	—	M	—	—	—	105.21	0.44
40	–29.1	1.2	M	—	M	—	—	—	110.96	0.24
41	–29.6	—	M	–835	—	—	—	15	110.96	0.21
42	–24.5	—	E	–837	M	—	—	61	111	0.5
43	–29.236	0.007	M	—	M	—	—	—	108.49	0.52
44	–28.4	—	M	–1080	—	—	—	50	114.37	0.69
45	—	—	—	—	—	—	—	—	108.4	0.5
46	–29	—	M	—	M	—	—	—	110.16	0.49
47	–29	0.1	M	—	M	—	—	—	109.83	0.54
48	–29.7	—	M	—	M	—	—	—	111.4	0.7
49	—	—	—	–770	—	—	—	40	110.04	0.56
49	—	—	—	–770	—	—	—	120	110.01	1.66
49	—	—	—	–775	—	—	—	45	110.13	0.63
50	–28.6	—	M	—	M	—	—	—	109.99	0.37
50	–28.6	—	M	—	M	—	—	—	110.92	0.38
51	–29	0.1	M	—	M	—	—	—	111.77	0.46
51	–28.5	0.1	M	—	M	—	—	—	110.52	0.39
51	–29.5	0.1	M	—	M	—	—	—	112.16	0.57
51	–29.2	0.1	M	—	M	—	—	—	110.6	0.46
52	–28.4	0.3	M	—	M	—	—	—	110.06	0.52
53	—	—	E	230	—	—	—	190	97.14	2.29
54	–29	0.5	M	—	M	—	—	—	110.22	0.571

Table A1.10 Full results for Sample J *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMCσ
55	–29.5	—	M	—	M	—	—	—	111.1	0.5
56	–28.9	0.2	M	—	M	—	—	—	109.2	1.1
57	–25	—	E	–853	—	—	—	41	111.2	0.51
58	–29.8	—	M	—	M	—	—	—	110.4	0.4
59	–29.2	—	M	–820	—	—	—	60	110.7	0.9
60	–30.56	—	M	—	M	—	—	—	111.5	0.4
61	–29.4	—	M	—	—	—	—	—	109.5	0.5
62	–29.83	0.05	M	–890	—	—	—	30	111.672	0.466
62	–29.4	0.05	M	–830	—	—	—	30	110.873	0.469
64	–29.3	—	M	—	—	—	—	—	111.4	0.53
65	–29.91	—	M	–842	—	—	—	26	111.05	0.36
66	–29.3	—	M	—	—	—	—	—	110.5	0.8
67	–25	—	E	—	—	—	—	—	112.277	—
68	–29.4	—	M	—	M	—	—	—	106.27	0.51
69	–27.5	0.2	M	—	—	—	—	—	106.1	1
70	—	—	—	—	—	—	—	—	108.8	8.16
70	—	—	—	—	—	—	—	—	101.3	7.5975
71	—	—	—	—	M	—	—	—	111.03	0.9
72	–28.9	—	M	—	—	—	—	—	111.5	0.4
73	–29.74	—	M	—	M	—	—	—	110.76	0.22
74	–27	—	M	—	M	—	—	—	110.72	0.31
75	—	—	—	—	—	—	—	—	110.8	0.6
76	–29.11	—	M	—	M	—	—	—	111.29	0.33
77	–29.33	—	M	—	M	—	—	—	111.342	—
77	–29.33	—	M	—	M	—	—	—	111.265	—
78	—	—	—	—	—	—	—	—	117.26	4.646
79	–28.18	—	M	—	M	—	—	—	109.62	0.55
81	–25	3	E	—	M	—	—	—	100.33	0.69
82	–27.38	—	M	–832.66	—	—	—	30.51	110.92	0.42

Table A1.10 Full results for Sample J *(Continued)*

Lab	$\delta^{13}C$	$\sigma\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMCσ
83	–29.1	—	M	–802	—	—	—	21	110.51	0.28
84	–28.6	—	M	—	M	—	—	—	110.61	0.46
84	–28.6	—	M	—	M	—	—	—	110.16	0.49
84	–28.6	—	M	—	M	—	—	—	110.28	0.46
85	–29.6	—	E	—	M	—	—	—	111.28	0.12
86	–30.8	—	M	–788	—	—	—	33	110.31	0.46
87	–29.22	—	M	–985	—	—	—	50	112.37	—
88	–28.8	—	M	–807	—	—	—	22	110.56	0.3
88	–28.8	—	M	–779	—	—	—	27	110.18	0.37
89	–29.16	—	M	–825	—	—	—	40	110.81	0.58
90	–30.82	—	M	–930	—	—	—	70	112.23	1.04
90	–29.93	—	M	–940	—	—	—	70	112.37	1.03
91	–28.6	1	M	–834	—	—	—	21	110.94	0.29

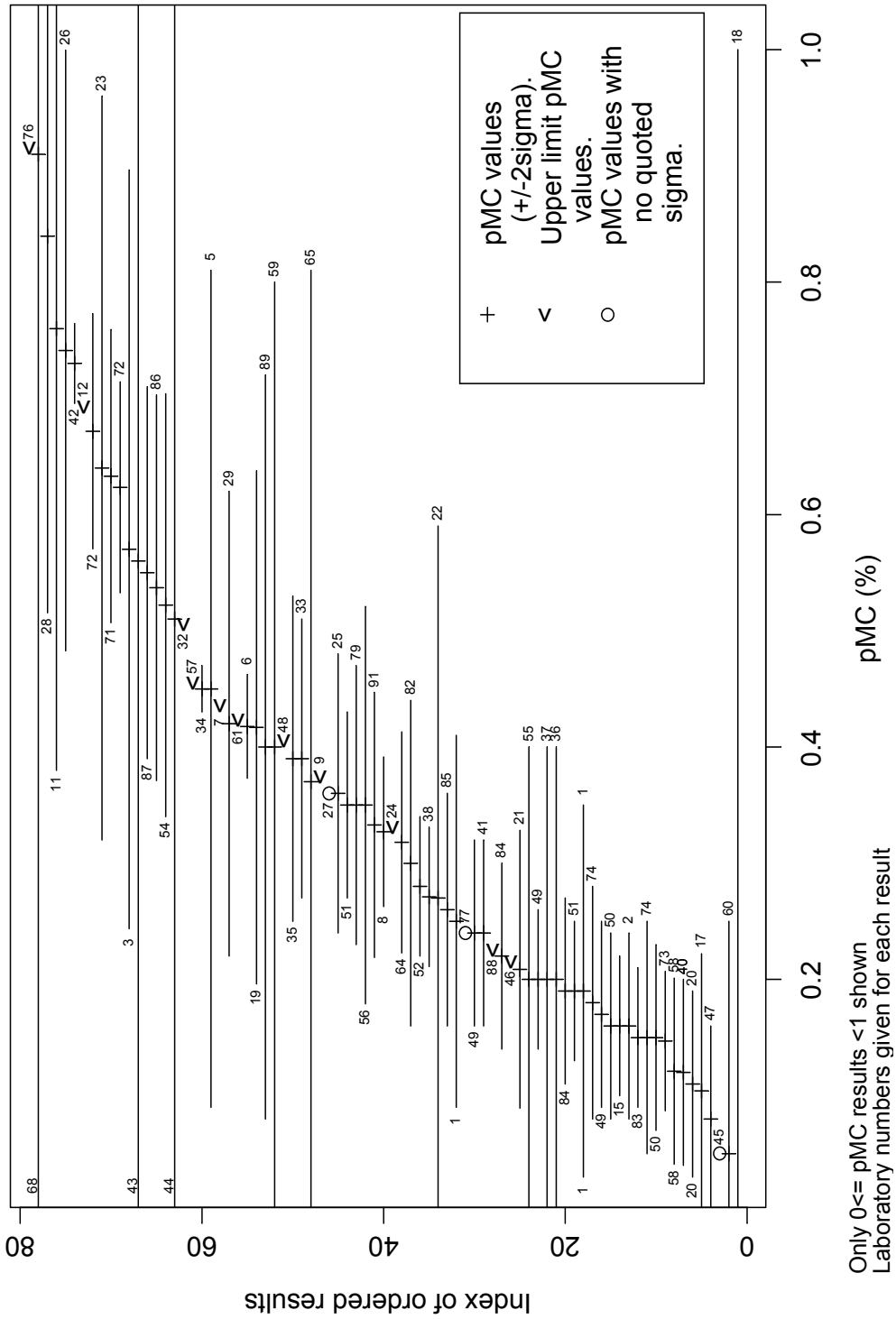

Figure A1.1 Distribution plot of pMC ±2 sigma for Kauri A (all laboratories)

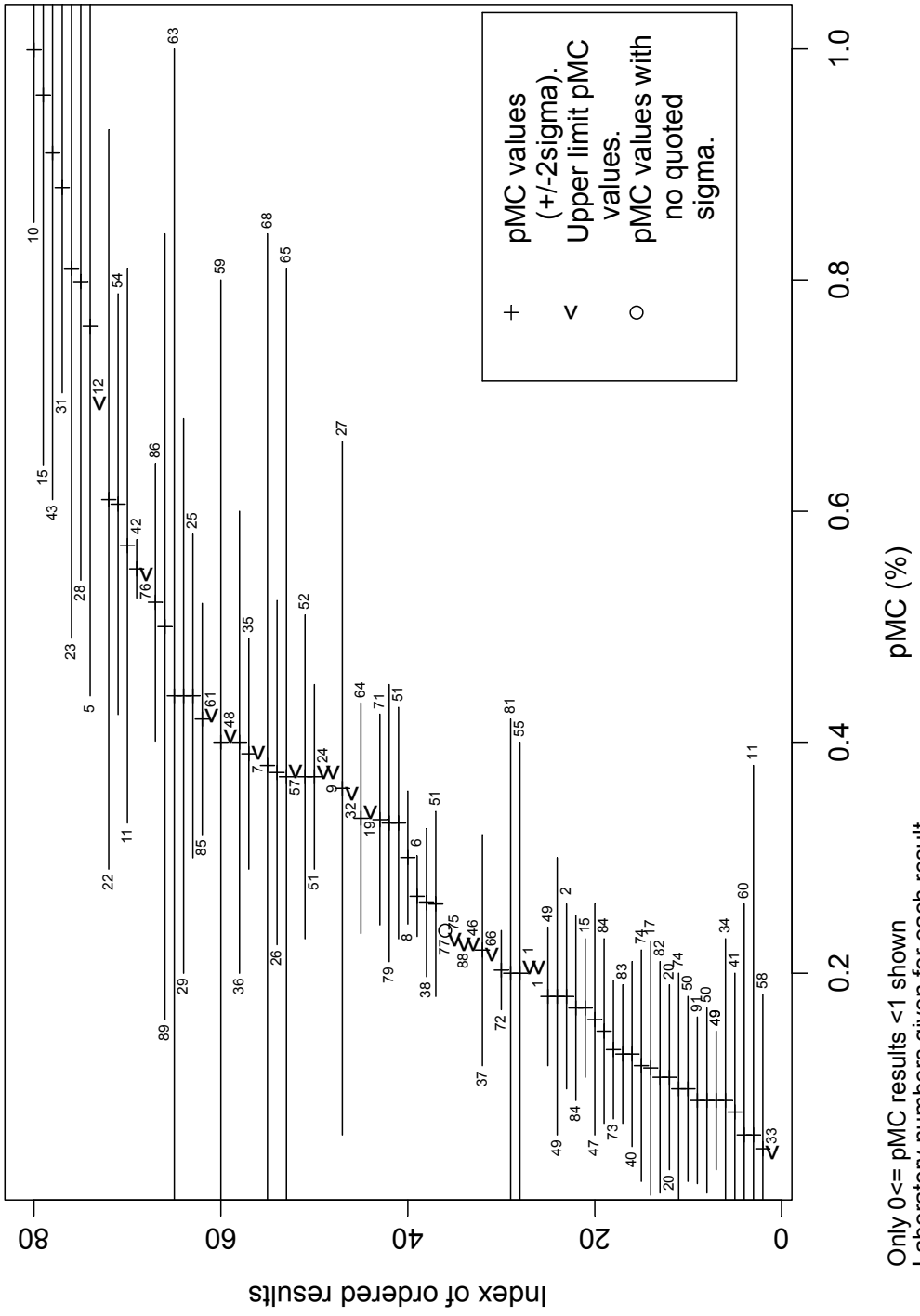

Figure A1.2 Distribution plot of pMC ±2 sigma for Kauri B (all laboratories)

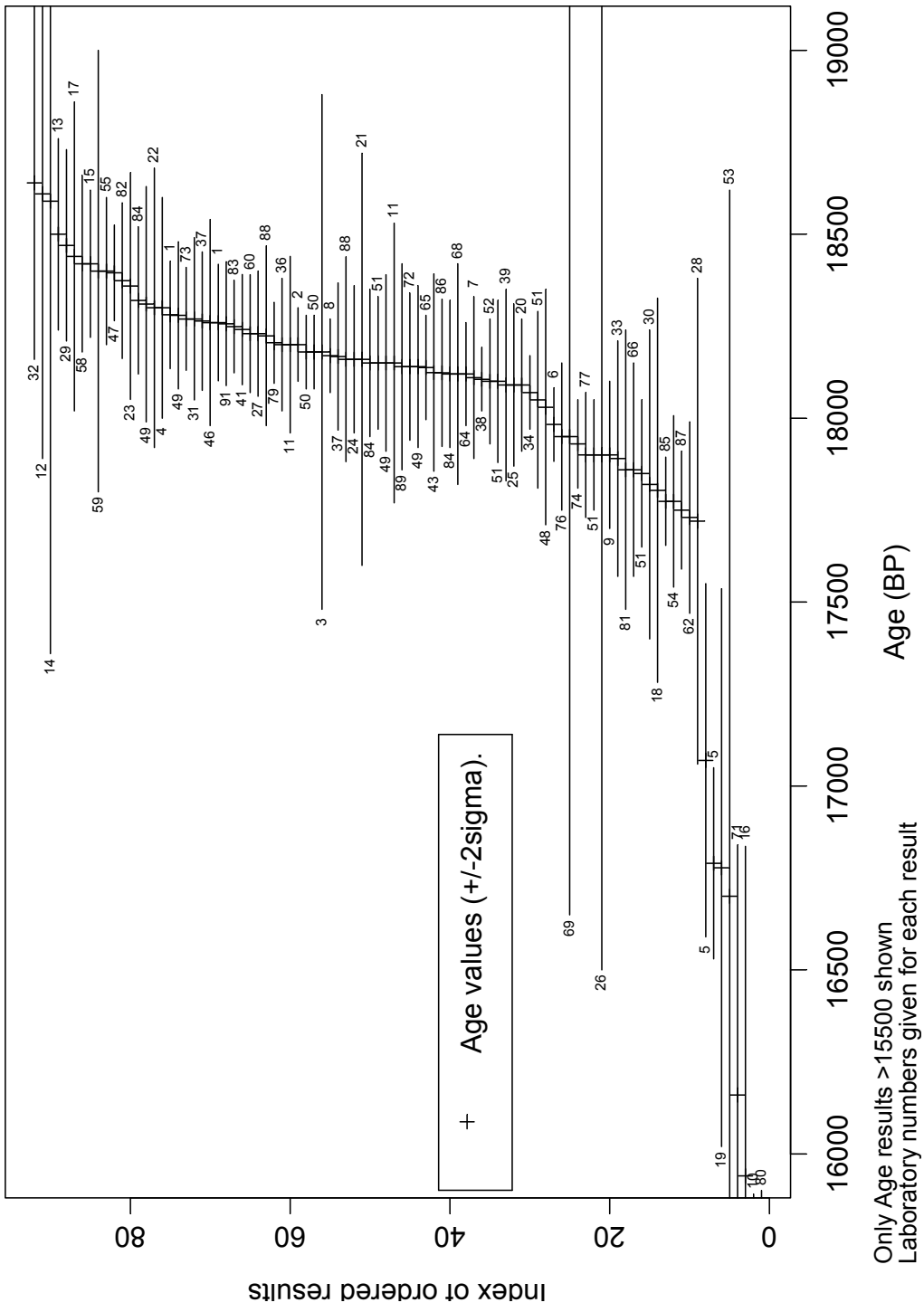

Figure A1.3 Distribution plot of Age ±2 sigma for turbidite, C, (all laboratories)

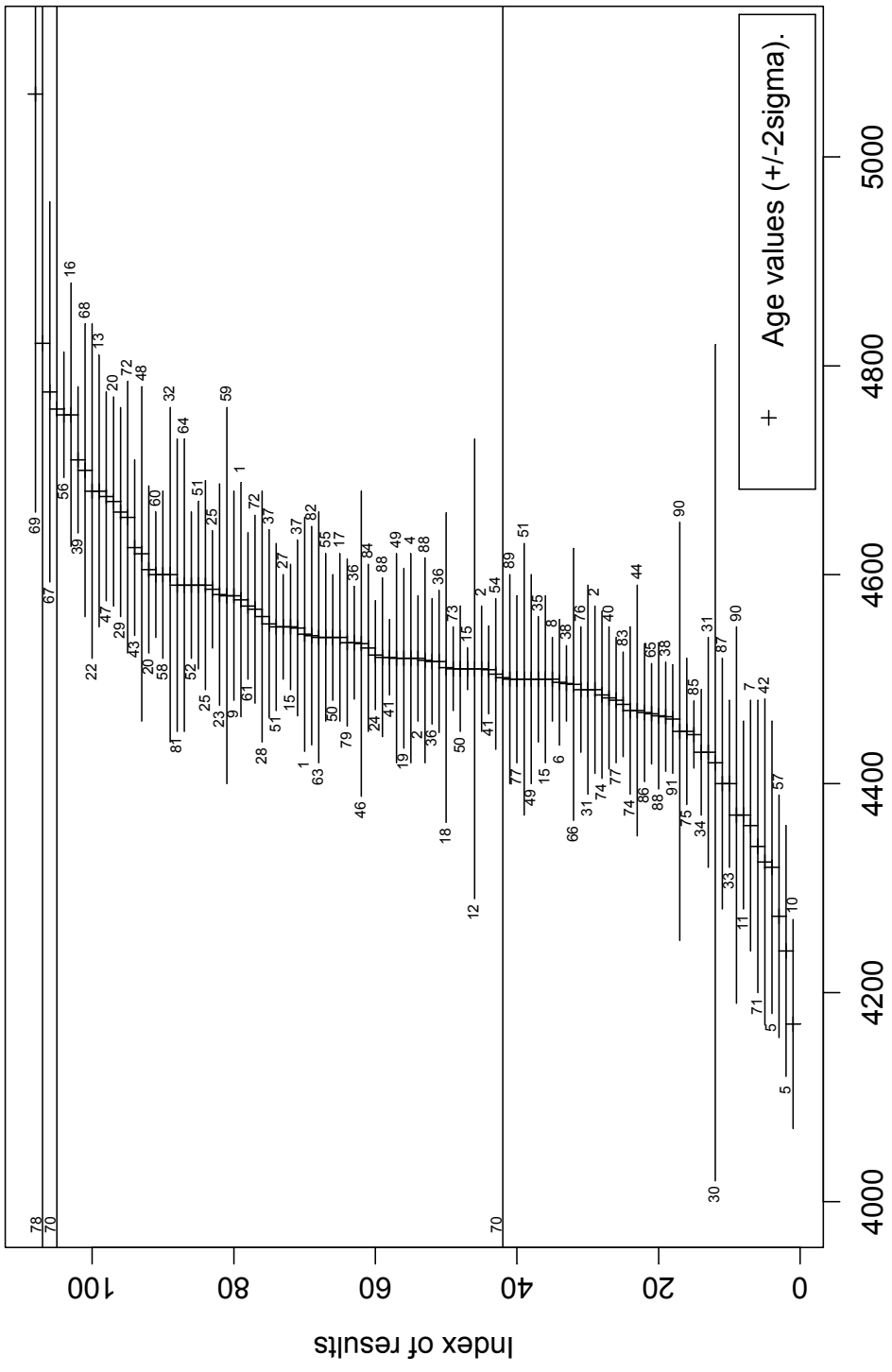

Figure A1.4 Distribution plot of Age ±2 sigma for dendro wood, D, (all laboratories)

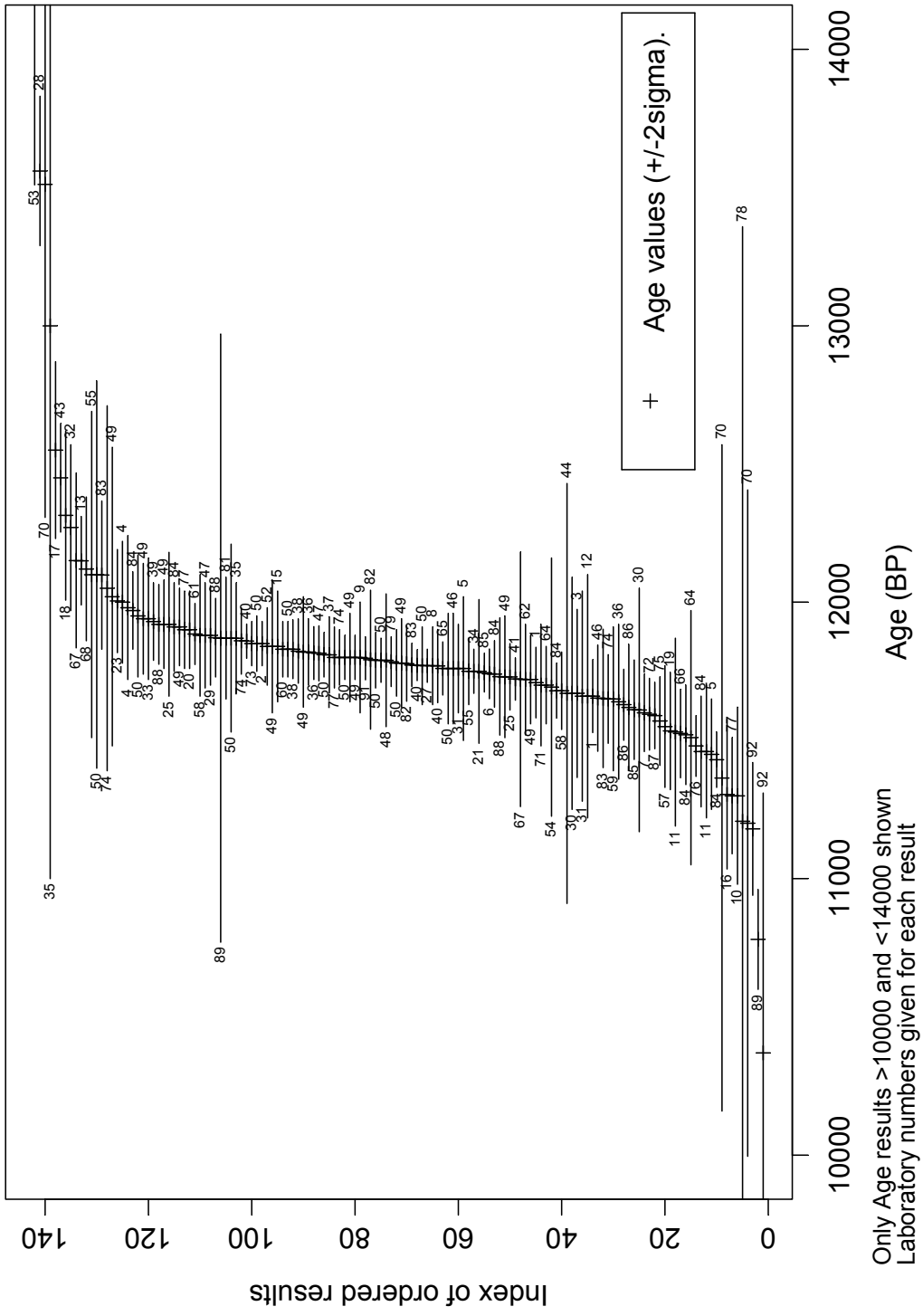

Figure A1.5 Distribution plot of Age ±2 sigma for humic acid, E, (all laboratories)

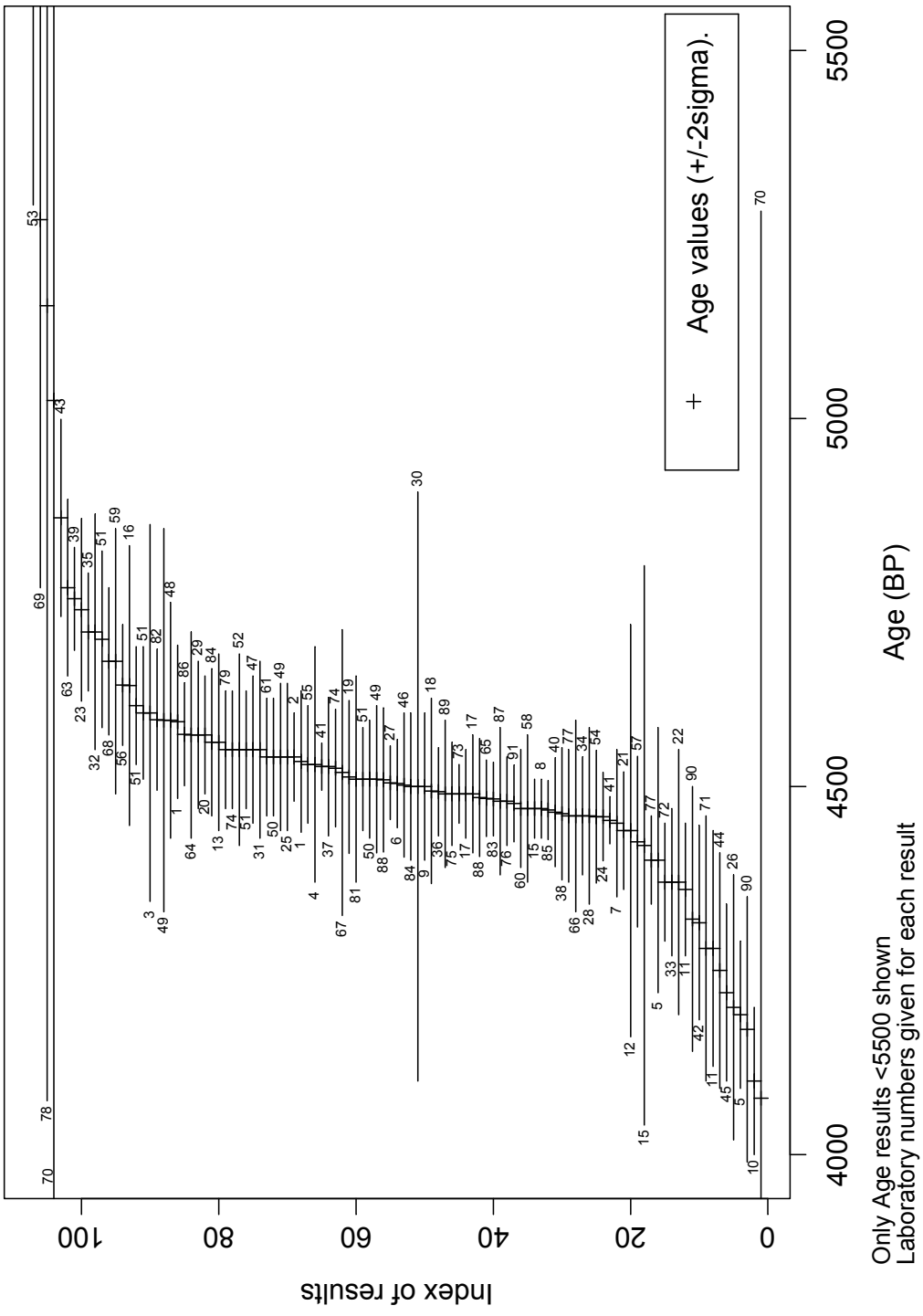

Figure A1.6 Distribution plot of Age ±2 sigma for dendro wood, F, (all laboratories)

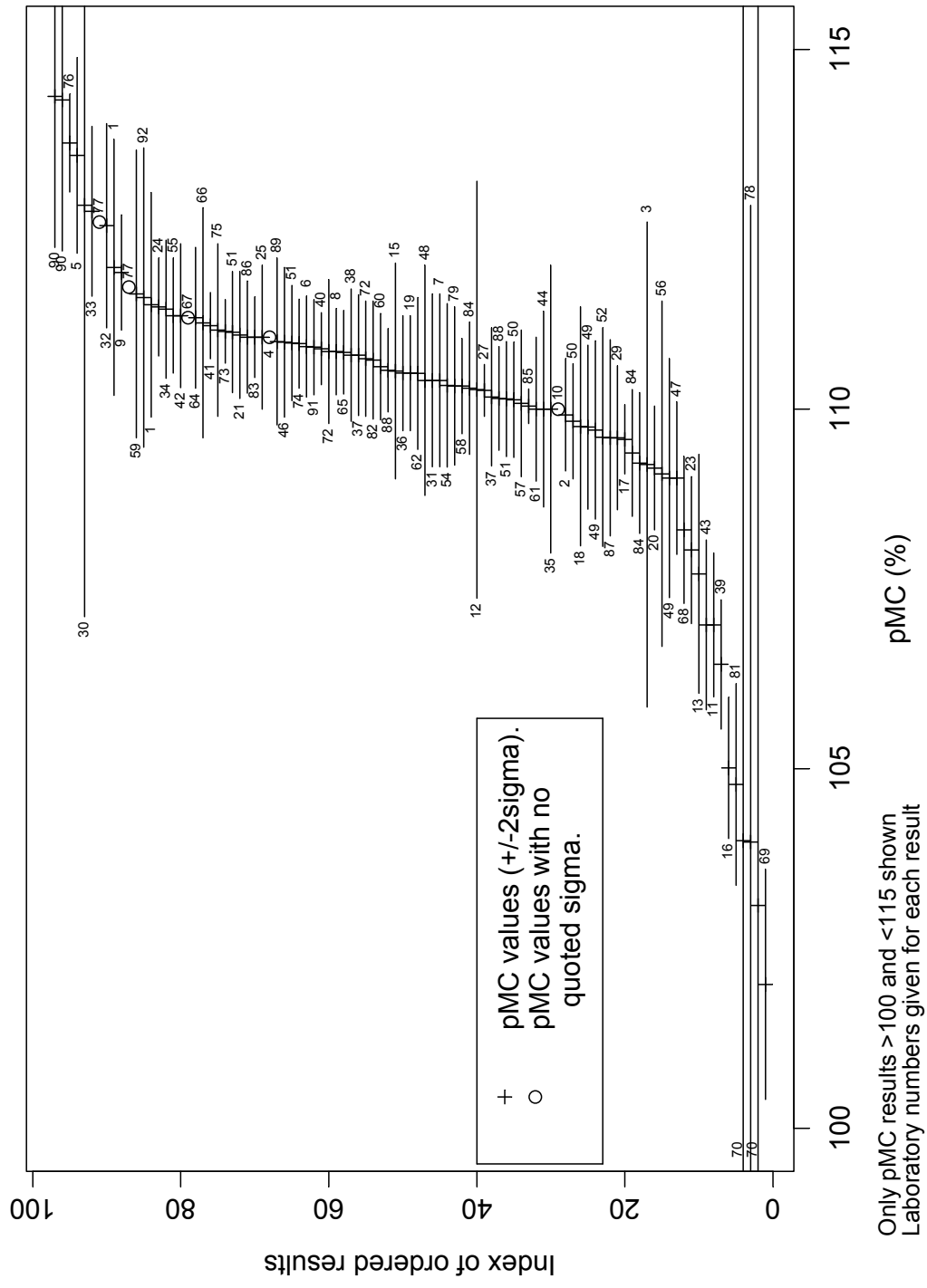

Figure A1.7 Distribution plot of pMC ±2 sigma for barley mash, G, (all laboratories)

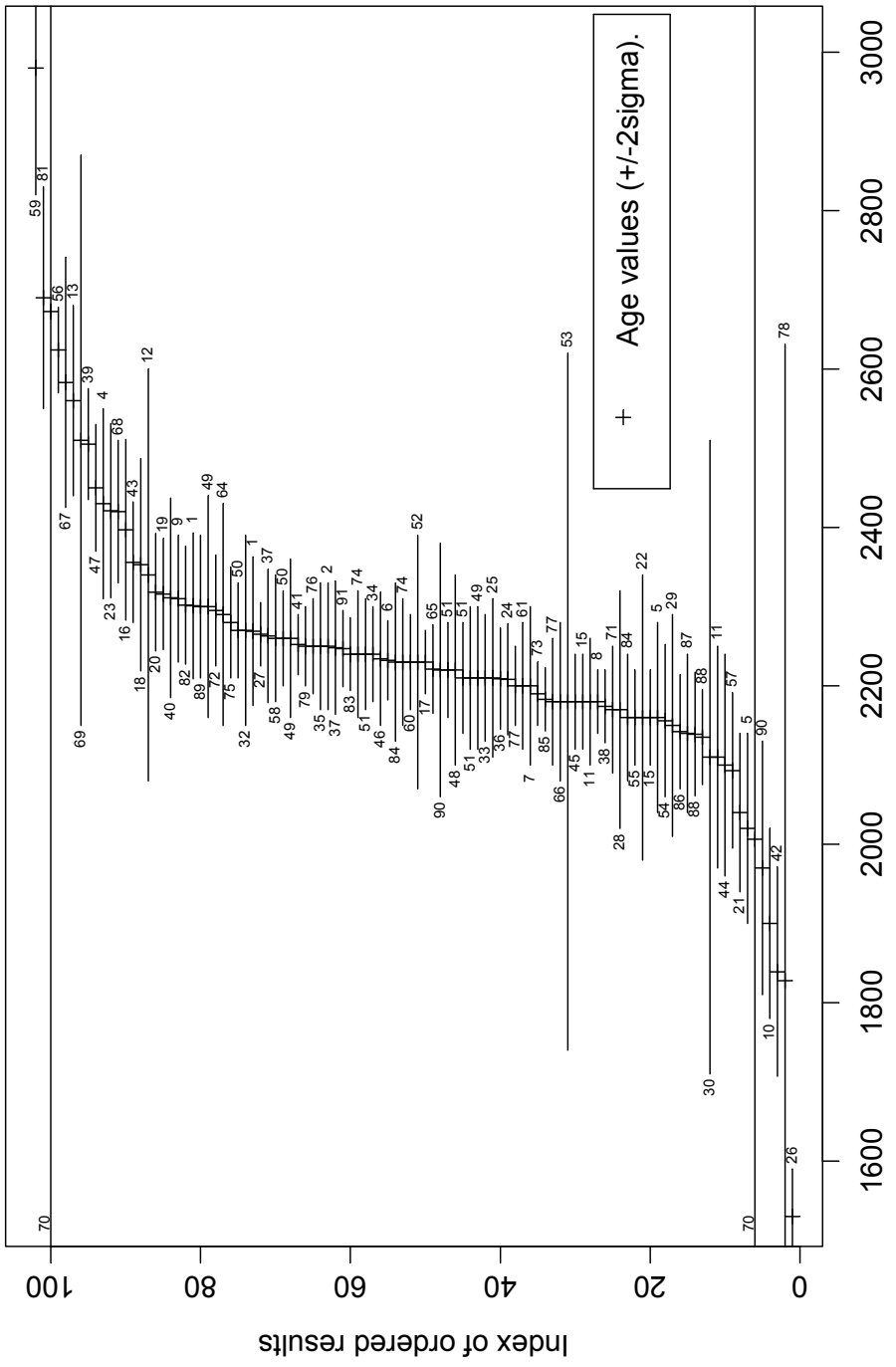

Figure A1.8 Distribution plot of Age ±2 sigma for dendro wood, H, (all laboratories)

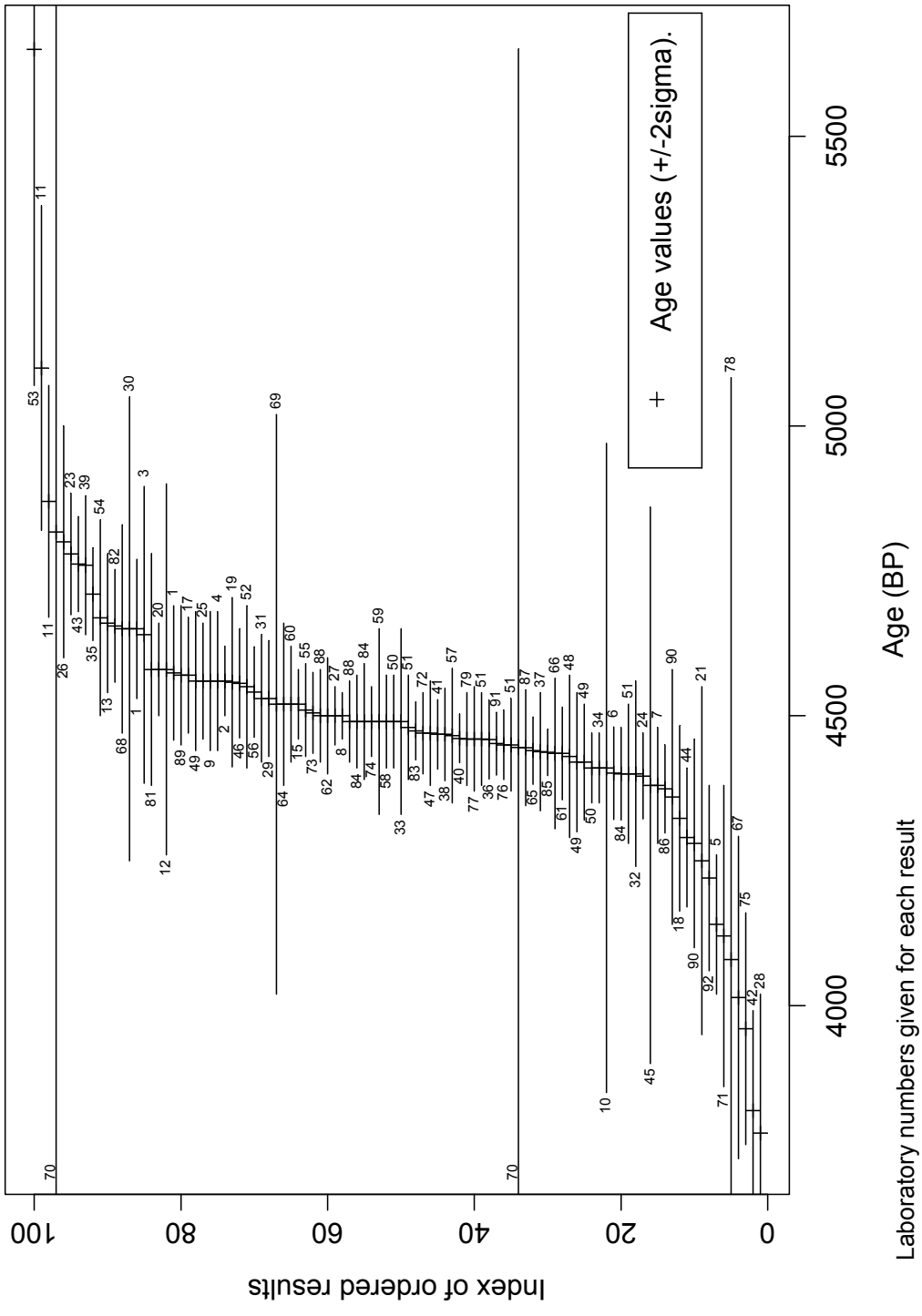

Figure A1.9 Distribution plot of Age ±2 sigma for cellulose, I, (all laboratories)

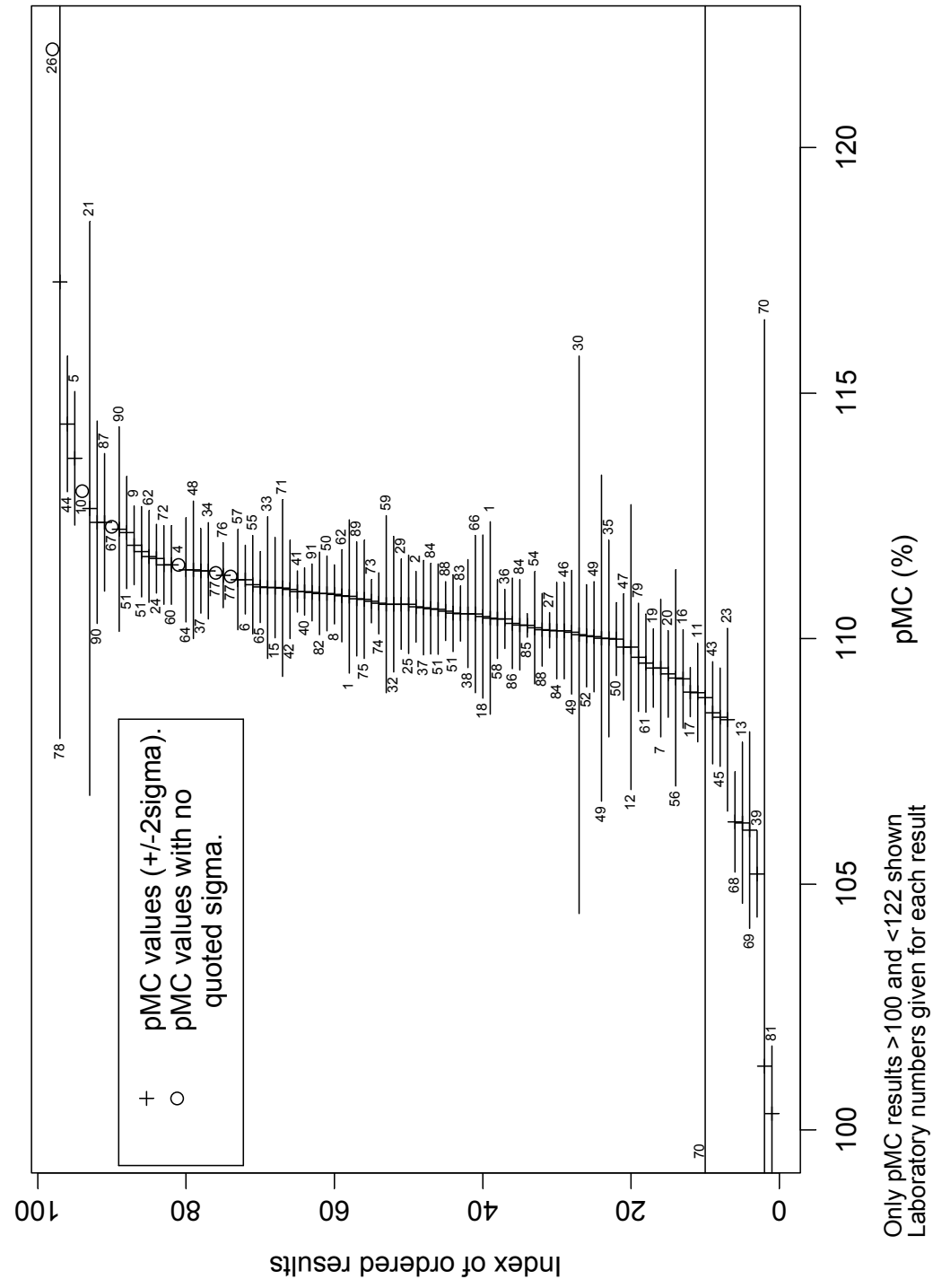

Figure A1.10 Distribution plot of pMC ±2 sigma for barley mash, J, (all laboratories)

APPENDIX 2: RESULTS FOR OPTIONAL SAMPLES (FIRI)

Table A2.1 Full results for Sample K

Lab	$\delta^{13}C$	M/E	Age	Limit	σ_L	σ_U	σ	pMC	pMCσ
18	–25	E	73	—	—	—	66	99.1	0.8
25	–24.9	M	80	—	—	—	50	—	—
30	–25	M	64	—	201	206	—	99.2	2.514
35	–23.5	M	310	—	—	—	40	96.2	0.6
55	–25.1	M	—	M	—	—	—	97.7	0.5
68	–25.45	M	190	—	—	—	70	—	—
76	–24	M	—	M	—	—	—	98.75	0.39
77	–26.04	M	—	M	—	—	—	100.482	—
77	–26.04	M	40	—	—	—	20	99.727	—

Table A2.2 Full results for Sample L

Lab	$\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
18	–25	E	2386	—	—	78	74.29	0.72
25	–25.3	M	2500	—	—	50	—	—
30	–25.5	M	2602	201	207	—	72.333	1.837
31	–25.6	M	2530	—	—	55	72.97	—
35	–24.8	M	2500	—	—	80	73.2	0.7
55	–26	M	2530	—	—	40	72.9	0.4
68	–25.47	M	2790	—	—	45	—	—
76	–25.63	M	2395	—	—	35	74.24	0.32
77	–26.66	M	2410	—	—	30	74.088	—
77	–26.66	M	2410	—	—	25	74.093	—

Table A2.3 Full results for Sample M

Lab	$\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
18	–27	E	11,413	—	—	129	24.14	0.39
25	–29.1	M	11,090	—	—	100	—	—
31	–29.4	M	11,090	—	—	85	—	—
31	–29.6	M	11,000	—	—	75	—	—
35	–28.8	M	10,710	—	—	50	26.3	0.1
50	–29.9	M	11,200	—	—	40	24.79	0.1
50	–30	E	11,180	—	—	40	24.85	0.11
50	–30	E	11,120	—	—	40	25.06	0.1
50	–29.9	M	11,070	—	—	40	25.21	0.1
55	–29.6	M	11,340	—	—	60	24.4	0.2
68	–29.46	M	11,330	—	—	120	—	—
76	–29.58	M	11,085	—	—	55	25.16	0.17
77	–30.61	M	11,300	—	—	70	24.619	—
77	–30.61	M	11,300	—	—	50	24.634	—
77	–29.55	M	10,850	—	—	165	24.174	—

Table A2.4 Full results for Sample N

Lab	$\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
35	–21.5	M	26,000	—	—	500	3.9	0.3
50	–20.1	M	28,530	—	—	210	2.87	0.07
50	–19.7	M	28,650	—	—	220	2.83	0.07
88	–20.6	M	28,574	—	—	418	2.85	0.15
88	–20.8	M	28,746	—	—	421	2.79	0.15

Table A2.5 Full results for Sample O

Lab	$\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
35	–20.5	M	34,700	—	—	1500	1.3	0.2
50	–20.4	M	37,980	—	—	670	0.88	0.07
50	–20.6	M	37,120	—	—	600	0.98	0.07
88	–20.7	M	40,504	—	—	1659	0.65	0.15
88	–20.8	M	38,773	—	—	1366	0.8	0.15

Table A2.6 Full results for Sample P

Lab	$\delta^{13}C$	M/E	Age	σ_L	σ_U	σ	pMC	pMCσ
35	–21.4	M	12,300	—	—	100	21.6	0.3
50	–20.8	M	12,600	—	—	40	20.84	0.1
50	–20.3	M	12,610	—	—	50	20.8	0.1
88	–21.1	M	12,696	—	—	65	20.59	0.17
88	–21.3	M	12,586	—	—	69	20.87	0.18

APPENDIX 3: STATISTICAL METHODS

There are a number of uses for statistical methods in the analysis of the results from TIRI and FIRI. They are exploratory and descriptive statistics, formal hypothesis testing (one-way *analysis of variance* to assess the significance of laboratory factors), estimation of consensus values (and errors), reliability analysis for assessment of Sample AB, measures of agreement (between duplicates), estimation of laboratory offsets, and error multipliers based on deviations. Our description of these techniques is only very brief and those interested in further detail are directed towards more specialized literature.

A3.1 EXPLORATORY AND DESCRIPTIVE METHODS

The summary statistics typically quoted are the mean (average), median (middle value or 50th percentile of the distribution in the ordered set of results), the lower and upper quartile (or the 25th and 75th percentile of the distribution), the interquartile range or IQR (which is the difference between the lower and upper quartile), the standard deviation, and the standard error of the mean (standard deviation / $\sqrt{n}$). The IQR shows the range over which the middle 50% of the data lie. Boxplots are commonly used throughout to present a visualization of the distribution of the results. A boxplot is simply constructed using the summary statistics of median, upper and lower quartile, and minimum and maximum; although in most statistical software, any extremes or outliers are first identified as shown below in Figure A3.1

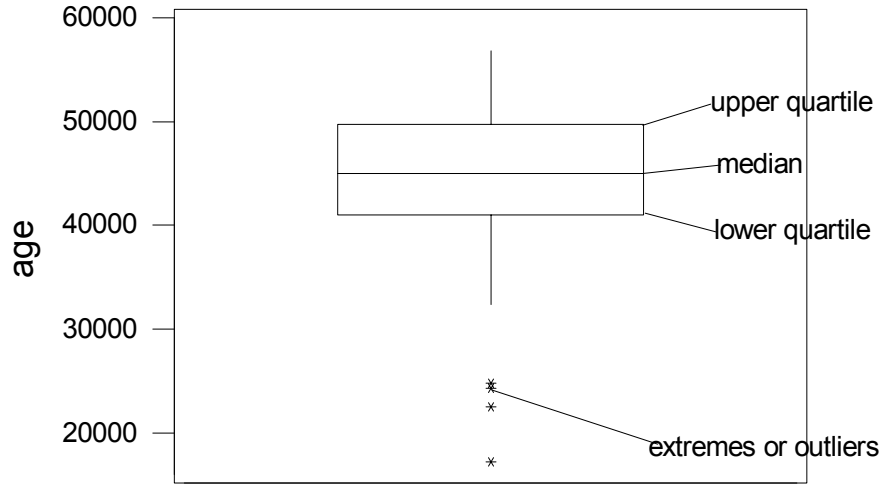

Figure A3.1 Construction of a boxplot

What is an outlier? In both TIRI and FIRI, we have used a statistical interpretation of the definition that an outlier is an unusual observation, either extremely small or extremely large. The definition is based on the quartiles and the interquartile range.

Outlying results are typically defined as those values which are greater than 3 interquartile ranges from the nearest of either the lower or upper quartiles. That is when a result is either greater than Q3 + 3(Q3–Q1) or less than Q1 – 3(Q3–Q1), where Q1 and Q3 are the lower and upper quartiles, respectively.

This interpretation was implemented in the screening of results before calculation of the consensus values.

A3.2 HYPOTHESIS TESTING (SPECIFICALLY, THE ONE-WAY ANALYSIS OF VARIANCE)

A statistical hypothesis test provides a decision rule for choosing between 2 competing hypotheses (one called the *null*, the other called the *alternative*). The decisions are "do not reject the null" or "reject the null." The evidence (data) is summarized in the value of the test statistic, and the chances of making the wrong decision (which in this case would be rejecting the null in favor of the alternative when the null is, in fact, true) is quantified in the *p-value* of the test.

Conventionally, p-values are considered to be statistically significant if they are less than 0.05. Thus, in the output from the test, if the p-value is less than 0.05, then we would reject the null hypothesis in favor of the alternative (see Table A3.1). Since the p-value is greater than 0.05, then we would not reject the null.

Table A3.1 Analysis of variance for age, using adjusted SS for tests

Source	DF	Seq SS	Adj SS	Adj MS	F	**P**
Type	2	99587	99587	49793	0.99	**0.375**
Error	105	5279518	5279518	50281		
Total	107	5379105				

In a one-way analysis of variance, the null hypothesis states that the mean age/activity is the same for all levels of the experimental factor (e.g., LSC, GPC, and AMS laboratories). The alternative states that the mean ages/activities are not all the same, but note that it does not specify where the differences might lie.

When the p-value is less than 0.05, multiple follow-up comparisons are often carried out to identify where the differences lie.

A3.3 ESTIMATION OF CONSENSUS VALUES

The algorithm for the calculation of the consensus values was given in Section 7. In the last stage, the weighted mean of the results and the associated error are calculated. The formulae used can be found in Rozanski et al. (1992), where the same algorithm was used. The basis for the calculation is a simple measurement model:

$$X_i \sim N(\alpha, s_i^2)$$

where X_i is the ^{14}C date, s_i is the quoted error, α is the true (or, in this case, consensus age), and $i = 1, \ldots, N$, the number of results (laboratories) to be used in the calculation.

Maximum likelihood (or least squares) estimates of α are given by

$$\alpha = \Sigma (x_i / s_i^2) / \Sigma(1 / s_i^2)$$

and the uncertainty on the estimate is given by

$$\sqrt{(1 / \Sigma[1 / s_i^2])}$$

A3.4 RELIABILITY (OR SURVIVAL ANALYSIS)

Analysis for Kauri A and B was modified because many of the results were not returned as a finite age. By this we mean, that for age, many results were simply quoted as "greater than" a specific age or indeed as "background" (both cases are described as *censored*). A *censored* datum is one for which the result is expressed as "> age" BP. This area of statistical analysis is very common in Medicine where survival after treatment is of importance and involves some specialized statistical techniques (a good general reference to this area is Cox DR and Oakes D. 1984. *Analysis of Survival Data.* London: Chapman and Hall).

Given the censored nature of the data, non-parametric methods of estimation of the mean age—used commonly in a survival or reliability analysis (in particular, the *Kaplan-Meier* survival estimator)—have been used to estimate the "mean" age of the sample. Reliability plots (or survivor curves) display the "survival" probabilities versus time, which in this context is the probability that the sample is greater than age *t*. Each point on the plot represents the proportion of results greater than age *t* and the non-parametric reliability curve is shown graphically as a step function. See Figure A3.2 for an illustration. In addition, common measures of the center and spread of the distribution of age are estimated. It should be noted that the mean is very sensitive to large ages, while the median, Q1 (25th percentile), Q3 (75th percentile), and the IQR (interquartile range) are resistant, so they are quoted in preference.

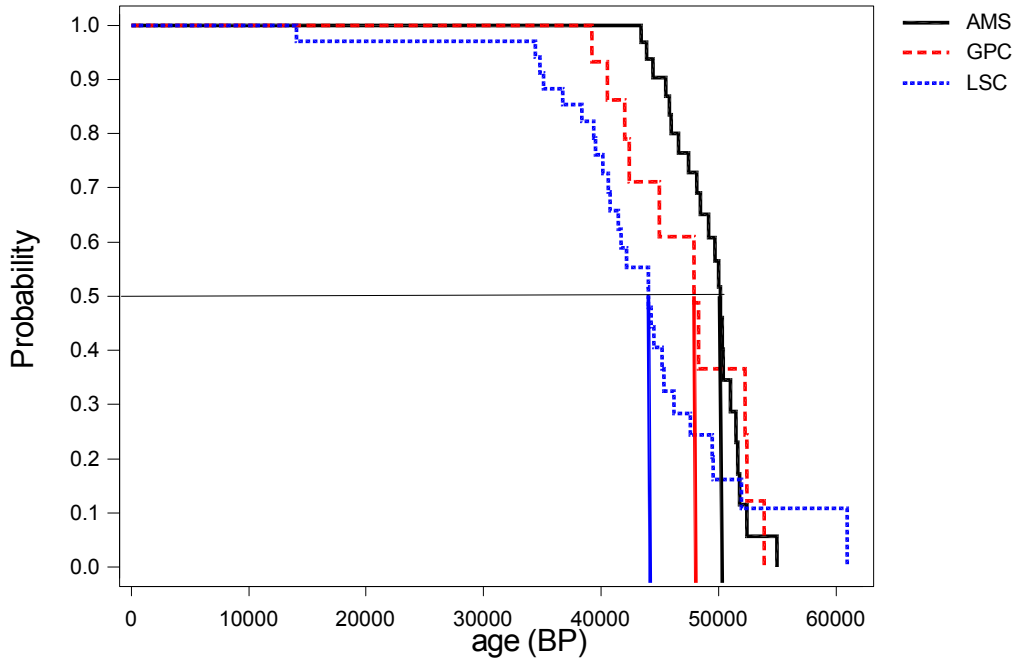

Figure A3.2 Reliability plot

In Figure A3.2, we can see the probability that the result is greater than 44,000 BP for an LSC laboratory is 0.5; for a GPC laboratory, the value is 49,000 BP; and for an AMS laboratory, it is 50,000 BP (from the intersection of the horizontal line at 0.5 with the 3 curves and then projection down onto the age axis).

We can also formally test whether the reliability (survival) curves are the same for the 3 laboratory types, and the evidence is summarized in the p-value of the test, which is interpreted in the same way as described previously in Section A3.2.

A3.4 MEASURES OF AGREEMENT

Duplicates (the same material measured as 2 independent replicates in the same laboratory, or the same material measured by 2 different techniques) are usually analyzed to assess the agreement between the results and, ultimately, precision. In the past, a correlation coefficient (which measures the degree of linear association between the pair) has often been used. We *a priori* expect the correlation coefficient to be high (since it is the same material), so in preference we have used a measure of agreement plot, which plots the difference in the 2 results against the average of the 2 results for each of the *n* laboratories. We expect to see the scatter of points to be uniform over the horizontal range and to be randomly scattered around zero in the vertical direction when the results are in agreement.

A further complication in the ^{14}C case is the role of the quoted errors. In this case, we have also chosen to calculate standardized deviations (as the difference between the duplicate results divided by the error on the difference). Such quantities should have a normal (or Gaussian) distribution with a mean of zero and a variance of 1. When squared, the normal distribution should have a Chi-squared distribution with a 1 degree of freedom *if* the 2 results are in agreement and the quoted errors represent all sources of variation in the results. This then forms the basis for a test of agreement between duplicates.

A3.5 LABORATORY OFFSETS AND ERROR MULTIPLIER ESTIMATION

For assessment of performance for an individual laboratory, we have assumed that the consensus values (or the dendro-dates for the known-age samples) can be treated as the "true" age/activity. Relative to the consensus values for each laboratory, an offset (and/or) an error multiplier, can be calculated. Error multipliers were used in TIRI, but were not used in FIRI.

In FIRI, the laboratory offset was defined as the average laboratory difference from the consensus profile (μ_i). The model used assumes that for a given laboratory, there is a potential systematic offset, α from the consensus profile, which we can again estimate by maximum likelihood or least squares. The form for α is that of a weighted average of the standardized deviations.

$$\alpha = (\Sigma(x_i - \mu_i)^2 / s_i^2) / \Sigma(1 / s_i^2)$$

We can then test whether α is plausibly zero (i.e. that the laboratory is, on average, accurate). The measurement model for the results from a laboratory underlying this estimation equation is:

$$X_i \sim N(\mu_i + \alpha, s_i^2)$$

where X_i is the ^{14}C age for sample *i*, μ_i is the true age, and s_i is the quoted error for i = 1,…, *n* the number of reference materials

In TIRI, a laboratory error multiplier was estimated for those laboratories where first there was no evidence against the null hypothesis that α was plausibly zero.

The measurement model in this case is:

$$X_i \sim N(\mu_i, \theta s_i^2)$$

where X is the ^{14}C age for sample i, μ_i is the true age, s_i is the quoted error, and $\sqrt{\theta}$ is the error multiplier.

$$\theta = \Sigma d_i^2 / J$$

where d_i is $X_i - \mu_i$, and J is the number of results submitted by the laboratory.

These 2 measures have been used in TIRI and FIRI (and previously in the 2 earlier intercomparisons, ICS and ISG) to provide a simple descriptive measure of individual laboratory performance.

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NOTE FOR INTERPRETATION OF DATA TABLES IN APPENDIX 4

All the results received (after any corrections) are reported in the following tables. Laboratories are identified by a code number. The laboratory type is also indicated (LSC=liquid scintillation, GPC=gas proportional, AMS=accelerator mass spectrometry, and CARB=direct absorption of CO_2). Laboratories which did not grant permission to publish have been removed from the tables (a total of 4).

Some laboratories have several entries with distinct laboratory codes. This reflects collaboration (generally) between a conventional and accelerator laboratory.

Some laboratories have multiple entries, and unless otherwise indicated, the tables show results for independent replicates. Occasionally, it may mean that results from different counters are reported.

- Some of the $\delta^{13}C$ values are estimated rather than measured; this is indicated in the tables by a *.
- Missing data is indicated by –9.
- For Samples A and C, *M* indicates results reported as "modern."

For Sample B, *B* indicates results quoted as "background."

TIRI: RESULTS FOR ALL SAMPLES

Table A4.1 TIRI A: Glengoyne barley mash

Lab	$\delta^{13}C$	$\delta^{14}C$	Error	$D^{14}C$	Error	pMC	Error
1	–27.4000	166.600	12.1000	172.200	13.3000	117.220	1.33000
2	–27.8100	*	*	168.000	2.0000	116.800	0.20000
3	–27.6000	160.600	0.9000	166.600	0.9000	116.660	0.09000
4	–26.2000	178.300	6.6000	181.100	6.6000	118.110	0.66000
5	–27.5000	*	*	148.800	8.8000	114.880	0.88000
5	–27.1000	*	*	172.200	8.6000	117.220	0.86000
6	–25.0000	316.010	19.7600	316.010	19.7600	131.601	1.97600
7	–25.0000	227.400	8.5100	227.400	8.5100	122.740	0.85100
8	–28.3000	*	*	181.000	8.0000	118.100	0.80000
9	–27.4000	165.750	8.0000	171.350	8.0000	117.135	0.80000
10	–26.4000	196.000	10.0000	199.000	10.0000	119.900	1.00000
11	–28.4000	149.200	5.3000	157.000	5.7000	115.700	0.57000
12	*	150.700	6.0000	150.700	*	115.070	*
13	–28.0000	159.800	4.9000	166.200	5.1000	116.620	0.51000
14	–24.7000	145.700	5.9000	145.000	5.9000	114.500	0.59000
15	–25.0000	171.430	15.5000	171.430	16.0900	117.143	1.60900
16	*	*	*	*	*	114.600	*
17	–28.2400	*	*	171.900	6.7000	117.190	0.67000
18	–29.7000	162.100	7.7000	173.000	7.7000	117.300	0.77000
19	–26.8000	172.200	6.3000	176.400	6.3000	117.640	0.63000
20	–27.0000	158.500	5.8000	163.100	5.8000	116.310	0.58000
21	–27.1300	154.000	15.0000	158.000	15.0000	115.800	1.50000
22	–26.3900	157.000	8.4000	160.000	8.6000	116.000	0.86000
22	–26.2200	157.000	7.2000	159.000	7.4000	115.900	0.74000
22	–26.6000	166.000	7.4000	169.000	7.6000	116.900	0.76000
23	–28.5000	163.000	4.8000	170.500	4.9000	117.050	0.49000
24	–25.8100	*	*	162.400	6.9000	116.240	0.69000
24	–26.3700	*	*	149.400	8.0000	114.940	0.80000
24	–27.3600	*	*	164.100	15.8000	116.410	1.58000
24	–25.6100	*	*	157.100	11.6000	115.710	1.16000
25	–27.3000	*	*	145.100	6.4000	114.510	0.64000
25	–27.3000	*	*	160.300	7.9000	116.030	0.79000
25	–27.3000	*	*	164.800	6.5000	116.480	0.65000
25	–27.3000	*	*	156.500	6.5000	115.650	0.65000
26	*	140.500	5.0000	*	*	*	*
27	–26.8000	*	*	160.300	4.6000	116.030	0.46000
28	*	*	*	172.850	6.7500	101.729	0.67500
29	*	188.000	13.0000	*	*	*	*
30	–25.7000	*	*	*	*	*	*
31	*	*	*	*	*	*	*
32	–28.8500	155.300	1.9800	164.180	1.9800	116.418	0.19800
33	*	*	*	*	*	*	*
34	–26.9000	161.900	3.7000	166.300	4.0000	116.630	0.40000
35	–26.0000	199.000	11.0000	201.000	11.0000	120.100	1.10000

Table A4.1 TIRI A: Glengoyne barley mash *(Continued)*

Lab	$\delta^{13}C$	$\delta^{14}C$	Error	$D^{14}C$	Error	pMC	Error
36	–28.7000	140.700	0.7000	149.200	0.8000	114.920	0.08000
37	–26.9000	154.900	15.9000	159.200	16.0000	115.920	1.60000
38	–26.6600	161.910	5.3900	165.760	5.4100	116.576	0.54100
39	–28.3400	149.700	4.7700	157.400	4.8100	115.740	0.48100
40	*	151.900	13.7000	*	*	*	*
42	–27.9000	131.600	9.6000	138.200	9.7000	113.820	0.97000
43	–27.5000	180.800	12.0000	186.800	12.0000	118.680	1.20000
44	–27.0000	156.500	5.1000	161.100	5.1000	116.110	0.51000
45	–26.8300	160.000	7.6000	165.800	7.6000	116.580	0.76000
46	*	151.500	0.8800	*	*	*	*
47	–27.0000	165.550	5.6700	170.210	5.6700	117.021	0.56700
48	–25.0000	179.000	8.0000	179.000	8.0000	117.900	0.80000
49	–27.5000	173.000	8.0000	179.000	8.0000	117.900	0.80000
50	–27.2000	*	*	*	*	*	*
51	–27.4000	*	*	*	*	*	*
52	–27.3000	160.370	7.8900	165.710	8.1100	116.571	0.81100
53	–27.0900	150.500	5.7000	155.400	5.7000	115.540	0.57000
53	–27.0900	152.500	5.4000	157.500	5.4000	115.750	0.54000
54	–27.4500	139.800	1.3000	145.400	1.3000	114.540	0.13000
56	–27.4100	172.010	5.8500	177.710	5.8800	117.771	0.58800
57	–28.0000	152.700	7.4000	159.800	7.5000	115.980	0.75000
58	–26.8000	150.070	5.9900	154.210	6.0200	115.421	0.60200
59	26.5000	159.500	6.4000	162.900	6.4000	116.290	0.64000
60	–27.8000	*	*	176.100	5.7000	117.610	0.57000
61	–25.0000	*	*	141.000	8.4000	114.100	0.84000
61	–25.0000	*	*	163.500	10.2000	116.350	1.02000
61	–25.0000	*	*	159.000	7.8000	115.900	0.78000
64	–24.5100	161.810	7.5900	160.640	7.5900	116.064	0.75900
65	*	*	*	*	*	*	*
66	–26.2000	164.500	6.8000	167.300	6.8000	116.730	0.68000
67	–27.8000	*	*	159.400	7.1000	115.940	0.71000
68	–27.8000	*	*	162.900	8.3000	116.290	0.83000
69	–27.6000	146.500	5.2000	152.500	5.2000	115.250	0.52000
71	–25.5000	*	*	*	*	*	*
72	–25.9000	*	*	161.000	6.0000	116.100	0.60000
75	–27.1100	170.000	0.1800	175.000	*	117.500	*

Table A4.2 TIRI B: Belfast pine

Lab	$\delta^{13}C$	$\delta^{14}C$	Error	$D^{14}C$	Error	Age	Error
1	–26.3000	–424.600	9.0000	–423.100	9.6000	4420	135
2	–25.6700	*	*	–430.000	2.0000	4521	20
3	–26.1700	–426.700	2.6000	–425.400	2.6000	4450	50
4	–25.1000	*	*	*	*	4472	44
5	–24.9000	*	*	–431.800	5.0000	4541	70
5	–24.4000	*	*	–430.200	5.2000	4518	73
5	–25.1500	*	*	–429.300	5.1000	4505	71
5	–25.2000	*	*	–422.600	4.8000	4412	67
6	–24.8200	–504.400	9.0800	–504.590	9.0800	5640	150
7	–24.8000	–438.000	3.9300	–438.210	3.9300	4630	60
8	–25.4000	*	*	–439.000	5.0000	4640	75
9	–26.3000	–408.660	4.4500	–407.120	4.4500	4200	60
10	–23.4000	–382.000	7.0000	–385.000	7.0000	3900	90
11	–25.9000	–437.700	2.6000	–436.600	2.7000	4610	40
12	*	–443.800	5.5000	*	*	4710	80
13	–25.1000	–414.500	5.2000	–414.300	5.2000	4300	55
14	–23.3000	–419.900	3.9000	–421.900	3.9000	4400	55
15	–25.0000	–429.590	7.1500	–429.590	7.1500	4510	50
16	*	*	*	*	*	4472	53
17	–24.8000	*	*	–427.500	3.6000	4480	50
18	–25.8000	–439.700	6.3000	–438.800	6.3000	4640	50
19	–24.8000	–423.600	4.1000	–423.800	4.1000	4430	73
20	–24.9000	–431.400	3.5000	–431.500	3.5000	4540	50
21	–27.0200	–431.000	8.0000	–429.000	8.0000	4500	115
22	–26.0900	–430.000	5.6000	–429.000	5.8000	4500	85
22	–26.0600	–429.000	4.7000	–428.000	4.8000	4490	70
22	–24.0300	–426.000	4.6000	–427.000	4.8000	4475	70
23	–25.0000	–410.900	4.8000	–410.900	4.8000	4250	65
24	–25.1500	*	*	–424.400	7.1000	4437	99
24	–24.8600	*	*	–439.000	11.100	4643	159
24	–25.1000	*	*	–429.100	4.9000	4503	69
24	–25.4400	*	*	–429.000	7.2000	4501	101
25	–23.9000	*	*	–426.300	4.9000	4460	70
25	–23.9000	*	*	–433.300	3.6000	4560	60
25	–23.9000	*	*	–429.400	3.6000	4510	60
25	–23.9000	*	*	–423.100	3.6000	4420	60
25	–23.9000	*	*	–431.100	3.6000	4530	60
25	–23.9000	*	*	–427.600	3.8000	4480	60
26	*	–428.800	18.900	*	*	4500	190
27	–25.0000	*	*	–429.100	4.4000	4500	60
28	*	*	*	*	*	4280	80
29	*	–426.700	7.2000	*	*	4460	100
30	–25.1000	*	*	*	*	4510	35
31	*	*	*	*	*	4465	40
32	–24.7200	–430.180	1.3100	–430.500	1.3100	4523	17

Table A4.2 TIRI B: Belfast pine *(Continued)*

Lab	$\delta^{13}C$	$\delta^{14}C$	Error	$D^{14}C$	Error	Age	Error
33	*	*	*	*	*	4390	20
34	–24.8000	–426.300	2.2000	–426.500	2.3000	4463	37
35	–25.3000	–395.000	7.0000	–395.000	7.0000	4040	100
36	–26.9000	–433.300	3.3000	–431.200	3.4000	4530	70
37	–26.0000	–426.800	10.400	–425.600	10.400	4452	101
38	–24.0100	–439.830	3.3700	–440.940	3.3600	4670	50
39	–26.6300	–437.250	3.2600	–436.540	3.2600	4610	50
40	*	–444.800	8.5000	*	*	4730	80
42	–25.8000	–426.100	6.0000	–425.100	6.0000	4577	86
44	–25.0000	*	*	–430.300	3.9000	4520	55
45	–25.2100	–414.400	5.3000	–414.100	5.3000	4296	72
46	*	–438.600	1.1800	*	*	4770	100
47	–25.0000	–434.170	3.5400	–434.170	3.5400	4580	40
48	–25.0000	–414.000	5.0000	–414.000	5.0000	4290	70
49	–25.5000	–424.000	9.0000	–424.000	6.0000	4420	80
50	–22.1000	*	*	*	*	4802	*
51	–23.7000	*	*	*	*	4465	125
51	–23.3000	*	*	*	*	4530	105
52	–25.8000	–426.940	5.1000	–426.020	5.1400	4460	70
53	–26.9700	–452.700	3.7000	–450.500	3.7000	4810	54
54	–26.6800	–427.300	3.8000	–425.300	3.9000	4580	60
56	–25.1600	–422.250	2.8900	–422.030	2.8900	4404	40
57	–24.5000	–416.800	3.3000	–418.000	3.3000	4345	45
58	–26.2000	–430.330	3.0200	–428.960	3.0300	4500	45
59	–25.1000	–432.600	4.0000	–432.500	4.0000	4551	57
60	–25.3000	*	*	–422.100	3.1000	4400	40
61	–24.5000	*	*	–435.300	5.1000	4590	73
61	–24.5000	*	*	–431.200	3.6000	4530	51
62	–25.3400	*	*	–426.730	2.4400	4470	35
62	–24.4900	*	*	–430.310	2.0600	4520	35
62	–24.3600	*	*	–431.690	2.7500	4540	40
64	–23.9600	–429.090	4.4100	–430.270	4.4100	4519	56
64	–23.9600	*	*	*	*	4530	45
66	–25.1000	–421.790	4.0300	–421.670	4.0300	4399	56
67	–25.3000	*	*	–427.300	4.1000	4480	60

Table A4.3 TIRI C: IAEA cellulose

Lab	$\delta^{13}C$	$\delta^{14}C$	Error	$D^{14}C$	Error	pMC	Error
1	–24.5000	292.600	14.0000	291.400	14.0000	129.140	1.40000
2	–24.5000	*	*	296.000	3.0000	129.600	0.30000
3	–24.5800	292.600	1.4000	291.500	1.4000	129.150	0.14000
4	–25.1000	305.800	5.7000	306.100	5.7000	130.610	0.57000
5	–24.5000	290.400	9.4000	289.100	9.4000	128.910	0.94000
6	–24.8300	291.250	21.8600	290.820	21.8500	129.082	2.18500
7	–23.6000	274.830	7.1900	271.190	7.1700	127.119	0.71700
8	–24.7000	*	*	304.000	9.0000	130.400	0.90000
9	–25.5500	298.600	11.0000	300.000	11.0000	130.000	1.10000
11	*	*	*	296.700	4.3000	129.670	0.43000
12	*	287.200	8.9000	*	*	*	*
13	–24.0000	292.600	7.7000	290.200	7.6000	129.020	0.76000
14	–24.9000	283.500	6.2000	283.200	6.2000	128.320	0.62000
15	–25.0000	302.010	11.4300	302.020	12.2100	130.202	1.22100
16	*	*	*	*	*	128.70	*
17	–25.1600	*	*	308.600	7.5000	130.860	0.75000
18	–25.3000	319.540	6.6000	320.100	6.6000	132.010	0.66000
19	–24.6000	295.500	4.2000	294.500	4.2000	129.450	0.42000
20	–24.5000	294.700	5.4000	293.400	5.4000	129.340	0.54000
21	–24.2700	316.000	16.0000	313.000	16.0000	131.300	1.60000
22	–25.4600	297.000	8.0000	298.000	8.0000	129.800	0.80000
22	–25.4600	300.000	8.1000	301.000	8.3000	130.100	0.83000
23	–24.0000	295.900	6.1000	293.400	6.1000	129.340	0.61000
24	–24.2700	*	*	303.000	8.3000	130.300	0.83000
24	–24.2700	*	*	296.300	7.2000	129.630	0.72000
24	–24.3200	*	*	307.200	9.1000	130.720	0.91000
24	–24.2500	*	*	295.100	6.3000	129.510	0.63000
24	–24.2800	*	*	303.600	13.5000	130.360	1.35000
25	–24.0000	*	*	283.400	7.2000	128.340	0.72000
25	–24.0000	*	*	292.500	9.0000	129.250	0.90000
26	*	240.600	7.3000	*	*	*	*
27	–24.8000	*	*	300.500	4.5000	130.050	0.45000
28	*	*	*	124.270	10.9300	112.427	1.09300
29	*	322.000	15.0000	*	*	*	*
30	–24.2000	*	*	*	*	129.52	*
31	*	*	*	*	*	129.60	0.5
32	–25.6800	293.850	2.1800	295.600	2.1800	129.560	0.21800
33	*	*	*	*	*	129.16	*
34	–24.9000	294.800	4.5000	294.500	4.7000	129.450	0.47000
35	–24.3000	290.000	11.0000	290.000	11.0000	129.000	1.10000
36	–24.8000	289.600	1.6000	289.100	1.6000	128.910	0.16000
37	–24.3000	301.600	17.5000	299.900	17.5000	129.990	1.75000
38	–24.8200	295.260	5.5900	294.800	5.5900	129.480	0.55900
39	–24.9500	300.900	5.2900	302.070	5.3000	130.207	0.53000
40	*	284.400	14.5000	*	*	*	*

Table A4.3 TIRI C: IAEA cellulose *(Continued)*

Lab	$\delta^{13}C$	$\delta^{14}C$	Error	$D^{14}C$	Error	pMC	Error
42	–25.1000	289.200	10.6000	289.400	10.6000	128.940	1.06000
43	–25.2000	311.000	12.0000	312.000	12.0000	131.200	1.20000
44	–24.9100	*	*	298.200	5.2000	129.820	0.52000
45	–24.3600	298.400	8.1000	298.500	8.1000	129.850	0.81000
46	*	287.550	0.8500	*	*	*	*
47	–25.0000	285.650	7.7600	285.650	7.7600	128.565	0.77600
47	–25.0000	292.751	5.7670	292.751	5.7670	129.275	0.57670
48	–25.0000	301.000	8.0000	301.000	10.0000	130.100	1.00000
49	–25.0000	305.000	8.0000	305.000	8.0000	130.500	0.80000
51	–24.4000	*	*	*	*	128.94	1.14
52	–25.0000	299.190	8.5500	299.190	8.5500	129.919	0.85500
53	–25.3900	277.300	4.7000	278.300	4.7000	127.830	0.47000
54	–25.8000	302.400	2.7000	304.500	2.8000	130.450	0.28000
56	*	297.320	6.4800	*	*	*	*
57	–24.2000	290.700	6.6000	288.700	6.5000	128.870	0.65000
58	–24.7000	302.880	6.4200	302.100	6.4300	130.210	0.64300
59	–24.2000	300.400	7.4000	298.300	7.5000	129.830	0.75000
60	–25.2000	*	*	308.700	6.0000	130.870	0.60000
61	–24.2000	*	*	323.300	13.4000	132.330	1.34000
61	–24.2000	*	*	283.900	9.9000	128.390	0.99000
61	–24.2000	*	*	305.300	7.4000	130.530	0.74000
62	–23.8400	*	*	295.750	5.8700	129.575	0.58700
64	–24.0700	294.700	8.5800	292.310	8.5800	129.231	0.85800
66	–25.1000	303.400	7.8000	303.700	7.8000	130.370	0.78000
67	–25.2000	*	*	304.400	8.0000	130.440	0.80000
68	–25.2000	*	*	295.800	8.2000	129.580	0.82000
69	–24.8600	261.900	5.8000	261.500	5.8000	126.150	0.58000
71	–25.0000	*	*	*	*	127.90	0.80
72	–23.3000	*	*	290.000	8.0000	129.000	0.80000
75	–25.5400	302.000	0.1600	303.000	*	130.300	*
75	–25.5400	303.000	0.1900	304.000	*	130.400	*

Table A4.4 TIRI D: Hekla peat

Lab	$\delta^{13}C$	$\delta^{14}C$	Error	$D^{14}C$	Error	Age	Error
2	–28.7300	*	*	–377.000	2.0000	3804	18
3	–29.2000	–382.800	2.4000	–377.600	2.4000	3810	50
4	–27.8000	*	*	*	*	3747	41
5	–28.8000	*	*	–375.300	5.2000	3780	66
5	–28.8700	*	*	–378.200	5.3000	3817	68
5	–28.9000	*	*	–380.500	5.9000	3847	77
6	–28.4800	–395.700	14.670	–391.490	14.8000	3990	200
7	–28.5000	–394.770	4.3700	–390.550	4.4000	3980	60
8	–29.3000	*	*	–375.000	6.0000	3790	75
9	–29.6500	–371.130	4.9000	–365.280	4.9000	3650	60
11	–24.6000	–407.200	4.0000	–407.800	4.1000	4200	55
12	*	–381.800	5.7000	*	*	3860	80
13	–29.2000	–383.500	6.6000	–378.300	6.6000	3820	65
14	–26.3000	–387.500	4.0000	–385.900	4.0000	3920	55
15	–27.0000	–358.220	7.5300	–355.650	13.980	3530	80
16	*	*	*	*	*	3834	61
17	–29.8100	*	*	–379.000	4.4000	3830	60
18	–29.4000	–378.200	8.7000	–372.800	8.7000	3750	70
19	–28.6000	–390.000	*	–385.500	*	3910	70
19	–28.5000	–374.900	4.2000	–370.500	4.2000	3710	70
20	–28.9000	–383.800	3.8000	–379.000	3.9000	3830	50
21	–27.5000	–362.000	9.0000	–359.000	9.0000	3570	114
22	–28.0700	–381.000	6.5000	–377.000	6.7000	3800	90
22	–28.8900	–380.000	6.5000	–376.000	6.7000	3785	90
23	–23.9000	–381.500	7.2000	–382.800	7.2000	3880	90
24	–28.7700	*	*	–390.600	4.8000	3979	63
24	–28.5600	*	*	–381.100	6.5000	3854	85
24	–28.4700	*	*	–377.200	6.4000	3804	83
24	–28.5600	*	*	–387.100	11.700	3932	153
25	–27.8000	*	*	–378.500	5.4000	3820	70
25	–27.8000	*	*	–374.600	3.9000	3770	60
26	*	–353.200	14.900	*	*	3500	175
27	–30.3000	*	*	–377.300	3.1000	3810	40
28	*	*	*	*	*	3550	60
29	*	–363.000	7.8000	*	*	3620	100
30	–28.6000	*	*	*	*	3885	35
31	*	*	*	*	*	3930	40
32	–29.1300	–385.450	1.7600	–380.380	1.7600	3845	22
33	*	*	*	*	*	3630	35
34	–27.9000	–379.800	2.7000	–376.200	2.8000	3790	36
35	–28.2000	–382.000	7.0000	–378.000	7.0000	3810	100
36	–29.7000	–384.300	2.4000	–378.500	2.4000	3820	70
37	–28.7000	–377.000	10.800	–372.500	10.900	3741	137
38	–29.3600	–372.430	5.9300	–366.960	5.9800	3670	80
39	–29.3600	–389.320	3.6600	–384.000	3.6900	3890	50

Table A4.4 TIRI D: Hekla peat *(Continued)*

Lab	$\delta^{13}C$	$\delta^{14}C$	Error	$D^{14}C$	Error	Age	Error
40	*	–380.000	13.700	*	*	3840	110
42	–29.5000	–373.200	6.5000	–367.600	6.6000	3788	86
44	*	*	*	–374.900	3.9000	3770	50
45	–28.3400	–375.400	5.4000	–370.400	5.5000	3720	70
46	*	–391.500	1.1400	*	*	4110	95
47	–27.0000	–373.710	4.4300	–371.210	4.4500	3730	40
48	–27.0000	–376.000	6.0000	–374.000	6.0000	3760	70
49	–28.6000	–365.000	9.0000	–360.000	6.0000	3590	80
50	–29.2000	*	*	*	*	4050	*
51	–28.3000	*	*	*	*	4065	120
52	–28.6000	–380.560	5.3200	–376.090	5.3700	3790	70
53	–28.8800	–388.700	3.0000	–383.800	3.0000	3890	39
53	–28.8800	–383.500	2.9000	–378.600	2.9000	3822	38
53	–28.8800	–387.500	3.9000	–382.600	3.9000	3875	51
54	–29.3100	–371.800	3.4000	–366.300	3.4000	3770	50
56	–29.2300	–387.210	3.0600	–382.080	3.0900	3867	40
57	–28.0000	–383.800	3.2000	–380.100	3.2000	3835	40
58	–28.5000	–379.930	3.3000	–375.390	3.3300	3780	45
59	–28.8000	–382.200	4.6000	–377.400	4.6000	3806	59
60	–28.4000	*	*	–370.000	3.6000	3710	40
61	–27.9000	*	*	–374.500	3.8000	3770	50
61	–27.9000	*	*	–376.800	3.7000	3800	50
61	–27.9000	*	*	–371.800	3.9000	3740	50
66	–27.8000	–386.150	4.1500	–382.610	4.1500	3874	54
67	–28.4000	*	*	–379.000	4.5000	3830	60
68	–28.4000	*	*	–379.400	6.0000	3780	80
69	–29.2200	–387.500	3.7000	–382.400	3.8000	3870	50
71	–29.0000	*	*	*	*	3595	*
72	–25.0000	*	*	–381.200	3.0000	3855	40
75	–28.6800	–387.000	0.1500	–383.000	*	3880	45
75	–28.6800	–379.000	0.1600	–374.000	*	3760	30

Table A4.5 TIRI E: Ellanmore humic acid

Lab	$\delta^{13}C$	$\delta^{14}C$	Error	$D^{14}C$	Error	Age	Error
1	–29.7500	–748.000	6.1000	–745.600	6.4000	11,000	204
3	–29.2800	–763.000	11.000	–761.000	11.000	11,500	120
4	–27.9000	*	*	*	*	10,898	69
5	–28.3000	*	*	–753.800	2.7000	11,258	87
5	–28.6000	*	*	–751.400	2.6000	11,180	83
5	–28.6300	*	0.0000	–751.700	2.9000	11,190	94
6	–29.5400	–791.180	7.9900	–789.290	8.0600	12,510	320
7	–29.1000	–715.640	2.8300	–713.310	2.8500	10,040	80
8	–29.0000	*	*	–748.000	5.0000	11,090	150
9	–29.7500	–738.750	4.7000	–736.240	4.7000	10,705	140
11	–28.4000	–753.500	2.6000	–751.900	2.6000	11,190	85
12	*	–757.800	3.5000	*	*	11,390	120
13	–29.0000	–741.700	6.3000	–739.600	6.2000	10,810	100
14	–26.1000	–681.100	3.5000	–680.400	3.5000	9160	90
15	–27.0000	–751.980	5.4600	–750.990	5.7300	11,170	140
16	*	*	*	*	*	10,993	88
17	–29.0800	*	*	–737.600	1.9000	10,750	60
18	–29.6000	–762.200	17.500	–759.900	17.500	11,400	140
19	–29.0000	–749.500	3.6000	–747.600	3.6000	11,060	120
20	–27.8000	–751.000	1.9000	–749.600	1.9000	11,120	60
21	–28.4700	–769.000	6.0000	–767.000	6.0000	11,710	210
22	–28.7800	–755.000	3.0000	–753.000	3.2000	11,250	95
23	–28.4000	–746.200	5.1000	–744.500	5.1000	10,960	160
24	–28.4100	*	*	–755.100	6.7000	11,300	219
24	–28.6100	*	*	–747.000	4.9000	11,040	157
24	–28.3000	*	*	–751.400	4.1000	11,180	132
24	–28.1800	*	*	–752.000	5.1000	11,202	165
24	–28.0700	*	*	–746.000	4.8000	11,009	153
25	–27.6000	*	*	–748.300	1.8000	11,080	60
25	–27.6000	*	*	–749.200	3.0000	11,110	100
26	*	–743.600	59.100	*	*	10,930	420
27	–28.8000	*	*	–750.200	2.0000	11,140	70
29	*	–742.000	4.5000	*	*	10,900	140
30	–28.7000	*	*	*	*	11,160	55
31	*	*	*	*	*	11,150	60
32	–28.6200	–752.640	1.2200	–750.850	1.2200	11,163	36
33	*	*	*	*	*	10,380	90
34	–28.3000	–742.900	2.3000	–741.200	2.3300	10,858	73
35	–28.3000	–732.000	4.5000	–730.000	4.5000	10,520	140
36	–28.8000	–750.000	7.6000	–748.200	7.7000	11,080	80
37	–28.0000	–682.100	8.5000	–680.100	8.5000	9153	151
38	–29.1200	–756.530	2.6100	–754.520	2.6300	11,280	90
39	–28.7800	–755.490	1.9600	–753.640	1.9700	11,250	70
40	*	–757.300	9.1000	*	*	11,380	140
42	–28.8700	–759.700	3.8000	–757.800	3.9000	11,723	131

Table A4.5 TIRI E: Ellanmore humic acid *(Continued)*

Lab	$\delta^{13}C$	$\delta^{14}C$	Error	$D^{14}C$	Error	Age	Error
44	–25.0000	*	*	–750.600	3.0000	11,150	100
45	–28.4500	–750.100	3.7000	–748.000	3.8000	11,070	120
46	*	–754.710	1.8700	*	*	11,600	155
47	–27.0000	746.050	2.5900	–745.030	2.6000	10,980	70
48	–27.0000	–779.000	11.000	–778.000	11.000	12,090	400
49	–29.1000	–743.000	13.000	–741.000	3.5000	10,860	110
50	–29.0000	*	*	*	*	*	*
51	–29.1000	*	*	*	*	11,200	230
52	–28.8000	–755.860	3.4900	–754.000	3.4900	11,260	110
53	–28.6600	–749.400	3.0000	–747.500	3.0000	11,060	100
53	–28.6600	–753.700	3.0000	–751.900	3.0000	11,200	100
54	–29.3700	–746.900	7.1000	–744.600	7.1000	11,300	220
56	–29.0300	–748.750	1.7600	–746.710	1.7800	11,031	56
58	–27.0000	–749.500	1.4300	–748.490	1.4400	11,090	50
59	–28.8000	–747.900	2.8000	–745.900	2.8000	11,005	88
60	–29.5000	*	*	–745.800	2.3000	10,930	70
61	–28.1000	*	*	–753.600	2.4000	11,250	78
61	–28.1000	*	*	–752.300	2.6000	11,210	84
62	–28.8400	*	*	–748.000	1.6700	11,070	50
64	–28.5600	–746.830	2.6900	–745.030	2.6900	10,979	78
66	–27.9000	–741.190	1.9800	–739.640	1.9800	10,810	61
67	–29.5000	*	*	–750.200	1.8000	11,140	60
68	–29.5000	*	*	–747.900	4.3000	11,070	140
69	–29.2800	–749.200	2.7000	–747.100	2.7000	11,040	90
71	–29.2000	*	*	*	*	11,265	*
72	–28.0000	*	*	–751.400	4.0000	11,180	60
75	–27.9600	–753.000	0.1200	–751.000	*	11,200	55
75	–27.9600	–750.000	0.1500	–749.000	*	11,100	55

Table A4.6 TIRI F: Icelandic doublespar

Lab	$\delta^{13}C$	$\delta^{14}C$	Error	$D^{14}C$	Error	Age	Error	pMC	Error
1	–3.80000	–995.00	3.90	–995.20	3.900	38,100	*	0.48000	0.3900
3	–3.74000	–982.00	260.7	–982.00	260.900	*	*	1.80000	26.0900
4	–3.40000	*	*	*	*	*	*	*	*
5	–3.54000	*	*	–999.00	0.200	55,364	1607	0.10000	0.0200
5	–3.53000	*	*	–997.70	0.400	48,673	1398	0.23000	0.0400
5	–3.50000	*	*	–998.40	0.200	51,714	1005	0.16000	0.0200
5	–3.42000	*	*	–998.50	0.200	52,106	1072	0.15000	0.0200
5	–3.42000	*	*	–998.00	0.200	49,795	805	0.20000	0.0200
5	–3.92000	*	*	–998.50	0.200	52,110	1072	0.15000	0.0200
5	–3.60000	*	*	–998.80	0.200	53,899	1340	0.12000	0.0200
5	–3.60000	*	*	–997.80	0.200	49,030	734	0.22000	0.0200
5	–3.65000	*	*	–998.00	0.200	49,796	806	0.20000	0.0200
5	–3.62000	*	*	–998.10	0.300	50,207	1270	0.19000	0.0300
6	–3.43000	*	*	*	*	*	*	*	*
7	–3.42000	*	*	*	*	*	*	*	*
8	–0.70000	*	*	–991.00	1.000	37,300	800	0.90000	0.1000
8	–0.70000	*	*	–999.00	1.000	49,950	800	0.10000	0.1000
9	–4.05000	*	*	*	*	46,700	*	*	*
10	–3.60000	–1004.0	3.00	–1004.0	3.000	*	*	–0.4000	0.3000
11	3.50000	*	*	–998.00	*	50,000	*	0.20000	*
13	–3.20000	–998.90	6.70	–998.90	6.700	43,780	*	0.11000	0.6700
14	–3.80000	–996.10	1.30	–996.30	1.300	39,800	1100	0.37000	0.1300
16	*	*	*	*	*	44,186	2159	*	*
17	–3.77000	*	*	–995.20	0.600	42,850	1000	0.48000	0.0600
18	–4.20000	–999.75	*	–999.75	*	41,000	*	0.02500	*
19	–3.60000	–999.19	3.50	–999.23	3.500	*	*	0.07700	0.3500
20	–4.00000	–998.60	1.80	–998.70	1.700	43,000	*	0.13000	0.1700
21	–3.81000	–989.00	4.00	–990.00	4.000	*	*	1.00000	0.4000
22	–3.70000	–999.44	3.00	–999.55	3.200	40,000	*	0.04500	0.3200
23	–3.10000	–1001.1	2.10	*	*	*	*	*	*
24	*	*	*	*	*	*	*	*	*
25	0.00000	*	*	–997.20	0.100	47,090	320	0.28000	0.0100
25	0.00000	*	*	–996.40	0.200	45,290	440	0.36000	0.0200
25	0.00000	*	*	–997.20	0.500	47,180	1370	0.28000	0.0500
25	0.00000	*	*	–996.90	0.100	46,370	320	0.31000	0.0100
26	*	*	*	*	*	*	*	*	*
27	–3.70000	*	*	–997.70	0.800	50,000	*	0.23000	0.0800
29	*	–1000.0	*	*	*	30,000	*	*	*
30	–3.00000	*	*	*	*	53,400	*	*	*
31	*	*	*	*	*	36,000	*	*	*
32	–3.59000	–999.85	0.190	–999.85	0.190	61,000	*	0.01500	0.0190
33	*	*	*	*	*	*	*	*	*
34	–3.50000	–997.70	1.350	–997.80	1.300	*	*	0.22000	0.1300
35	–3.70000	–997.00	3.000	–997.00	3.000	45,000	*	0.30000	0.3000
36	–3.50000	–996.20	290.80	–996.40	290.600	45,100	2800	0.36000	29.0600

Table A4.6 TIRI F: Icelandic doublespar *(Continued)*

Lab	$\delta^{13}C$	$\delta^{14}C$	Error	$D^{14}C$	Error	Age	Error	pMC	Error
37	–3.70000	–1005.5	7.000	–1005.3	6.600	37,000	*	–0.5300	0.6600
38	–3.77000	–997.29	1.930	–997.40	1.840	*	*	0.26000	0.1840
39	–3.35000	*	*	–999.97	0.610	54,000	*	0.00300	0.0610
40	*	–998.60	17.200	*	*	*	*	*	*
41	–2.86000	–998.10	3.500	–998.18	3.500	*	*	0.18200	0.3500
42	–5.23000	–997.10	1.300	–997.30	1.300	*	*	0.27000	0.1300
44	*	*	*	–1000.0	*	*	*	0.00000	*
45	–3.33000	–997.20	2.400	–997.30	2.400	*	*	0.27000	0.2400
46	*	–1000.9	*	*	*	37,800	*	*	*
48	–8.00000	–997.00	3.000	–997.00	3.000	29,000	*	0.30000	0.3000
49	–3.90000	–996.00	415.00	–997.00	1.500	45,600	4600	0.30000	0.1500
50	–4.40000	*	*	*	*	*	*	*	*
51	–3.40000	*	*	*	*	*	*	*	*
52	–3.80000	*	*	–1002.5	2.120	*	*	–0.2460	0.2120
53	–3.86000	–999.60	1.900	–999.60	1.900	*	*	0.04000	0.1900
53	–3.86000	–998.30	2.000	–998.40	2.000	*	*	0.16000	0.2000
54	–2.37000	–1000.0	*	–1000.0	*	*	*	0.00000	*
55	–3.82000	*	*	–999.20	0.900	*	*	0.08000	0.0900
55	–3.59000	*	*	–998.70	0.900	53,400	*	0.13000	0.0900
56	–3.62000	*	*	*	*	*	*	*	*
57	–3.67000	–1000.6	2.000	*	*	*	*	*	*
58	–4.80000	–1000.2	0.420	–1000.2	0.440	62,000	*	–0.0170	0.0440
59	–3.70000	*	*	*	*	*	*	*	*
60	–4.10000	*	*	–1000.00	0.500	47,000	*	0.00000	0.0500
61	–3.40000	*	*	–1001.00	0.700	60,000	*	–0.1000	0.0700
61	–3.40000	*	*	–999.40	0.200	60,000	2700	0.06000	0.0200
64	–3.39000	–1000.3	4.550	–1000.3	4.550	*	*	–0.0320	0.4550
66	*	*	*	*	*	*	*	*	*
67	–4.10000	*	*	–997.60	0.100	48,000	*	0.24000	0.0100
69	–3.73000	*	*	*	*	*	*	*	*
71	–3.50000	*	*	*	*	*	*	*	*
72	–2.90000	*	*	–996.00	*	44,000	*	0.40000	*
75	–2.60000	–997.00	0.110	–997.00	*	37,000	*	0.30000	*
75	–2.60000	–1001.0	0.130	–1001.0	*	43,000	*	–0.1000	*

Table A4.7 TIRI G: Fugla Ness wood fragments

Lab	$\delta^{13}C$	$d^{14}C$	Error	$D^{14}C$	Error	Age	Error
3	–25.90	–991.00	76.70	–990.90	76.70	37,780	620
7	–27.51	–998.15	0.80	–998.14	0.80	50,510	3480
11	–27.68	–992.00	2.00	–992.00	2.00	38,000	2000
14	–23.50	–9.00	–9.00	–980.80	1.40	31,800	600
15	–25.00	–992.65	1.23	–992.64	1.23	> 40,000	–9
19	–27.60	–998.40	3.00	–998.40	3.00	B	–9
19	–27.60	–996.00	2.70	–996.00	2.70	B	–9
19	–27.60	–998.50	2.90	–998.50	2.90	B	–9
22	–27.00	–9.00	–9.00	–9.00	–9.00	B	–9
23	–26.30	–1000.50	2.40	–1000.50	2.40	> 41,000	–9
28	–9.00	–9.00	–9.00	–993.06	–9.00	39,910	1200
30	–26.40	–9.00	–9.00	–9.00	–9.00	> 50,000	–9
31	–26.68	–991.93	7.60	–991.90	7.61	> 35,000	–9
33	–9.00	–9.00	–9.00	–9.00	–9.00	37,140	260
34	–27.90	–997.10	0.80	–997.10	0.80	46,940	2590
36	–26.40	–996.60	201.90	–996.70	226.50	45,700	1800
37	–27.38	–1000.30	3.80	–1000.30	3.80	> 37,000	–9
38	–27.52	–995.12	2.09	–995.09	2.10	42,710	4500
39	–27.68	–996.36	0.54	–996.34	0.54	45,200	1500
40	–9.00	–1000.10	3.40	–9.00	–9.00	> 40,000	–9
42	–25.37	–996.00	1.50	–996.00	1.50	> 45,000	–9
43	–27.70	–995.80	2.80	–995.70	2.80	43,900	6300
43	–26.90	–995.30	2.80	–995.30	2.80	43,100	5500
45	–24.58	–981.70	2.50	–981.70	2.50	35,150	1170
47	–25.00	–998.75	0.82	–998.75	0.82	> 51,560	–9
49	–25.40	–9.00	–9.00	–998.80	0.70	> 47,000	–9
51	–26.50	–9.00	–9.00	–9.00	–9.00	> 40,835	–9
53	–26.90	–999.60	2.80	–9.00	–9.00	B	–9
53	–26.90	–994.20	3.90	–9.00	–9.00	B	–9
53	–26.90	–1000.00	2.50	–9.00	–9.00	B	–9
56	–26.99	–9.00	–9.00	–9.00	–9.00	> 40,000	–9
57	–26.60	–998.00	1.60	–998.00	1.60	> 45,000	–9
58	–24.30	–998.50	0.40	–998.60	0.40	> 51,700	–9
69	–28.06	–996.90	2.00	–996.80	2.00	> 40,200	–9
71	–25.30	–9.00	9.00	–9.00	–9.00	> 42,000	–9
72	–26.49	–996.00	0.11	–996.00	–9.00	> 35,000	–9
72	–26.49	–998.00	0.19	–998.00	–9.00	> 38,000	–9
74	–26.10	–9.00	–9.00	–999.00	0.60	> 54,025	–9

Table A4.8 TIRI H: Ellanmore whole peat

Lab	$\delta^{13}C$	$d^{14}C$	Error	$D^{14}C$	Error	Age	Error
3	–28.56	–756.20	7.30	–754.50	7.20	11,280	80
7	–29.08	–749.75	2.70	–747.71	2.70	11,130	90
8	–28.80	–9.00	–9.00	–753.50	3.50	11,265	110
14	–26.20	–9.00	–9.00	–749.10	3.10	11,060	100
15	–27.00	–732.12	1.90	–731.00	4.57	10,550	140
19	–28.10	–749.30	2.90	–747.70	2.90	11,060	100
19	–28.10	–756.20	2.60	–754.70	2.60	11,290	160
19	–28.10	–760.30	3.00	–758.90	3.00	11,430	140
22	–28.46	–756.00	1.87	–754.00	1.88	11,272	62
23	–28.00	–772.90	3.40	–771.50	3.40	11,860	120
28	–9.00	–9.00	–9.00	–761.63	–9.00	11,510	80
31	–29.14	–745.15	1.85	–743.04	1.86	10,915	58
33	–9.00	–9.00	–9.00	–9.00	–9.00	10,630	25
34	–28.80	–752.20	4.30	–750.30	4.34	11,146	139
36	–28.60	–747.40	7.20	–745.60	7.30	11,000	80
37	–28.22	–723.90	3.70	–722.10	3.70	10,280	108
39	–29.02	–759.96	2.09	–758.03	2.10	11,400	70
42	–28.31	–754.10	4.20	–752.50	4.30	11,544	141
43	–28.70	–735.90	5.10	–733.90	5.20	10,640	160
43	–29.10	–746.80	6.40	–744.80	6.50	10,970	200
47	–27.00	–750.68	1.15	–749.68	1.15	11,130	40
49	–28.80	–9.00	–9.00	–754.00	3.00	11,270	100
49	–28.80	–9.00	–9.00	–756.00	3.00	11,330	110
50	–28.90	–9.00	–9.00	–749.40	17.40	11,120	580
51	–28.50	–9.00	–9.00	–9.00	–9.00	10,895	105
53	–27.90	–746.80	3.10	–745.30	3.10	10,990	100
53	–27.90	–743.40	3.50	–741.90	3.50	10,880	110
53	–27.90	–743.20	4.50	–741.60	4.60	10,870	140
58	–27.90	–752.71	1.17	–751.27	1.18	11,180	40
69	–28.75	–746.00	2.80	–744.10	2.80	10,950	90
71	–28.70	–9.00	–9.00	–9.00	–9.00	11,170	200
72	–29.20	–756.00	0.12	–754.00	–9.00	11,300	65
72	–29.20	–745.00	0.20	–743.00	–9.00	10,900	60
74	–28.60	–9.00	–9.00	–755.30	2.20	11,310	70
74	–28.60	–9.00	–9.00	–760.90	2.20	11,490	70

Table A4.9 TIRI I: travertine

Lab	$\delta^{13}C$	$d^{14}C$	Error	$D^{14}C$	Error	Age	Error
7	–9.81	–754.65	2.60	–762.15	2.60	11,540	90
11	–10.02	–741.00	3.00	–748.00	3.00	11,100	100
14	–6.20	–9.00	–9.00	–750.40	3.00	11,100	100
19	–9.90	–737.90	3.50	–745.80	3.50	11,000	140
19	–9.90	–735.90	3.60	–743.90	3.60	10,940	120
19	–9.90	–732.80	3.80	–740.80	3.80	10,850	120
19	–9.90	–740.60	3.00	–748.40	3.00	11,090	130
22	–8.80	–740.00	1.86	–748.00	1.80	11,075	57
23	–9.60	–745.20	3.30	–753.10	3.20	11,235	105
28	–9.00	–9.00	–9.00	–711.92	–9.00	9990	60
31	–9.95	–735.60	2.27	–743.57	2.28	10,932	71
33	–9.00	–9.00	–9.00	–9.00	–9.00	10,430	35
36	–9.90	–735.50	7.00	–743.50	7.00	10,930	80
37	–9.69	–740.60	4.70	–748.50	4.60	10,835	145
39	–10.08	–755.14	2.16	–762.45	2.09	11,550	100
41	–9.50	–741.18	6.80	–749.20	6.60	11,109	214
42	–9.70	–730.40	4.50	–738.60	4.40	11,092	142
43	–10.01	–738.90	5.20	–745.90	5.10	11,000	160
43	–9.96	–735.00	5.20	–743.00	5.00	10,910	160
47	–4.10	–740.12	2.63	–750.98	2.52	11,170	80
49	–9.95	–9.00	–9.00	–748.00	3.00	11,070	100
49	–9.90	–9.00	–9.00	–749.00	3.00	11,120	100
50	–9.80	–9.00	–9.00	–738.40	16.90	10,770	530
50	–9.80	–9.00	–9.00	–755.50	16.80	11,310	570
51	–10.70	–9.00	–9.00	–9.00	–9.00	11,075	125
53	–10.20	–738.80	3.30	–746.50	3.20	11,030	100
53	–10.20	–745.40	3.10	–753.00	3.00	11,230	100
53	–10.20	–737.50	3.70	–745.30	3.60	10,900	120
56	–9.73	–9.00	–9.00	–747.30	2.60	11,049	80
57	–9.50	–740.90	1.80	–748.70	1.70	11,065	55
58	–9.60	–742.03	1.24	–749.97	1.27	11,135	40
71	–9.90	–9.00	–9.00	–9.00	–9.00	11,320	70
72	–9.69	–739.00	0.12	–747.00	–9.00	11,000	60
72	–9.69	–746.00	0.15	–753.00	–9.00	11,200	55

Table A4.10 TIRI J: Crannog wood

Lab	$\delta^{13}C$	$d^{14}C$	Error	$D^{14}C$	Error	Age	Error
3	–27.28	–200.30	1.60	–196.60	1.60	1760	70
7	–27.00	–175.30	5.70	–172.01	5.70	1550	50
8	–26.70	–9.00	–9.00	–180.50	6.30	1595	65
11	–22.49	–170.00	6.00	–173.00	6.00	1530	60
14	–25.40	–9.00	–9.00	–166.80	4.70	1420	45
15	–25.00	–186.67	3.34	–186.67	3.34	1660	40
19	–25.30	–187.00	3.50	–186.60	3.50	1660	70
19	–27.40	–192.70	3.90	–188.80	3.90	1680	80
19	–25.30	–185.00	4.20	–184.40	4.20	1640	80
19	–25.30	–189.00	3.80	–188.60	3.80	1680	50
22	–26.65	–181.00	2.86	–178.00	2.87	1574	30
23	–26.60	–197.50	4.90	–194.80	4.90	1740	50
28	–9.00	–9.00	–9.00	–157.29	–9.00	1370	60
30	–25.70	–9.00	–9.00	–9.00	–9.00	1610	35
31	–27.45	–170.20	3.00	–166.13	3.01	1460	29
33	–9.00	–9.00	–9.00	–9.00	–9.00	1550	10
34	–28.20	–185.00	3.00	–179.80	3.10	1592	30
36	–27.00	–183.60	1.00	–180.40	1.00	1600	70
37	–27.62	–167.00	6.80	–162.60	6.80	1423	66
38	–26.25	–211.88	4.49	–209.91	4.50	1890	45
39	–27.30	–202.36	4.12	–198.70	4.14	1780	80
40	–9.00	–199.10	8.40	–9.00	–9.00	1780	80
42	–26.56	–149.70	8.40	–147.10	8.50	1315	82
47	–25.00	–179.33	3.75	–179.33	3.75	1590	40
51	–26.80	–9.00	–9.00	–9.00	–9.00	1610	65
53	–27.40	–188.10	4.00	–184.10	4.10	1634	40
53	–27.40	–176.30	3.80	–172.20	3.80	1520	37
53	–27.40	–172.80	4.90	–168.70	5.00	1480	50
56	–27.00	–9.00	–9.00	–167.30	4.20	1471	40
58	–26.40	–186.35	3.66	–184.07	3.67	1635	40
69	–27.97	–186.60	4.40	–181.80	4.40	1610	40
71	–27.50	–9.00	–9.00	–9.00	–9.00	1670	30
72	–27.47	–186.00	0.14	–182.00	–9.00	1610	25
72	–27.47	–176.00	0.17	–172.00	–9.00	1510	35
74	–26.40	–9.00	–9.00	–177.30	3.90	1570	40
74	–26.40	–9.00	–9.00	–178.20	4.00	1580	40

Table A4.11 TIRI K: turbidite

Lab	$\delta^{13}C$	$d^{14}C$	Error	$D^{14}C$	Error	Age	Error
7	1.77	–889.39	1.80	–895.31	1.80	18,130	130
11	0.89	–887.00	3.00	–893.00	3.00	18,000	200
13	2.00	–882.8	6.50	–889.1	6.50	17,670	210
14	7.30	–9.00	–9.00	–885.80	1.80	17,400	130
19	0.90	–897.80	2.70	–903.20	2.70	18,760	310
19	0.90	–898.90	2.60	–904.10	2.60	18,830	360
22	1.26	–891.00	1.45	–897.00	1.38	18,260	106
23	1.40	–890.00	2.30	–895.70	2.20	18,160	170
28	–9.00	–9.00	–9.00	–863.29	–9.00	15,980	80
31	0.86	–888.13	1.47	–893.91	1.48	18,022	112
33	–9.00	–9.00	–9.00	–9.00	–9.00	16,520	100
36	1.10	–889.50	14.50	–895.30	15.10	18,130	130
37	1.10	–876.30	3.60	–882.80	3.40	16,774	235
38	0.87	–895.74	2.07	–901.13	1.96	18,590	160
39	0.69	–894.46	1.45	–899.88	1.38	18,500	110
41	1.30	–886.99	4.97	–892.93	4.70	17,943	361
47	0.00	–893.41	1.80	–898.74	1.81	18,400	140
50	0.60	–9.00	–9.00	–933.10	15.20	21,700	2100
51	1.00	–9.00	–9.00	–9.00	–9.00	18,320	205
53	0.60	–896.20	2.50	–901.40	2.50	18,610	200
53	0.60	–891.10	4.30	–896.60	4.30	18,230	320
53	0.60	–897.30	3.40	–902.50	4.30	18,700	270
57	1.00	–890.70	1.60	–896.10	1.50	18,190	120
58	1.50	–889.52	1.20	–895.37	1.26	18,135	100
69	1.10	–886.30	2.40	–892.30	2.20	17,900	170
71	2.20	–9.00	–9.00	–9.00	–9.00	18,750	170
72	1.52	–891.00	0.15	–896.00	–9.00	18,200	100
72	1.52	–889.00	0.11	–895.00	–9.00	18,100	105
74	1.20	–9.00	–9.00	–894.60	1.50	18,080	120
74	1.20	–9.00	–9.00	–893.70	1.40	18,010	110

Table A4.12 TIRI L: whalebone

Lab	$\delta^{13}C$	$d^{14}C$	Error	$D^{14}C$	Error	Age	Error
8	–19.40	–9.00	–9.00	–796.00	2.60	12,785	90
13	–13.40	–783.00	5.00	–788.10	5.00	12,450	110
15	–13.20	–795.97	5.16	–800.78	7.42	12,960	300
18	–14.10	–9.00	–9.00	–9.00	–9.00	12,680	70
22	–13.98	–800.00	2.32	–804.00	2.27	13,091	93
29	–14.77	–786.10	4.80	–790.50	4.80	12,600	180
29	–14.77	–788.40	5.90	–792.80	5.90	12,640	230
30	–14.50	–9.00	–9.00	–9.00	–9.00	12,810	100
31	–15.22	–792.64	2.19	–796.69	2.20	12,797	87
33	–9.00	–9.00	–9.00	–9.00	–9.00	11,050	70
36	–14.80	–787.20	11.40	–791.60	11.60	12,600	120
37	–14.55	–762.00	4.60	–767.00	4.50	11,691	154
38	–14.42	–795.37	2.42	–799.70	2.37	12,920	90
39	–17.99	–796.05	1.44	–798.91	1.42	12,900	60
40	–9.00	–799.20	4.70	–9.00	–9.00	12,900	180
45	–14.79	–786.80	3.50	–791.20	3.50	12,580	130
47	–13.20	–786.19	1.17	–791.23	1.63	12,580	60
49	–16.20	–9.00	–9.00	–788.00	8.00	12,440	310
51	–14.20	–9.00	–9.00	–9.00	–9.00	12,875	145
52	–17.90	–777.71	3.39	–780.87	3.40	12,190	130
58	–15.30	–795.40	0.99	–799.37	1.01	12,900	40
69	–15.31	–792.50	3.10	–796.50	3.10	12,790	120

Table A4.13 TIRI M: Icelandic peat

Lab	$\delta^{13}C$	$d^{14}C$	Error	$D^{14}C$	Error	Age	Error
7	–28.70	–215.70	5.40	–210.60	5.40	1900	50
8	–29.00	–9.00	–9.00	–175.60	12.00	1550	120
11	–29.80	–186.00	6.00	–178.00	6.00	1580	60
15	–27.00	–361.38	4.44	–358.79	10.82	3570	120
19	–27.80	–190.10	4.30	–185.60	4.30	1650	70
19	–27.80	–190.20	4.20	–185.60	4.20	1650	80
23	–28.50	–235.70	5.00	–230.20	5.10	2100	50
31	–29.75	–187.29	4.39	–179.56	4.40	1590	43
33	–9.00	–9.00	–9.00	–9.00	–9.00	1550	30
36	–28.80	–195.00	1.30	–188.90	1.30	1680	70
37	–27.55	–169.40	6.20	–165.20	6.20	1448	59
38	–28.77	–218.32	5.53	–212.43	5.57	1920	60
39	–28.36	–206.80	4.05	–201.47	4.08	1810	80
40	–9.00	–224.80	7.00	–9.00	–9.00	2045	70
42	–27.95	–174.30	6.80	–169.40	6.80	1534	68
43	–26.60	–279.90	19.00	–277.60	19.00	2610	220
43	–26.60	–187.70	11.00	–185.10	11.00	1645	110
43	–27.00	–225.30	24.00	–222.20	24.00	2020	250
50	–29.10	–9.00	–9.00	–233.80	36.60	2140	240
50	–28.10	–9.00	–9.00	–195.30	20.30	1750	200
50	–27.30	–9.00	–9.00	–179.50	35.90	–9	–9
53	–27.80	–197.90	5.00	–193.30	5.00	1730	50
53	–27.80	–195.90	3.80	–191.30	3.80	1710	38
53	–27.80	–193.20	5.10	–188.70	5.20	1680	55
57	–28.10	–217.80	3.80	–212.80	3.80	1920	40
58	–28.80	–216.91	3.41	–210.96	3.44	1905	35
71	–28.60	–9.00	–9.00	–9.00	–9.00	1755	30
72	–29.21	–196.00	0.14	–189.00	–9.00	1690	35
74	–28.20	–9.00	–9.00	–187.80	4.20	1670	45
74	–28.20	–9.00	–9.00	–184.50	4.50	1640	45

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