

QUALITY ASSESSMENT OF ^{14}C DATES

A report from international studies and the workshop in 1989 in Glasgow

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ABSTRACT

For at least 25 years concern has been expressed at the ^{14}C conferences that the ^{14}C measurements are often less reliable than would be expected from the statistical uncertainties given together with the dates. With well collected, stored and pretreated samples it should have been possible satisfactorily to measure a background sample and the common international standard sample or possibly a secondary standard sample, related to the international standard. This came into use after the ^{14}C conference 1959 in Groningen. International comparisons of samples distributed before the New Zealand conference in 1972 and thereafter have revealed discrepancies, which should not occur.

Introduction

The ^{14}C age, T , of a sample is calculated according to the formula

$$T = T_{1/2} \cdot \frac{1}{\log 2} \cdot \log \frac{S_0 - B}{S_T - B}$$

where $T_{1/2}$ is the half-life, S_0 the count rate of the standard, S_T that of the sample, and B the background of the detector. No detector for radioactivity can be constructed to have no background. To obtain the net activity the background must be subtracted from the count rate. At conventional measurements, using gas or scintillation counting, it is assumed that all necessary normalizations are performed, such as correction of the background value for barometric-pressure changes unless these are eliminated by a good shield (Olsson 1958, 1988, de Vries et al 1959). The same amount of the sample as of the standard should be used although normalization to the same amount can be achieved if the amount is well measured and the correction factors are known (Olsson 1982a, 1988). The conditions at the measurements should be the same, e.g. the high voltage used, but here too normalizations can be performed for small deviations from the normal situation. Differences in the purity of the gas at gas counting and of the liquid at scintillation counting must also be compensated.

The conditions for accelerator measurements are similar insofar as the ratio between two $^{14}\text{C}/^{12}\text{C}$ ratios is measured, and several physical parameters must be carefully controlled. The background problem is physically different from that at conventional measurements. Instead of measuring the radioactive decay the ratio between the number of ^{14}C atoms and the stable atoms is measured by counting the atoms after mass spectrometer separation enabled by a strong acceleration of negative ions from the source and positive ions after the acceleration.

All measurements require that a normalization for the isotopic fractionation in nature be performed. The heavier isotopes are enriched when carbon dioxide is dissolved in water and depleted when assimilated by plants. Because of the secular variations of the $^{14}\text{C}/^{12}\text{C}$ ratio of the atmospheric carbon dioxide the ^{14}C ages should also be calibrated.

The pretreatment of all samples is essential for reliable results at conventional and accelerator measurements. The procedures always followed in the Uppsala conventional dating laboratory are given in the introduction of the date lists and by Olsson in numerous papers, e.g. Olsson (1972a, b, 1979, 1982b, 1983a, b, c, 1985a, b, 1989) and Olsson & Florin (1980). A survey of possible uncertainties at gas counting was presented by Olsson et al (1962) and Olsson (1983b, 1988). A similar detailed discussion

of the uncertainties at scintillator counting was given by Pearson et al (1977) and Pearson (1979, 1980).

Although the given formula for age calculation indicates that the measured ages of any sample should, within the limits of estimated uncertainties, agree between different laboratories, this is not always the case. A common standard sample and an "infinitely old" sample should be available to determine the actual values S_0 and B for the particular laboratory. Then it only remains to measure the sample net activity. This would be the ideal case but the ^{14}C community realized already long ago that the deviations of the results from laboratory to laboratory are larger than indicated by the estimated uncertainties. This fact has made international comparisons necessary.

It must also be stated that no result can be reliable unless the sample is carefully collected. At the discussions at the ^{14}C conference in 1965 somebody even suggested that a sample should be split and sent to 100 laboratories to test their standards but then professor Flint said that we also could send out 100 archaeologists to collect one sample each to date an event and let one laboratory date all samples to test the archaeologists.

Standard activity

The need of a common standard was clear since we learned in the early fifties that combustion of fossil fuels had diluted the radioactive carbon of the atmosphere, that the ^{14}C activity of the atmospheric carbon dioxide increased as a consequence of nuclear-weapon tests, that Münnich (1957) published findings on the ^{14}C activity of tree rings indicating natural variations of the atmospheric $^{14}\text{C}/^{12}\text{C}$ ratio and that de Vries (1958) added more results and made the first attempt to explain the variations by correlating them to climatic changes. At the ^{14}C symposium in Groningen in 1959 three laboratories presented a joint paper on the ^{14}C activity of tree rings from a *Sequoia* trunk (Willis et al 1960). This *Sequoia* series covered the period from AD 659 to AD 1859 and 25 samples were taken. The sample from AD 1859 was chosen as the standard for all three laboratories. Most results were within $\pm 2\%$ from the standard after correction for radioactive decay from the growing time to AD 1859. The standard deviation of the determinations was $\pm 0.6\%$ but of the nine samples measured in two laboratories as many as three showed a difference of more than 2% between the two laboratories involved in that particular comparison. Each laboratory thus measured 10, 11 or 12 samples and of these each of the two other laboratories measured three. Besides the obvious discrepancies, the variations within ± 2

% confirmed that a common standard was needed instead of the different wood standards used at ^{14}C dating. At this time it was decided that an oxalic-acid sample from the National Bureau of Standards in Washington (NBS) should be used by all laboratories as the primary standard. (NBS is now NIST - National Institute of Standards and Technology).

To fit the natural activity shortly before the disturbance from the combustion of fossil fuels it was also decided that the standard activity should be 95% of the activity of this oxalic acid in 1950. Moreover that the $\delta^{13}\text{C}$ value must be fixed to eliminate errors due to isotopic fractionation in the laboratories. Craig (1961) measured the $\delta^{13}\text{C}$ for all laboratories whereupon a decision was reached that the normal value should be -19% in the PDB scale. The zero in this scale is determined by the ^{13}C content of a belemnite from the Pee Dee formation in South Carolina.

Since the first lot of oxalic acid was almost finished by the end of the 1960's discussions were initiated to develop a new standard. Sucrose was produced in huge amounts (Polach & Krueger 1972). It originates from sugar cane grown in Queensland, Australia from September 1969 to June 1971 and thus has an activity about 1.5 times that of the oxalic-acid standard activity. The sucrose is, however, difficult to burn and the spread of the ^{14}C results from different laboratories too large to be accepted as a primary or secondary standard used in all laboratories. Any laboratory, familiar with the combustion technique for this substance may of course use the sucrose as a laboratory standard related to the official NBS oxalic-acid standard.

A new oxalic-acid sample was prepared from French beet molasses from 1977. Material from this was sent to some laboratories to measure the activity related to the old oxalic-acid sample (Cavallo & Mann 1980). The results were published by Mann (1983). The old oxalic acid is also called SRM-4990 and the new RM-49. The $\delta^{13}\text{C}$ for SRM-4990 is now accepted to be between -19.2 and -19.3% and that for RM-49 would be -17.7 to -17.8% . The value for RM-49 is based on a difference from that for SRM-4990 of 1.5% , although the mean in this comparison for SRM-4990 was -19.1% and both samples exhibited a non-normal distribution of the $\delta^{13}\text{C}$ values. The ^{14}C community decided at the Seattle conference in 1982 that the new oxalic acid should be normalized to $\delta^{13}\text{C} = -25\%$, and that the ratio between the standard activities should be 0.7459 when the new oxalic acid was normalized to -25 and the old to -19% (Stuiver 1983). The inverted value is 1.3407 ± 0.001 . The old oxalic-acid sample activity is multiplied by 0.95 to yield the defined primary ^{14}C standard activity, right in 1950.

Table 1. Some samples dated in several laboratories before the oxalic-acid standard was accepted and the corrected dates according to the Index

Laboratory number and comments	Published date \pm	Corrected date \pm	Reference
<i>Allrød/Younger Dryas boundary. Wood submitted by H. Tauber</i>			
K-101, solid carbon	10 890 240	11 090 240	Anderson et al (1953)
K-101 bis, K-102 bis, K-103 bis mean, solid carbon	11 030 200		Tauber (1960)
W-82	10 260 200		Suess (1954)
W-84	10 510 180		
H-105-87	11 500 300		Münnich (1957)
St-18, solid carbon	10 200 370	10 145 370	Östlund (1957)
BM-19	11 333 200		Barker & Mackey (1959)
U-20	10 830 130	10 950 130	Olsson (1959)
U-75	10 680 130	10 810 140	Olsson (1959)
GrN-454, originally Gro	10 995 250		de Vries et al (1958)
R-64, old value of R-64 discarded	11 200 145		Alessio et al (1965)
<i>Lago di Nemi. Wood from Roman ships attributed to Emperor Caligula's reign (AD 37-41) submitted by Corten Bella</i>			
R-1, solid carbon	2 125 75		Bella & Cortesi (1957)
R-1, gas counting	1 990 85		Alessio et al (1964)
T-9	1 880 130		Nydal & Sigmond (1957)
St-103A	1 940 70	1 885 70	Östlund (1957)
St-103B	2 090 75	2 035 75	Östlund (1957)
BM-15	2 080 150		Barker & Mackey (1959)
U-68	1 980 70	2 110 80	Olsson (1959)
U-239, same gas as U-68	2 120 80		Godwin & Willis (1959)
Q-112	1 904 95		Olsson (1964)
<i>St Walburgkerk. Wood from a church in Groningen submitted by de Vries</i>			
Gro, mean value	980 50		de Vries & Barendsen (1954)
T-29	1 050 100		Nydal & Sigmond (1957)
H8-7	1 245 130		Münnich (1957)
A-81A&B, mean, solid C	900 160		Shutler & Damon (1959)
A-81 bis	1 080 140		Damon & Long (1962)
L-292	1 250 150		Olson & Broecker (1959)
U-69	1 095 70	1 220 80	Olsson (1959)
K-143, solid carbon	1 380 120		Tauber (1960)

Problems with the oxalic acid

The measurements of the $\delta^{13}\text{C}$ values for the old oxalic acid (Craig 1961) revealed discrepancies which to some extent seemed correlated to the technique used at the combustion. Polach (1972) found that he obtained acceptable values for the ^{14}C activity only when he had small deviations from the accepted $\delta^{13}\text{C}$ value of -19 ‰. Several scientists (Grey et al 1969, Kim 1970, Polach & Krueger 1972, Valastro et al 1977) have found that an appreciable isotopic fractionation easily occurred at the combustion of the oxalic acid with far different $\delta^{13}\text{C}$ values in the beginning and at the end of the combustion. Thus the combustion should be complete with a yield very close to 100 % for obtaining a reliable standard.

Early international comparisons

Many laboratories which started ^{14}C dating in the fifties measured three samples of different ages

made available in large quantities. When the Uppsala conventional laboratory had finished these measurements a summary was compiled (tab 1) from published date lists. It is reproduced here to demonstrate the unsatisfactory agreement and the influence on early results by the correction to the common standard according to the agreement on NBS oxalic acid as the standard sample. In 1967 *The American Journal of Science* issued *Radiocarbon Measurements: Comprehensive Index, 1950-1965* with previously published results corrected for this change of standard and other possible factors. Obviously the corrections may change some results far more than the given statistical uncertainties. It is recommended to check in literature what standard was used for every result from 1965 or earlier and then, if oxalic acid was not used, search the Index for what result should be used.

An international cross-calibration exercise was

initiated almost 20 years ago with some 15 laboratories involved. Two samples were distributed; namely AD 1850 wood from Arizona and ANU sucrose from the Australian National University. The results (Currie & Polach 1980) were related to the oxalic acid standard. The consensus value for the deviation for the wood was $-2.5 \pm 1.5 \text{ ‰}$ and for sucrose $+508.1 \pm 2.0 \text{ ‰}$, when both samples were normalized to $\delta^{13}\text{C} = -25 \text{ ‰}$, but only the wood age corrected for the decay to 1950. Consensus values were used partly because there were three outliers with deviations more than four times the given uncertainty in the set of values for sucrose. About half of the laboratories reported uncertainties of 4% or less and all but one of the others, 5 to 8%. The $\delta^{13}\text{C}$ values for oxalic acid and wood exhibited a greater spread than those for the sucrose.

Numerous interlaboratory comparisons and duplicate measurements in the laboratories have been performed but may be difficult to trace in the literature. The conventional laboratory in Uppsala has exchanged single samples with several laboratories and made many duplicate measurements. Six samples were dated in Lund and Uppsala (Olsson 1981) since both laboratories were involved in the measurements of samples from Gårdlösa (Olsson & Håkansson 1981). Polach (1973) published many interlaboratory comparisons and duplicates from his own laboratory.

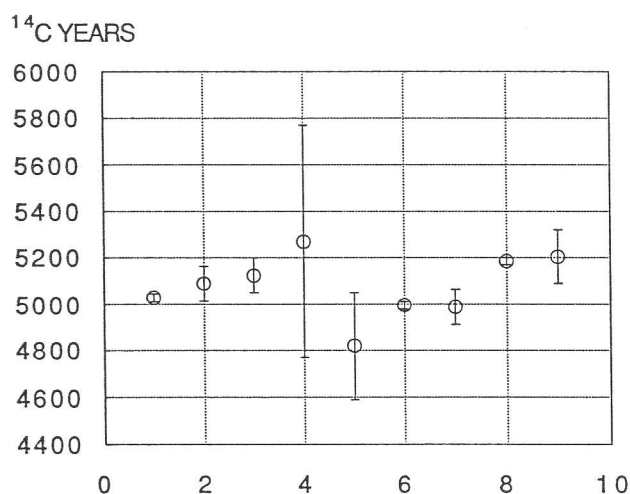


Fig 1. A Comparison between the consensus value (1) and INSI (wood insoluble in NaOH), INS2 (wood insoluble in NaOH after a second treatment), and the fraction extracted with HCl and that with NaOH for rings 205-214, values 1-5; a comparison between the consensus value (6) and INS for rings 147-156, values 6-7; a comparison between the consensus value (8) and the fraction extracted with NaOH for rings 2-11, values 8-9.

An international comparison on eight tree-ring samples

Twenty ^{14}C laboratories participated in an interna-

tional comparison organized by a group of scientists in Glasgow (International Study Group 1982). Eight samples, each of 10 rings width, cut from a wood section covering 200 years, were used. Originally more laboratories were free to participate. Fourteen laboratories submitted results on all eight samples. A cellulose fraction should be extracted according to a submitted recipe, although some laboratories used their own procedures for the pretreatment. The laboratories in Uppsala and La Jolla tested the pretreatment and found that this was not critical for these samples. The Uppsala comparisons between the cellulose and various fractions for three samples are given in figure 1. The "standardized residuals" were calculated, by the organizers, for the laboratories for each sample as the age difference from the mean value divided by the error quoted by the laboratory. Very few residuals should fall outside the range -2 to $+2$. If each laboratory had given eight results the number of dates would have been 160. The problem arose whether the mean value should be calculated from all results for each sample or from the results from a selected group of laboratories. The results were treated using three differently calculated mean values for each sample and thus three baselines were received. Besides the residuals a bias was calculated for each laboratory for the different baselines. Six laboratories had a significant bias (5% level) and for three or four, dependent on the choice of baseline, the bias exceeded 100 years. Similarly four or six laboratories should multiply their given uncertainties with a factor larger than 2 to obtain realistic uncertainties. A similar number of laboratories obtained a factor less than 1; since the factor should scatter around 1 and very seldom exceed 2 half to three quarters of the laboratories had acceptable results although it must be remembered that the number of samples dated by each is too small for conclusive judgements. The results were also broken down according to the type of laboratory. The three high-precision laboratories apparently had, as a group, overestimated the results, since a bias was detected between them. The three laboratories reporting uncertainties ≥ 80 years seemed to have realistic estimates considering that some results should appear too good at a statistical analysis. The nine non-high precision gas-counting laboratories had far better estimates of the uncertainties than the eight non-high precision liquid-scintillation laboratories. It is important to recall that the described study yields an idea of the general performance 10 years ago of a small fraction of the laboratories producing ^{14}C dates. The results do not allow a statement that a consumer of ^{14}C dates should regard the uncertainties quoted by an individual laboratory to be too small. Since anonymity was requested the customers must find out from other sources how a particular laboratory performs.

The study mainly yields information *re* ^{14}C measurements but essentially nothing about the quality of the pretreatment since the samples apparently were unusually easy to pretreat.

The described study involved a study of the bias. This and the error multiplier should be differentiated in certain investigations. ^{14}C variations within a series of results can be detected even if the whole series suffers from a bias against other laboratories. Such a bias may derive from a value in error of the standard activity. In a laboratory there may be a bias between the detectors, and the bias may vary with time. The conventional ^{14}C laboratory in Uppsala submitted separate results from two detectors. Only one set of values was included in the printed report (International Study Group 1982). The two sets are, however, compared in a diagram by Olsson (1990).

The recent international interlaboratory comparison

A three-stage comparison comprising 16 samples was organized from Glasgow (Scott et al 1989, 1990). The Nordic laboratories were represented by those of Copenhagen, Espoo, Helsinki, Lund, Trondheim and the two in Uppsala. The first samples were received in September 1986 and the last in March/April 1988. The results and related questions were discussed in Glasgow in September 1989. As many as 52 laboratories of 80 invited participated in the first stage by returning results. Nineteen laboratories used carbon-dioxide gas-counting, five other gases, 20 liquid-scintillation counting and eight accelerator measurements. The number of laboratories participating in stage 2 was 37. There was a reduction with six laboratories using CO_2 gas counting, six using liquid-scintillation counting and three using accelerators. The number of laboratories participating in stage 3 was 38.

The aim was to check the counting process in the first stage. Two duplicate samples were supplied and there was a choice between carbonate, suitable for the 32 laboratories using gas counting and accelerator measurements, and benzene, suitable for scintillation counting. Some of the scintillation-counting laboratories measured the carbonate samples. The young ages were modern and about 900 years for the carbonate and benzene samples respectively. The old ages were about 3600 and 6900 respectively. No pre-treatment was included in the laboratory process. The aim of stage 2 was similar to stage 1, thus no pretreatment, but involved a combustion and preparation of the gas, liquid scintillator or the source needed for the measurements. Two samples out of three could be chosen (humic acid, cellulose and carbonate as the marine alga *Lithothamnion*).

The aim of stage 3 was to include the whole preparation, including pretreatment, and measurement in the laboratory. Three samples (wood, shell and peat) were submitted in duplicate and besides these six samples two dendrochronologically dated wood samples were included in this stage. All samples were younger than about 3400 years. The peat sample was the same as that from which humic acid was extracted in stage 2.

The author has some comments and some questions on the samples and the results, mostly not yet discussed with the organizers:

a) The consensus value for humic acid was 3390 and that for the peat 3395. It is a seemingly excellent agreement, but the normal pretreatment for peat is to remove the humic acid with sodium hydroxide and date the insoluble remains. Usually, but not always, the insoluble fraction is older than the soluble fraction since young humic acid often penetrates down into lower levels from higher levels. The question is whether there is a difference between the laboratories in the pretreatment and, if so whether this resulted in different ages. Scott et al (1990) write that humic acid was chosen since a homogeneous sample could easily be obtained because of the solution in an intermediate stage of the preparation.

b) The humic acid yielded scattered values (fig 2) but only 41 results from 21 laboratories were recorded.

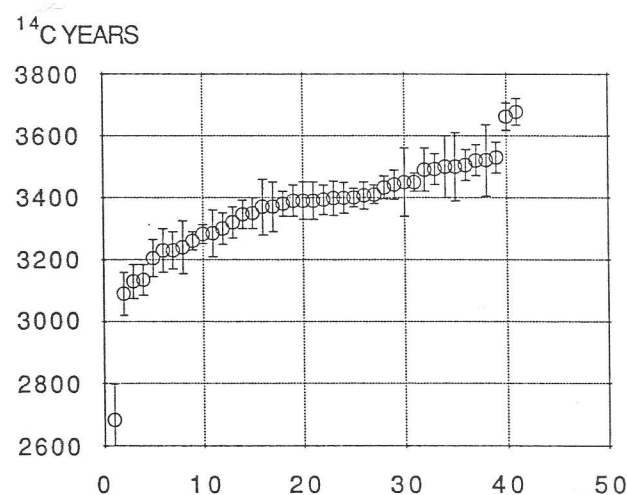


Fig 2. All results for humic acid in stage 2.

c) The mean value of the shells, *Anadara antiquata*, was 637 and the median value 670 years. The youngest mean value for a laboratory was -24, thus 700 years too young, and the oldest 1040, thus about 400 years too old. Because of the young age of the shells any contamination by atmospheric carbon dioxide would hardly be significant in such a short time bet-

ween the dispatch of the samples and the dating. The shells received in Uppsala weighed about 15 g each with a surface of almost 200 mm² so certainly have an age of their own. No information on this was given, nor any about the context. What age distribution could be expected in such a collection of samples? Disregarding the few outliers, deriving from liquid-scintillation counting laboratories, it is difficult to see any systematic difference in quality of the shell dates from that for other samples. The $\delta^{13}\text{C}$ values are not yet available for the participants in stage 3. A private discussion with another participant, however, revealed that this had normalized the result not to $\delta^{13}\text{C} = -25\text{‰}$, but to 0. Thus two results should be changed with about 410 years. This means a small shift of the mean value if only one laboratory has made that mistake.

d) Crushed carbonate must be regarded as a very hazardous material unless the greatest care is taken during the crushing and later in the laboratory. The material should be homogeneous, but if the carbonate is exposed to the atmosphere for some time it could be contaminated; this is more easily detected for old samples than for young. Figures 3 and 4 reproduce all results for the two samples submitted in the first stage. It is seen that the tails of the distribution of the results indicate a larger offset for the younger than the older ages for the old sample. This is also obvious for the young carbonate. The range for the old carbonate was wider than for the young. It is difficult to draw any conclusion whether the scatter is due to the sample itself, to contamination or to the measurement.

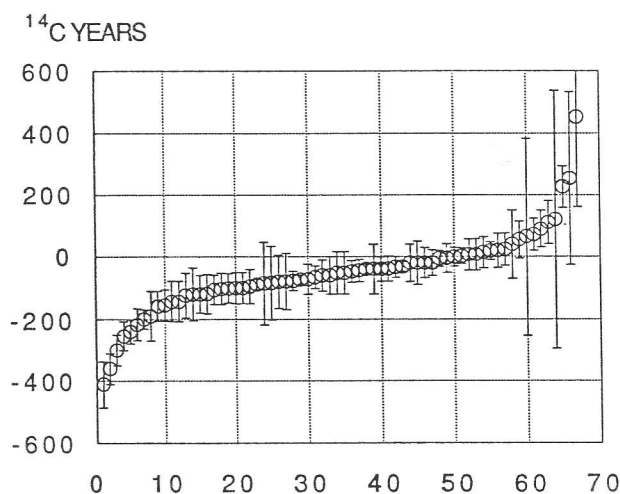


Fig 3. All results for the young carbonate in stage 1.

The conclusions in the report (Scott et al 1990) are:

a) The duplicate samples allowed an evaluation of the internal error multiplier, IEM, for the 38 laboratories which participated in at least two stages one of which should be stage 3. Five gas-counting, and

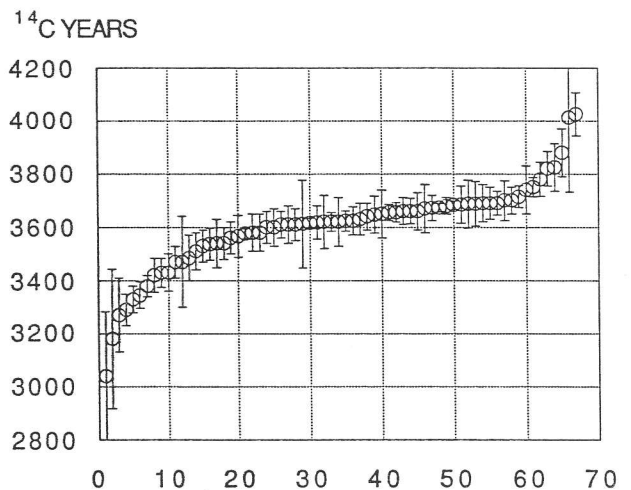


Fig 4. All results for the old carbonate in stage 1.

five liquid-scintillation counting laboratories failed to meet the criterion that the estimated range should cover the value 1.

b) The systematic bias was calculated relative to a baseline defined by all the study results. In some cases the baseline seemed to shift appreciably between the stages. Here six gas-counting laboratories, five liquid-scintillation laboratories and one accelerator laboratory failed.

c) The external error multiplier, EEM, which is dependent on the IEM and the bias was also calculated for each laboratory. The numbers failing were 14, 13 and 3 respectively. Six gas-counting laboratories, no liquid-scintillation counting laboratory and two accelerator laboratories quoted adequate uncertainties.

d) The effect of different pretreatments must be investigated. Since such a study seemed interesting and the pretreatment very often affects the final result the Uppsala laboratory submitted results from an intermediate and an inner fraction of each shell sample; the intention was also to date the extract of the humic acid from the peat although the time did not allow that. It is recalled that the author always tries to obtain an opportunity to date two or more fractions of a shell sample.

e) The variability and its components should be further studied.

f) The influence of ^{13}C should be examined.

The Glasgow workshop

The proceedings and discussions of the workshop will appear in No 3 of *Radiocarbon*, vol 32 1990.

It was apparent that the whole exercise had helped some laboratories to find the weaknesses of their procedures.

The consumers of dates have been very concerned from knowing too little about the quality of the results they have obtained and the sponsors of the datings have also expressed disquiet. Comparisons like that organized from Glasgow could help the laboratories to improve the general standard. It was also suggested that the laboratories which were very successful in this exercise should form a Help Squad to assist with advice when requested and those in need thereof should be encouraged to ask for help.

The completed international comparisons usually suffer from lack of very old or "infinitely old samples". It was determined that IAEA should acquire a set of different types of sample covering the whole age range for radiocarbon dating. The homogeneity is very important and must be carefully tested. These samples should be distributed to interested laboratories for blind tests at first. This was done and the results are due at the end of November 1990. The results should then be carefully scrutinized. Finally the samples should be available in Vienna for the ^{14}C community for frequent tests. If the results are sent to Vienna they will be published, but it should not be compulsory to send any. Since the samples are limited as to the amount subsamples should be available in each laboratory in order to avoid exhausting the supply in Vienna too early.

It was also suggested that Glasgow should continue to arrange for comparisons. Even if the samples are free each laboratory contributes generously by dating them. The two extensive comparisons organized from Glasgow were anonymous at the request of some participants, but others are pleased that the results from the Vienna samples may be published. The comparison of 1850 wood and sucrose about 20 years ago was also anonymous, but the comparison of the two oxalic acids was open.

Austin Long should write a paper on certain routine work in the laboratories such as calculating uncertainties, keeping a logbook on the samples, storing samples and results for a certain time after dating the samples. The first version should be circulated to allow remarks and suggestions for changes. The author wrote several comments but has not seen the final version. Some of the points are given in the following paragraph.

It should be recalled that the principles for calculating uncertainties have been discussed for years. Some laboratories have used only the statistical uncertainties calculated from the registered number

of decays. Others, among them the conventional laboratory in Uppsala, have tried to estimate all uncertainties connected with the physical measurement such as the influence from the small uncertainty in the filling pressure, the choice of voltage, the $\delta^{13}\text{C}$ measurement, and the barometric-pressure dependence of the background. The uncertainty in the value of the half-life, yearly included by many ^{14}C workers, was thus never incorporated in the results released from the conventional ^{14}C laboratory in Uppsala. Since the calibration curves are drawn with the same half-life any uncertainty in the half-life will lack influence on the final value when the age is given in ^{14}C years.

Similarly, the uncertainty should never be arbitrarily increased to cover uncertainties due to the ^{14}C activity in nature. This correction of the uncertainty should not be included until the discussions of the results. This applies not only to the secular variations but also the reservoir effect. Better knowledge at a later occasion then allows a re-evaluation. Similarly the procedures and results of the pretreatment should be declared to allow a discussion of the validity of the results. The international comparisons have demonstrated the need for information about long-term stability, statistical analyses of repeated measurements of background, standards and samples.

At the Glasgow workshop it was decided that the sample documentation should be improved, so that detailed information about physical and chemical treatments, yields etc, should be available and easily traced in every laboratory. The results of the background and standard samples should be available with the sigma values to illustrate the system's reliability. The standard samples should be measured frequently. Age determinations should not be released until a fair picture of the reliability could be ascertained from graphs and statistical analyses.

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