

## **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 (Rozanski 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  $^{14}\text{C}$  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 “malting” 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 <sup>14</sup>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<sub>2</sub> 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<sub>3</sub>) 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<sub>2</sub> 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 <sup>14</sup> C Lab	Estonia	LSC
Kraków	Poland	LSC
Illinois Geological Survey	USA	LSC
Ruder 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 Radiosotope	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 <sup>14</sup> 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  $\delta^{14}\text{C}$ ,  $\Delta^{14}\text{C}$ , 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  $\delta^{14}\text{C}$ ,  $\Delta^{14}\text{C}$ , 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

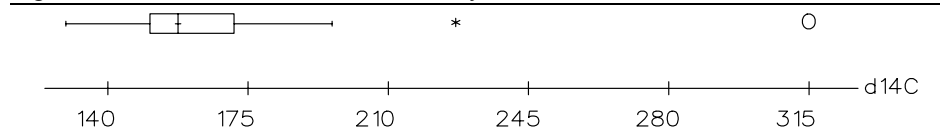


Figure 3.1a Boxplot for  $d^{14}C$

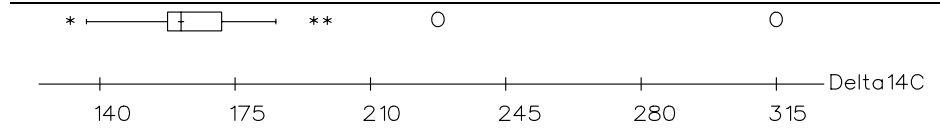


Figure 3.1b Boxplot for  $D^{14}C$

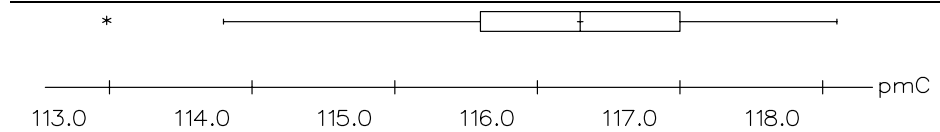


Figure 3.1c Boxplot for pMC

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

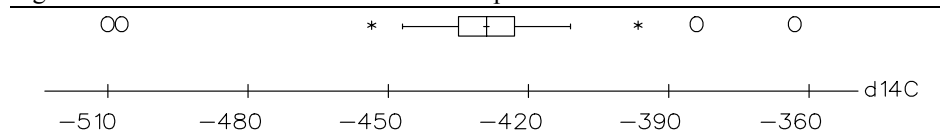


Figure 3.2a Boxplot for  $d^{14}C$

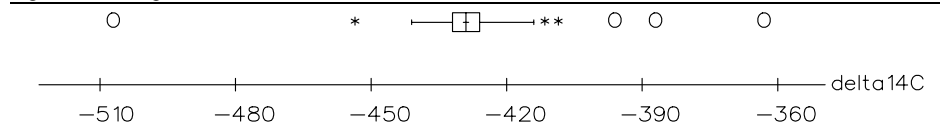


Figure 3.2b Boxplot for  $D^{14}C$

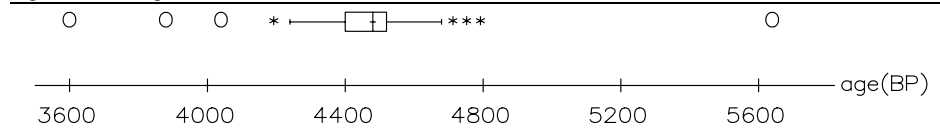


Figure 3.2c Boxplot for age (yr BP)



Table 3.3 Numerical summary for TIRI-B

	N	Mean	Median	Q1	Q3
d <sup>14</sup> C	47	-428.52	-427.30	-434.17	-421.79
D <sup>14</sup> C	63	-426.93	-428.96	-434.40	-423.10
Age	78	4485	4500	4420	4540

**Comments:** The mean <sup>14</sup>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

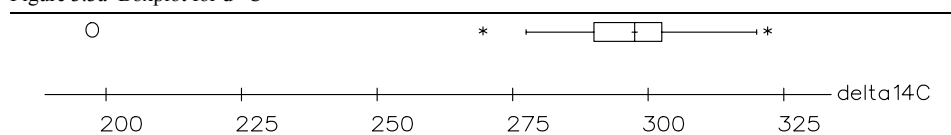
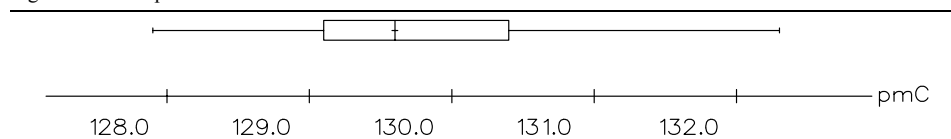
Figure 3.3a Boxplot for d<sup>14</sup>CFigure 3.3b Boxplot for D<sup>14</sup>C

Figure 3.3c Boxplot for pMC

Table 3.4 Numerical summary for TIRI-C

	N	Mean	Median	Q1	Q3
d <sup>14</sup> C	45	295.42	295.50	290.2	302.1
D <sup>14</sup> 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

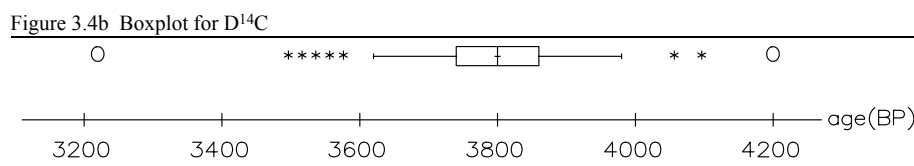
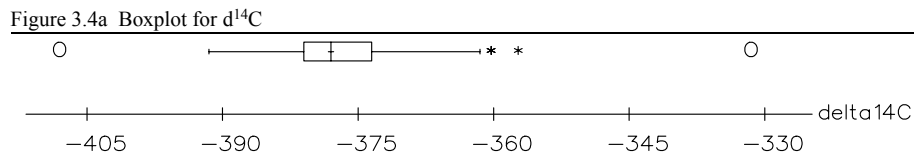
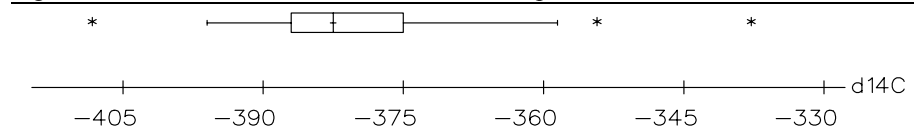


Table 3.5 Numerical summary for TIRI-D

	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

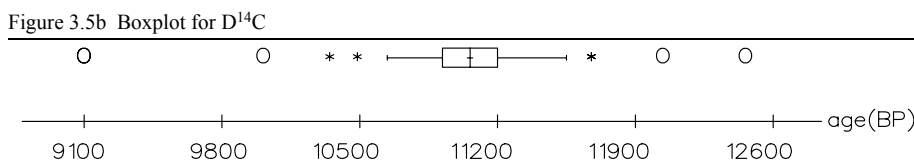
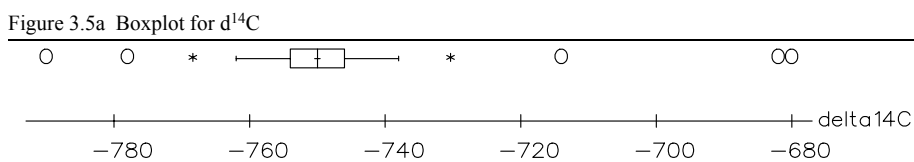
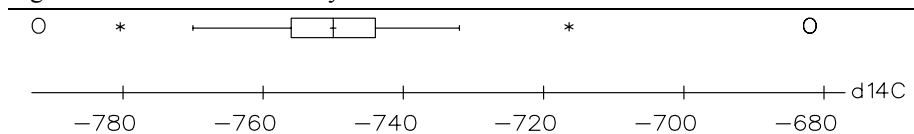


Table 3.6 Numerical summary for TIRI-E

	N	Mean	Median	Q1	Q3
d <sup>14</sup> C	43	-747.7	-749.5	-755.5	-743.0
D <sup>14</sup> 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

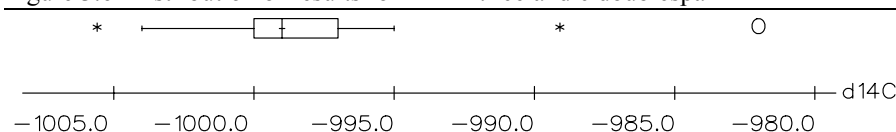
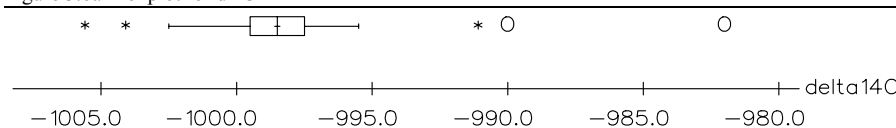
Figure 3.6a Boxplot for d<sup>14</sup>CFigure 3.6b Boxplot for D<sup>14</sup>C

Table 3.7 Numerical summary for TIRI-F

	N	Mean	Median	Q1	Q3
d <sup>14</sup> C	33	-997.9	-998.6	-1000.0	-996.6
D <sup>14</sup> 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  $^{14}\text{C}$  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  $^{14}\text{C}$  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 ( $\text{sd} = [\text{result} - \text{rcv}] / \text{quoted error}$ ). 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  $\pm 2$  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 ( $1 \sigma$ )
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  $\pm 2 \sigma$  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  $\pm 2$  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.

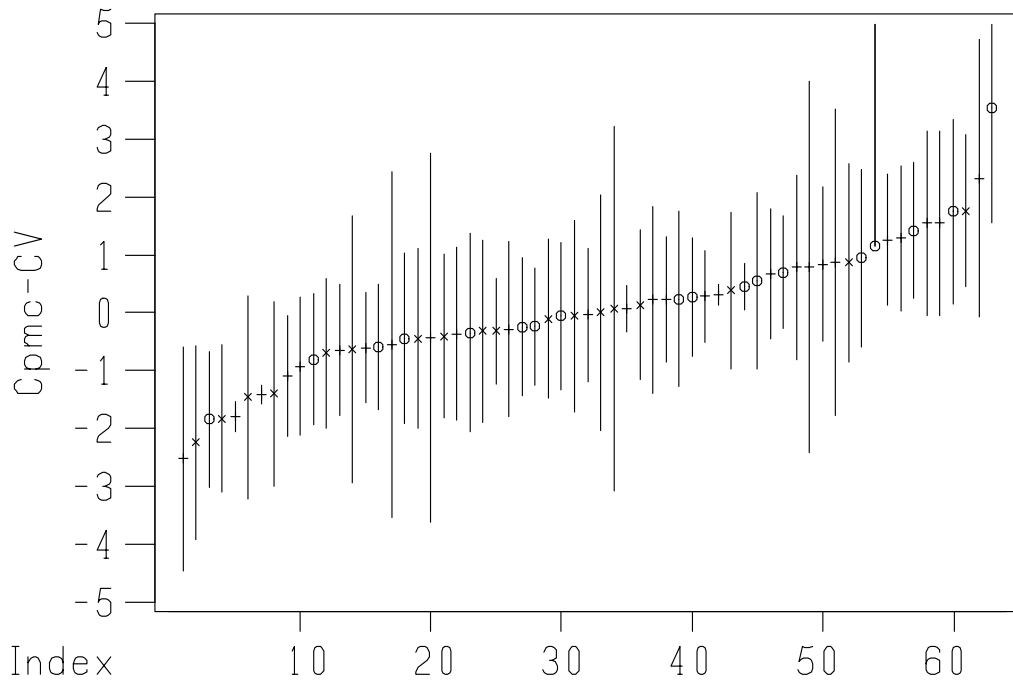


Figure 3.7 Sample A: Glengoyne barley mash

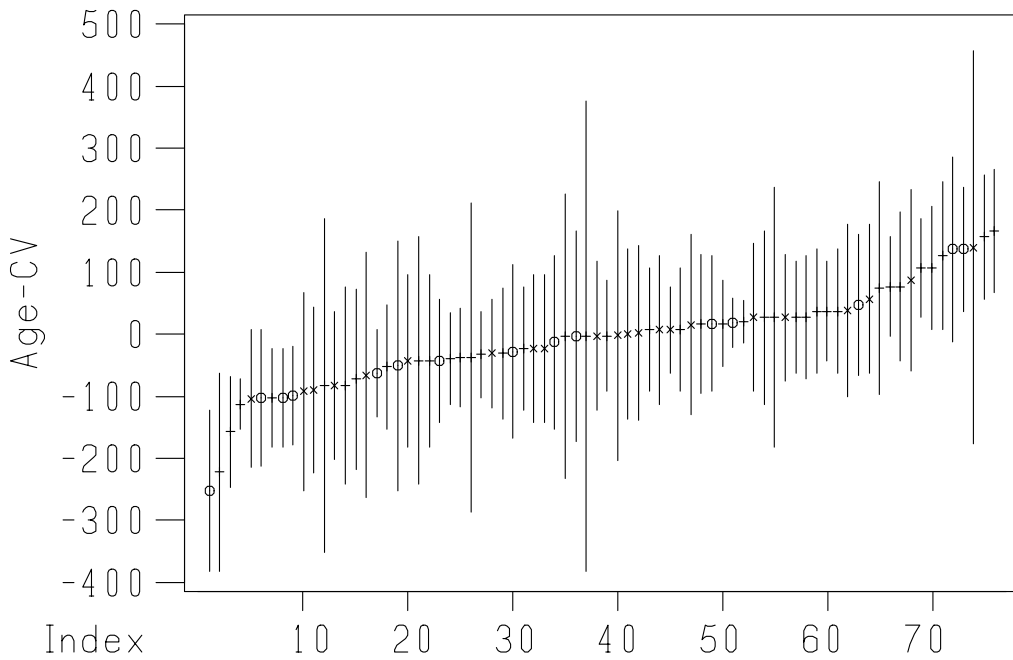


Figure 3.8 Sample B: Belfast wood

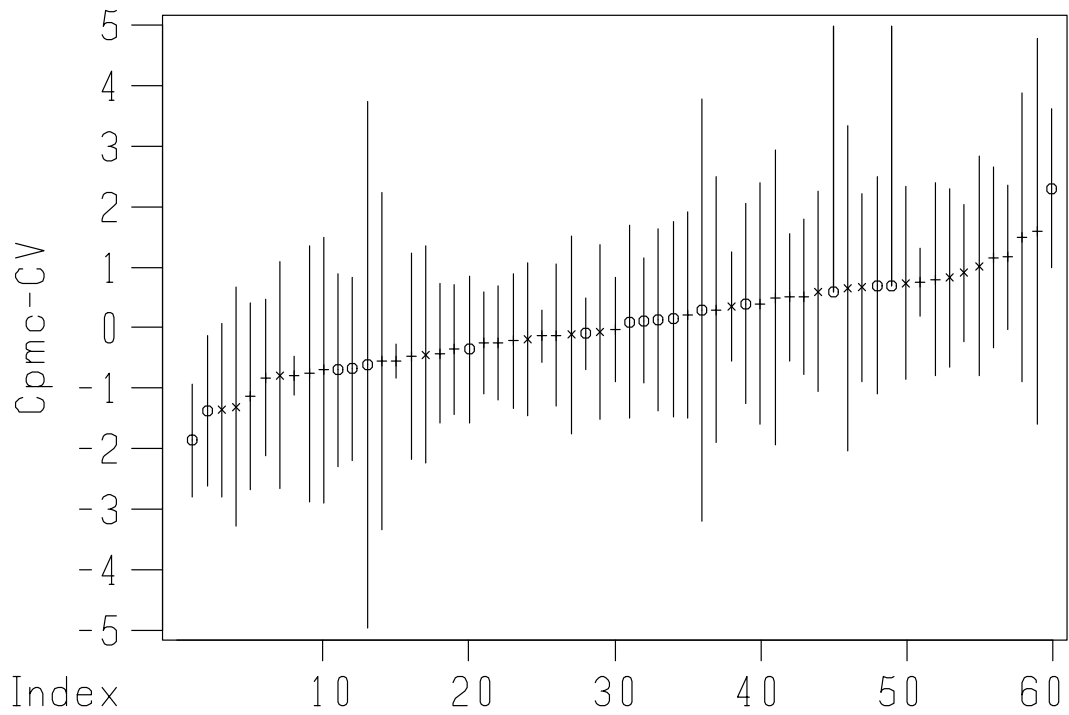


Figure 3.9 Sample C: IAEA cellulose

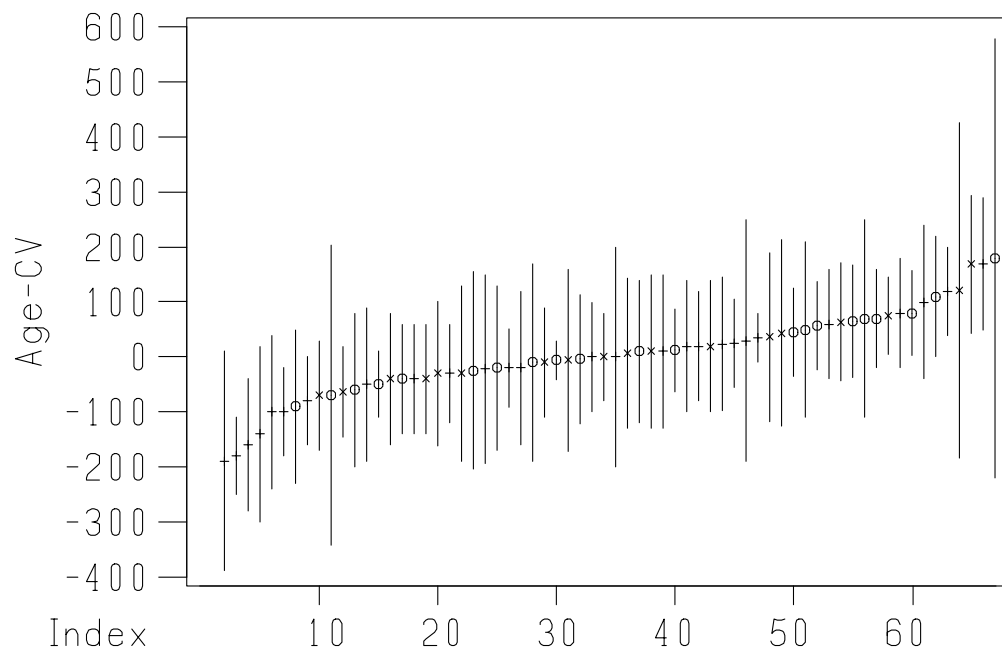


Figure 3.10 Sample D: Hekla peat

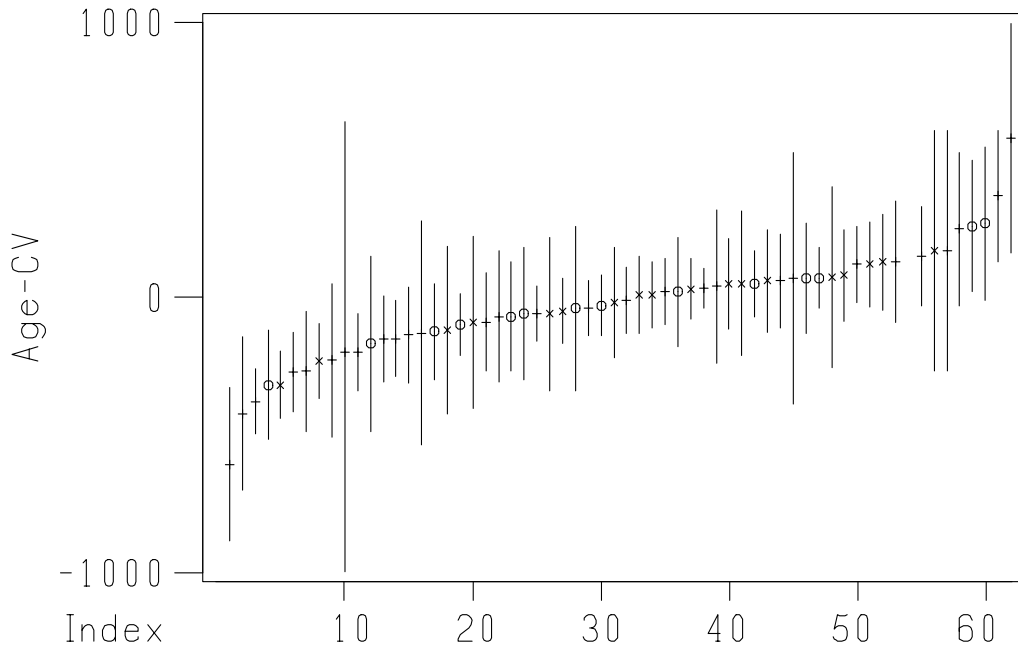


Figure 3.11 Sample E: Ellanmore humic

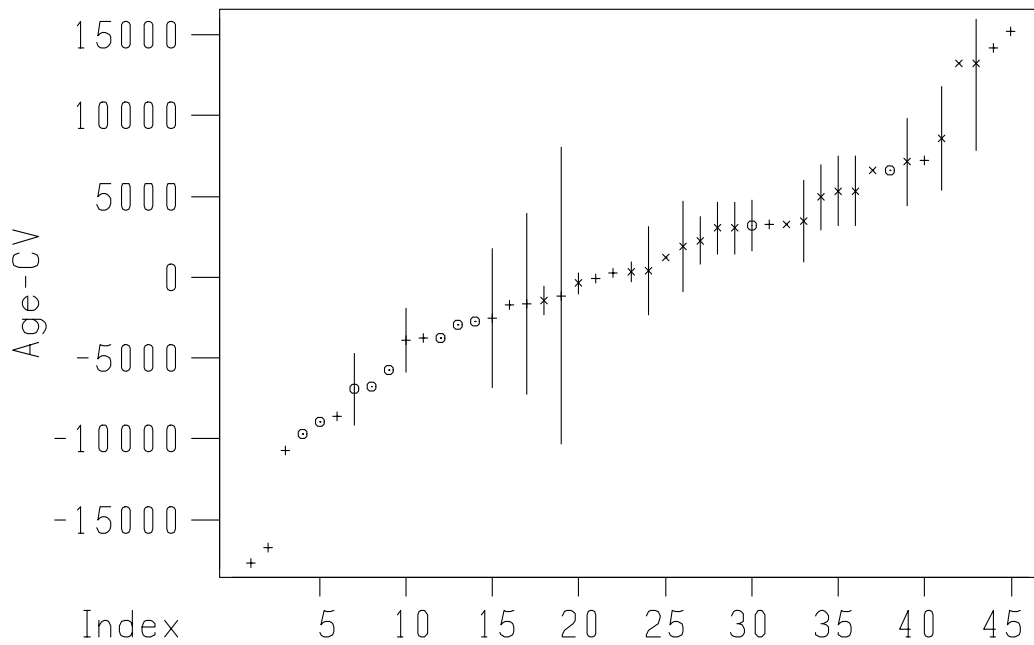


Figure 3.12 Sample F: Icelandic doublespar (yr BP)

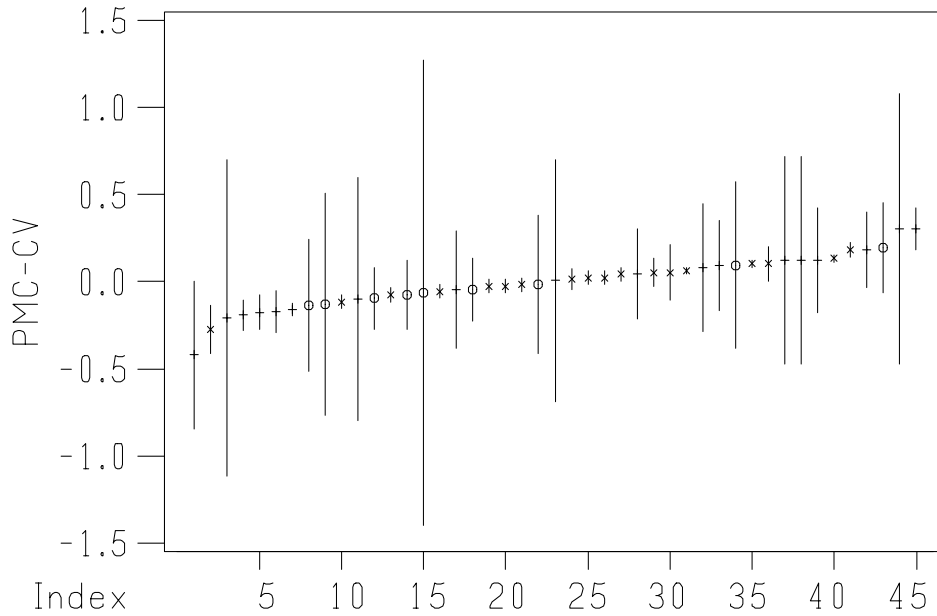


Figure 3.13 Sample F: Icelandic doublespar (PMC)

Further, to allow a comparison of the scatter of results for the different samples, deviations have been calculated, where the deviation is defined as:

$$\text{Deviation} = (\text{laboratory result} - \text{consensus value}) / \text{quoted uncertainty}$$

We would anticipate that deviations should generally lie between  $\pm 2$ , (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.

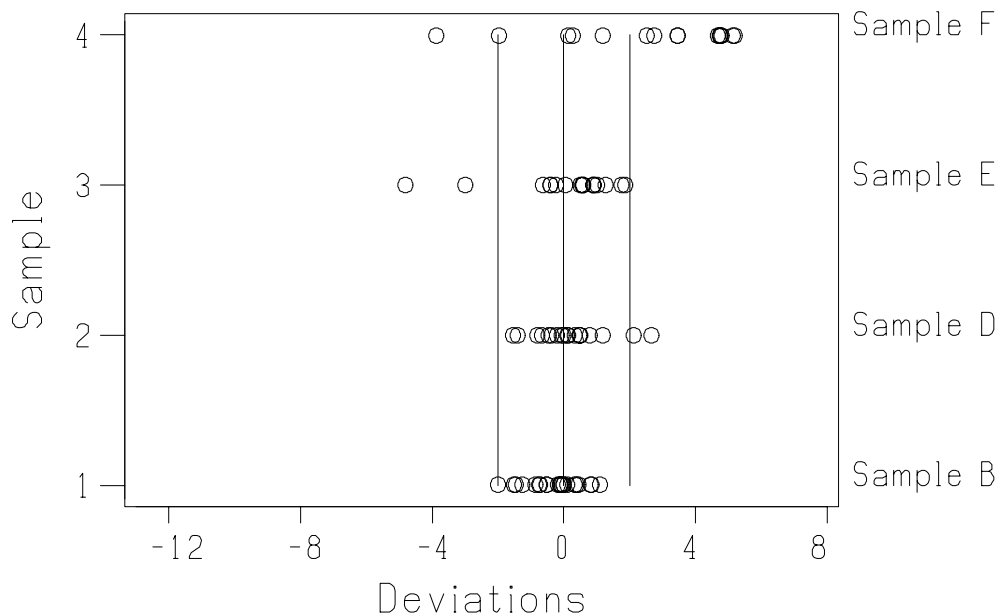


Figure 3.14 Age deviations for AMS Laboratories by sample



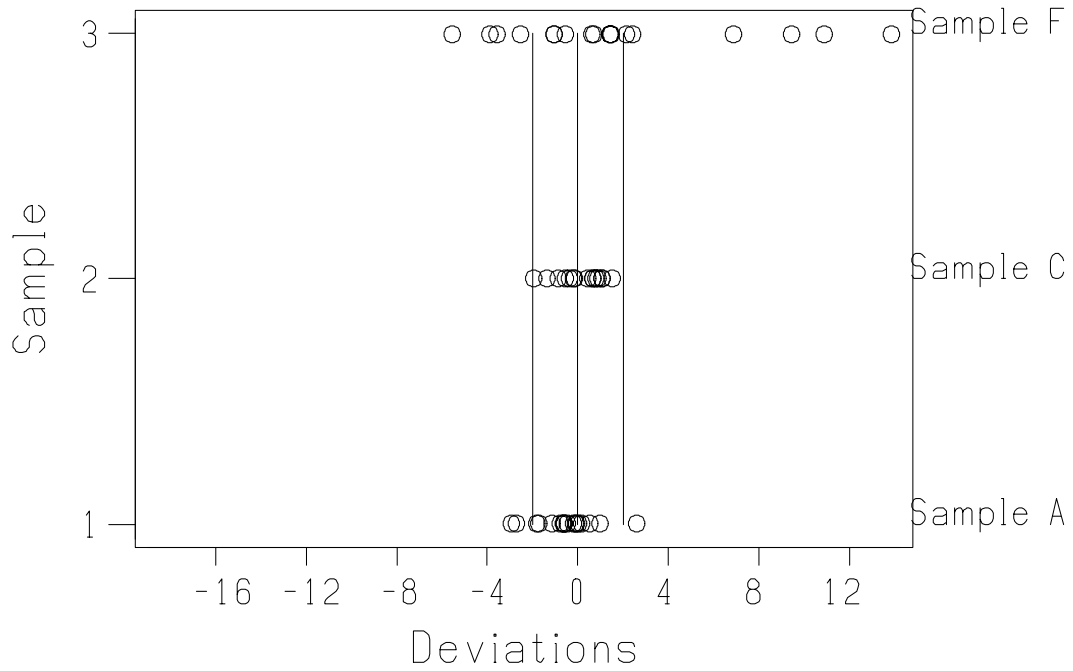


Figure 3.15 pMC deviations for AMS Laboratories by sample

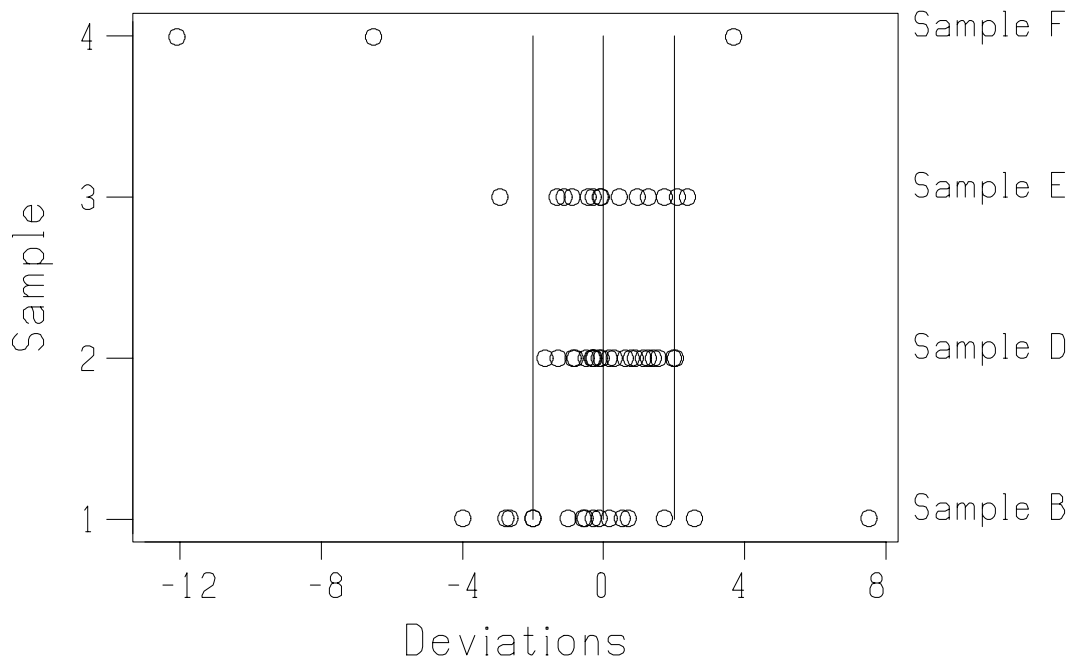


Figure 3.16 Age deviations for GPC laboratories by sample

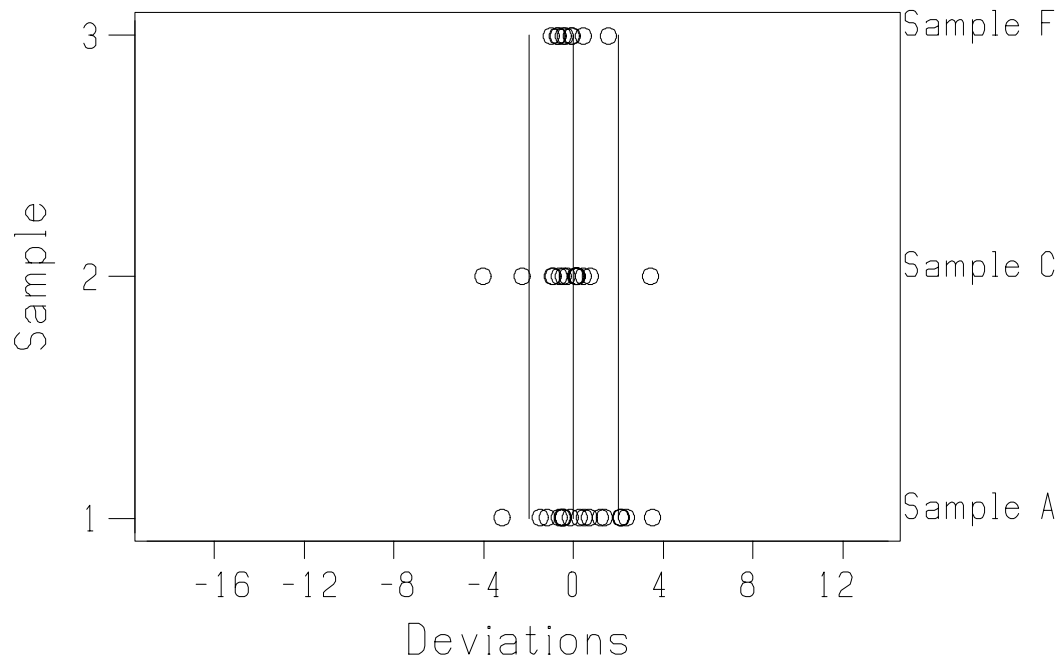


Figure 3.17 pMC deviations for GPC laboratories by sample

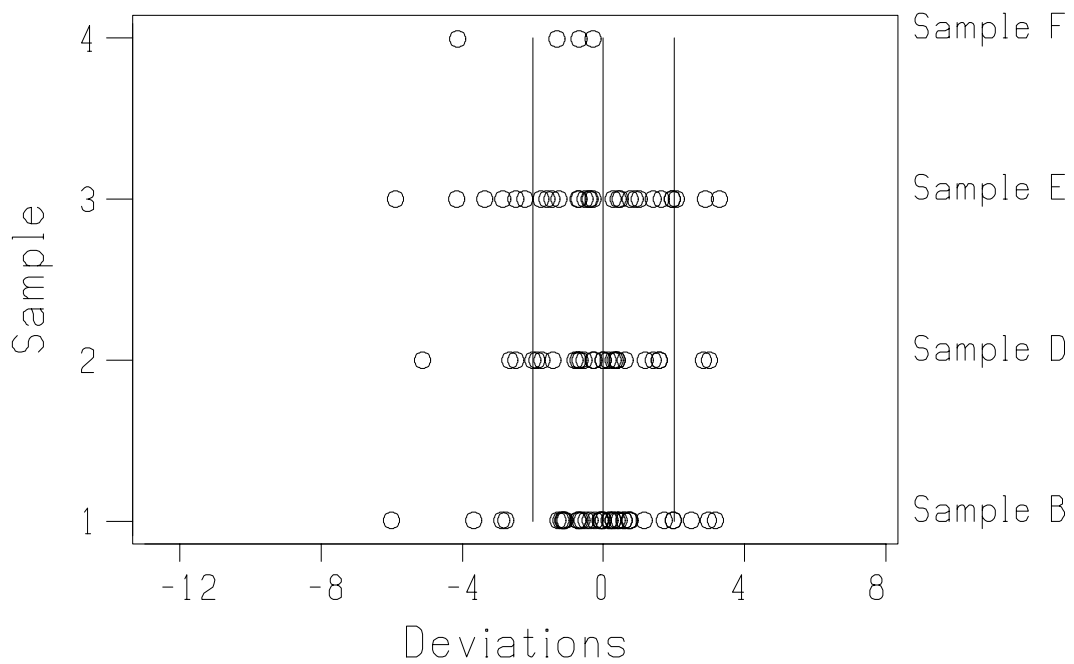


Figure 3.18 Age deviations for LSC laboratories by sample

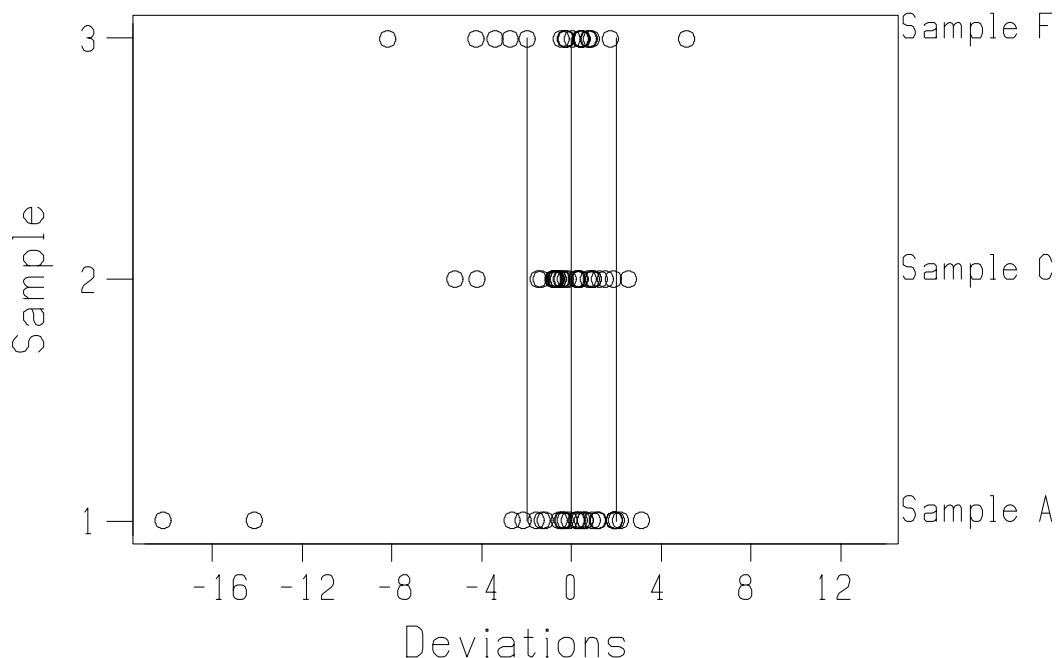


Figure 3.19 pMC deviations for LSC laboratories by sample

#### 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<sub>2</sub> 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 <sup>14</sup> 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 <sup>14</sup> 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
$\delta^{13}\text{C}$	35	3	-26.518	-26.680	1.122	0.190	-28.060	-23.500	-27.520	-25.900
$\delta^{14}\text{C}$	28	10	-996.20	-996.75	3.86	0.73	-1000.50	-981.70	-998.50	-995.16
$\Delta^{14}\text{C}$	28	10	-995.39	-996.52	4.72	0.89	-1000.50	-980.80	-998.47	-993.57

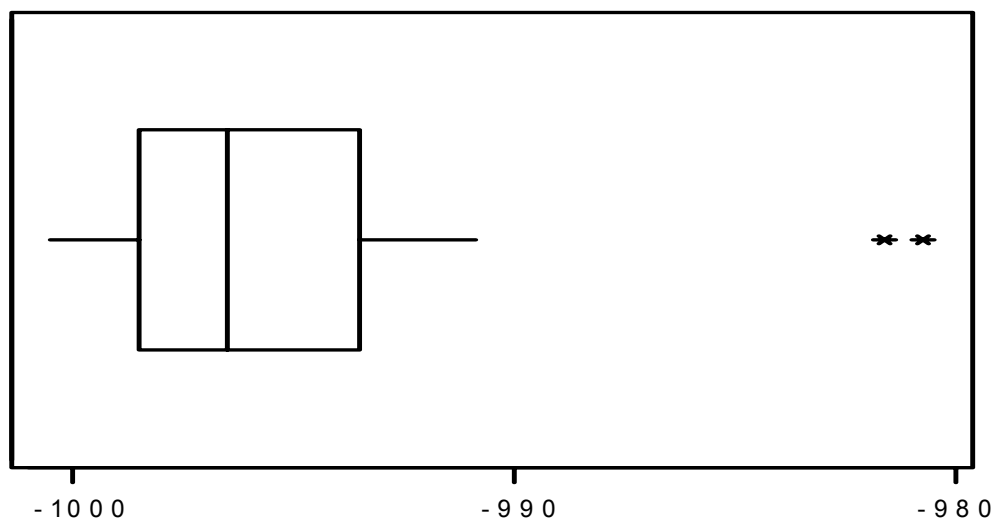


Figure 4.1 Distribution of  $\Delta^{14}\text{C}$  for Sample G

### 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
$\delta^{13}\text{C}$	33	2	-28.392	-28.600	0.679
$\delta^{14}\text{C}$	24	11	-749.25	-749.53	9.95
$\Delta^{14}\text{C}$	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
$\delta^{13}\text{C}$	0.118	-29.200	-26.200	-28.800	-28.050
$\delta^{14}\text{C}$	2.03	-772.90	-723.90	-756.00	-745.04
$\Delta^{14}\text{C}$	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.

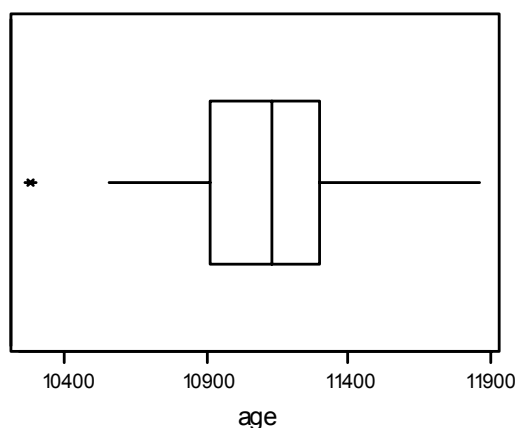


Figure 4.2 Distribution of age for Sample H

#### 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
$\delta^{13}\text{C}$	32	2	-9.556	-9.900	1.224
$\delta^{14}\text{C}$	24	10	-740.42	-740.06	5.82
$\Delta^{14}\text{C}$	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
$\delta^{13}\text{C}$	0.216	-10.700	-4.100	-9.958	-9.690
$\delta^{14}\text{C}$	1.19	-755.14	-730.40	-741.82	-736.30
$\Delta^{14}\text{C}$	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.

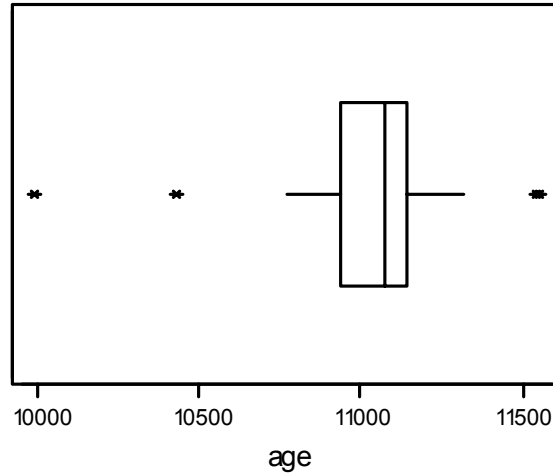


Figure 4.3 Distribution of age for TIRI-I

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
$\delta^{13}\text{C}$	33	3	-26.579	-26.800	1.147
$\delta^{14}\text{C}$	26	10	-184.03	-185.50	12.88
$\Delta^{14}\text{C}$	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
$\delta^{13}\text{C}$	0.200	-28.200	-22.490	-27.400	-25.975
$\delta^{14}\text{C}$	2.53	-211.88	-149.70	-189.93	-175.82
$\Delta^{14}\text{C}$	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).

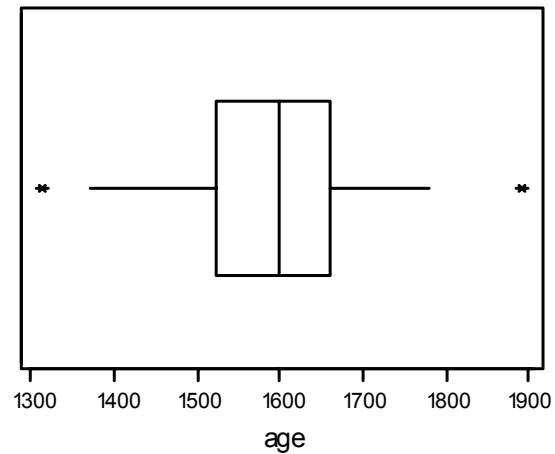


Figure 4.4 Distribution of age for TIRI-J

#### 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
$\delta^{13}\text{C}$	28	2	1.321	1.100	1.260
$\delta^{14}\text{C}$	22	8	-890.57	-890.35	5.21
$\Delta^{14}\text{C}$	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
$\delta^{13}\text{C}$	0.238	0.000	7.300	0.863	1.475
$\delta^{14}\text{C}$	1.11	-898.90	876.30	-894.78	-887.85
$\Delta^{14}\text{C}$	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.

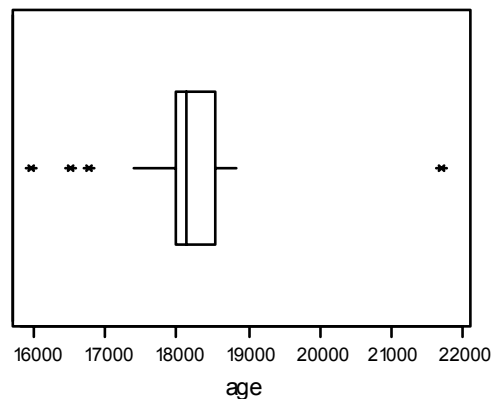


Figure 4.5 Distribution of age for TIRI-K



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
$\delta^{13}\text{C}$	21	2	-15.06	-14.770	1.602
$\delta^{14}\text{C}$	17	6	-789.04	-789.10	9.18
$\Delta^{14}\text{C}$	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
$\delta^{13}\text{C}$	0.35	-19.400	-13.200	-15.305	-14.15
$\delta^{14}\text{C}$	2.23	-800.00	-762.00	-795.68	-786.15
$\Delta^{14}\text{C}$	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.

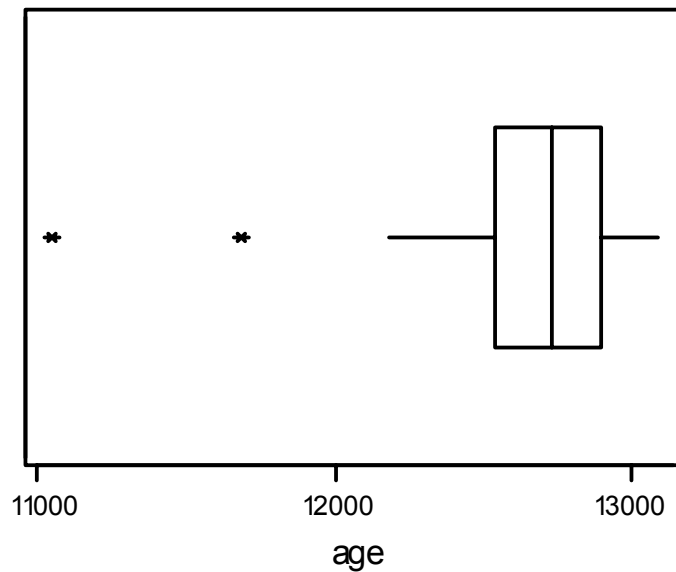


Figure 4.6 Distribution of age for TIRI-L

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
$\delta^{13}\text{C}$	28	2	-28.178	-28.150	0.841
$\delta^{14}\text{C}$	22	8	-212.07	-196.95	41.05
$\Delta^{14}\text{C}$	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
$\delta^{13}\text{C}$	0.159	-29.800	-26.600	-28.792	-27.800
$\delta^{14}\text{C}$	8.75	-361.38	-169.40	-219.94	-189.50
$\Delta^{14}\text{C}$	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.

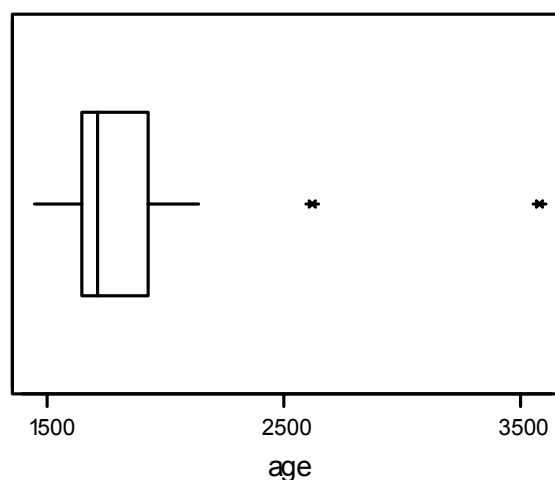


Figure 4.7 Distribution of age for TIRI-M

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  $\text{StDev} / \text{Mean} \times 100$ ), 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 $\sigma$ )
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.

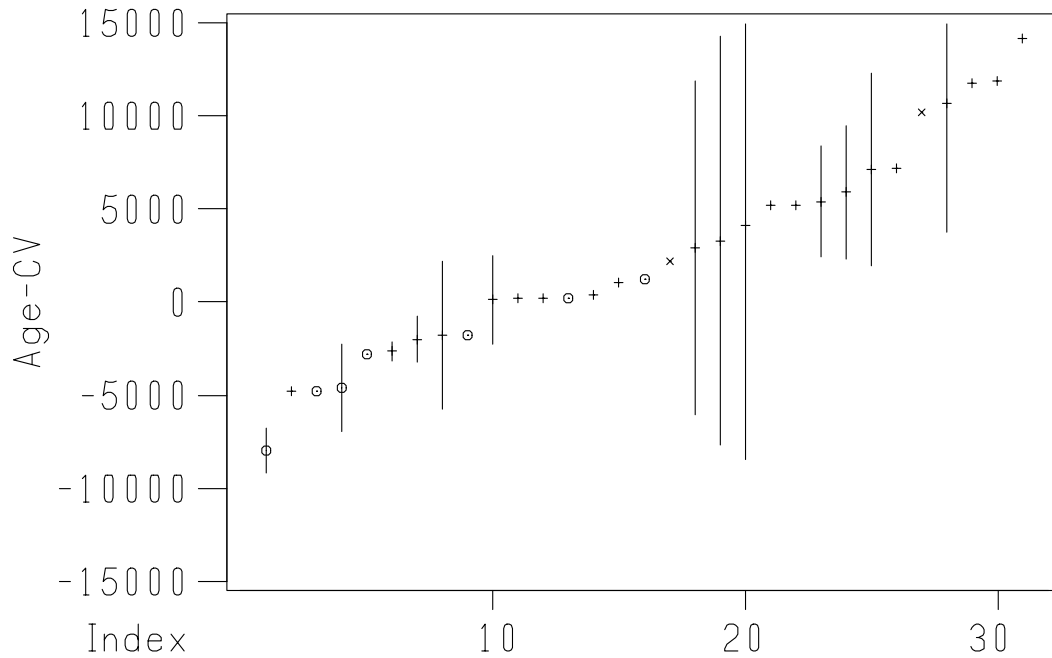


Figure 4.8 Sample G: Fuglaness wood

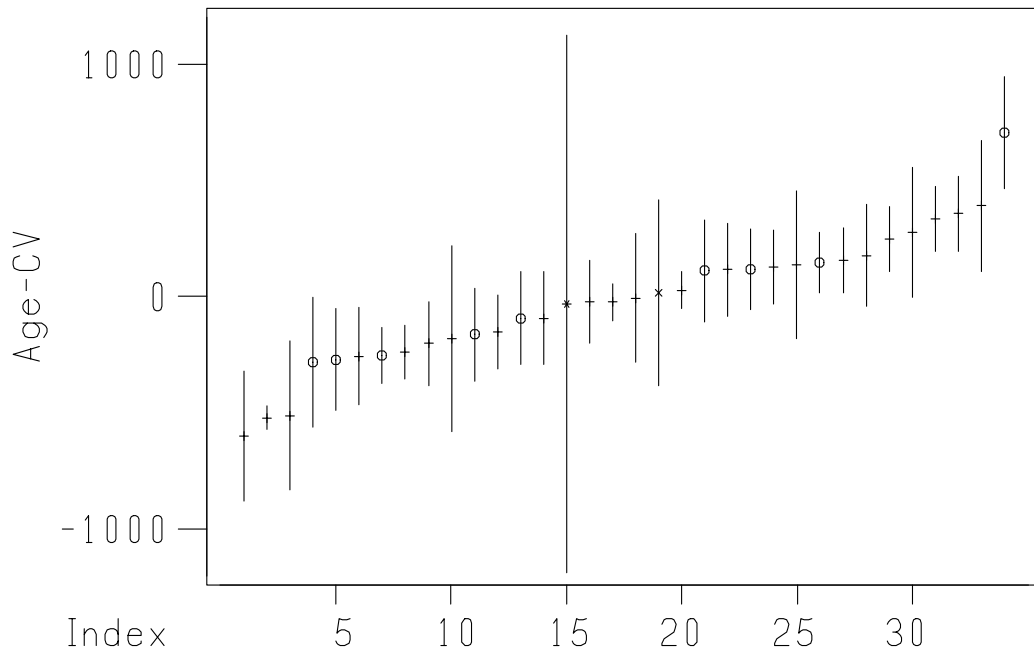


Figure 4.9 Sample H: Ellanmore peat

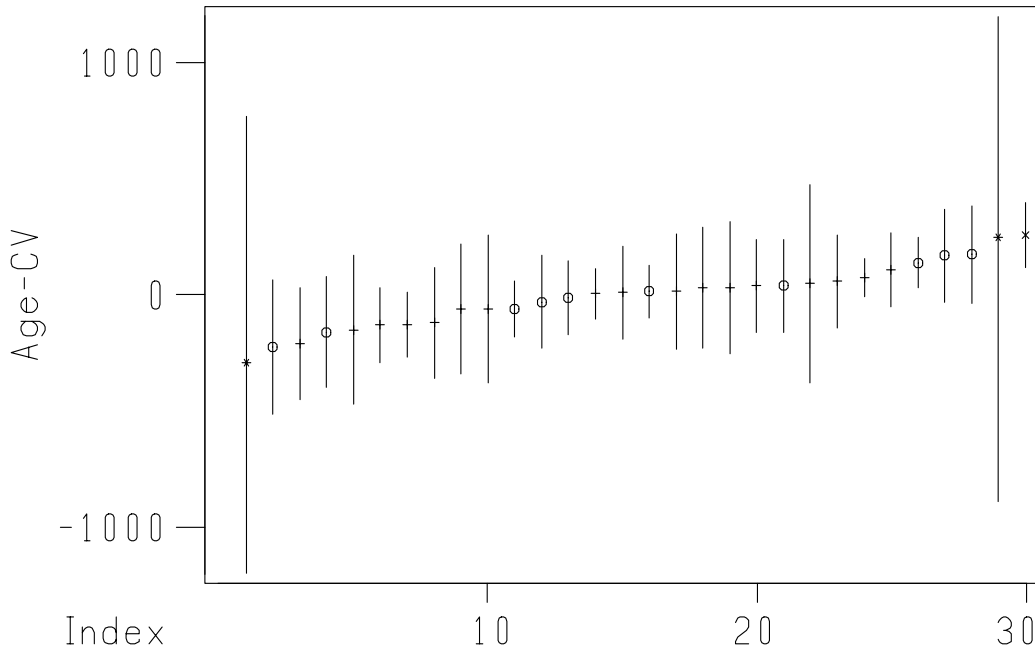


Figure 4.10 Sample I: travertine

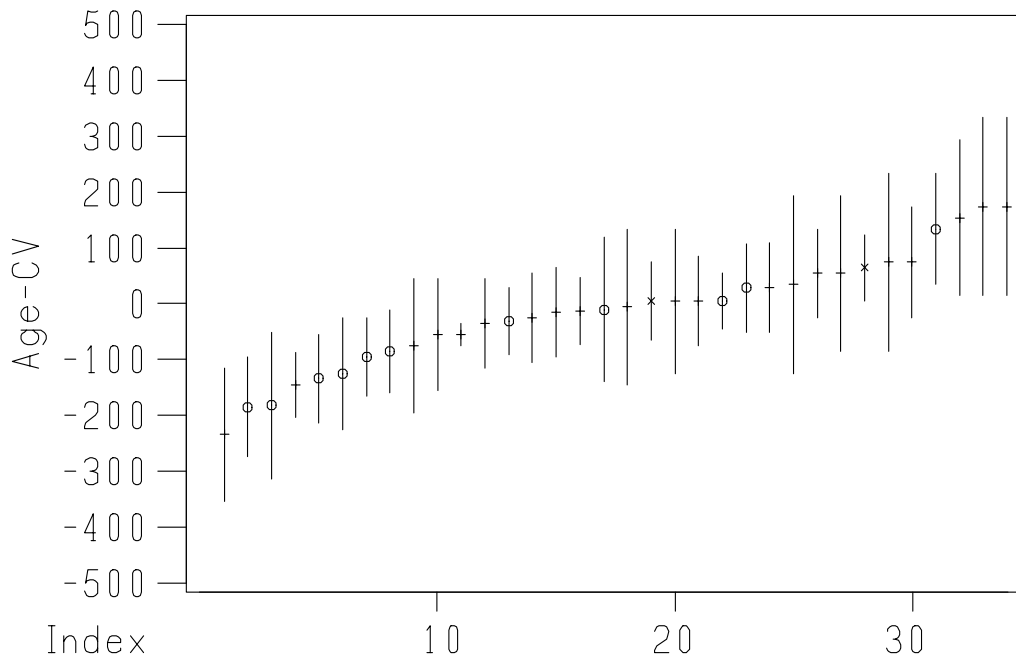


Figure 4.11 Sample J: wood (Buiston Crannog)

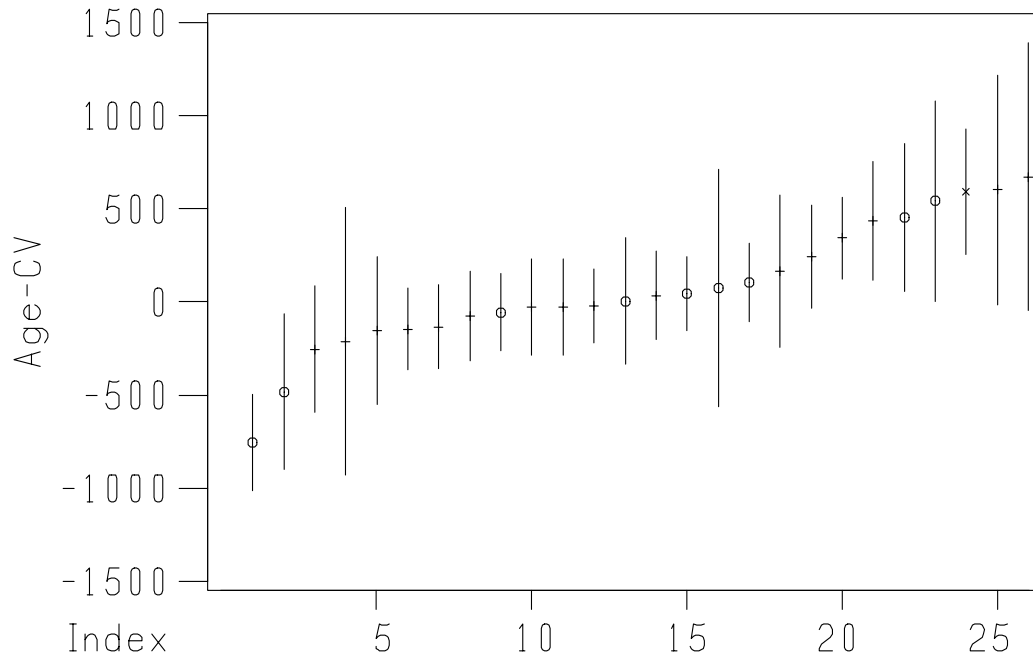


Figure 4.12 Sample K: turbidite carbonate

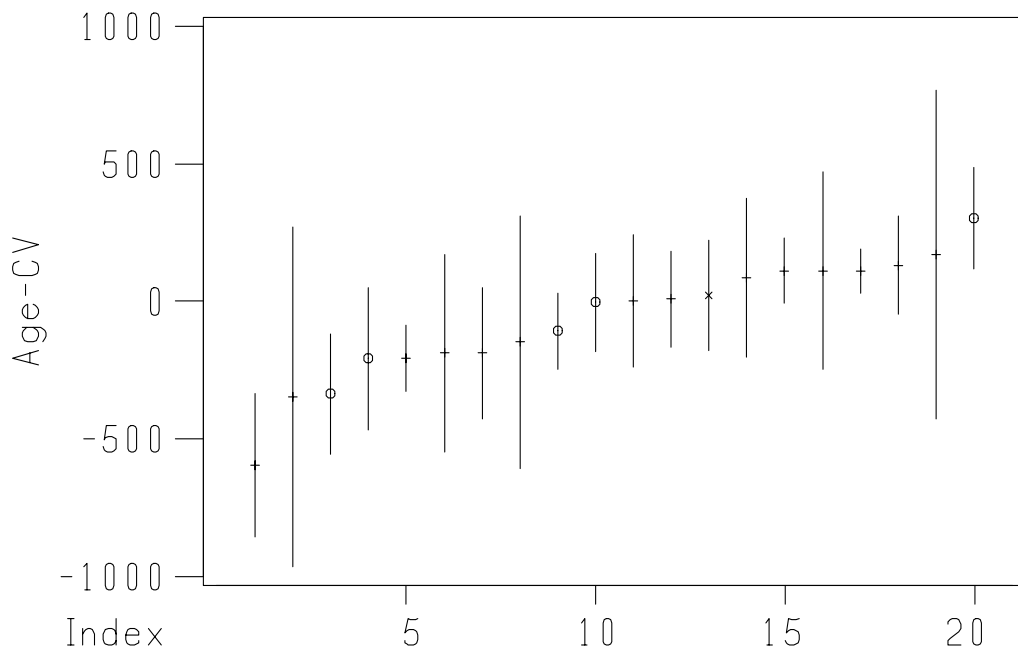


Figure 4.13 Sample L: whalebone

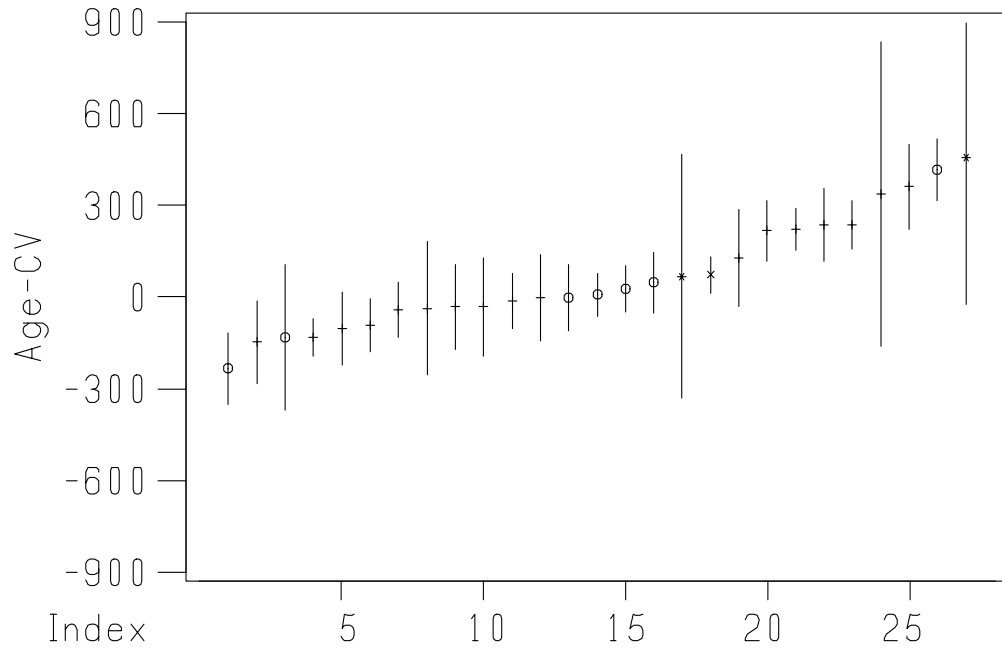


Figure 4.14 Sample M: Icelandic peat

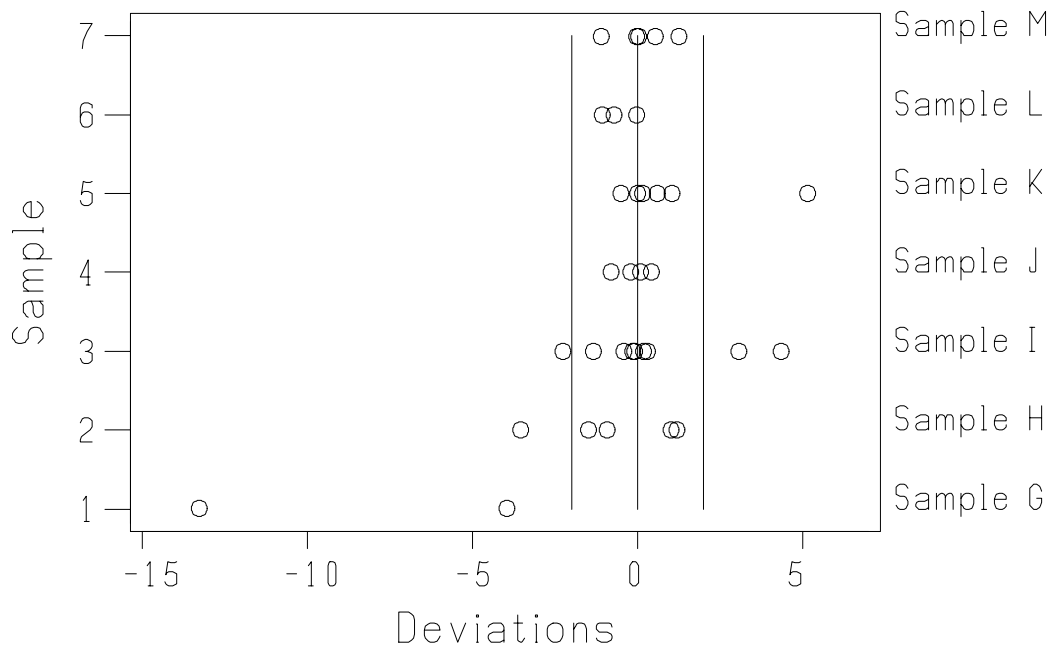


Figure 4.15 Deviations for GPC laboratories by sample

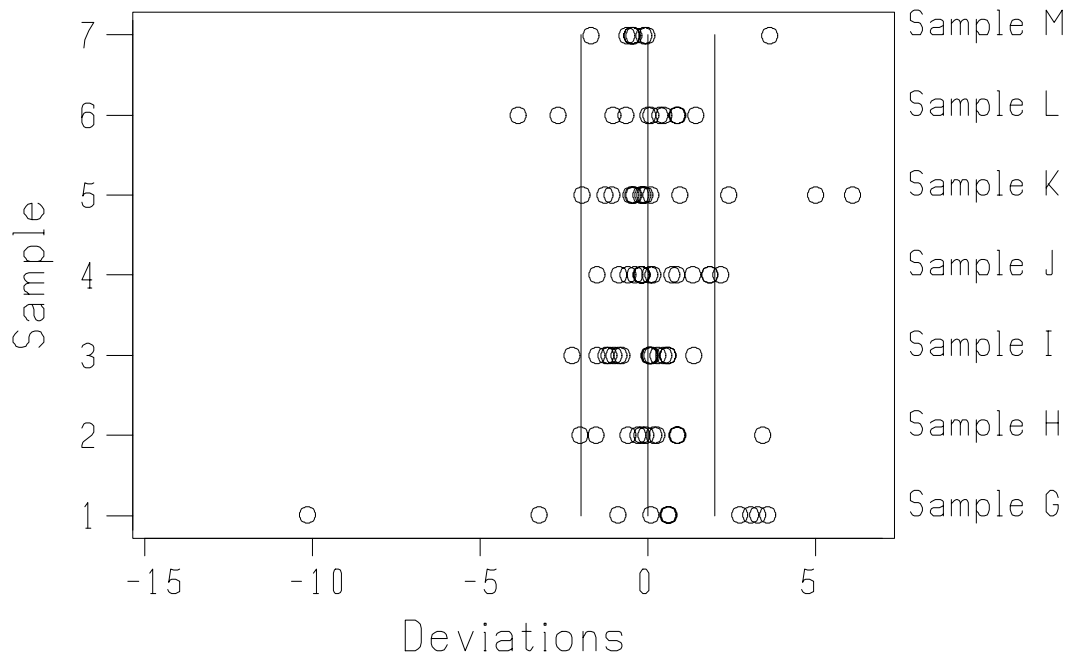


Figure 4.16 Deviations for LSC laboratories by sample

## 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:

$$X_{ij} = \text{Consensus value}_i + \varepsilon_{ij} \text{ for } i = 1, \dots, N \text{ (number of labs) and } j = 1, \dots, J \text{ (number of samples)}$$

where  $X_{ij}$  is the  $^{14}\text{C}$  age for sample  $i$  given by lab  $j$  and  $\text{Consensus value}_i$  is the consensus value for sample  $i$ .

We further assume that  $\varepsilon_{ij}$  is normally distributed with mean 0 and variance =  $S_{ij}^2 \sigma_{ij}^2$

where  $S_{ij}$  is the quoted uncertainty and  $\sigma_{ij}$  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  $\sigma_{ij}$  is calculated based on a  $\chi^2$  distributional result.



For those labs without relative bias, the error multiplier,  $\sigma_i$ , has been calculated under the model, assuming no bias, shown below as:

$$\sigma_{ij}^2 = 1 / J \sum d_{ij}^2$$

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.

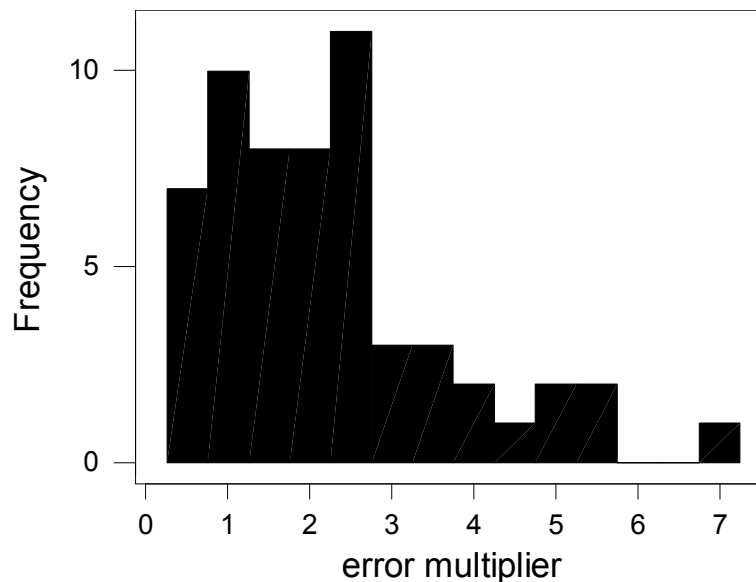


Figure 5.1 Histogram of error multipliers

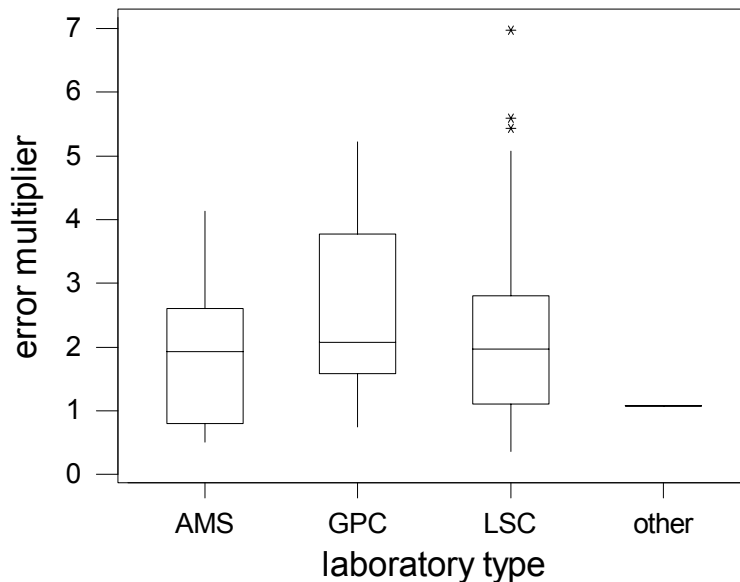


Figure 5.2 Distribution of error multipliers by laboratory type

## 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  $^{14}\text{C}$  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  $^{14}\text{C}$  dating community.

Table 6.1a Consensus values for Stage 1 TIRI samples

Sample	Consensus value	Estimated precision ( $1\sigma$ )
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 ( $1\sigma$ )
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 ( $1\sigma$ )
TIRI-B: Belfast dendro-dated wood	4503 yr BP	3200–3239 BC ( $^{14}\text{C}$ age 4495 BP)	6
FIRI-D, F: Belfast dendro-dated wood	4508 yr BP	3200–3239 BC ( $^{14}\text{C}$ 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.

## REFERENCES

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