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ANALYTICAL PROTOCOL AND QUALITY ASSURANCE FOR ^{14}C ANALYSES: PROPOSAL FOR A FURTHER INTERCOMPARISON

E. M. SCOTT,[1] D. D. HARKNESS[2] and G. T. COOK[3]

ABSTRACT. We present a proposal for a further intercomparison exercise following discussions at the 16th International Radiocarbon Conference in Groningen in 1997. This new intercomparison will build on previous exercises by making use of both reference materials already characterized and additional known-age material. For this comparison, we describe two separate but essentially related protocols that are meant to satisfy the different priorities of radiometric and AMS laboratories. The new intercomparison is planned to begin in mid-1998.

INTRODUCTION

A laboratory intercomparison serves two important functions: to permit individual laboratories to check their procedures and results, and to provide the wider user community with an objective demonstration of comparability of results. It aids harmonization amongst laboratories and meets all requirements for an independent and verifiable component of any laboratory's quality assurance. From the statistical analysis of the results, individual laboratories can identify any systematic offsets and quantify any additional sources of variation (beyond those already encapsulated in the laboratory's definition of analytical confidence). Such information is vital to the appropriate interpretation of the scientific evidence provided by ^{14}C analyses in many fields (*e.g.*, the reconstruction of past environments, calibration of chronologies and archaeology, and quantification of the processes of ongoing environmental change).

Previous Intercomparisons

^{14}C laboratories worldwide have already participated in several intercomparisons; however, these have not included all procedures, and some have used nontypical samples, *i.e.*, materials that were not representative of those routinely dated by a majority of participants. With few exceptions, the previous intercomparisons have shown evidence of some systematic biases, and variation additional to that incorporated in the laboratory quoted error.

A number of reference materials have been made available to the ^{14}C community on routine request following on the 1990 IAEA intercomparison (Rozanski *et al.* 1992). The most recent large-scale intercomparison was the Third International Radiocarbon Intercomparison (TIRI), whose experimental work was completed in 1994. TIRI, in common with the earlier intercomparisons, reported the existence of bias and additional variation in the results (Gulliksen and Scott 1995; Scott, Harkness and Cook 1998). Since then, several smaller, more specialized intercomparisons have taken place (Milton *et al.* 1998; Le Clercq *et al.* 1998). The time seems appropriate to undertake a further, larger study, open to all laboratories that wish to participate.

METHODS

General Objectives

We propose these objectives for an analytical intercomparison:

[1]Department of Statistics, University of Glasgow, Glasgow G12 8QW, Scotland
[2]NERC Radiocarbon Laboratory, Scottish Enterprise Technology Park, East Kilbride, G75 0QU, Scotland
[3]Scottish Universities Research and Reactor Centre, Scottish Enterprise Technology Park, East Kilbride, G75 0QU, Scotland

1. providing direct evidence of the comparability or otherwise of the results from different laboratories, which will benefit both laboratory and user community;
2. quantifying the extent and possible causes of any significant interlaboratory variation;
3. assessing the comparability of radiometric and accelerator mass spectrometry (AMS) techniques when subsamples (*e.g.,* different chemical fractions or physical components) are extracted from bulk material.

The achievement of these objectives requires careful consideration of the study design, particularly the choice of sample material. We describe the proposed structure in the following sections, inviting comments and suggestions from intending participants.

Comparison Design

The planned intercomparison builds on the results and lessons already learned from previous intercomparisons but goes beyond them in introducing a third objective, *viz.* point 3 in the preceding section. As in previous intercomparisons, we will offer a set of core samples, which all laboratories will receive, plus optional samples by request and to reflect areas of special interest.

The intercomparison will include two specialized themes—one specifically addressed to radiometric laboratories (those using gas counting and liquid scintillation techniques), the other to AMS laboratories—but both types of laboratory will be linked through common sample material. Direct links to two previous ^{14}C studies (TIRI (Gulliksen and Scott 1995; Scott, Harkness and Cook 1998) and IAEA (Rozanski *et al.* 1992)) will be maintained through the use of existing reference materials. This is an important feature. However, as intimated above, the current proposal introduces some new design aspects, specifically the separation into two separate but related programs for AMS and radiometric laboratories and the emphasis on examining natural and sampling variations (which is only possible with the small-sample capabilities of the AMS laboratories).

Sample Materials and Numbers

The samples used will be representative of material routinely submitted by users and their activities will span the ^{14}C timescale. They will be natural, requiring pretreatment in most cases. The menu of possible samples includes known-age wood (from Germany and Ireland), shells, bone and peat.

The total number of samples in the intercomparison suite is balanced between the requirements for meaningful statistical analysis of the data and, of course, the realistic practical commitments of the participating laboratories. It is intended that the study will include a limited degree of replication (with the identity of replicates withheld from the participating laboratories) to allow a direct assessment of within-lab variation (or repeatability).

Homogeneity of Samples

An essential requirement when using a naturally occurring material as the basis for analytical intercomparison is homogeneity, which is particularly important when sample weight requirements vary by several orders of magnitude. All bulk material will be homogenized and checked by replicate analyses on randomly selected aliquots before distribution. In some instances this will require that the material be chemically homogenized. However, the raw material will also be provided within the core sample suite 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.

Planned Structure of the Intercomparison

AMS laboratories. One of the main objectives here is to explore the variation in results of repeated sampling from a bulk material; therefore, only a limited set of samples will be used. Laboratories would receive 5 samples, and be asked to make several replicate analyses on each sample (up to 3).

Radiometric laboratories. Radiometric laboratories would receive 6 samples, several of which will be in common with those received by the AMS laboratories. Again, a limited number of replicate analyses would be requested. For both AMS and radiometric laboratories, some materials will be provided in both "raw" and pretreated forms.

These would comprise the core samples sent to all participating laboratories, which could be supplemented by optional samples of more specialized interest such as bone and shell.

In total, AMS laboratories would be asked to provide a maximum of 15 analyses, while radiometric laboratories would be required to complete a maximum of 10. It is hoped that the available wood samples will be of known age (dendro-dated) and form part of the international calibration set.

Organizational Details

As in previous studies, the program will be coordinated by the authors, but in this instance several others have agreed to act as scientific advisors. They are Steinar Gulliksen of the Norwegian University of Science and Technology, Tim Jull of the NSF–University of Arizona AMS facility, Alan Hogg of Waikato University and Ellen Druffel of the University of California at Irvine.

Provisional Timetable

A provisional timetable of activities is shown in Table 1.

TABLE 1. Timetable of Intercomparison Activities

Timepoint	Objective
Late 1998	Preparation and distribution of samples
End of 1999	Results returned by laboratories, draft report of comparison completed and circulated to laboratories
July 2000	Formal presentation of findings and workshop during the 17th International ^{14}C Conference

Presentation of Results

The results and findings of the project will be available to the wider scientific community.[4] A final report will be circulated to all participants including all results, and a summary report of the findings will be published. The latter summary will not identify individual laboratories, but is intended rather to discuss the general findings. The results of the intercomparison will also be presented at the 17th International Radiocarbon Conference in Israel in 2000.

Participants

Following the 16th International Radiocarbon Conference, laboratories listed in *RADIOCARBON* were contacted and asked if they were willing to participate in a further intercomparison. To date, over 80

[4]Internet WWW site for project information and results: http://www.stats.gla.ac.uk/~marian/intercomp.html

laboratories have expressed their willingness. If you have not been reached directly and would like to be a registered participant, then please contact Marian Scott at the address given on the first page of this article, or e-mail her at marian@stats.gla.ac.uk.

Conclusion

The provisional plans are presented here to the radiocarbon community for discussion and comment. Funding is being sought to allow the intercomparison to proceed by covering costs of selecting and testing sample material, and shipment and distribution.[5]

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