ELSEVIER

Nuclear Instruments and Methods in Physics Research B 172 (2000) 355–358

NIM B
Beam Interactions with Materials & Atoms

www.elsevier.nl/locate/nimb

Sample requirements and design of an inter-laboratory trial for radiocarbon laboratories

Charlotte Bryant [a], Israel Carmi [b], Gordon Cook [c], Steinar Gulliksen [d], Doug Harkness [h], Jan Heinemeier [e], Edward McGee [f], Philip Naysmith [c], Goran Possnert [g], Marian Scott [h,*], Hans van der Plicht [i], Mark van Strydonck [j]

[a] *NERC Radiocarbon Laboratory, Scotland, UK*
[b] *Weizmann Institute, Israel*
[c] *SUERC, Scotland, UK*
[d] *NUST, Norway*
[e] *University of Aarhus, Denmark*
[f] *University College Dublin, Eire, UK*
[g] *University of Uppsala, Sweden*
[h] *Department of Statistics, University of Glasgow, Glasgow G12 8QW, Scotland, UK*
[i] *University of Groningen, The Netherlands*
[j] *KIK, Belgium*

Abstract

An on-going inter-comparison programme which is focused on assessing and establishing consensus protocols to be applied in the identification, selection and sub-sampling of materials for subsequent ^{14}C analysis is described. The outcome of the programme will provide a detailed quantification of the uncertainties associated with ^{14}C measurements including the issues of accuracy and precision. Such projects have become recognised as a fundamental aspect of continuing laboratory quality assurance schemes, providing a mechanism for the harmonisation of measurements and for demonstrating the traceability of results.

The design of this study and its rationale are described. In summary, a suite of core samples has been defined which will be made available to both AMS and radiometric laboratories. These core materials are representative of routinely dated material and their ages span the full range of the applied ^{14}C time-scale. Two of the samples are of wood from the German and Irish dendrochronologies, thus providing a direct connection to the master dendrochronological calibration curve. Further samples link this new inter-comparison to past studies.

Sample size and precision have been identified as being of paramount importance in defining dating confidence, and so several core samples have been identified for more in-depth study of these practical issues. In addition to the core samples, optional samples have been identified and prepared specifically for either AMS and/or radiometric laboratories. For AMS laboratories, these include bone, textile, leather and parchment samples. Participation in the study requires a commitment to a minimum of 10 core analyses, with results to be returned within a year.

* Corresponding author. Tel.: +44-141-3305125; fax: +44-141-3304814.
E-mail address: marian@stats.gla.ac.uk (M. Scott).

0168-583X/00/$ - see front matter
PII: S0168-583X(00)00098-7

Keywords: Inter-comparison; Radiocarbon

1. Introduction

1.1. Why another inter-comparison?

The quality of applied science depends fundamentally on the quality of the measurements made in support of the scientific investigation. Radiocarbon by its ubiquitous nature is widely used in many applied science fields and there are many laboratories capable of providing the detailed analyses required. Therefore it is crucial that the quality of the measurements be assured. Laboratory quality assurance has a number of components, including the use of in-house reference materials, measurement of international standards, development and implementation of detailed procedural documentation and regular participation in laboratory inter-comparisons. This latter aspect of laboratory quality assurance provides an independent check on laboratory performance.

Participation in such inter-comparisons is a significant effort on the part of most laboratories, therefore, the time interval between these exercises requires to be sufficiently long so as not to perturb the laboratory working pattern. It has been five years since the previous large ^{14}C inter-comparison and in that time there has been a shift in laboratory demography, with the establishment of additional AMS laboratories and with target preparation representing an increasing proportion of ‘business’ for some existing radiometric laboratories. Technical developments have also meant development of a capability to measure smaller and smaller samples (including compound specific analyses). From the user perspective, there continues to be a perceived desire for more and more precise analyses on smaller and smaller samples. Thus it seems an appropriate time to undertake a new inter-comparison.

1.2. The past

During the past 15–20 years there have been several, large inter-comparison studies, and in each, an increase in AMS participation has been apparent. In those first inter-comparisons, one of the key questions was whether the AMS results were directly comparable to those from the radiometric laboratories. Their design also focussed on the radiometric measurement style and samples were provided in large quantities (in AMS terms). Many of the inter-comparisons [1–4] used natural samples, although, artificially produced samples were also occasionally included. The focus in sample selection was also typically on the archaeological applications of ^{14}C dating. The results of the inter-comparisons showed evidence of significant variation in results and of some laboratory biases but no evidence of a difference in performance due to laboratory type.

In summary, samples have been typically provided in large quantities and in good condition, not necessarily reflecting the day-to-day reality of the laboratory. Nonetheless, for the individual laboratory, participation has proved invaluable and many laboratories have been able to identify and correct problems.

1.3. What is new and how must we respond?

^{14}C dating still remains a key tool for the archaeologist, but its applications are widening, with increasing focus on palaeo-environments and palaeo-climate and anthropogenic enhancements. Small samples are increasingly becoming the ‘norm’ rather than the exception and the move toward compound specific analyses simply reflects the increased sensitivity required to answer the scientific questions posed.

These developments in how the scientific question is phrased, when linked to the technological developments, introduce practical issues of what should be sampled and how that sample relates to the event being investigated. This shift in scientific direction has also raised awareness of some of the measurement issues. For users, sample size has become an increasingly important issue. As a result, for proper interpretation of the radiocarbon age, an understanding of the nature of the sample becomes crucial (not least the magnitude of any natural in-homogeneity of that material).

The current ^{14}C inter-comparison has been designed, at least in part to reflect these scientific priorities. However, the primary objectives are still to answer the fundamental measurement questions relating to accuracy and precision of the analyses, questions which are still equally appropriate for both radiometric and AMS laboratories.

2. Aims and objectives

The fundamental aims and objectives of the Fourth International Radiocarbon Inter-comparison (FIRI) reflect a continuing commitment to the issues of accuracy and precision in basic ^{14}C research and can be simply summarised.

- Demonstration of the comparability of routine analyses of 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 study therefore was conceived with a number of design and sample selection criteria [5].

The design structure is rather simple: the inter-comparison will include core (which all laboratories will measure) and optional samples representing 'typical' materials.

The sample selection criteria are relatively simple to express but more difficult to satisfy due to the quantity of material required. The criteria are that (i) all samples should be natural and several should be dendrochronologically dated wood; (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; (vi) most materials should be suitable for measurement by both AMS and radiometric laboratories and (vii) finally, a fundamental property of any sample is that of homogeneity in ^{14}C activity either as a natural property or artificially induced. This has translated into (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 or (iii) samples that have been chemically treated and physically homogenised in bulk.

3. Current status

The current status of the programme is that all core samples have been identified; they comprise a number of dendrochronologically dated wood samples, cellulose, barley mash, humic acid and a marine turbidite. The optional samples have also been identified and include bone, parchment and textile samples for AMS as well as whole peat, and further wood and cellulose samples suitable for both AMS and radiometric analysis.

Samples have been pre-treated where necessary and tested for homogeneity (by eight replicate analyses performed in a minimum of two laboratories, of which one is an AMS laboratory).

As of September 1999, over 120 laboratories had expressed a willingness to participate and the sample sets were dispatched to all laboratories in October 1999.

4. Conclusions

The overwhelming support by the ^{14}C community for this inter-comparison reflects the clear and continuing commitment to ensuring the quality of ^{14}C measurements as used in every field of application. This is not a static programme, it is one which has evolved as the ^{14}C field has evolved and it will continue to do so. Assuring the quality of the measurement remains an essential laboratory function and the ^{14}C inter-comparison is, and will continue to be, an important part of laboratory quality assurance procedures, providing an independent check on measurement capabilities.

Acknowledgements

This project is supported by NERC grant GR/09/03389 and EC project SMT4-CT98-2265. Mike Baillie, Marco Spurk, Roy Switsur, Glengoyne Distilleries, KhA Arslanov, Steinar Gulliksen, Israel Carmi and Mark van Strydonck are thanked for their help in acquiring some of the samples.

References

[1] International Study Group, Nature 198 (1982) 619.
[2] E.M. Scott, T.C. Aitchison, D.D. Harkness, G.T. Cook, M.S. Baxter, Radiocarbon 32 (3) (1990) 309.
[3] K. Rozanski, W. Stichler, R. Gonfiantini, E.M. Scott, R.P. Beukens, B. Kromer, J. van der Plicht, Radiocarbon 34 (3) (1992) 506.
[4] S. Gulliksen, E.M. Scott, Radiocarbon 37 (2) (1995) 820.
[5] E.M. Scott, D.D. Harkness, G.T. Cook, Radiocarbon 39 (3) (1997) 347.