

NOTES AND COMMENTS

A QUALITY ASSURANCE PROTOCOL FOR RADIOCARBON DATING LABORATORIES¹

Compiled by AUSTIN LONG, with advice and consent from many colleagues
Department of Geosciences, The University of Arizona
Tucson, Arizona 85721

The purpose of this Quality Assurance (QA) protocol is to summarize guidelines that have been accepted by the majority of directors of radiocarbon dating laboratories throughout the world, and by the International Atomic Energy Agency (IAEA). Laboratories that carefully adhere to this protocol will produce consistently reliable data which will be comparable in accuracy to all other laboratories following this or any other equally rigorous quality assurance program. This statement does not, however, pertain to samples with ¹⁴C activities highly sensitive to method or degree of pretreatment, as pretreatment techniques vary among laboratories.

The newly-formed Association of Carbon-14 Laboratories (ACL) has authorized laboratories following this protocol to state in data reports that "These analyses were performed according to ACL-approved quality assurance protocol." Radiocarbon date consumers should understand that ACL cannot guarantee the accuracy of the date, as a variety of factors, many of which are beyond the laboratory's control, can affect accuracy.

BASIC ELEMENTS OF THE QUALITY ASSURANCE (QA) PROTOCOL

- I. Sample Documentation, Traceability
- II. Written Procedures
- III. Analysis of Primary Standards
- IV. Replication of Secondary Standards
- V. Recognition and Correction of Problems
- VI. Establishment of Total Analytical Precision

I. *Sample Documentation and Traceability*

- A. It should be possible for anyone who is unfamiliar with the laboratory, using the laboratory's written and computer records, to reconstruct what happened to any sample, and when it happened, and who did it, from the sample's arrival to the report of the data and the ultimate disposition of the remains of the sample.
 1. *Log Book.* All samples, upon arrival, must be logged-in with a sequential number. The log book will contain the sample lab number, an identification code the submitter gave it, a brief description of the physical or chemical nature of the sample, the name of the submitter, and the date of arrival in the lab. The lab number will follow the sample through the lab.
 2. *Procedures.* The lab personnel will keep up-to-date records of all operations performed on each sample (for example, type of pretreatment performed, comments on pretreatment, CO₂ yields, benzene yields, counter performance, purity corrections, age calculation details and copies of relevant correspondence.)
 3. *Sample Archival.* Remaining sample material, if any, should either be kept in laboratory, returned to submitter or discarded after an established length of

¹ The author welcomes suggestions from readers which will be incorporated into the final version of this protocol and published later this year as "A Suggested Quality Assurance Protocol for Radiocarbon Dating Laboratories" by Austin Long and RM Kalin in the Glasgow Proceedings.

time. Each laboratory must have a policy on sample archival and maintain records of the final disposition of each sample.

- B. A minimum of primary records should be kept in perpetuity. This minimum is the information required for publication in *RADIOCARBON* or the International Radiocarbon Data Base (IRDB), plus laboratory processing data and counting and calculation summary. Some laboratories even retain primary count-rate data.
 - C. Laboratories should retain primary counting data for samples and the graphs of standards and backgrounds (or blanks, in the case of AMS) as long as the particular analysis equipment is in service, and for at least five years after the data appear in publication.
- II. *Written Procedures*
- A. An up-to-date procedures notebook, containing detailed steps with diagrams of equipment, must be in the laboratory while the analysis is underway.
 - B. Records must indicate the nature and dates of all changes in procedures, replacement, repair, modification and adjustment of equipment.
- III. *Analysis of Counting Background, Chemical Blanks and Primary Standards*
- A. Establish the count rates of background and NBS Oxalic Acid at regular intervals and immediately after replacement, repair, modification and adjustment of measurement equipment.
 - B. Time intervals between routine measurements of background and NBS primary standard will vary with general stability of equipment and frequency of measurement of secondary standards, but should not exceed one month.
 - C. Plots of these data ($\pm 1\sigma$) should be on calendric scales, with annotations explaining adjustments of equipment or procedures, that accompanied aberrations and discontinuities in the linearity of the plot. Annotations will also explain adjustments in data (for example, atmospheric pressure corrections, purity compensation). These graphs will be available to illustrate the system's reliability.
- IV. *Replication of Secondary Standards*
- A. Three or four standard materials will soon be available from the IAEA. The results of several laboratories' analyses of these materials will also be available soon. For details, see Long and Kra (1990).
 - B. The purposes of repeat analysis of these known-age materials at regular intervals are:
 1. Continual monitor of analytical accuracy
 2. Recognition of analytical problems before they propagate to the release of erroneous data
 3. Establishment of analytical precision of procedures
 - C. Technicians will run each of these samples through each combustion/hydrolysis/purification/catalysis/counting system in the laboratory at least twice a year. One of the younger secondary standards should be run more often, at least at first, in order to establish analytical precision and error multiplier (see below). New or modified equipment should be tested more frequently until steady operation is proven.
 - D. New personnel should run these test samples until they can "solo", and be tested more frequently than experienced personnel.
 - E. Under routine operation, 20 to 30% of counting time will be devoted to quality assurance activity (background, primary standard, secondary standards). Change in equipment, procedures or personnel will require a more intensive quality assurance effort.

- V. *Recognition and Correction of Problems*
- A. All QA analyses should be plotted on calendric graphs and examined for deviations beyond statistical expectation.
 - B. Frequent analysis of background and primary and secondary standards should reveal problems before affected dates are released.
 - C. Trouble-shooting is beyond the scope of these guidelines. However, considerable expertise is available within the ^{14}C dating community, and several of our most experienced have expressed surprise at never having been consulted for advice.
- VI. *Establishment of Total Analytical Precision*
- A. Radiocarbon dating convention (Stuiver & Polach 1977) requires dates to be reported with the \pm figure reflecting only the counting statistics. In practice, only some laboratories adhere to this convention. Some laboratories arbitrarily increase this figure; AMS laboratories usually report an uncertainty based on replication of data.
 - B. The error figure most relevant to the consumer of ^{14}C dates is the Total Analytical Precision obtained by repeat analysis, through the entire chemical and physical system in the laboratory, of a homogeneous material similar to many samples of unknown age normally run through the lab.
 - C. For β -counting systems:
 1. Calculate the Total Analytical Precision from the standard deviation of repeat ^{14}C analyses of the known-age test samples available from the IAEA. Analyses included in this calculation should be all those analyzed within the past year with the following exception. Do not include analyses originally affected by some analytical problem now recognized and corrected before release of erroneous data. Analyses included in this calculation should have about the same standard deviation. Laboratories that produce data that, for the same age range, have significantly different counting statistics for whatever reason (longer or shorter counting times, different counters or pressures, dilution), should carry out separate standard deviation calculations for each set of data grouped by similar ($\pm 20\%$) values of counting statistics errors.
 2. Calculate the error multiplier factor (Stuiver & Pearson 1986) for each data set characterized by size of the counting statistics error. The error multiplier for a particular data set is the ratio of the standard deviation of the ages within a data set (σ_i) to the average counting error of the individual dates (σ_c). This error multiplier, called “K” by Stuiver and Pearson, should be equal to or greater than 1.0. It will also depend on the value of σ_c . Laboratories should re-evaluate this factor annually, and after significant changes in equipment, procedures or personnel.
 3. Laboratories should either release the relevant K value along with dates in publications and with an explanation of its application, or publish the laboratory analytical error instead of the conventional \pm figure. In either case, the laboratory responsible for the date should clarify which error figure is presented.
 - D. For AMS systems:

It is not practical to use counting statistics as a ‘conventional error’ in AMS ^{14}C dating. We recommend that AMS laboratories report \pm values on their ^{14}C measurements based on reproducibility of test samples within a single loading of a batch of targets, and on repeat samples run over several months. This would be comparable to the Total Laboratory Precision in β -counting laboratories. Laboratories should specify how the error is calculated (see Donahue *et al* 1990).
 - E. All \pm values reported should be based on 1σ standard deviations.

REFERENCES

- Donahue, DJ, Linick, TW and Jull, AJT 1990 Isotope-ratio and background corrections for accelerator mass spectrometer radiocarbon measurements. *Radiocarbon*, this issue.
- Long, A and Kra, RS, eds 1990 International Workshop of Intercomparison of Radiocarbon Laboratories, Proc. *Radiocarbon* 32(3) in press.
- Stuiver, M and Pearson, GW 1986 High-precision calibration of the radiocarbon time scale, AD 1950–500 BC. *In* Stuiver, M and Kra, RS, eds, International ^{14}C Conf, 12th, Proc. *Radiocarbon* 28(2B):805–838.
- Stuiver, M, and Polach, HA 1977 Discussion: Reporting of ^{14}C data. *Radiocarbon* 19(3):355–363.