SIXTH IAEA INTERCOMPARISON OF LOW-LEVEL TRITIUM MEASUREMENTS IN WATER (TRIC2000)

REPORT

prepared by

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Vienna, October 2001

1. INTRODUCTION

It is now 36 years since the isotope intercalibration programmes of the International Atomic Energy Agency (IAEA) were initiated. Regular exercises are conducted for stable isotopes of light elements and for tritium, the radioactive isotope of hydrogen. The Agency's environmental isotope intercalibration programme is now much larger than originally conceived, being a component of the Analytical Quality Control Services (AQCS). The purpose of AQCS is to assist laboratories engaged in various fields of research to check and maintain measurement quality.

Five previous tritium intercomparisons were conducted [1-6](Cameron, 1967; Florkowski, Payne and Sauzay, 1970; Taylor, 1978; Hut, 1986a, 1986b: Östlund, Scott and Taylor, 1995). Basic details of these and of the recent one are summarized below:

Table 1: Details of conducted IAEA tritium interlaboratory comparison exercises.

Year	Number of participants, (approached)	Water samples distributed and tritium concentration [TU]
1965	12 (29)	Sample A: < 100 TU Sample B: 200 - 800 TU Sample C: 1000 – 6000 TU
1970	35 (38)	A: ~ 10 TU B: ~ 50 TU C: ~ 250 TU
1975	41 (79)	A: < 0.2 TU B: 8.37 ± 0.04 TU C: 33.1 ± 0.1 TU D: 678.5 ± 2.2 TU
1985	57 (85)	A: < 0.2 TU B: 5.94 ± 0.03 TU C: 25.66 ± 0.13 TU D: 100.2 ± 0.5 TU
1994	57 (91)	A: ~ 0 TU B: ~ 1 TU C: ~ 5 TU D: ~ 19 TU
2000	86 (102)	T3: 0 TU T2: 2.152 TU T4: 5.252 TU T1: 10.112 TU T5: 26.118 TU T6: 505.83 TU

The quoted precise values for 1975, 1985 and 2000 refer to the chosen reference dates for these intercomparisons. Bracketed 2nd column no. are the numbers of laboratories approached.

In each case, a set of water samples was distributed, covering the concentration range then prevailing in environmental waters. In 1965, 1970 and 1995, the tritium values were known only approximately. In 1975 and 1985, samples were prepared by gravimetric dilution of tritiated standard water NBS4926 (now referred to as SRM4926) with water of near-zero tritium concentration. In the present exercise of 2000, samples were prepared by gravimetric dilution of tritated standard water SRM4927F with virtually tritium-free water.

Laboratories were advised of the approximate concentrations in advance, and were requested to provide detailed information on techniques and procedures. Results of the first two exercises were reported with identification of the laboratories, but anonymity has applied since 1975.

Participation in IAEA intercomparison exercises is voluntary. However, leading laboratories of recognized experience and quality are usually requested to participate, and some may also be involved in the data evaluation. Results are often discussed at meetings of invited experts, from which decisions and recommendations are passed to the scientific community.

Most laboratories applying environmental isotopes in geochemistry, hydrology, oceanography, agriculture, plant physiology, life sciences and other environmental studies have participated in the Agency's intercomparison programmes. These are valuable opportunities to check measurements against those of other laboratories, to discover the occurrence of errors and systematic deviations, to stimulate improvements, and to promote confidence in data from international research projects encompassing the efforts of several laboratories.

Table 1 reveals a continuing increase in the number of laboratories measuring tritium at environmental levels. A further increase of participants occurred in this 6th intercomparison (code-named TRIC2000). The increase has occurred despite the decline of tritium concentrations in rainfall to very low levels, and the elapse of 3 half-lives since maximum levels were recorded in the mid-1960s following atmospheric H-bomb tests. The present low tritium environment has persisted now for about 15 years, and levels in the ocean and groundwater will continue to decline. Levels in the southern hemisphere are only marginally above pre-bomb levels. These markedly lower concentrations impose much greater technical difficulty in measurement. It is therefore important to judge from the present exercise how well laboratories have improved their sensitivity and accuracy.

In 1982, IAEA recommended a fixed tritium scale based on reference standard water NBS4926C (now SRM4926C, US National Institute of Standards and Technology, (NIST)) and half-life 4540 days, (12.43 years) [7]. With SRM4926C now entirely used up, a new set of NIST tritiated standard waters has been prepared and issued, and massic activity ratios have been measured for all their previous and present standards [8,9]. It is very opportune that NIST has simultaneously conducted a comprehensive review and critical evaluation of the tritium half-life [10]. This has demonstrated that the previously adopted half-life is too long, and evaluated a new value of 4500 ± 8 days. The coincidence of these two projects allows all the scale parameters to be readjusted simultaneously. This is believed to be the best possible approach to end the continuing diversity of standardization applied in different laboratories. A recommendation to this effect is being prepared for publication. Some details are necessary for the present report; these are considered in Appendix B.

102 laboratories declared their willingness to participate in this 6th interlaboratory comparison and have received the set of six water samples. Results on their tritium concentration have

been received in 89 independent data sets from 86 laboratories at the time of writing this report. Six samples of different tritium concentrations were prepared at the Agency's laboratory. One was a virtually tritium-free water, collected from the same deep artesian aquifer at Grafendorf (near Graz, Austria), which was used in earlier IAEA tritium interlaboratory comparisons. The other samples were prepared by accurate gravimetric dilution of the new NIST tritiated water standard SRM4927F, as described below.

2. SAMPLE PREPARATION

As was the case in the earlier intercomparison exercises, the submitted results indicate that various tritiated standard waters and half-lives are still adopted by the laboratories to calibrate their measurements, despite the 1982 recommendation to adopt a fixed tritium scale. In light of the new robust tritium half-life assessment [10], and the associated re-evaluation of all NIST tritiated water standards [9], we have decided on this occasion to calculate the prepared values and their uncertainties based on the newly determined half-life 4500 ± 8 days and the NIST tritiated water standard SRM4927F. To achieve this, we have incorporated four uncertainty sources: (a) the calibration uncertainty attached to the certified NIST standard tritiated water SRM4927F (calibration date 3 Sep 1998) used for sample preparations; at a stated standard uncertainty of 0.36%, this is the dominant contributor; (b) the small uncertainty in correcting the values to our reference date (1 July 2000) using the half-life 4500 ± 8 days; (c) the smaller errors of our dilution procedures; (d) the calculated uncertainties have been broadened by including an upper limit for a possible tritium concentration for sample T3, which was used in preparing the other five samples. As detailed below, this upper limit resulted from statistical analysis of the submitted results for that sample.

Throughout this report, the results on the sample tritium concentrations are expressed in terms of their massic tritium activity [Bq/kg] and as their tritium/hydrogen isotope ratio in terms of TU (tritium unit). Strictly speaking, this is not a "unit" but a ratio and may be referred to more rigorously as tritium ratio. The tritium ratio of 1 TU corresponds to a tritium/hydrogen ratio of $T/H=10^{-18}$. The massic tritium activity is related to the tritium ratio via the following relationship:

1 Bq/kg corresponds to 8.390 ± 0.015 TU [11], using the new value for the tritium half-life [10] and the CODATA 1998 value for the Avogadro constant [12].

Adoption of half-life 4500 days means that results from laboratories using other half-lives for their calibrations will be biased relative to the "true" tritium values and ranges of the samples as calculated for this exercise. For example, those laboratories calibrating their measurements by the former NIST water standard SRM4926C (calibration date 9 Sep 1982) and 4540 day (12.43 year) half-life, should bear in mind that their results will be positively biased by 1.55% relative to the prepared values we provide here, based on SRM4927F (calibration date 3 Sep 1998). Some results may therefore appear to be marginally high outliers, but would be within the correct range if adjusted to the new half-life. Similarly, some results, which are within the ranges given here, may actually be marginal outliers on the negative side. We recommend that laboratories make their own final assessments of the quality of their results. We have added Appendix B, which provides help to make the adjustments according to published procedures [7]. A different approach for this adjustment is suggested in [13].

2.1 Massic Activity of SRM4927F at Reference Date

NIST's calibration certificate gives the massic activity of SRM4927F as 634.7 kBq.g^{-1} on 3 September 1998, with a combined standard uncertainty of 0.36%; this uncertainty is the square root of the sum of squares of all error components in the calibration. We have applied the tritium half-life value 4500 ± 8 days evaluated by NIST [10] to calculate the massic activity at our intercomparison reference date (1 July 2000). The standard uncertainty of the half-life introduces an additional error component, which increases with time elapsed since 3 September 1998. Expressed as a percentage uncertainty, to be added in quadrature with the combined standard uncertainty (0.36%), this error is $2.378\times10^{-5}\times t$, where t is the elapsed time in days; this is only 0.016% to our reference date 1 July 2000 (667 days). The massic activity of SRM 4927F at that date is 572.7 ± 2.1 kBq.g^{-1}.

The website http://srmccatalog.nist.gov/srmcatalog/certificates/srm-4927f.htm provides full details of the SRM4927F calibration.

2.2 Sample Dilution Procedure

SRM4927F was diluted in 3 stages to obtain a daughter water of appropriate tritium activity to serve for common laboratory purposes (see Table 4 in Appendix A for details). The diluting water was a de-ionised sample from an artesian well in Grafendorf, Austria, which was used as "tritium-free" water in earlier interlaboratory comparisons and is used in the IAEA Isotope Hydrology Laboratory for the daily routine work. The third dilution of SRM48927F served as a common mother water for a final stage of dilution to obtain samples T1, T2, T4, T5, T6 (see Table 5 in Appendix A). Sample T3 taken in June 2000 from the same artesian well in Grafendorf as mentioned above was used as the diluting water for this final stage. Full details of the dilution procedures are given in Appendix A. All dilutions were performed using calibrated balances, checking the zero reading and a test weight before and after each weighing process. The process consisted of the weighing of (a) the empty containers, (b) the filled containers and, in case of the tritium standard transfer, (c) the empty transfer container (syringe). The combined uncertainty of the weighing procedure was calculated according to the law of uncertainty propagation, taking into account balance precision, maximum drift and display uncertainty. No buoyancy correction was applied.

2.3 Tritium Concentration of Diluting Water (T3)

Sample T3 was used as diluting water to prepare the five other samples. This water was collected in 150 litre stainless steel barrels and was not de-ionised. Its tritium concentration was assessed using results submitted by the participating laboratories. Only the 35 results with stated uncertainties less than 0.25 TU were considered in the evaluation as "high precision" laboratories. Obvious outliers were removed before evaluating a first arithmetic mean value with its standard deviation. A range of acceptability to judge individual results was based on the mean value ± twice its standard deviation. Results differing by more than twice the stated laboratory measurement uncertainty from those upper or lower limits were discarded, and a second mean value evaluated. The process was repeated until further repetition produced no more changes to the outlier population. The 26 remaining accepted results provided a mean value of 0.028 ± 0.022 TU (standard error of the mean) (see Table 2). This value is indistinguishable from zero, which is certainly its value at the source. Therefore it was decided to set the tritium ratio for T3 to 0 TU with an expanded upper uncertainty limit. Under this condition its upper limit is $2\times0.022 + 0.028 = 0.072$ TU at the two-σ level. This value for the tritium ratio of the diluting water T3 has been applied in calculating the tritium ratios of the other five samples, whose stated ranges thereby include all components of uncertainty at the two-σ level.

The same evaluation procedure for the tritium ratio of sample T3 can be used to compare results based on the three individual measurement methods, liquid scintillation counting (LSC), gas proportional counting (GAS) and ^{3}He mass spectrometry (3HE) for the same sub-set of laboratories. In addition, in the third column of Table 2 similar results are calculated including all participating laboratories.

Table 2: Evaluated mean tritium ratios with standard errors of the mean for sample T3, obtained from the sub-set of "high precision" laboratories (left column) and by all laboratories (right column). In brackets, the number of accepted/reported results is given. Note: For the further evaluation a value of 0 TU ± 0.072 TU at 2-σ level is used (see text).

	Tritium ratio and standard error of the mean for "high precision" laboratories [TU]	Tritium ratio and standard error of the mean for all participating laboratories [TU]
All data	0.028 ± 0.022 (26/35)	-0.004 ± 0.093 (63/80)
LSC	0.006 ± 0.029 (16/26)	0.067 ± 0.120 (52/66)
GAS	0.063 ± 0.074 (3/3)	-0.033 ± 0.086 (7/7)
3HE	0.020 ± 0.014 (5/6)	0.020 ± 0.014 (5/6)

The results of all data sets in Table 2 are consistent with each other within their evaluated standard errors of the mean. All these mean values are indistinguishable from zero (tritium free water) at the 2-σ level.

2.4 Reference Values for the Prepared Samples T1 to T6

The reference tritium ratios and combined standard uncertainties for the prepared six samples T1 to T6 can now be calculated from the combination of all dilution factors for the original standard solution SRM4927F. The uncertainties are not symmetrical around the reference values, but larger at the upper side. By considering all possible sources of uncertainty we have ensured that the true tritium ratios lie with 95% probability within the calculated upper and lower uncertainty limits.

The calculated reference values and their ranges are given in Table 3. They are believed to be the best possible approximation of the true tritium values and are consequently used in the further data evaluation, in the graphical display and for the determination of outliers in the data sets.

Table 3: Reference values and uncertainty ranges for the tritium ratio of samples T1 to T6 (see also Table 4 in Appendix A).

Sample Name	**Reference Value [TU]**	Lower Uncertainty Limit (at 2-σ level) [TU]	Upper Uncertainty Limit (at 2-σ level) [TU]
T1	**10.112**	10.038	10.258
T2	**2.152**	2.136	2.240
T3	**0**	0	0.072
T4	**5.252**	5.214	5.362
T5	**26.118**	25.928	26.380
T6	**505.83**	502.15	509.58

All data evaluation and especially the outlier determination was performed with the PASCAL-based software ICE (InterComparisonEvaluation, version 4.5), specially developed at IAEA for the purpose of providing a standardized evaluation tool for the interlaboratory comparisons and proficiency tests performed in the field of stable isotopes and tritium. The program was tested and results were verified by simulated data sets and comparison with the output of the same algorithms used with commercial spreadsheet programs. The main advantage was found to be the easy application of outlier detection procedures and robustness of ICE for different data sets.

3. PRESENTATION OF RESULTS

The submitted results are listed in Table 6 in Appendix C, with columns listing laboratory identification number, reported tritium ratio and reported measurement uncertainty in TU for the six samples, the used measurement method, and remarks on any necessary data modification. Results are ordered according to laboratory code number. Used measurement methods include Liquid Scintillation Counting, Gas Proportional Counting and ^{3}He Mass Spectrometry. Empty cells indicate data not submitted. Some results could not be assessed due to failure to submit measurement uncertainties. Laboratories which submitted 2 sets of results by applying different methods have suffixes a and b attached after their lab code numbers (except for 93a and 93b, where two laboratories measured the same set of samples).

Additionally, results for each sample are depicted graphically, in increasing order of tritium ratio, using S-plots (Figs.1-6); these show the relation to the reference value and its uncertainty range, represented by the lower and upper uncertainty limits from Table 3. Error bars are added at 2 times the submitted uncertainties, enabling easy visual identification of outliers (open markers instead of filled ones). The laboratory code numbers (Lab ID) appear along the x-axes. In all figures, the suffix “-D” added to Lab ID’s signalises direct counting without prior tritium enrichment for samples T1 to T5.

All the S-plots are asymmetric around the reference values and their uncertainty limits, with more results on the high side. A part of this asymmetry is likely to be due to the use of older standard waters and higher half-lives (e.g. 12.43 years = 4540 days) by many laboratories for their calibrations, whereas the reference values for the prepared samples are based on the new SRM4927F standard water and half-life 4500 ± 8 days (see section 2). However, this cannot completely account for the overall biases of the population means from the reference tritium ratios for the five tritium bearing samples. Those biases decrease with increasing tritium ratio from +20.3% (T2), +7.6% (T4), +3.3% (T1) and +2.1% (T5) to +0.9% (T6). The underlying reasons for the systematic biases towards higher tritium values cannot be interpreted conclusively from the available information.

Because of the asymmetry of the S-plots and the above-mentioned underlying reasons, average results for the samples calculated from the whole population of laboratories are of limited usefulness, and are therefore not reported here.

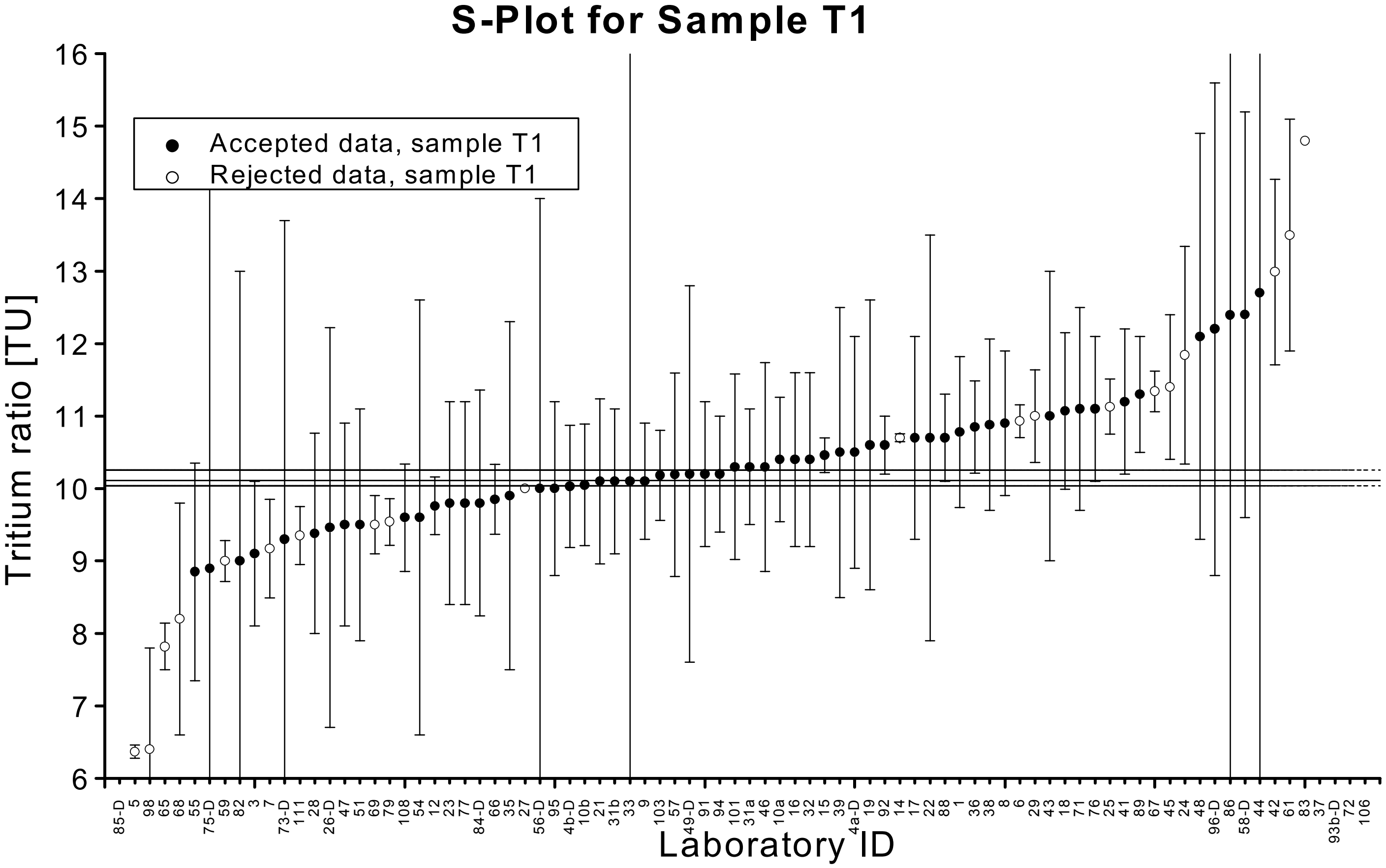

Fig.1: S-plot for sample T1. Laboratory results at the utmost left and right sides are outside the chosen scale. Rejected values are marked with open symbols.

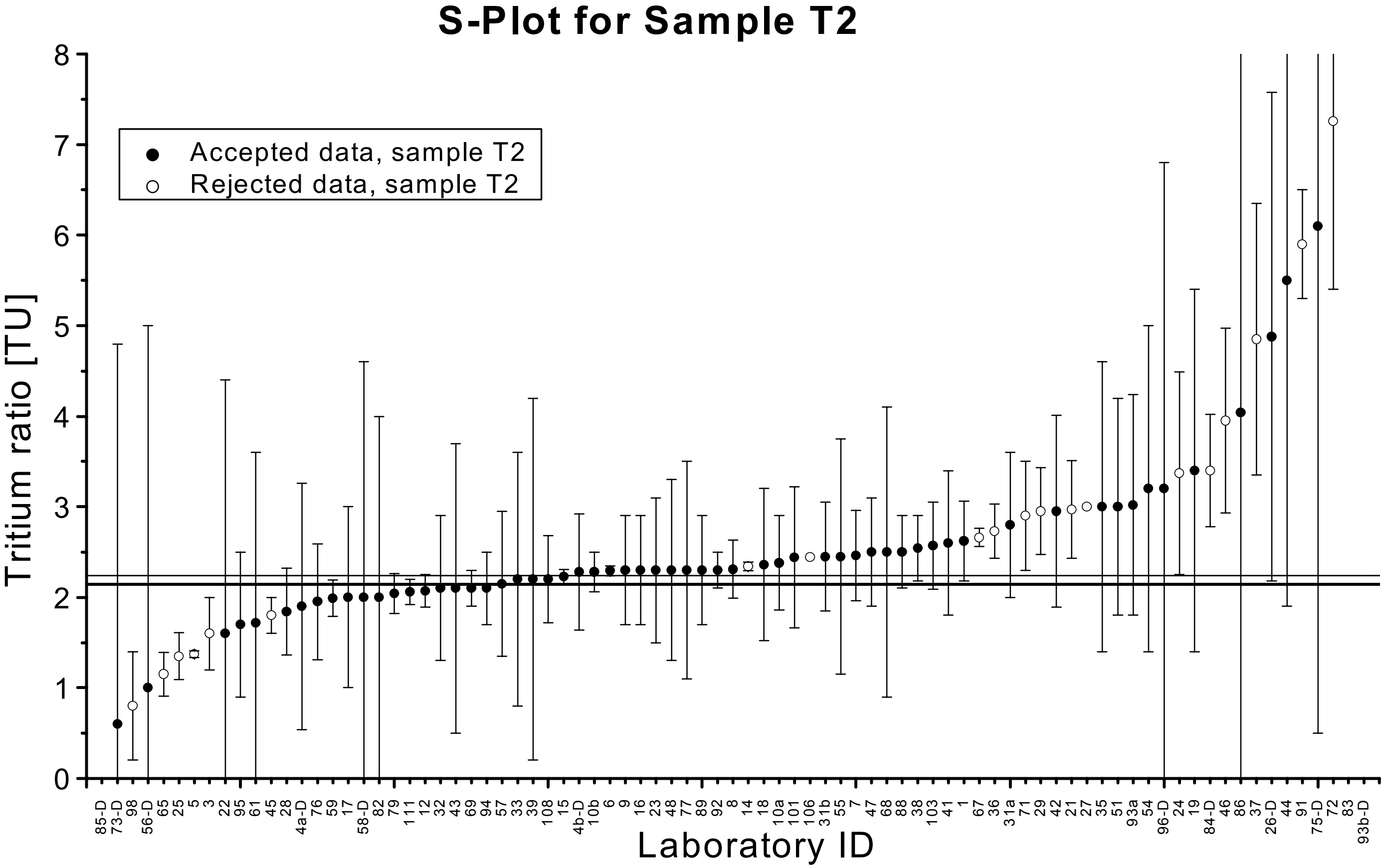

Fig.2: S-plot for sample T2. Laboratory results at the utmost left and right sides are outside the chosen scale. Rejected values are marked with open symbols.

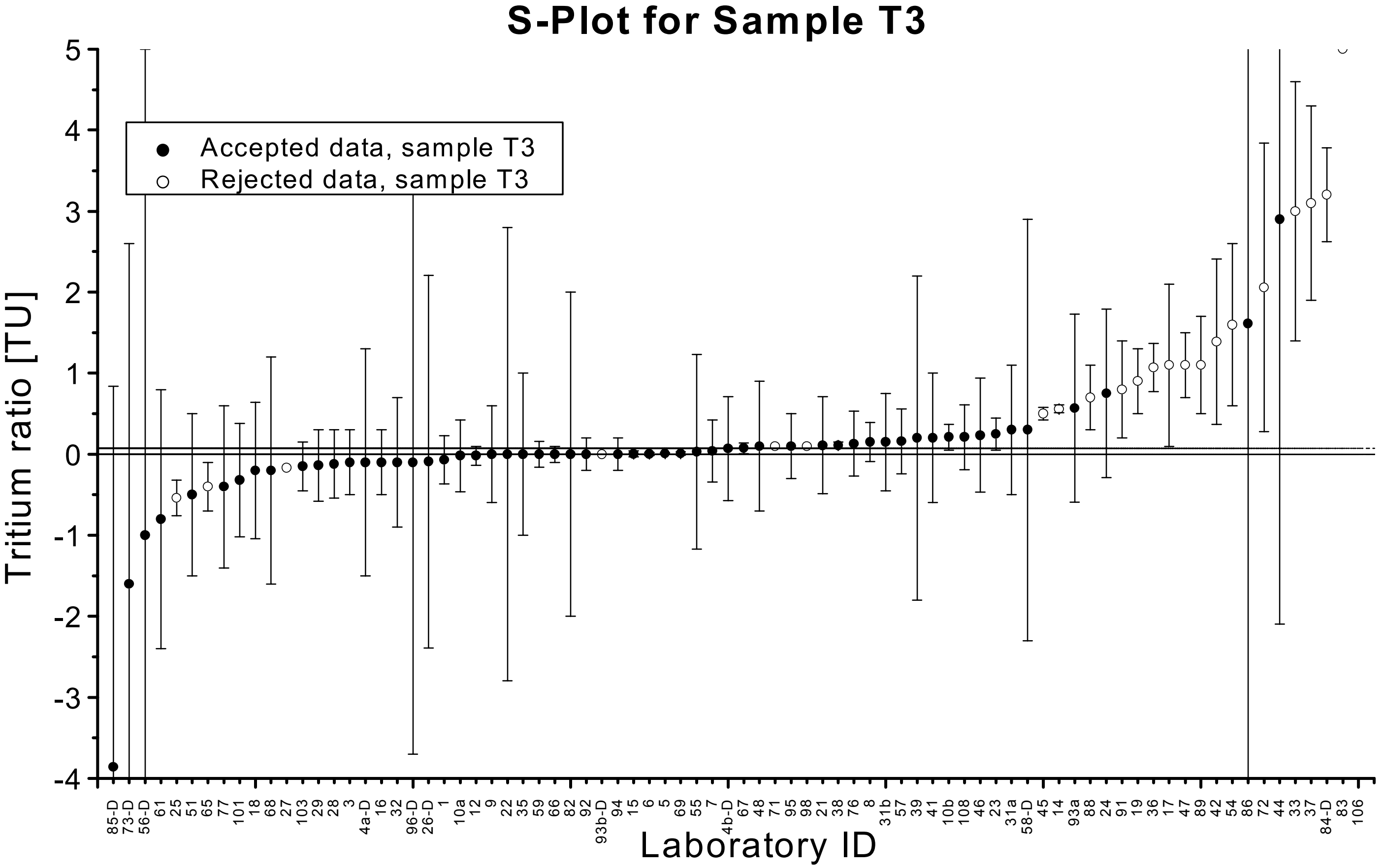

Fig.3: S-plot for sample T3. Laboratory results at the utmost left and right sides are outside the chosen scale. Rejected values are marked with open symbols.

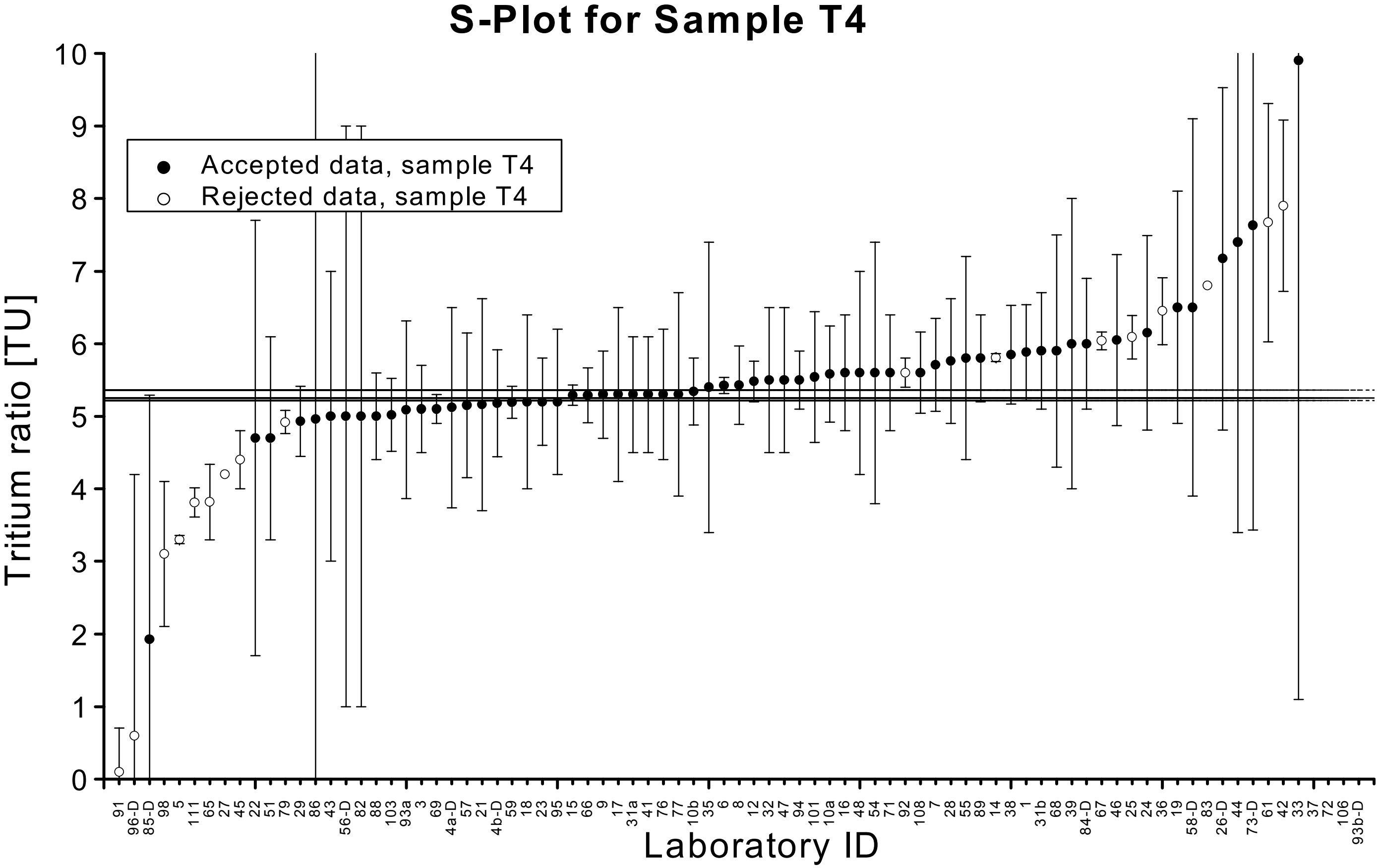

Fig.4: S-plot for sample T4. Laboratory results at the utmost left and right sides are outside the chosen scale. Rejected values are marked with open symbols.

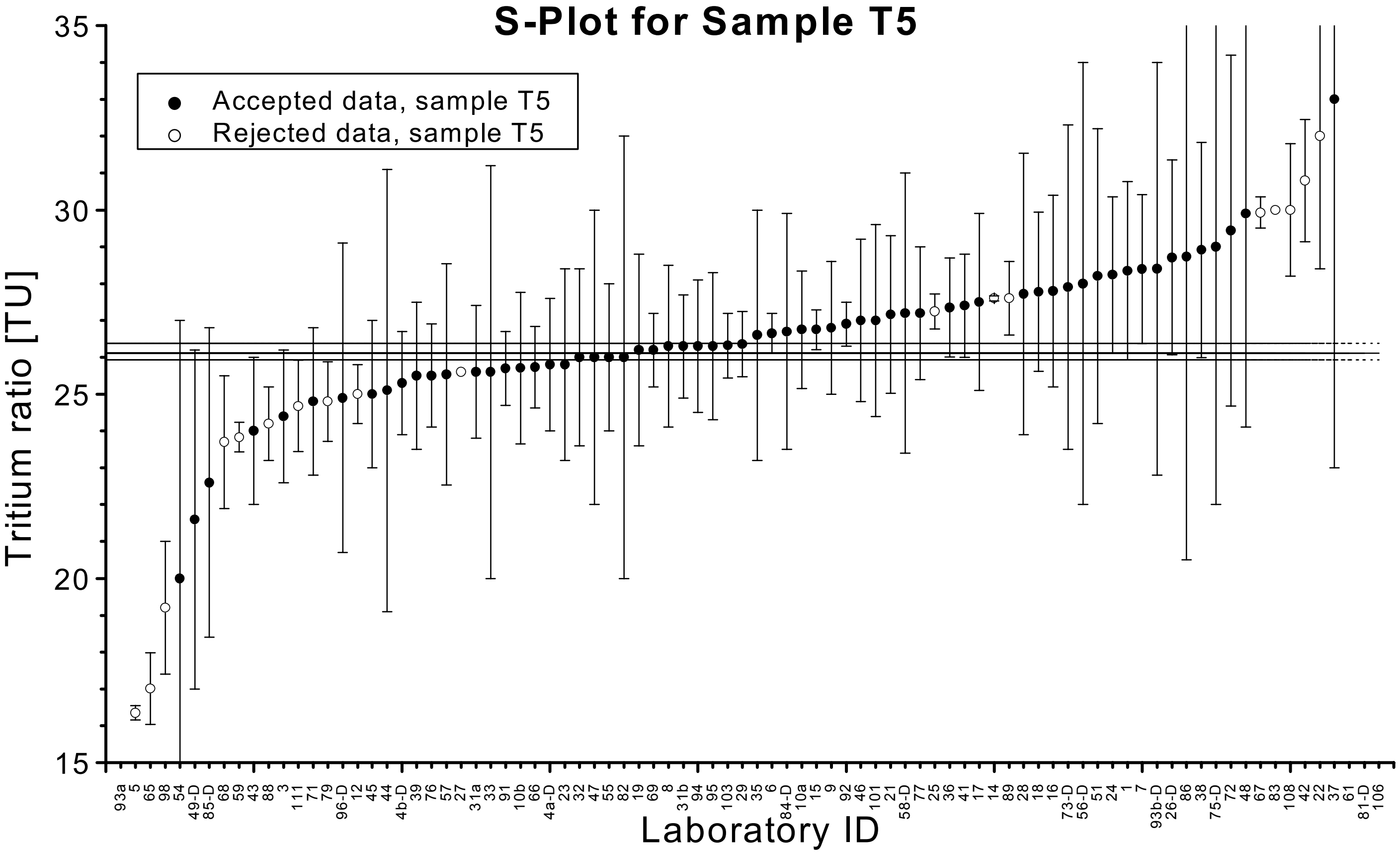

Fig.5: S-plot for sample T5. Laboratory results at the utmost left and right sides are outside the chosen scale. Rejected values are marked with open symbols.

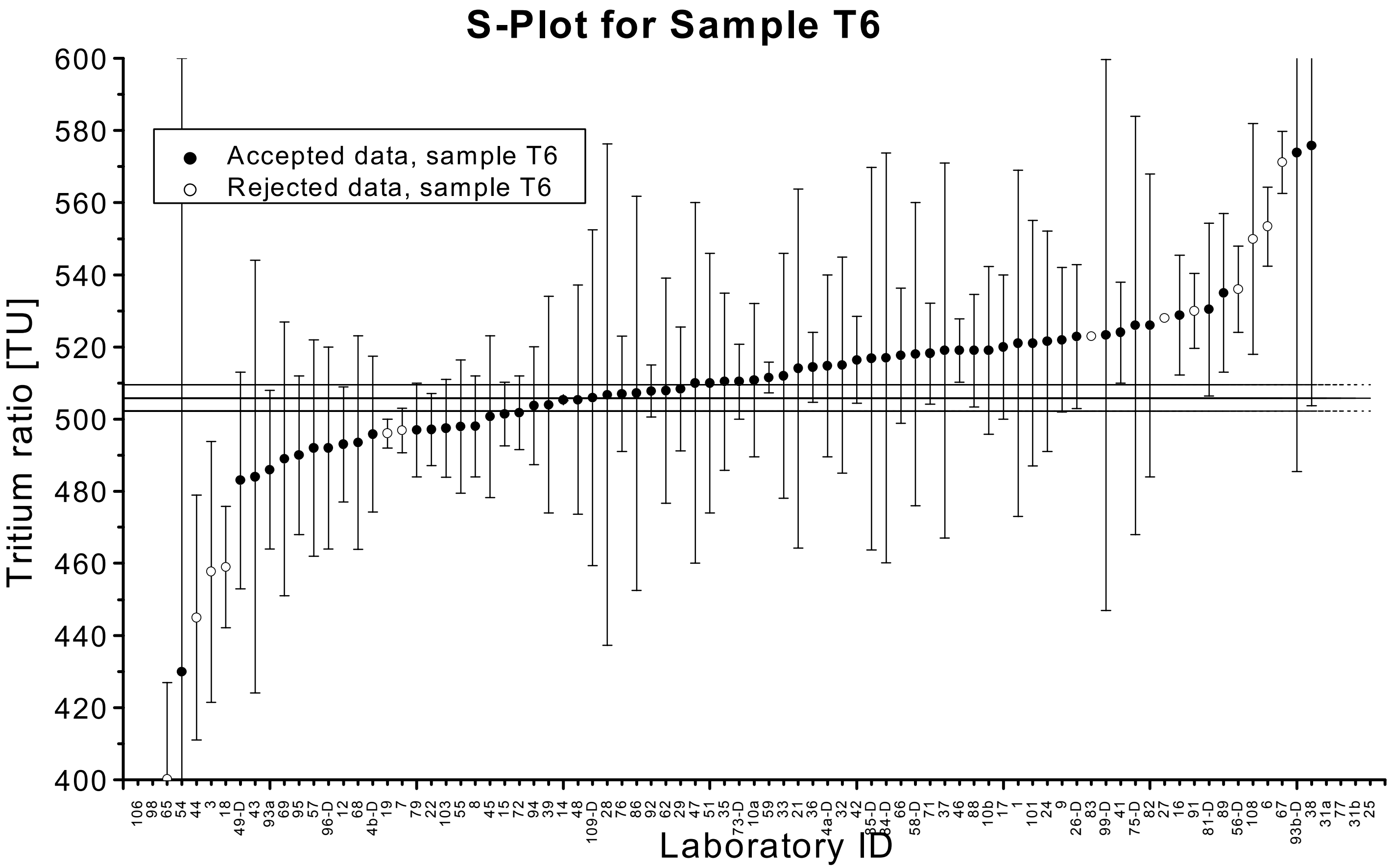

Fig.6: S-plot for sample T6. Laboratory results at the utmost left and right sides are outside the chosen scale. Rejected values are marked with open symbols.

3.1 Implications of Results for Sample T3

Due to the very low range of environmental tritium concentrations now prevailing, sample T3 has special importance in this exercise. Results for the equivalent sample in the 5th intercomparison revealed that slight contamination was prevalent at several laboratories. The overall mean average of the results, after removal of outliers, was slightly higher by about 0.1 TU, suggesting a small degree of contamination on average; 7 out of 51 results (14%) were obvious outliers.

In a similar assessment for T3 in the present exercise, 80 laboratories submitted values, but 5 could not be used in the evaluation, due to absence of the requested measurement uncertainty. Of the remaining 75 results, 15 (20%) were outliers on the high side. 2 negative results were obviously incorrect, being negative even if the reported measurement errors were taken into account. The results for sample T3 for all accepted laboratories yielded a mean value of zero (-0.004 ± 0.093 TU), see Table 2. However, this mean value is biased by one laboratory (no.85) reporting a quite negative value and shifting the mean value considerably towards zero.

In nearly all cases, high outliers must be due to contamination of the sample during storage and/or processing. 14 of the 15 high outlier laboratories applied sample pre-distillation and electrolytic enrichment before counting. Handling in a contaminated atmosphere is the most likely cause of such results. To achieve satisfactory results at such low levels, the tritium concentrations of atmospheric vapour in processing laboratories or sample storage should not noticeably exceed that of outside uncontaminated air. At all laboratories, exposure of the samples to atmosphere during storage and processing should be rigidly kept to the absolute minimum. However, some laboratories are known to be unsuitably sited in contaminated areas; this now severely limits their effectiveness for hydrological research. A very common cause of laboratory contamination is the insidious influence of tritiated vapour escaping from luminous watches. To avoid this contamination, it is essential to exclude the use of such watches by laboratory personnel, and to prevent emitted vapour from entering the laboratory from other areas of buildings, particularly via air-conditioning. Policing of entry, and regular and thorough monitoring of vapour samples within and outside laboratories is the only way to combat this problem. Ideally, a tritium laboratory should be housed in its own building, allowing better control. Prolonged contamination within a building takes a long period to decay, as vapour is absorbed by many materials, and only very slowly released after the sources are identified and removed.

Other possible causes of high results for near-zero samples include contamination of electrolytes (usually hygroscopic Na_2O_2 or NaOH), sample storage in insecure bottles which allow evaporation, or memory effects in enrichment cells and gas preparation lines.

A conforming result for sample T3 is not necessarily evidence of lack of contamination if the measurement uncertainty is high. This may not be of too much concern to some laboratories, but regular measurements of a tritium-free water as control sample would soon show up any problem.

A likely cause of negative outliers (counting methods) is the use of tritium-free background water which has become contaminated.

3.2 Results for Sample T6

This sample can be measured accurately by direct gas or liquid scintillation counting, and was presumably treated this way by the participants. Although the spread of the submitted results can be slightly greater due to the various used calibrating standard waters and half-lifes, it should have been easily possible to achieve conformity with the prepared value. However, as in previous intercomparisons, a disappointingly high number of laboratories submitted results which were gross outliers. 82 laboratories provided results with errors attached, with 16 (20%) identified as outliers. 14 of these applied liquid scintillation counting, and 2 used the ^{3}He method. 5 results were on the low side (range 341 – 459 TU) and 9 were too high (530 – 631 TU). 2 further outliers are close to the reference value, but indicate an overestimation of measurement precision by the laboratories concerned.

Several calibrating “standard” waters were reported for these 16 outliers, including NIST standards in 6 cases. High results can be due to storage of standard waters over time in insecure bottles (shift to lower concentrations due to net evaporation into an atmosphere of lower concentration). Low results can be due to a variety of handling or calculation errors, or perhaps reliance on standard waters obtained at second or third hand without performing adequate cross-calibration checks.

3.3 Comparison of Reported Uncertainties

To enable laboratories to judge how their reported precision compares with that of other participating laboratories, we have compiled two distributions of the reported uncertainties, one for samples T1 to T5 (Fig.7) and one for sample T6 (Fig.8).

Several parameters, which are highly variable among laboratories, have to be considered in judging these two diagrams:

(a) it is by no means certain that all the laboratories have correctly assessed their measurement uncertainties by combining all contributing components according to the Guide on the Uncertainty of Measurements (GUM) [14]; although several reported values were considered much too low to be acceptable (laboratory 14 at the sensitive end of all the diagrams is such a case), all have been included in the diagrams;

(b) longer counting times (accumulation times for ^{3}He), lower and more stable background count rates and better counting sensitivity significantly improve the uncertainty;

(c) several laboratories counted directly the samples without prior tritium enrichment.

The laboratories which counted without prior enrichment for samples T1 – T5, have been identified in the figures by “-D” attached to the laboratory ID number; this has not been done for T6, because the sample was presumably counted directly by nearly all participants. In Fig. 7, the cumulative reported uncertainties for five samples are stated. In cases where less than five samples were measured, the number of samples is indicated in brackets at the laboratory ID number. As could be expected, most of the direct counting laboratories are near the upper end of the uncertainty distributions. However, they are grouped among laboratories using enrichment before counting. This may give cause for thought to the latter, which are applying much more effort and expense for no obvious gain in precision.

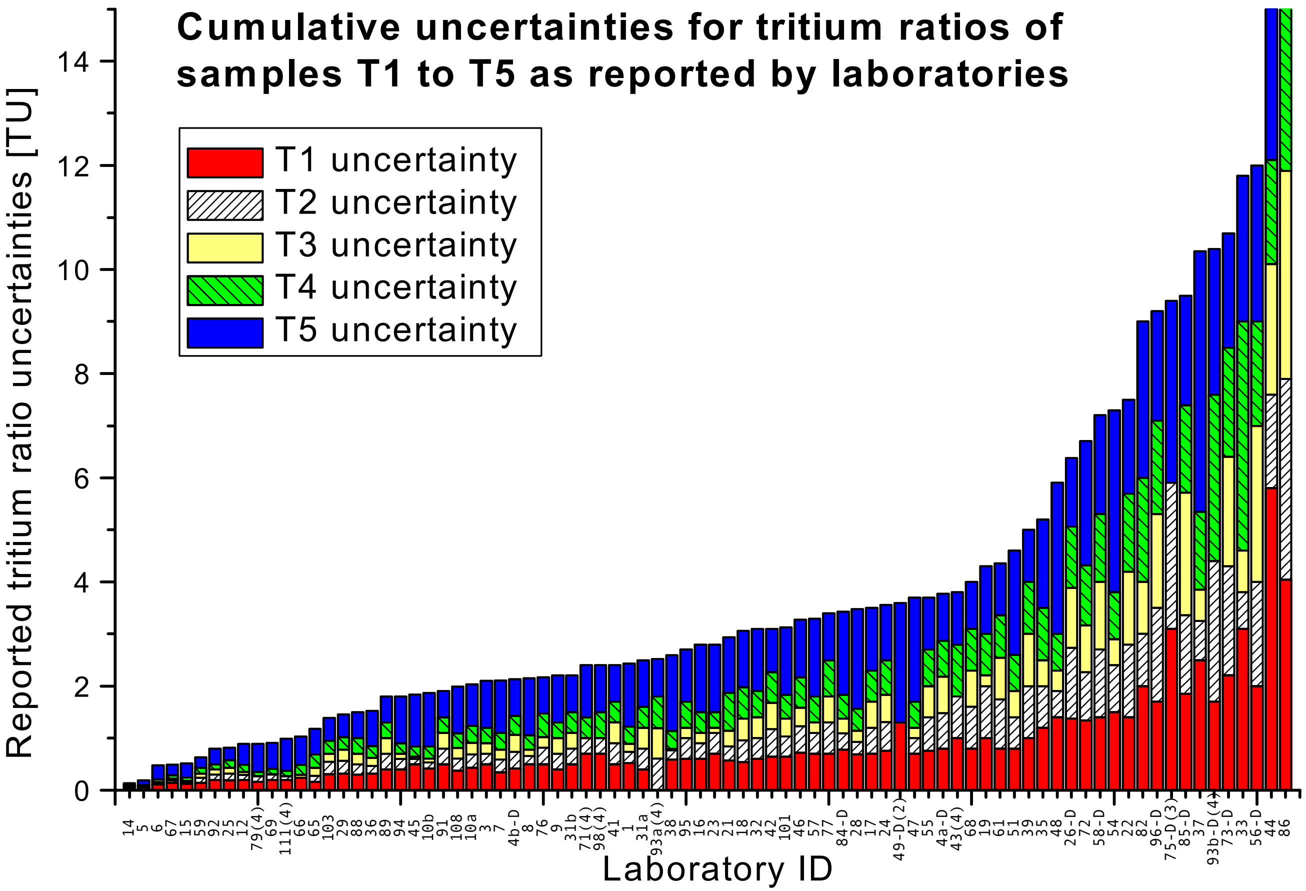

Fig.7: Cumulative uncertainties for samples T1 to T5 as reported by laboratories in increasing order. Laboratories using direct measurements without enrichment are marked with the suffix “-D” at the Lab ID. For laboratories with less than 5 analysed samples (biasing the ranking), the respective number is added in brackets to the Lab ID.

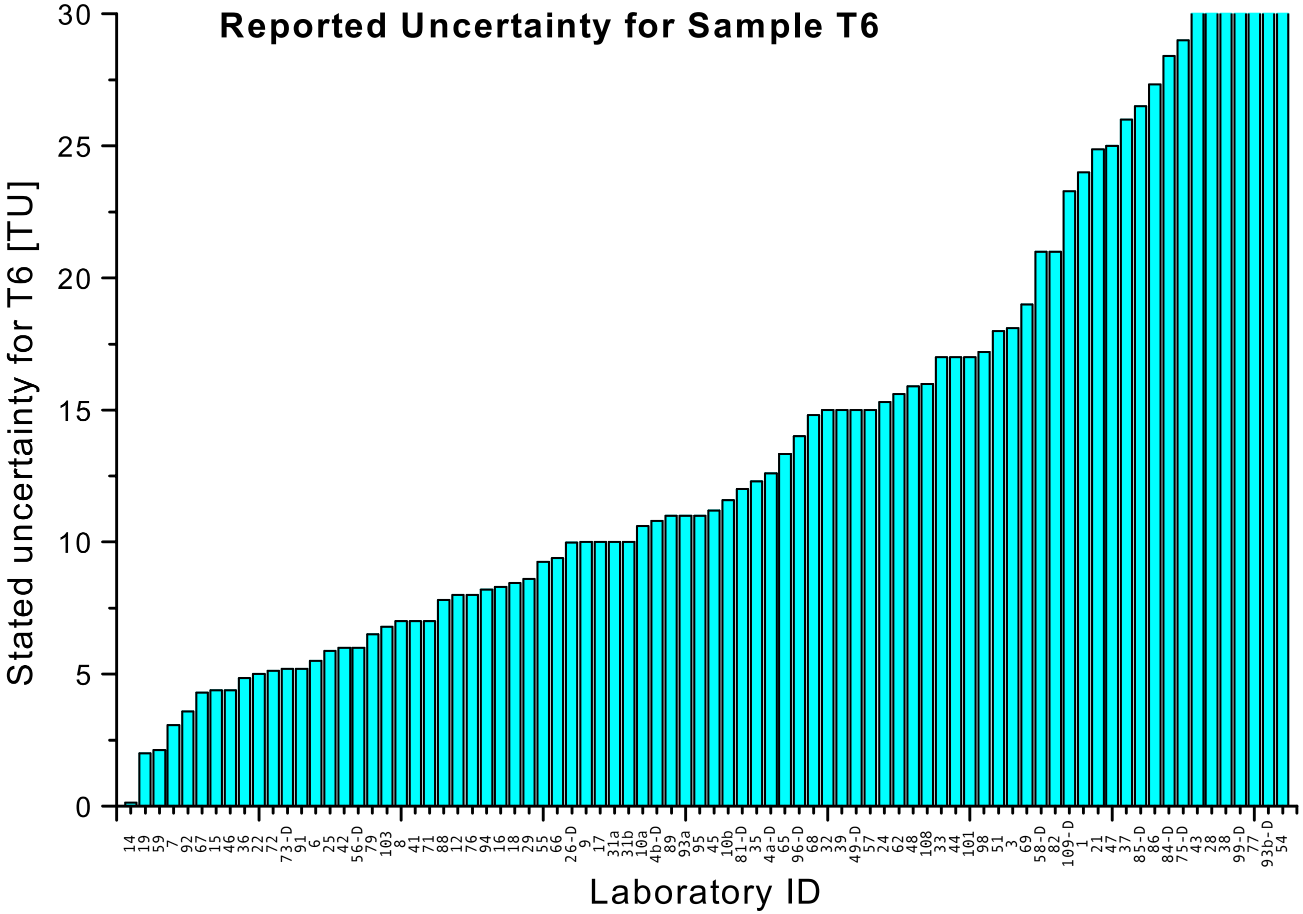

Fig.8: Reported uncertainty for sample T6 as stated by laboratories.

3.4 Laboratory Performance Indicators – Accuracy and Precision

An attempt was made to provide a rough estimate of laboratory performance by judging their results versus the well-determined reference values of the samples as prepared by the IAEA.

3.3.1 Accuracy:

The most obvious performance ranking can be performed by calculating the deviation of the tritium ratio reported by the laboratory from the reference value in TU. The sum of deviations for samples T1 to T5 for each laboratory is shown in Fig. 9 (sorted according to absolute sum of deviations). This provides a good estimate for the accuracy of values reported for an individual laboratory within the span of covered tritium ratios in TRIC2000.

Several features can be derived from Fig. 9. The sum of deviations from the reference value ranges from 0.5 TU (lab 94) to 64 TU (lab 93b), not considering lab 106 with a deviation of more than 300 TU. The majority of laboratories show distinct deviations and bias in positive or in negative direction for all samples. A balanced distribution of deviations around the reference values (zero in the plots), – as to be expected for random errors following a normal distribution – occurs only for a minority of about 30 laboratories. This indicates problems with the calibration of measurements. Obviously, the ranking of laboratories in Fig. 9 is only correct for laboratories having measured all five samples T1 to T5. Those labs with less than five results are consequently marked at the Lab ID with the respective number of analysed samples indicated in brackets.

Due to the different activity level of sample T6 and the commonly used direct measurement approach without enrichment and with much higher uncertainties, the deviations from the reference tritium ratio for sample T6 are presented separately in Fig.10.

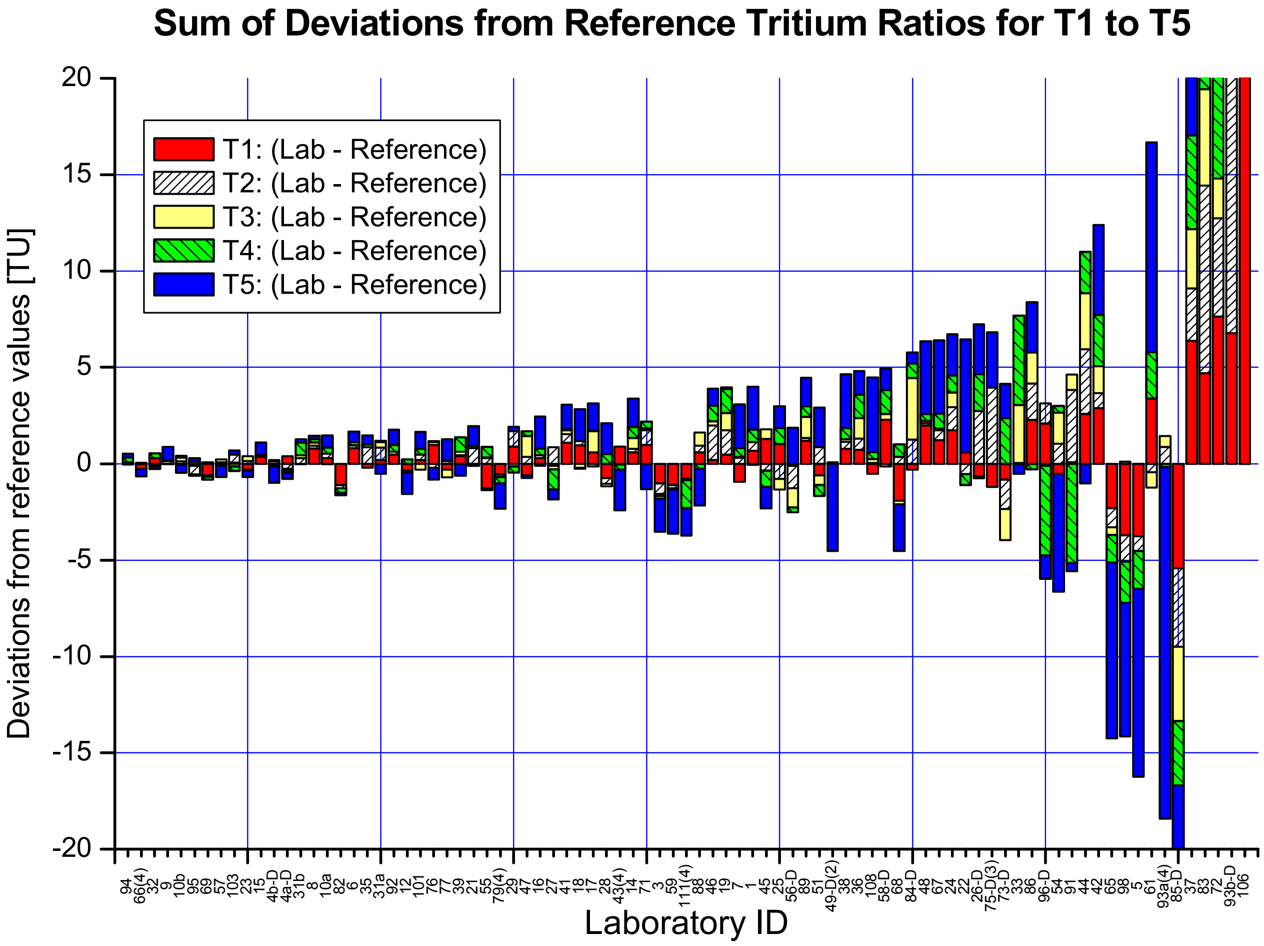

Fig.9: Sum of deviations from the reference tritium ratios for samples T1 to T5 in increasing absolute order in TU.

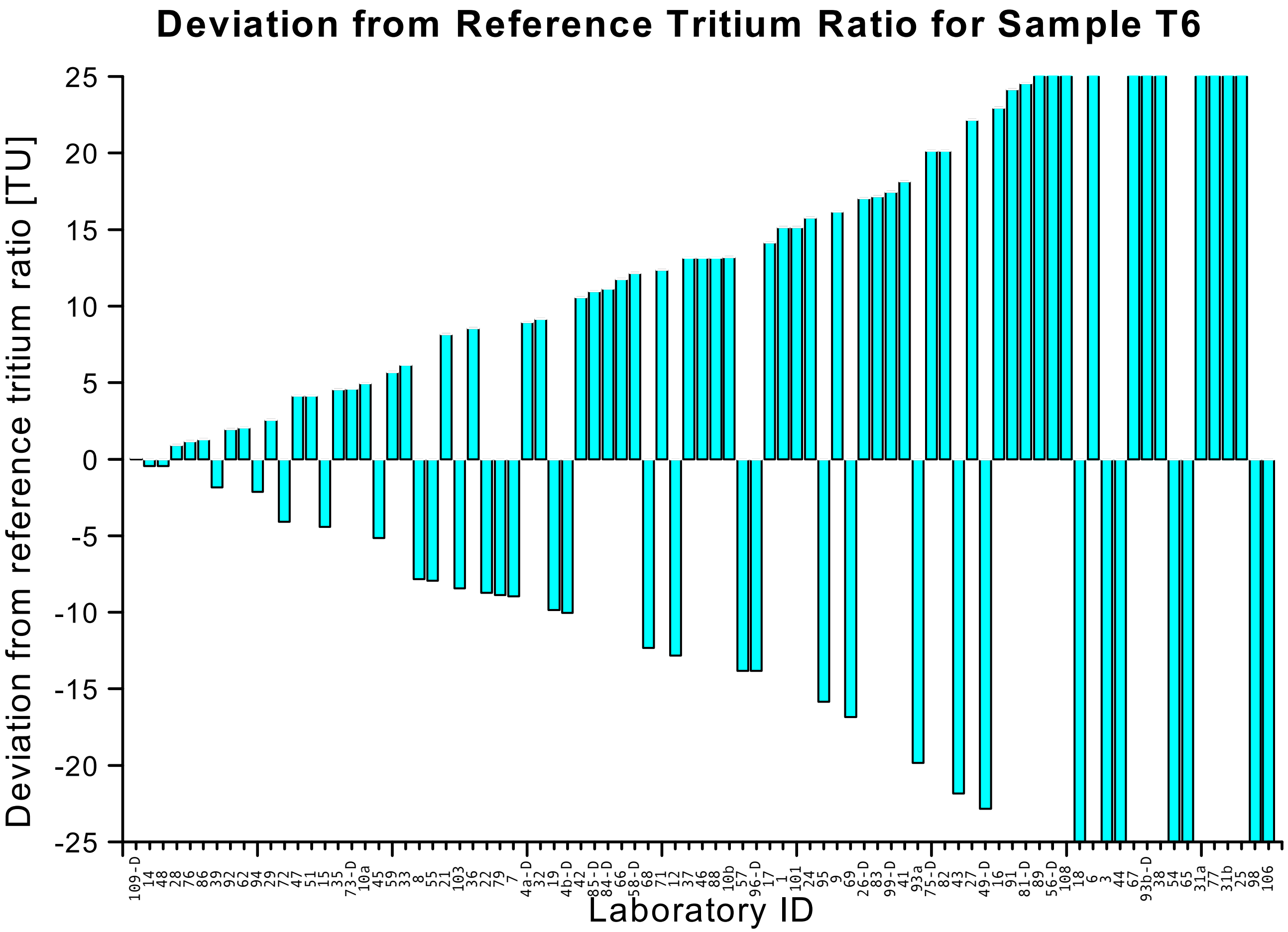

Fig.10: Deviation from the reference tritium ratio for sample T6 in increasing order

3.3.2 Precision (Reliability of Precision Statement):

An additional aspect of visualising laboratory performance is the actual precision stated for performed measurements. This approach focuses on the reported uncertainty associated with each measurement and on the assessment of its reliability. The absolute deviation for each sample is again calculated, and then divided by the reported uncertainty. Therefore, the deviation for each sample is expressed in a sigma scale, directly comparing the reported 1σ-uncertainty for each sample of a laboratory with the actual deviation from the reference value. Each laboratory's "sigma-deviations" for the six samples T1 to T6 are presented in Fig. 11, ordered by increasing sum of absolute "sigma-deviation" values.

Assuming the absence of any systematic bias for each individual laboratory, all results should be normal distributed. Under this assumption the respective references value should lie with 95% probability within 2-σ uncertainty ranges around the reported values. As this holds for all six samples following in each case a normal distribution, the average standard deviation at a 1σ-level (67% probability) for a set of six measurements should be close to one; or expressed differently, the sum of all absolute standard deviations for six samples should be close to six sigma in Fig. 11.

As a subjective quality criterion, for six samples reported, the sum of deviations should not exceed 8σ and it should not fall below 4σ. As it can be seen from Fig. 11, about 50% of all laboratories exceed significantly the value of 8σ, which clearly is an indicator for an underestimation of uncertainty in those laboratories (including the possibility of systematic biases). On the other side, about 25% of all laboratories provided data deviating in sum less then 4σ from the reference values of samples T1 to T5, which signalises their quite conservative approach in stating significantly too high uncertainties.

It has to be stressed, however, that the results of Fig. 11 do not tell anything about the actual suitability of reported data for a certain purpose e.g. for hydrology. This is illustrated in Fig. 12, where a similar plot as in Fig. 11 is presented for samples T1 to T5. In addition to the sum of absolute "sigma-deviations" for the five samples, the cumulated reported uncertainty for the five samples is plotted in the lower part of the figure. Obviously, no correlation exists between the magnitude of uncertainty stated by the laboratories and its reliability as approximated by the sum of absolute "sigma-deviations".

Comparing the 12 laboratories with lowest reported cumulative uncertainties for samples T1 to T5 from Fig. 7 with their position in Fig. 12 reveals that 11 out of these 12 laboratories do considerably overestimate their precision. This statement even holds true when considering a possible +1.55% positive bias of laboratories in case of using a higher tritium halflife and old standards. Seven labs show average offsets of more than 5% from the reference tritium ratios. Five of these 12 laboratories are among the dozen of laboratories with the largest sigma-deviations from the reference values (lab 5 and 14 deviating both by more than 100σ), all with average offsets from the reference tritium ratios between 5 to 35%.

This indicates the strong need to further improve the evaluation and uncertainty statements for tritium measurements. An IAEA advisory group meeting was conducted in 1998 to discuss uncertainties of nuclear analytical measurements. A contribution discussed in detail the uncertainty evaluation for tritium at environmental low-level activities using electrolytic enrichment and liquid scintillation counting [10], providing numerical examples to facilitate the application of the described principles.

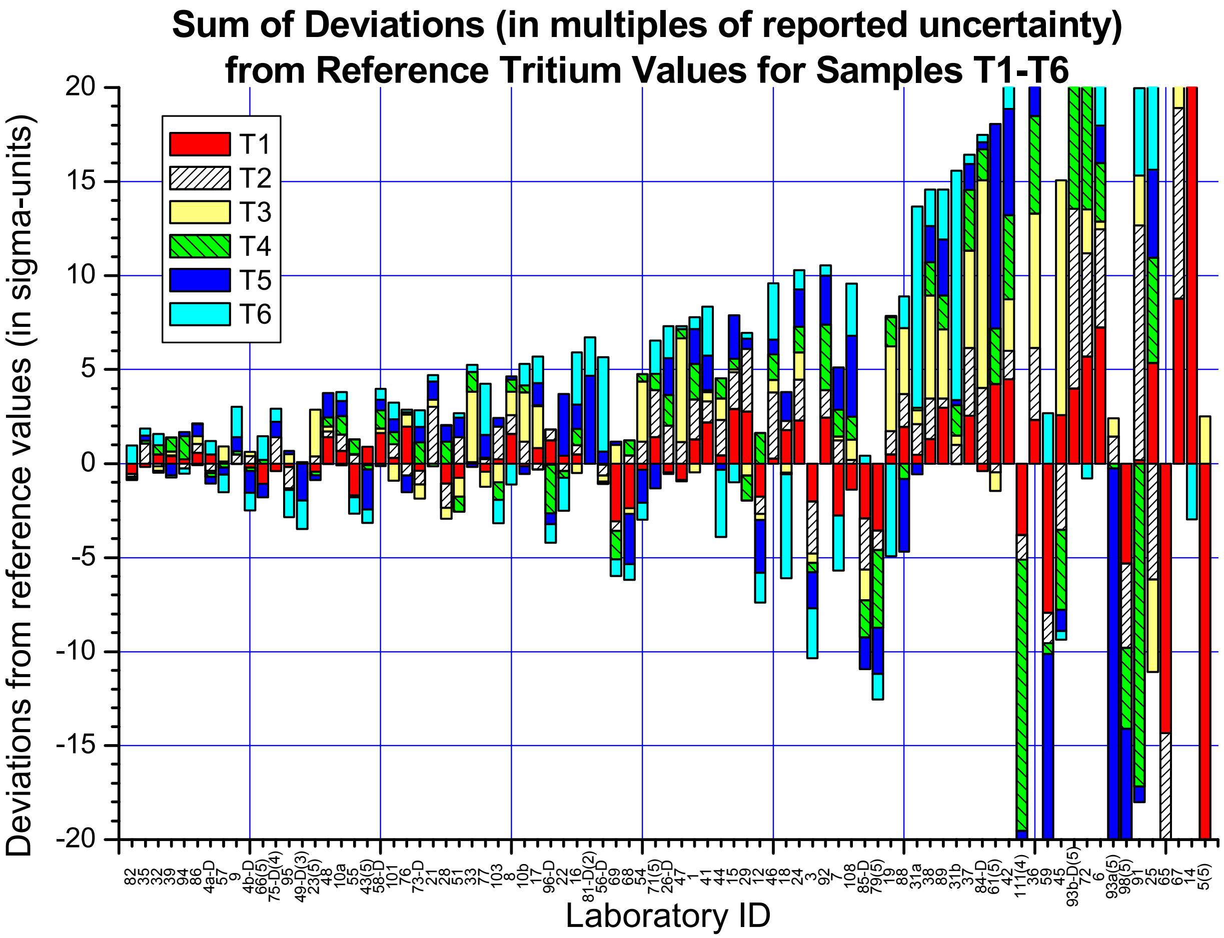

Fig.11: Sum of deviations from the true values for samples T1 to T6 in increasing absolute order, here reported as sigma-deviation by dividing the calculated deviation for each sample by the stated uncertainty. For details see text.

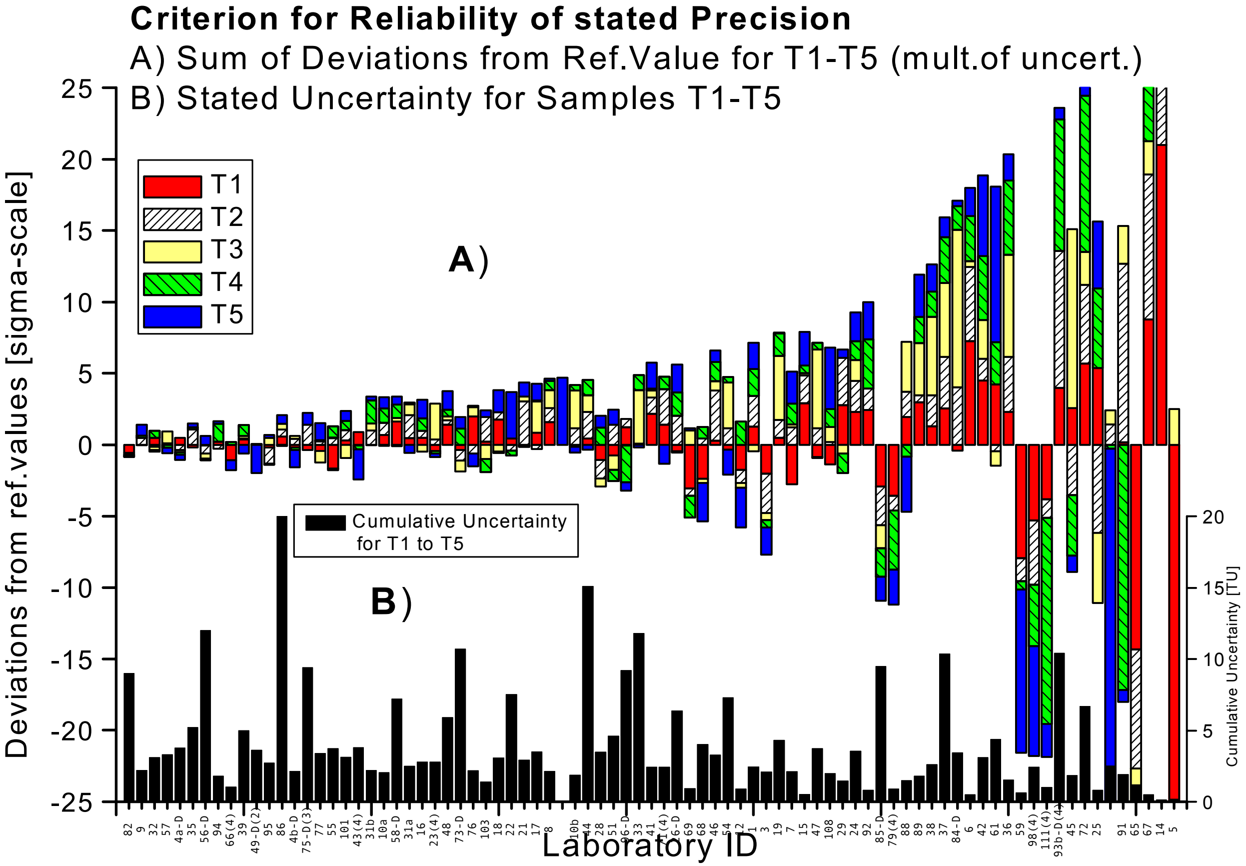

Fig.12: Sum of deviations from the reference value for samples T1 to T5 in increasing absolute order, reported as sigma-deviation by dividing the calculated deviation for each sample through the stated uncertainty.

3.5 Contrast of Reported Uncertainties in 5th and 6th Tritium Intercomparisons

It is worthwhile to assess whether there has been any indication of a general improvement in sensitivity and accuracy since the 5th intercomparison in 1994. This is attempted here by examining the error distributions for the zero samples (A in 1994 and T3 in 2000, Fig.13) and samples of moderate concentration (D in 1994 and T5 in 2000, Fig.14).

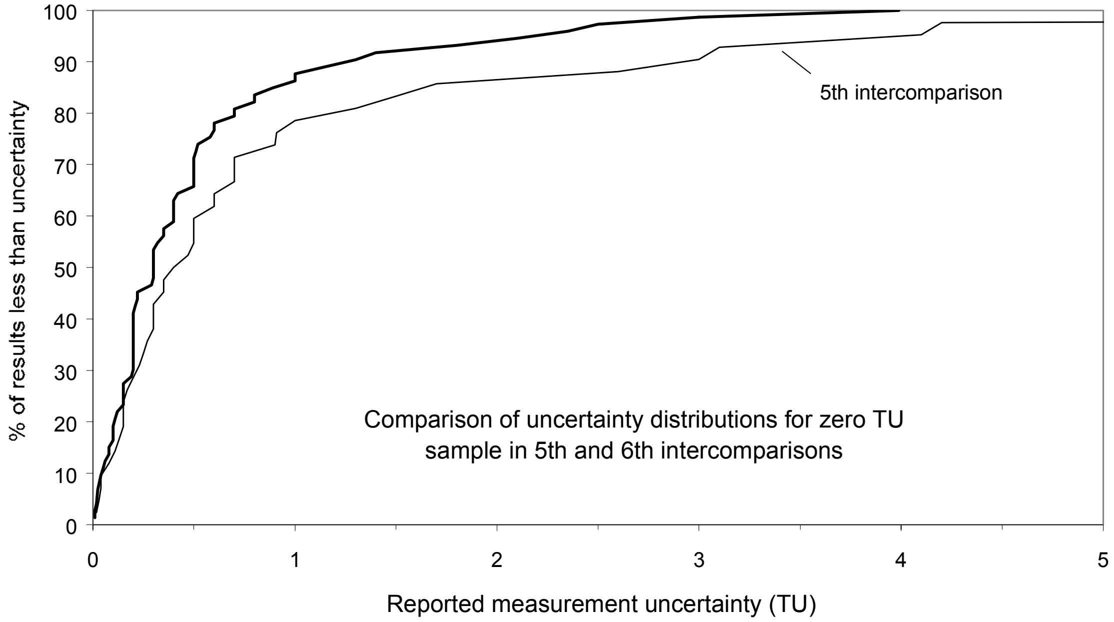

Fig.13: Uncertainty distributions for tritium-free samples in the 5th and 6th tritium intercomparisons. The upper line denoting the recent exercise indicates a slightly better performance (as percentage of all participating laboratories).

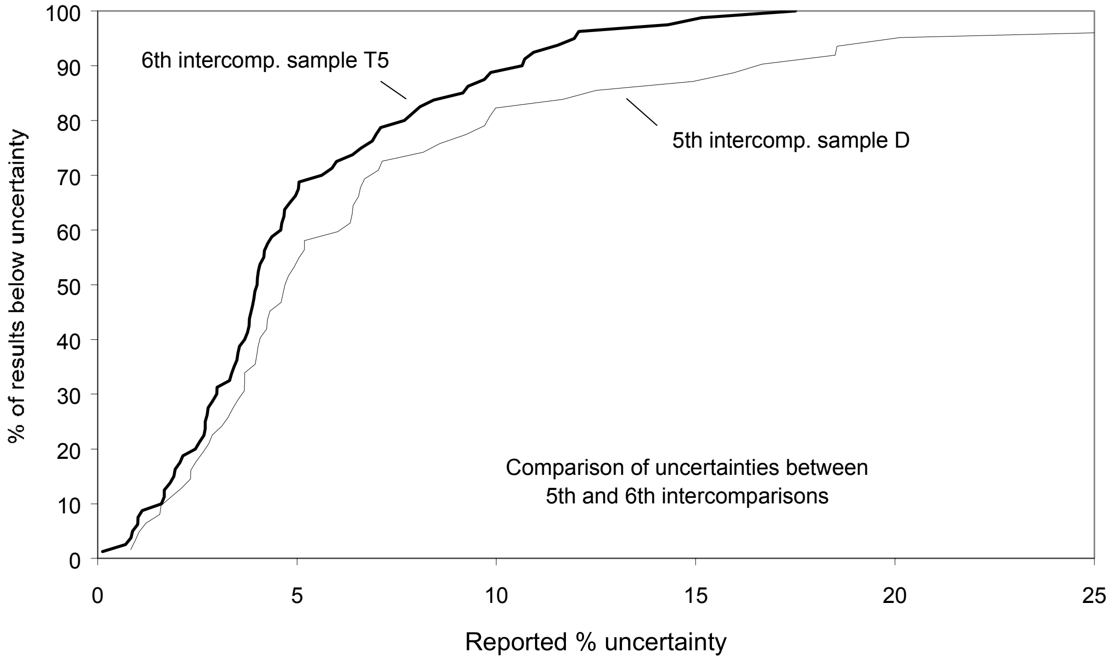

Fig.14: Uncertainty distributions for samples with approx. 25 TU in the 5th and 6th tritium intercomparisons. The upper line denoting the recent exercise indicates a slightly better performance (as percentage of all participating laboratories).

For the zero samples there is little difference between the distributions at uncertainty values less than 0.25 TU; because more laboratories are involved in 2000, this indicates that more laboratories are reporting low detection limits. At greater uncertainties, the sensitivities have improved slightly overall. However, it should be realised that the choice of limit 0.25 TU for this comparison was arbitrary. It is possible to achieve standard measurement uncertainty significantly lower than that. Very low detection limits have become necessary for studies involving very low concentrations, as for example ocean measurements.

Samples D and T5 had slightly different tritium ratios (sample D: 19.2 TU; T5: 26.1 TU). The comparison (Fig. 14) was therefore made using the percentage uncertainties. The distributions again reveal some overall improvement in TRIC2000.

This comparison definitely includes a number of incorrectly calculated or reported uncertainties. In both intercomparisons, a few laboratories grossly underestimated their measurement uncertainty, while some others did not include all uncertainty components. Therefore, the indications are of a very general nature.

3.6 Reporting of Results

Methods of reporting results are often neither consistent nor appropriate. Many results in TRIC2000 were reported with different numbers of decimal points for result and error. Others had too few decimal points, and some too many. Details of standardisation were unclear in many cases, which has caused problems in looking for factors influencing the data individually and overall. The following ideas may help to improve this situation for the future.

Results should always be expressed in an appropriate number of decimal places, with the same number applying to result and error. The appropriate number can be judged by the relative magnitudes of the reported value and measurement error, and the range of tritium concentrations pertaining to samples handled by the laboratory. As an example, consider a laboratory operating normally now within a range lower than a tritium ratio of 20 TU, and able to achieve 3% standard measurement uncertainty for all but near-zero results. A result of 10 TU would bear an uncertainty of 0.30 TU; one of 5 TU would have an uncertainty of 0.15 TU. In this case, it is important to report results and uncertainties to 2 decimal places. Moreover, for results less than 1 TU, where the % error increases sharply, reporting to 3 decimal places becomes appropriate. If samples of order 100 TU are measured reporting to 2 decimal places is no longer of any advantage, 1 being sufficient. (Note: Due to the very small uncertainties associated to the reference values in TRIC2000, those data are reported with more decimal places than recommended for routine samples).
Similar considerations should be applied in determining the appropriate number of decimal places for results reported as massic activities (e.g. Bq.kg^{-1}).

The sample date to which a result refers, should always be reported.

All laboratories should be able to clarify the basis of their calibrations, and even include this as a footnote to results in published papers or reports to clients. Many laboratories reported 4500 days as their applied half-life. This value was stipulated for decay correction of the TRIC2000 results to 1 July 2000, but it is unlikely that that very recently determined and published half-life was the basis of any calibration procedures. Only few laboratories stated to use already the “new generation” of NIST standards with calibration date 3 Sep 1998. The necessary fundamental information comprises the identity and certified calibration (with date) of the tritiated water standard which is the cornerstone of the calibration procedures, and the half-life applied to estimate its decay over the years. A number of laboratories reported to use “Packard” or “Wallac” tritium standards; these were probably sealed control cocktails monitoring counter stability during routine liquid scintillation counting, which are not relevant for calibration purposes. Tritium calibration involves counting cocktails with daughter water standards of appropriate count rates and background waters, both having identical composition to the unknown sample cocktails.

3.7 Suggestions for Improved Sensitivity

Given the need for optimum sensitivity, it is useful to examine whether any of the results and technique details provided by the laboratories indicate that they are individually getting the best value from equipment available to them. For liquid scintillation counting, the background count rate and its stability are very important factors. A more stable background count rate will reduce the uncertainty for the same counting time, or allow samples to be counted for shorter times, enabling greater sample throughput. Background is not only counter-dependent, but it also depends on the material of the sample vials and the cocktail constituents. From the available data provided by the participants, the only two counter groups giving assessable background information were those laboratories using Wallac type Quantulus and Packard type 2770/3170TR counters. These counters are well protected against cosmic ray variations (including altitude dependence) and against other factors influencing many other counters. 26 respectively 12 laboratories reported using these counter types mentioned above, with backgrounds ranging from 0.44 to 1.38 cpm (Quantulus) and 0.89 to 1.90 cpm (2770/3170TR). The lower values give a huge advantage, while the upper values seem to be perhaps unnecessarily high, indicating not-optimised settings. Slightly higher backgrounds can be expected for other instrument types, or those without all the electronic options aiding background reduction, but spectral analysis capability allows all counters of these types to be tuned carefully to give the best compromise between detection sensitivity of tritium decay events and background count rate. Spectral analysis capability also enables the selection of counting vials and cocktails with lowest background from available commercial options, and determination of the most advantageous water/cocktail proportions. Low-level tritium samples should now be counted in polyethylene or teflon vials (i.e. not glass, which has a higher background), using a cocktail type which does not diffuse through the vial wall; teflon vials are expensive, and there are several types of cheaper polyethylene vial which are equally effective.

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APPENDIX A. DETAILS OF PROCEDURES TO PREPARE SAMPLES

Because SRM4927F is an extremely active tritiated water, the first two dilution stages were not performed in the low-level Isotope Hydrology Laboratory, but at the IAEA Laboratories at Seibersdorf. A de-ionised "tritium-free background water" was used for these 3 stages. This water dates from 1992 and was used for the previous tritium interlaboratory comparison; it is from the same Grafendorf well which was re-sampled to provide the water for the final dilution stage (sample T3). Repeat analyses of the de-ionised water indicated that its tritium ratio is less than 0.1 TU. Its massic activity is less than 5×10^{-7} that of the resulting daughter water after 3 dilution stages, and its contribution to the prepared values can therefore be neglected.

Table 4: Dilution procedure for the tritiated water standard SRM4927F

Dilution stage	1	2	3
Weight of tritiated water (in brackets uncertainty) [g]	4.9063 (0.0003)	20.9142 (0.0003)	83.585 (0.003)
Weight of added T3 dilution water (uncertainty) [g]	994.38 (0.07)	999.89 (0.07)	8453.95 (0.05)
Dilution factor (relative uncertainty)	203.674 (0.009%)	48.8091 (0.007%)	102.142 (0.004%)
Combined dilution factor (relative uncertainty)	203.674 (0.009%)	9941.12 (0.012%)	1015409 (0.012%)

A sufficient supply of diluting water for the final dilution stage was sampled from the Grafendorf well. This is sample T3. The possible tritium concentration range of this water was judged to be between 0 - 0.072 TU, using the submitted results.

The diluting water T3 was not distilled or de-ionised, in order to ensure that the samples would be put through the routine pre-measurement distillation step by the participating laboratories. In the final dilution stage, the measured weights of diluting water were corrected for the dissolved solid content; this correction is 218 mg/kg water, i.e. –0.0218%.

The dilution stages of the daughter water and the calculated TU values are summarised in the accompanying table. Numbers in brackets are 1 standard uncertainty. The factor 8.390 was applied to convert Bq/kg to TU.

Table 5: Dilution procedure for the six samples T1 to T6 used in this interlaboratory comparison exercise, using as tritiated standard the 'Dilution Stage 3' of Table 4. Values in brackets are uncertainties at the 1-σ level.

Step	T1	T2	T4	T5	T6
Weight of SRM4927F daughter [g]	166.52 (0.03)	33.451 (0.003)	82.400 (0.003)	412.19 (0.03)	1806.37 (0.03)
Weight of T3 water [g]	77782.9 (0.2)	73541.7 (0.2)	74173.3 (0.2)	74286.9 (0.2)	15096.3 (0.2)
Correction of above T3 weight for TDS [g]	77765.9 (0.2)	73525.7 (0.2)	74157.1 (0.2)	74270.7 (0.2)	15093.0 (0.2)
Dilution factor	468.006 (0.018%)	2199.01 (0.009%)	900.965 (0.004%)	181.186 (0.007%)	9.35543 (0.004%)
Total dilution factor [×10^{-8}] relative to SRM4927F	4.75218 (0.022%)	22.3289 (0.015%)	9.14848 (0.013%)	1.83978 (0.014%)	0.0949959 (0.013%)
Massic tritium activity at 1 July 2000 [Bq/kg]	1.20519 (0.363%)	0.25650 (0.363%)	0.62604 (0.363%)	3.11303 (0.363%)	60.2899 (0.363%)
Tritium ratio at 1 July 2000 [TU]	10.112 (0.037)	2.152 (0.008)	5.252 (0.019)	26.118 (0.095)	505.83 (1.84)
Possible range of added tritium [TU] due to diluting water T3 (additional uncertainty component)	(0 - 0.072)	(0 - 0.072)	(0 - 0.072)	(0 - 0.072)	(0 - 0.072)
Combined uncertainty range of tritium ratio at 1 July 2000 [TU] at 2-σ level	10.038 - 10.258	2.136 - 2.240	5.214 - 5.362	25.928 - 26.380	502.15 - 509.58

APPENDIX B: ADJUSTMENT OF RESULTS TO THE PREPARED VALUE RANGE

The previously recommended tritium scale [7] was based on two primary parameters. The first was the certified massic activity (Bq/kg) of the primary tritiated water standard on 3 September 1978. This standard water was suitable for dilution by environmental laboratories to provide daughter standard waters for calibration of routine measurements (for enrichment, counting and ^{3}He mass spectrometry). The second adopted parameter was the former tritium half-life 4540 days. These were fixed parameters, i.e. defined to be error-free. Their actual uncertainties (for example for massic activity as quoted by NIST on accompanying standard certificates) were not relevant for intercomparability with data obtained using other standards and half-life values. In fact, it was not possible till now to judge the uncertainty of any half-life value, making the fixed parameter scale the only option. The new NIST half-life (4500 ± 8 days) is the first to provide such a confident uncertainty estimate. Absolute measurements (not based on fixed parameter scale) must take into account reliably determined uncertainties in standard calibration and half-life value.

The two primary parameters defined the old scale for massic activity measurement, but a conversion factor had to be applied to yield TU. This factor was deduced from the molar weight assumed for water, the Avogadro constant (determining the number of hydrogen atoms in 1 kg of water) and the assumed half-life, yielding 1 Bq/kg = TU = 8.464 (old scale!).

The new conversion factor 8.390 is based on the newly determined half-life (4500 days), the present value of the Avogadro constant and the molecular weight of water.

Laboratories can adjust their intercomparison results to the prepared values using the following conversion equation.

$$\frac{\mathrm{TR(adjusted)}}{\mathrm{TR(original)}} = \frac{n_{1s}}{n_{2s}} \times \frac{a_{s2}(t_{02})}{a_{s1}(t_{01})} \times \frac{\exp(-\lambda_2(t_c - t_{02}))}{\exp(-\lambda_1(t_c - t_{01}))}$$

Here the quantities $a_{s1}(t_{01})$, $a_{s2}(t_{02})$ are respectively the massic activities of the laboratory's standard water and SRM4927F at their calibration dates t_{01}, t_{02}.. λ_1, λ_2 are the old, new radioactive decay constants, and t_c is the intercomparison reference date. n_{1s} is the count rate of the daughter standard used to calibrate the original measurement. n_{2s} is the count rate which would have been recorded by an equally-diluted daughter of the SRM4927F. It must be emphasised that data adjustment to the prepared values **must involve an accurately measured ratio** n_{1s}/n_{2s}. Luckily ratios are available for all old and newer NIST standards [9]. It would be extremely useful if other used tritiated water standards could be compared against NIST tritiated standard waters.

Very few laboratories would have used any of the very recent NIST standards or the new half-life to calibrate their measurements for TRIC2000 (only seven laboratories indicated having used a NIST standard of the new series: LabIDs 1,12,29,31a/b,57,75,103). Many reported values using the SRM4926C/half-life 4540 days fixed parameter scale. Adjustment to the values calculated for the intercomparison samples involves incorporation of the NIST-determined massic activity ratio [9]

SRM4927F/SRM4926C = 576 758 ± 0.066%

into the above equation. Converted values should have this uncertainty component added in quadrature to the original % measurement error. In nearly all cases, this will not change the already higher uncertainty, but corrections from standard waters not originated from NIST may involve larger uncertainties in the ratio n_{1s}/n_{2s}. Laboratories which used other standard waters and/or half-lives can evaluate and apply the appropriate factors in the above conversion equation.

APPENDIX C: LISTING OF RESULTS FOR THE SIX SAMPLES

In Table 6, all submitted results from the 86 laboratories participating in TRIC2000 are listed and sorted according to the assigned laboratory ID number. The results are presented with as many digits as originally reported (only in one case, a fourth digit was omitted). Necessary alterations to the raw data are marked under the column ‘Remarks’. The gray-shaded fields are data which were regarded as outliers. Three criteria for outlier-detection were applied:
1. all data without stated uncertainty could not be evaluated and were regarded as outliers irrespective of the reported value (see the part requested information in the TRIC2000 invitation); 2. all data strongly deviating from the remaining data set were regarded as outliers (interquartile criterion); 3. all data deviating from the stated limits around the reference value by more than twice their reported uncertainty.
In Table 7 a list of correction data is reported which were received after the reporting deadline and after an indication of possible problems in the sample analysis, was sent to 26 laboratories. For this reason, those data were not included in the original data set.

Table 6: All sample results as reported by the individual participating laboratories. The uncertainties **u(**sample**)** were requested to be stated at the 1-σ level; only obvious changes were applied (see remarks). Blank fields indicate not-submitted data. The three measurement methods are indicated: LSC – Liquid Scintillation Counting; Gas – Gas Proportional Counting; 3He – Mass Spectrometric ^{3}He analysis. All outlier values in the tables are gray-shaded, for details on the outlier determination procedure see text. The TRIC2000 reference values are stated in the header lines.

Lab	T1	u(T1)	T2	u(T2)	T3	u(T3)	T4	u(T4)	T5	u(T5)	T6	u(T6)	Meas. Method	Remarks
Ref.	**10.112**		**2.152**		**0**		**5.252**		**26.118**		**505.83**			Reference value for TRIC2000
	All tritium values and uncertainties are stated as tritium ratio in [TU]													
1	10.78	0.52	2.62	0.22	-0.07	0.15	5.88	0.33	28.35	1.21	521	24	LSC	
3	9.1	0.5	1.6	0.2	-0.1	0.2	5.1	0.3	24.4	0.9	457.7	18.1	LSC	See also revised data (T6)
4a-D	10.5	0.8	1.90	0.68	-0.10	0.70	5.12	0.69	25.8	0.9	514.8	12.6	LSC-D	
4b-D	10.03	0.42	2.28	0.32	0.07	0.32	5.18	0.37	25.3	0.7	495.8	10.8	Gas-D	
5	6.367	0.045	1.370	0.019	0.010	0.004	3.301	0.028	16.352	0.094			3He	Values+uncertainties adjusted for ref.date
6	10.931	0.113	2.293	0.027	0.004	0.010	5.424	0.055	26.654	0.270	553.4	5.5	3He	See also revised data (T6)
7	9.17	0.34	2.46	0.25	0.04	0.19	5.71	0.32	28.39	1.01	496.88	3.06	LSC	
8	10.9	0.5	2.31	0.16	0.15	0.12	5.43	0.27	26.3	1.1	498	7.0	LSC	
9	10.1	0.4	2.3	0.3	0.0	0.3	5.3	0.3	26.8	0.9	522	10	LSC	
10a	10.4	0.43	2.38	0.26	-0.02	0.22	5.58	0.33	26.75	0.8	510.8	10.6	LSC	
10b	10.05	0.42	2.28	0.11	0.21	0.08	5.34	0.23	25.71	1.03	519.04	11.59	Gas	
12	9.76	0.20	2.07	0.09	-0.02	0.06	5.48	0.14	25.0	0.40	493	8	Gas	
14	10.70	0.028	2.34	0.024	0.56	0.024	5.81	0.027	27.60	0.033	505.40	0.145	LSC	Probably relative uncertainty stated?
15	10.46	0.12	2.23	0.04	0.003	0.018	5.29	0.07	26.75	0.27	501.4	4.4	LSC	
16	10.4	0.6	2.3	0.3	-0.1	0.2	5.6	0.4	27.8	1.3	528.8	8.3	LSC	
17	10.7	0.7	2.0	0.5	1.1	0.5	5.3	0.6	27.5	1.2	520	10	LSC	
18	11.07	0.54	2.36	0.42	-0.20	0.42	5.20	0.60	27.78	1.08	459.00	8.44	LSC	
19	10.6	1.0	3.4	1.0	0.9	0.2	6.5	0.8	26.2	1.3	496	2	LSC	
21	10.10	0.57	2.97	0.27	0.11	0.30	5.16	0.73	27.16	1.07	514.01	24.87	LSC	
22	10.7	1.4	1.6	1.4	0.0	1.4	4.7	1.5	32.0	1.8	497.1	5	LSC	See also revised data (T5)
23	9.8	0.7	2.3	0.4	0.25	0.1	5.2	0.3	25.8	1.3			3He	
24	11.84	0.75	3.37	0.56	0.75	0.52	6.15	0.67	28.24	1.06	521.6	15.3	LSC	
25	11.13	0.19	1.35	0.13	-0.54	0.11	6.09	0.15	27.24	0.24	631.23	5.88	LSC	
26-D	9.46	1.38	4.88	1.35	-0.09	1.15	7.17	1.18	28.71	1.32	522.88	9.99	Gas-D	
27	10		3.0		-0.17		4.2		25.6		528		LSC	
28	9.38	0.69	1.84	0.24	-0.12	0.21	5.76	0.43	27.72	1.91	506.76	34.75	LSC	

Lab	T1	u(T1)	T2	u(T2)	T3	u(T3)	T4	u(T4)	T5	u(T5)	T6	u(T6)	Meas. Method	Remarks
Ref.	**10.112**		**2.152**		**0**		**5.252**		**26.118**		**505.83**			Reference value for TRIC2000
	All tritium values and uncertainties are stated as tritium ratio in [TU]													
29	11.00	0.32	2.95	0.24	-0.14	0.22	4.93	0.24	26.36	0.44	508.4	8.6	LSC	
31a	10.3	0.4	2.8	0.4	0.3	0.4	5.3	0.4	25.6	0.9	613	10	LSC	
31b	10.1	0.5	2.45	0.3	0.15	0.3	5.9	0.4	26.3	0.7	628	10	LSC	
32	10.4	0.6	2.1	0.4	-0.1	0.4	5.5	0.5	26.0	1.2	515	15	LSC	
33	10.1	3.1	2.2	0.7	3.0	0.8	9.9	4.4	25.6	2.8	512	17	LSC	See also revised data (T4)
35	9.9	1.2	3.0	0.8	0.0	0.5	5.4	1.0	26.6	1.7	510.4	12.3	LSC	
36	10.85	0.32	2.73	0.15	1.07	0.15	6.45	0.23	27.35	0.67	514.39	4.85	LSC	
37	16.5	2.5	4.85	0.75	3.1	0.6	10.1	1.5	33	5	519	26	LSC	See also revised data set (T1-T6)
38	10.88	0.59	2.54	0.18	0.11	0.02	5.85	0.34	28.91	1.46	575.9	36.1	LSC	See also revised data set (T5,T6)
39	10.5	1	2.2	1	0.2	1	6.0	1	25.5	1	504	15	LSC	
41	11.2	0.5	2.6	0.4	0.2	0.4	5.3	0.4	27.4	0.7	524	7	LSC	
42	12.99	0.64	2.95	0.53	1.39	0.51	7.9	0.59	30.8	0.83	516.4	6	LSC	
43	11	1	2.1	0.8			5	1	24	1	484	30	LSC	Reported: T3<0.8
44	12.7	5.8	5.5	1.8	2.9	2.5	7.4	2.0	25.1	3.0	445	17	LSC	See also revised data set (T1-T6)
45	11.4	0.5	1.8	0.1	0.5	0.04	4.4	0.2	25.0	1.0	500.7	11.2	LSC	
46	10.3	0.72	3.95	0.51	0.235	0.352	6.05	0.59	27.0	1.1	519	4.4	LSC	Reported relative uncertainty converted
47	9.5	0.7	2.5	0.3	1.1	0.2	5.5	0.5	26.0	2.0	510	25	LSC	
48	12.1	1.4	2.3	0.5	0.1	0.4	5.6	0.7	29.9	2.9	505.4	15.9	LSC	
49-D	10.2	1.3							21.6	2.3	483	15	LSC-D	
51	9.5	0.8	3.0	0.6	-0.5	0.5	4.7	0.7	28.2	2.0	510	18	Gas	
54	9.6	1.5	3.2	0.9	1.6	0.5	5.6	0.9	20	3.5	430	85	LSC	
55	8.85	0.75	2.45	0.65	0.03	0.60	5.80	0.70	26.00	1.00	497.90	9.25	LSC	
56-D	10	2	1	2	-1	3	5	2	28	3	536	6	LSC-D	See also revised data (T6)
57	10.19	0.7	2.15	0.4	0.16	0.2	5.15	0.5	25.54	1.5	492	15	LSC	
58-D	12.4	1.4	2.0	1.3	0.3	1.3	6.5	1.3	27.2	1.9	518	21	LSC-D	
59	9.00	0.14	1.99	0.10	0.00	0.08	5.19	0.11	23.83	0.20	511.52	2.13	Gas	
61	13.5	0.8	1.72	0.94	-0.80	0.80	7.67	0.82	37.0	1.0			LSC	
62											507.9	15.6	LSC	
65	7.82	0.16	1.15	0.12	-0.40	0.15	3.82	0.26	17.01	0.49	400.26	13.33	LSC	See also revised data set (T1,T5,T6)
66	9.85	0.24			0.00	0.05	5.29	0.19	25.73	0.55	517.6	9.4	3He	
67	11.34	0.14	2.66	0.05	0.075	0.032	6.04	0.06	29.93	0.21	571.2	4.3	3He	See also revised data set (T5,T6)

Lab	T1	u(T1)	T2	u(T2)	T3	u(T3)	T4	u(T4)	T5	u(T5)	T6	u(T6)	Meas. Method	Remarks
Ref.	**10.112**		**2.152**		**0**		**5.252**		**26.118**		**505.83**			Reference value for TRIC2000
	All tritium values and uncertainties are stated as tritium ratio in [TU]													
68	8.2	0.8	2.5	0.8	-0.2	0.7	5.9	0.8	23.7	0.9	493.5	14.8	LSC	
69	9.5	0.2	2.1	0.1	0.01	0.01	5.1	0.1	26.2	0.5	489	19	3He	
71	11.1	0.7	2.9	0.3	0.1		5.6	0.4	24.8	1.0	518.2	7.0	Gas	
72	17.75	1.34	7.26	0.93	2.06	0.89	17.92	1.16	29.44	2.38	501.75	5.13	LSC	
73-D	9.3	2.2	0.6	2.1	-1.6	2.1	7.63	2.1	27.9	2.2	510.43	5.2	LSC-D	
75-D	8.9	3.1	6.1	2.8					29	3.5	526	29	LSC-D	See also revised data (T2)
76	11.1	0.5	1.95	0.32	0.13	0.20	5.3	0.45	25.5	0.7	507	8.0	LSC	
77	9.8	0.7	2.3	0.6	-0.4	0.5	5.3	0.7	27.2	0.9	615	40	LSC	
79	9.54	0.16	2.04	0.11			4.92	0.08	24.80	0.54	496.96	6.51	3He	Reported : T3<0.4
81-D									37.8	2.5	530.4	12.0	LSC-D	
82	9	2	2	1	0	1	5	2	26	3	526	21	LSC	
83	14.8		11.9		5.0		6.8		30.0		523		LSC	
84-D	9.8	0.78	3.4	0.31	3.2	0.29	6.0	0.45	26.7	1.60	517	28.4	LSC-D	Reported relative uncertainty converted
85-D	4.70	1.85	-1.93	1.51	-3.86	2.35	1.93	1.68	22.6	2.1	516.8	26.5	LSC-D	Reported values in Bq/kg converted to TU
86	12.391	4.044	4.038	3.858	1.612	3.989	4.960	4.014	28.728	4.11	507.150	27.33	LSC	
88	10.7	0.3	2.5	0.2	0.7	0.2	5.0	0.3	24.2	0.5	519.0	7.8	LSC	See also revised data set (T1,T4)
89	11.3	0.4	2.3	0.3	1.1	0.3	5.8	0.3	27.6	0.5	535	11	LSC	
91	10.2	0.5	5.9	0.3	0.8	0.3	0.1	0.3	25.7	0.5	530.0	5.2	LSC	
92	10.6	0.2	2.3	0.1	0.0	0.1	5.6	0.1	26.9	0.3	507.8	3.6	LSC	
93a			3.02	0.61	0.57	0.58	5.09	0.61	7.86	0.72	486	11	LSC	
93b-D	16.9	1.7	28.0	2.7	0		34.7	3.2	28.4	2.8	573.9	44.2	LSC-D	Reported relative uncertainty converted
94	10.2	0.4	2.1	0.2	0.0	0.1	5.5	0.2	26.3	0.9	503.7	8.2	LSC	Reported negative uncertainty (T3) corrected
95	10	0.6	1.7	0.4	0.1	0.2	5.2	0.5	26.3	1	490	11	LSC	
96-D	12.2	1.7	3.2	1.8	-0.1	1.8	0.6	1.8	24.9	2.1	492	14	LSC-D	
98	6.4	0.7	0.8	0.3	0.1	0.0	3.1	0.5	19.2	0.9	341	17.2	LSC	
99-D											523.30	38.20	LSC-D	Reported relative uncertainty converted
101	10.3	0.64	2.44	0.39	-0.32	0.35	5.54	0.45	27.0	1.3	521	17	??	Average value of two data sets calculated
103	10.18	0.31	2.57	0.24	-0.15	0.15	5.02	0.25	26.32	0.44	497.4	6.8	LSC	
106	110.69		2.442		10.581		23.60		229.11		244.98		LSC	
108	9.6	0.37	2.2	0.24	0.21	0.20	5.6	0.28	30	0.9	550	16	LSC	
109-D											505.91	23.27	LSC-D	Reported relative uncertainty converted

Lab	T1	u(T1)	T2	u(T2)	T3	u(T3)	T4	u(T4)	T5	u(T5)	T6	u(T6)	Meas. Method	Remarks
Ref.	**10.112**		**2.152**		**0**		**5.252**		**26.118**		**505.83**			Reference value for TRIC2000
	All tritium values and uncertainties are stated as tritium ratio in [TU]													
111	9.35	0.20	2.06	0.07			3.81	0.10	24.68	0.62			3He	Reported: T3<0.05; relative uncertainties converted

Table 7: Corrections to the original submitted data of Table 6. The data were not used for any compilation, as they were received after the deadline passed and nearly all only after a letter was sent to the laboratories to alert them on probable problems with their results. The TRIC2000 reference values are stated in the header lines.

Lab	T1	u(T1)	T2	u(T2)	T3	u(T3)	T4	u(T4)	T5	u(T5)	T6	u(T6)	Meas. Method	Remarks
Ref.	**10.112**		**2.152**		**0**		**5.252**		**26.118**		**505.83**			Reference value for TRIC2000
	All tritium values and uncertainties are stated as tritium ratio in [TU]													
3											501.3	18.5	LSC	Correction after indication of possible problem
6											507.0	5.0	3He	Correction after indication of possible problem
22									29.0	1.8			LSC	Correction after indication of possible problem
33							7.3						LSC	Correction after indication of possible problem
37	12.4	2.6	0.7	1.1	-1	1	6.0	1.7	28.5	5	519	26	LSC	Correction after indication of possible problem
38									21.61	1.09	537.98	21.52	LSC	Correction after indication of possible problem
44	3.59	3.4	9.52	2.3	6.80	2.5	13.2	3.8	25.1	3.0	328	30	LSC	Correction after indication of possible problem
56-D											513	5	LSC-D	Correction after indication of possible problem
65	8.536	0.188							17.667	2.044	484.81	9.644	LSC	Correction after indication of possible problem
67									25.84	0.58	538.7	9	3He	Correction after indication of possible problem
75			<6.7										LSC-D	Correction after indication of possible problem
88	10.3	0.3					24.8	0.9					LSC	Re-analysis confirming former result

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APPENDIX E: LISTING OF ANNOUNCEMENT, DATA REPORTING INSTRUCTIONS, REPORTING FORM

E1: Invitation letter with requirements:

ANNOUNCEMENT

Sixth IAEA Intercomparison Exercise of Low-Level Tritium Measurements in Water - TRIC2000

The Isotope Hydrology Laboratory of the International Atomic Energy Agency is organizing the sixth interlaboratory comparison exercise of tritium analysis of water at environmental activity levels (TRIC2000). All laboratories and institutes having analytical capabilities for low-level tritium measurements (gas proportional counting, liquid scintillation counting, He3 ingrowth method) are invited to take part in the exercise. Purpose of the intercomparison exercise is to give the participating laboratories the opportunity to check the agreement of their measurements with those of other laboratories, to discover the occurrence of errors and systematic deviations and, eventually, to improve the quality and reliability of their analytical procedures.

A set of six water samples has been prepared for distribution to the interested laboratories:

Codes: TRIC2000-T1 to TRIC2000-T5, 5 samples, 500ml each with tritium concentrations between 0 and about 25 TU (0-3 Bq/kg)
and
TRIC2000-T6, 1 sample, in a marked 100ml bottle with a tritium concentration of about 500 TU (120 Bq/kg)

In addition to the range from 0-25 TU (T1-T5) covered by previous intercomparison exercises we will provide one sample (T6) with a tritium concentration of approximately 500 TU. This sample should be analyzed by direct counting without enrichment and thus should allow to check the proper calibration of the used laboratory standards as well as the precision of laboratories engaged in the analysis of higher tritium concentration levels.
The samples will be prepared by spiking a tritium-free water from an Artesian well (Steiermark/Austria) with well specified dilutions of a NIST tritiated water standard (NIST 4927F). The samples have not been distilled and will be shipped in polyethylene bottles.

The laboratories will be requested to report for each sample the results of the tritium measurement in TU and its assigned standard uncertainty at the 1 sigma level. It would be useful to specify in addition the individual uncertainty components as assessed in the individual laboratories (standardization, enrichment, counting, etc). Mandatory information include the used equipment and analytical procedures as well as the calibration method (specifying the used tritium standards and dilutions). Any other information which is considered relevant and useful will be appreciated. The respective forms including detailed information and instructions for preparation of the report will be sent out together with the samples. The results will be evaluated in the IAEA and each participating laboratory will receive a report with the individual data, the statistical evaluation of all results and their graphical display. TRIC2000 is open to all low-level tritium laboratories with full confidentiality with respect to link between identity of participants and results.

We particularly request mass-spectrometric laboratories using the He3 ingrowth method to participate in the intercomparison exercise. In these cases we will take into account that the analytical procedure might take longer than for the beta-counting laboratories. Please specify in your response if you do not want to receive the higher-TU sample.

Participation in the exercise will be limited to approximately 120 laboratories. If you are interested in taking part in the intercomparison, please respond directly BY EMAIL at your earliest convenience, but not later than August, 15th, to the TRIC2000 account (isotope.hydrology.lab@iaea.org) specifying the subject TRIC2000. Please indicate also in your response your full address for shipment of the samples as well as your email and fax number for further correspondence.

In case email is not available, please send your information to the following address:

Isotope Hydrology Laboratory
"TRIC2000"
International Atomic Energy Agency
P.O. Box 100
Wagramerstrasse 5
A-1400 Vienna
Austria
Fax +43-1-26007
Tel: +43-1-2600-21766
Email: isotope.hydrology.lab@iaea.org

Time schedule

Deadline for registration of your participation: August, 15th
Shipment of samples: August/September 2000
Deadline for submission of the results to IAEA: December, 31th.

We are looking forward to your active participation in the forthcoming exercise.
Best regards,

Manfred Groening / Gisela Winckler

For more information please refer to the home page of the IAEA Isotope Hydrology Laboratory
http://www.iaea.org/programmes/rial/pci/isotopehydrology/

E2: Instructions for Data Reporting (attached to shipped samples):

الوكالة الدولية للطاقة الذرية
国 际 原 子 能 机 构
INTERNATIONAL ATOMIC ENERGY AGENCY
AGENCE INTERNATIONALE DE L'ENERGIE ATOMIQUE
МЕЖДУНАРОДНОЕ АГЕНТСТВО ПО АТОМНОЙ ЭНЕРГИИ
ORGANISMO INTERNACIONAL DE ENERGIA ATOMICA

ISOTOPE HYDROLOGY LABORATORY
WAGRAMER STRASSE 5, P.O. BOX 100, A-1400 VIENNA, AUSTRIA
TELEPHONE: (+43 1) 260021766, FACSIMILE: (+43 1) 26007 21763, E-MAIL: isotope.hydrology.lab@iaea.org INTERNET: http://www.iaea.org

September 1st, 2000

Dear Colleague:

Thank you very much for your interest in the 6th IAEA Tritium Interlaboratory Comparison Exercise TRIC2000.
Enclosed please find the set of six samples which we prepared for this exercise. The samples T1-T5 cover the range between 0 and 25 TU (0-3 Bq/kg). Note that the consecutive numbering of the samples T1-T5 does not imply increasing tritium concentrations, i.e. the samples are in random order. Sample T6 has a higher tritium activity of ca. 500 TU.

An **Laboratory Identification Number (Lab ID)** is assigned to each laboratory and is given on the bottle labels. This identification number ensures the confidentiality and will be used throughout the exercise, i.e. it will allow you to identify your data in the final report of the exercise.

Reporting your results

For analysing the samples, we ask you to follow the routine procedure as practised in your laboratory. You are kindly requested to provide detailed information on the measurement technique applied as well as on the calibration methods used (measurement scale, standardisation procedure, half-life value, etc.). Please also note the **Annex** providing information on the newly determined tritium half life and its implications for future tritium work.

We kindly ask you to carefully complete the **Reporting Sheet**. We would ask you to submit the results by e-mail in order to avoid typing errors and to simplify the evaluation procedure. We will send the electronic version of the Reporting Sheet (tric2000.doc, MicrosoftWord 6.0 for Microsoft Windows) as an e-mail attachment. In case you do not use Microsoft Windows or/and Microsoft Word the Reporting Sheet will also be sent to you as plain text by e-mail

(ASCII). In addition, both electronical versions of the Reporting Sheet are available for download on our Internet site.
You are kindly requested to fill out the electronic version and send it back to us **by e-mail** (Word6.0-file attachment preferred). For your convenience, a printed copy of the Reporting Sheet is included in the sample shipment. The deadline for sending back the results will be December, 31th.

Statement of uncertainty

Please give a careful statement of the uncertainty of your data. The uncertainty should not only include the standard uncertainty of the individual measurement (counting). It rather means an assessment of all uncertainty components of the complete analysis procedure including all steps. For details please refer to the **draft paper by Rozanski & Gröning** which will be shortly sent to you by email and will be available to download from our Internet site.

Evaluation of the results/Quantitative Assessment

The exercise is designed as a proficiency test. All samples were prepared by dilution of a NIST water standard (NIST 4927F) by tritium-free water. The assigned value of each sample (the best estimate of the "true" activity) is the tritium activity as calculated from the dilution procedure.
For evaluation of the results we will use the following procedure:
(i) The submitted data and uncertainties will be compared to the assigned value allowing a direct assessment of the performance of each laboratory.
(ii) In a second approach, a two-stage statistical treatment which has been adopted in previous IAEA interlaboratory exercises will be applied to the population of the submitted results allowing the identification of obvious outliers (stage 1) and examination of the deviation of the individual results from the mean value of all laboratories.

If you have any questions concerning the exercise or the completion of the report sheet, please do not hesitate to directly contact us. Information related to the exercise as well as documents for download will be provided on our Internet site at **http://www.iaea.org/programmes/rial/pci/isotopehydrology/ic.htm.**

We understand that participating in the exercise means a lot of work for you and, therefore all the more are grateful for your kind co-operation;

Yours sincerely,

G. Winckler M. Gröning

Annex: Implication of the newly determined tritium half-life for future tritium work

ANNEX

Implication of the newly determined tritium half-life for future tritium work

It is highly desirable that all laboratories participating in the exercise should aim to produce data which are consistent with that of other laboratories, i.e. to express their results on the same measurement scale.

In the past, the main reference has been a scale based on the NIST standard water NBS 4926 C (calibration date 3 September 1978) which was proposed by C.B. Taylor and W. Roether in 1982 [1]. As the stock of this NIST standard generation is now depleted, the US National Institute of Science and Technology has prepared a new set of standard waters (SRM4926E, SRM4927F from September 1998, [2]). Furthermore, NIST has re-evaluated all tritium half—life determinations and has provided a new half-life value for tritium of 4500 ±8 days [3].

On the basis of these new developments, the IAEA Isotope Hydrology Laboratory recommends **for future work** to adopt the new NIST standard generation as the new scale for low-level tritium work and to use the new value for the tritium half-life. In this scale, 1TU is 0.11919 ± 0.00021 Bq/kg H2O.

Compatibility of older data (based on an old scale) with the new standard scale

Calibration of the data on the new scale leads to slightly but significantly different values. However, we would like to point out that adopting the new scale means a complete re-calibration of all standard solutions used in the laboratory [2]. For consistency reasons, only laboratories that completely re-calibrate their standard procedures should use the new conversion equation given above. For details concerning the adjustment to the new scale please contact us directly. A short note on this issue is under preparation by C.B. Taylor and M. Gröning.

References

[1] C.B. Taylor and W. Roether (1982). A uniform scale for reporting low-level tritium measurements in water. Applied Radiation and Isotopes, Vol. 33, 377-382.

[2] L.L. Lucas (2000). Massic activity ratios of the NBS/NIST tritiated-water standards issued between 1954 and 1999. Journal of Research of the National Institute of Standards and Technology, Vol. 105.

[3] L. L. Lucas and M.P. Unterweger (2000). Comprehensive review and critical evaluation of the half-life of tritium. Journal of Research of the National Institute of Standards and Technology, Vol. 105.

E3: Data Reporting Sheet (attached to shipped samples):

Sixth IAEA Intercomparison Exercise of Low-Level Tritium Measurements in Water - TRIC2000

Reporting Sheet

Participant Information

Participating Laboratory	**Lab ID** (identical to the number on the sample bottles)

Method applied

☐ Liquid scintillation counting
☐ gas proportional counting
☐ He3/T ingrowth (no need to follow the query below)

Standardization/Calibration

Which standard do you use ? (e.g. from NIST, PTB, IRMM, other; Please specify name, activity and certification date)	
Which lab standard do you use?	
How was your lab standard prepared?	
Please specify the dilution procedure	
Which tritium half-life value do you use for calculations of tritium activities	

General Lab information

Elevation of the laboratory [in m above sealevel]	
Counter location [ground floor/sub floor/etc.]	
Enrichment procedure used? [Y/N]	
Number of analyses per year	
Range of typ. tritium values measured	

Description of applied procedures

First destillation

Predistillation of samples [Yes/No]	
If yes, distillation at atmospheric pressure or under vacuum	
Conductivity after pre-destillation [microS/cm]	
Remarks:	

Enrichment

Which kind of anode do you use (e.g. steel)	
Which kind of kathode do you use (e.g. Nickel)	
Number of cells	
Volume of the electrolytic cell [ml]	
Initial volume of sample water [ml]	
Final volume of sample water [ml]	
Temperature during enrichment [C]	
Coolant medium during enrichment	
Which type of electrolyte do you use ? (e.g. NaOH, Na_2O_2)	
Which amount of electrolyte [g] ?	
how long do you run the electrolysis? [hours]	
Which current do you apply [A] if different sequences, please note	
Total charge used [Amperehours]	
Number of enrichment runs [per year]	
Average enrichment factor	
How do you determine the enrichment factor?	
Remarks:	

Second destillation and Neutralization

neutralization after enrichment [Yes/No]	
Type of chemical added [e.g. PbCl2]	
Amount of chemical added [g]	
Final distillation [Yes/No]	
If yes, distillation at atmospheric pressure or under vacuum	
Remarks:	

Counting

For Laboratories using proportional counters

How do you prepare the hydrogen gas (e.g. reduction on Mg turnings)	
temperature during reduction process	
which counting gas do you use?	
Geometric volume of prop counter [l]	
Effective Volume of counter [l]	
Voltage applied [V]	
pressure [atm]	
Temperature during counting [C]	
Counting time [min]	
Counting efficiency [%]	
Calibration factor [TU/cpm]	
Background countrate [cpm]	
Used background water (origin etc.)	
Detection limit [TU]	

For laboratories using liquid scintilation counters

Type/Model of counter	
Type of vials (e.g. glass, polyethylene)	
Volume of the counting vials [ml]	
Volume of water in the vial [ml]	
Type of scintillation cocktail	
Amount of scintillation cocktail	
Quench indicating parameter	
Counting efficiency for an	

unquenchend standard [%]	
Counting efficiency for a quenched standard [%]	
Calibration factor [TU/cpm]	
Temperature during counting [C]	
Counting time [min]	
Sequential/cycle counting or in block?	
If sequential, specify sequences	
Composition of each enrichment run, please specify how many samples, blanks, spikes and standards per run	
Background countrate [cpm]	
Used background water (origin etc.)	
Detection limit [TU]	

Uncertainty Assessment

How do you determine the uncertainty of your tritium results?	
Which uncertainty components contribute to your over all error?	
Uncertainty of enrichment factor [%]	
Uncertainty of background countrate [%]	
Uncertainty of standardization [%]	
Uncertainty of other components	

Measurement

Please read this section carefully before you fill out the results table.

1. Please calculate the tritium values for the date of sample collection (July, 1st of 2000).
2. Please express your results in TU. If you use other units (e.g., Bq/kg), please specify it!
3. Please present your numerical TU values and errors even if they are negative and/or below the usually stated limit of detection. Negative numbers can occur because the net tritium count rate is in principle the difference between the count rate of the sample and that of a tritium-free sample (background count or blank sample). The negative values should be reported as such and will allow us an unbiased statistical treatment of sets of the data. Please do NOT give any numbers using the < symbol (e.g., < 1 TU) as these data cannot be used in the statistical analysis!
4. Please give the uncertainty of your results as standard uncertainty at 1 sigma-level
5. If you perform **multiple analyses** of the samples, please report in Table 1 the **mean value** and specify the number of analyses in the respective column in Table 1.
6. Please supply the results of the multiple measurements in the extra table below.

Final Results

Code	Tritium activity [TU]	Standard uncertainty (one sigma)	date of measurement	Number of Analyses
T1				
T2				
T3				
T4				
T5				
T6				

Used Half-Life:
If you used any conversion equation (e.g., from Bq/kg to TU), please specify:

In case you performed multiple measurement of the samples please add the results of the individual measurements here

Individual Results of multiple analyses

	T1	T2	T3	T4	T5	T6
1.						
2.						
3.						

Laboratory Manager	
Date	

Please send the Reporting Sheet (electronic version strongly preferred) to:

email: isotope.hydrology.lab@iaea.org

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TRIC2000
International Atomic Energy Agency
P.O. Box 100
A-1400 Vienna
Austria
Tel: +43-1-2600 21766
Fax: +43-1-2600721763

E4:Problem-indicating email to 26 laboratories

After the deadline had passed the submitted data were sreeened and obvious outlying measurements identified. Emails similar as stated below were sent to altogether 26 laboratories. 20 replies and 12 corrected datasets were received

Evaluating your TRIC2000 report we found
the T6 value out of range
Please check your calibration.
Best regards,
Isotope Hydrology Laboratory
International Atomic Energy Agency
P.O. Box 100
A-1400 VIENNA
Austria

Evaluating your TRIC2000 report we found
the T1,T2,T3 and T5 value out of range
Please check for offset.
Best regards,
Isotope Hydrology Laboratory

Evaluating your TRIC2000 report we found
the T1,T4,T5 and T6 values out of range
Please check your calibration.
Best regards,
Isotope Hydrology Laboratory