

# **SEVENTH IAEA INTERCOMPARISON OF LOW-LEVEL TRITIUM MEASUREMENTS IN WATER (TRIC2004)**

## **REPORT**

**prepared by**

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### 1. INTRODUCTION

It is now 40 years since the first tritium interlaboratory comparison exercise of the International Atomic Energy Agency (IAEA) was published. Six large tritium intercomparison exercises were conducted previously by the IAEA [1-6]. Basic details of these interlaboratory comparisons and of the recent one are summarized below in table 1.

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 exercise of 2000 as in the present one (code-named TRIC2004), samples were prepared by gravimetric dilution of the tritiated standard water SRM4927F with virtually tritium-free water. That reduced the calculated uncertainty for prepared samples and allows for the data evaluation to concentrate fully on the laboratory performance without added interpretation ambiguities due to sample preparation effects.

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

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Year	Number of final participants, (no. of laboratories receiving samples)	Reference date	Water samples distributed, tritium activity and standard uncertainty [7] at 1 $\sigma$ - level at chosen reference date
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 $\pm$ 0.04 TU C: 33.1 $\pm$ 0.1 TU D: 678.5 $\pm$ 2.2 TU
1985	57 (85)		A: < 0.2 TU B: 5.94 $\pm$ 0.03 TU C: 25.66 $\pm$ 0.13 TU D: 100.2 $\pm$ 0.5 TU
1994	57 (91)		A: ~ 0 TU B: ~ 1 TU C: ~ 5 TU D: ~ 19 TU
2000	86 (102)	2000-07-01	T1: 10.112 TU +0.073 / -0.037 TU T2: 2.152 TU +0.044 / -0.008 TU T3: 0 TU +0.036 / -0 TU T4: 5.252 TU +0.055 / -0.019 TU T5: 26.118 TU +0.131 / -0.095 TU T6: 505.83 TU +1.88 / -1.84 TU
2004	70 (74)	2004-07-01	T7 0 TU +0.050 / -0 TU T8 10.920 TU +0.064 / -0.040 TU T9 1.738 TU +0.050 / -0.006 TU T10 5.433 TU +0.054 / -0.020 TU T11 25.408 TU +0.105 / -0.092 TU T12 1011.58 TU +3.68 / -3.68 TU T12 1011.58 TU +3.68 / -3.68 TU

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 of laboratories was kept since 1975.

Participation in IAEA inter-comparison 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.

Table 1 reveals a continuously high number of laboratories measuring tritium at environmental levels. This is remarkable 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. Tritium levels in the southern hemisphere are only marginally above pre-bomb levels. These low-level concentrations impose much greater technical difficulty in measurement than two decades ago. It is therefore important to judge from the present 7th inter-comparison exercise how well laboratories have improved their sensitivity and accuracy.

In 2000, the US National Institute of Standards and Technology (NIST) published simultaneously the results of a re-analysis of its tritium standards [8, 9] as well as a comprehensive review and critical evaluation of the tritium half-life [10]. The coincidence of these two projects allowed all the scale parameters to be readjusted simultaneously. This is believed to be the best possible data set to end the continuing diversity of standardization applied in different laboratories. The results demonstrated that the previously adopted tritium half-life was too long, and evaluated a new value for the tritium half-life of  $4500 \pm 8$  days. This value is strongly recommended to be used by all laboratories. It is also the basis for calculations in this and the previous exercise TRIC2000 [6].

74 laboratories declared their willingness to participate in this 6th interlaboratory comparison TRIC2004 and have received the set of six water samples end of 2004. Results on their tritium activity have been received in 70 independent data sets from 67 laboratories during 2005. This is the highest relative response rate ever in tritium interlaboratory comparisons organized by the IAEA.

Six samples of different tritium activity were initially prepared at the Agency's laboratory. One was a virtually tritium-free water, collected from the artesian well G3 at Grafendorf (near Graz, Austria), located in the same well nest as the well G4 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 with water from the artesian well Grafendorf G3, as described below.

## 2. SAMPLE PREPARATION

As it 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. In light of the new robust tritium half-life assessment [10], and the associated re-evaluation of all NIST tritiated water standards [9], we based all calculations of reference tritium values and their uncertainties on the newly determined half-life  $4500 \pm 8$  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% (the dominant contributor); (b) the small uncertainty in correcting the values to our reference date (1 July 2004) using the half-life  $4500 \pm 8$  days; (c) the small uncertainty of our dilution procedures; (d) an maximum tritium activity offset for each sample by assuming a residual tritium offset in the used “tritium-free” water sample T7, which was used in preparing the other five samples. As explained below, this upper limit was derived from a statistical analysis of the submitted high-precision results for that sample.

Throughout this report, the results of 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, the latter is is not a “unit” but an atom ratio and may be referred to more rigorously as tritium ratio [11]. The tritium ratio of 1 TU corresponds to a tritium/(total 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  $8.390 \pm 0.015$  TU [12, 13], using the new value for the tritium half-life [10] and the CODATA 1998 value for the Avogadro constant [14].

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. The necessary adjustments are discussed elsewhere [6, 11].

### 2.1 Massic Activity of SRM4927F at Reference Date

NIST’s calibration certificate gives the massic activity of SRM4927F as  $634.7 \text{ kBq}\cdot\text{g}^{-1}$  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  $4500 \pm 8$  days evaluated by NIST to calculate the massic activity at our intercomparison reference date (1 July 2004). 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  $2.378 \times 10^{-5} \times t$ , where t is the elapsed time in days; this is only 0.05% at our reference date 1 July 2004 (2128 days). The massic activity of SRM 4927F at that date is  $457.3 \pm 1.7$  kBq.g<sup>-1</sup>.

The website <http://srmcatalog.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 during a previous interlaboratory comparison exercise to obtain a daughter water of appropriate tritium activity to serve for common laboratory purposes [6](see Table 4 therein for details). The diluting water was a de-ionised sample from the artesian well G4 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.

In the recent exercise the second dilution “D2-2000” was used to spike tritium free water T7 to produce the other five samples. In this manner the samples T8, T9, T10, T11 and T12 were produced. Sample T7 was taken in June 2004 from the artesian well G3 in Grafendorf, Austria. It served 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 (T7)

As a first step of any evaluation for the prepared samples, the tritium activity of the used dilution water has to be determined. Such a determination is not easy, since of course all efforts have been taken to use a water sample without any residual tritium activity. The artesian well Grafendorf G3 showed no significant tritium activity in high sensitive tritium measurements performed at the IAEA Isotope Hydrology Laboratory ( $0.02 \pm 0.08$  TU, see lab 10b). Together with the relatively low <sup>14</sup>C activity of  $45.4 \pm 0.8$  pMC and the high helium contents, especially the absence of any detectable CFC-11, CFC-12 and CFC-113 concentrations in triplicate samples of G3 is a strong indicator in support of the detected lack of tritium.

The final test for any residual activity of the dilution water is provided by the results of the participating laboratories for sample T7 (equaling Grafendorf G3). The procedure used all reported results with a stated uncertainty smaller than  $\pm 0.25$  TU, which were considered as high precision data. The results were evaluated using the same two step

statistical evaluation as in former tritium interlaboratory comparison exercises. In Table 2 the results of the evaluation are presented.

Table 2: Average tritium concentration for the used “tritium-free” water T7 from well Grafendorf G3 evaluated from results of high precision laboratories with stated uncertainties below 0.25 TU.

Step	Mean [7]	Stddev [7]	no. of labs	Std.error of the mean [7]	Weighed Mean [7]	Weighed Stddev [7]
Raw data	0.185	0.417	30(30)	0.076	0.180	0.375
Stage I	0.091	0.220	28(30)	0.042	0.106	0.189
Stage II	0.043	0.127	26(30)	0.025	0.045	0.040

During the evaluation process, four of thirty results were rejected. The resulting 26 results provided a mean value of 0.043 TU, which is indistinguishable from zero at a  $2\sigma$ -level of its standard error of the mean (0.025 TU).

Therefore a value of 0 TU was assumed for the dilution water used for preparation of all other five water samples (“tritium free background water”). Its upper level  $1\sigma$  uncertainty was calculated to 0.050 TU by taking the square root of squares of the standard error of the mean and the calculated mean value.

#### 2.4 Reference Values for the Prepared Samples T7 to T12

The five distributed samples T8 to T12 were prepared by spiking the sample water T7 with carefully weighed amounts of gravimetric dilutions of a primary tritium standard (NIST SRM 4927F). All sample tritium activities were corrected for the contents of total dissolved solids in the T7 water ( $262 \pm 7$  mg/kg), so the tritium activity values calculated from the dilution factors were multiplied with 1.000262. The reference values are presented in Table 3.

Table 3: Tritium activity of the six prepared samples for the reference date 1 July 2004, as calculated from gravimetric dilutions (T11 and T12: last digit not significant, included only for calculation purposes). ese= estimated standard error of the mean.

Sample name	Tritium activity [7]	Total combined lower side standard uncertainty [7]	Total combined upper side standard uncertainty [7]	Uncertainty of the standard dilution [7]	Offset of background tritium activity [7]	ese of the background tritium activity [7]
T7	<b>0</b>	<b>0</b>	<b>0.050</b>	n.a.	0.043	0.025
T8	<b>10.920</b>	<b>0.040</b>	<b>0.064</b>	0.040	0.043	0.025
T9	<b>1.738</b>	<b>0.006</b>	<b>0.050</b>	0.006	0.043	0.025
T10	<b>5.433</b>	<b>0.020</b>	<b>0.054</b>	0.020	0.043	0.025
T11	<b>25.408</b>	<b>0.092</b>	<b>0.105</b>	0.092	0.043	0.025
T12	<b>1011.58</b>	<b>3.68</b>	<b>3.68</b>	3.68	0.043	0.025

## 2.5 Data Evaluation Procedure

All data evaluations are based on the procedure as adopted for earlier interlaboratory comparison (IC) exercises organized by the IAEA ([5, 6, 15-18]).

A three step evaluation is performed as follows:

Step Zero: Obvious outlier laboratories with offsets from the mean values larger than ten times a generally acceptable limit for adequate precision are excluded from the evaluation (here only lab 67 with offsets from 100 to more than 600 TU from the true value), in order not to disturb a meaningful statistical evaluation.

The two other steps are based on a separate assessment for each of the samples.

Stage I: Using an interquartile criterion, the 25% and 75% percentiles are evaluated and the range between them used as outlier rejection criterion. All those results are marked as outliers, which are more than 2.58 times the interquartile range outside of the two percentile values. This quite conservative criterion removes only the obvious outliers and takes a similar role as the initial expert judgment for outlier removal. From the remaining accepted data set a provisional mean is evaluated.

Stage II: For each of the six samples the reference tritium value (Table 2) is taken as reference and the individual laboratory results compared against it using the individual stated uncertainties from each laboratory. A laboratory value is rejected if its offset from the reference value is larger than the combined uncertainty at a 2.58 sigma level of the two individual uncertainties combined according to the law of error propagation. This criterion replaces the formerly used procedure of simple addition of the two uncertainties at the 2 sigma level (see all former exercises, especially TRIC2000) and provides comparable results with slightly (20%) more relaxed acceptance criteria. This results in a higher percentage of acceptable results compared to earlier exercises, but does not significantly changes conclusions drawn by the exercise for individual laboratories.

The new approach is harmonized with the evaluation of interlaboratory comparison exercises performed by other groups at the IAEA.

All calculations were performed with the software ICE (InterComparisonEvaluation, version 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.

### 3. PRESENTATION OF RESULTS

#### 3.1 Individual Laboratory Data Display

The submitted results are listed in Table 4 in Appendix B, with columns listing the laboratory identification number, the reported tritium ratio and reported measurement uncertainty at  $1\sigma$ -level in TU for the six samples and the used measurement method. Results are ordered according to laboratory code number. Used measurement methods include Liquid Scintillation Counting, Gas Proportional Counting and  $^3\text{He}$  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.

Additionally, results for each sample are depicted graphically, laboratories sorted 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 2. Error bars are added at 2 times the submitted uncertainties. Outliers are visually identified (open markers instead of filled ones). The laboratory code numbers (Lab ID) appear along the x-axes.

Due to the new evaluation procedure ( $2.58\sigma$  of combined sample-reference uncertainties) some results are accepted despite  $2\sigma$  uncertainty bars not overlapping with those of the reference.

All the S-plots are asymmetric around the reference values and their uncertainty limits, with more results on the high side. Because of this asymmetry of the S-plots, average results for the samples calculated from the whole population of laboratories are of limited usefulness, and are therefore not reported here.



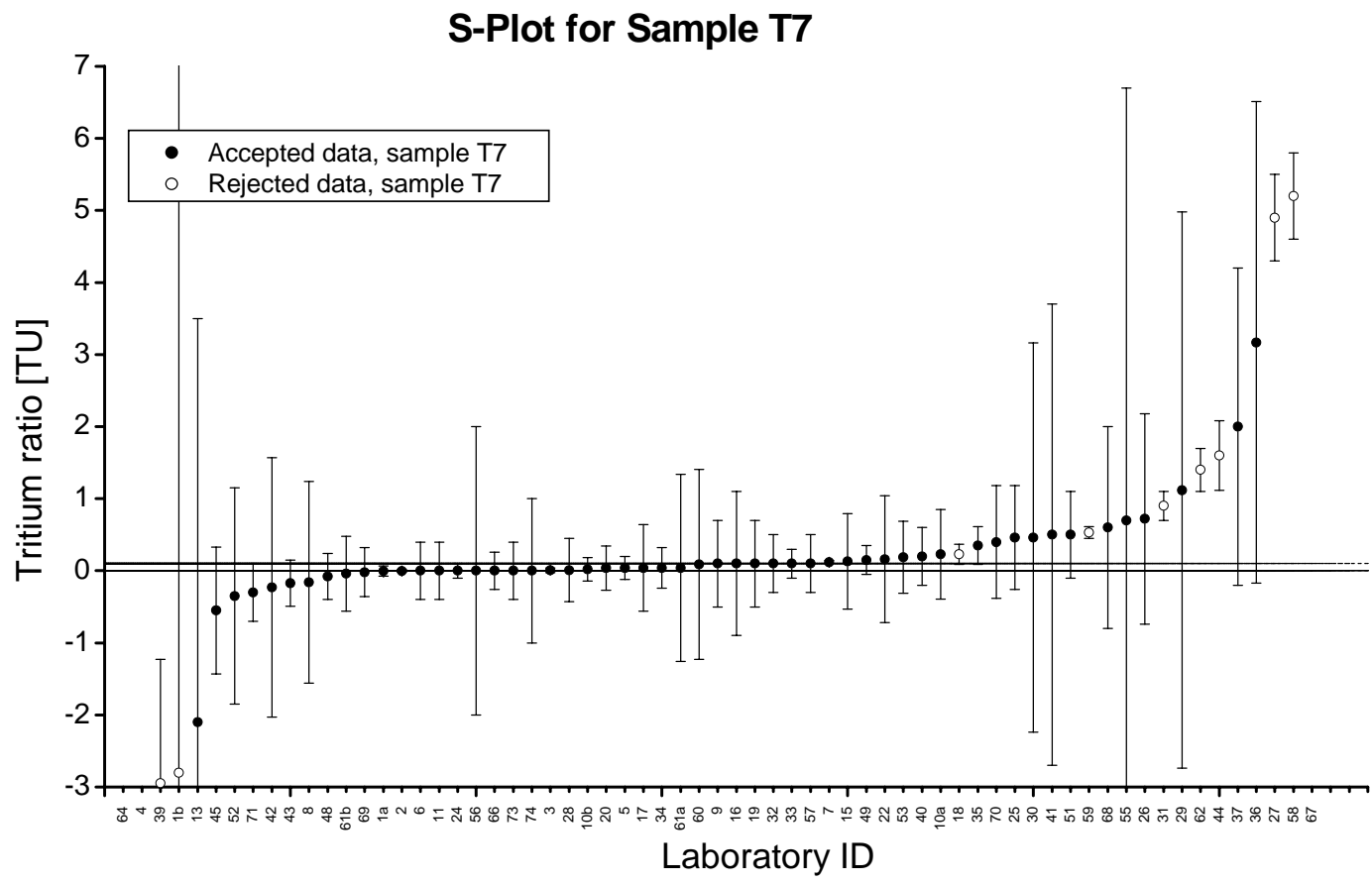


Fig.1: S-plot for sample T7. Laboratory results at the utmost left and right sides are outside the chosen scale. Rejected values are marked with open symbols.

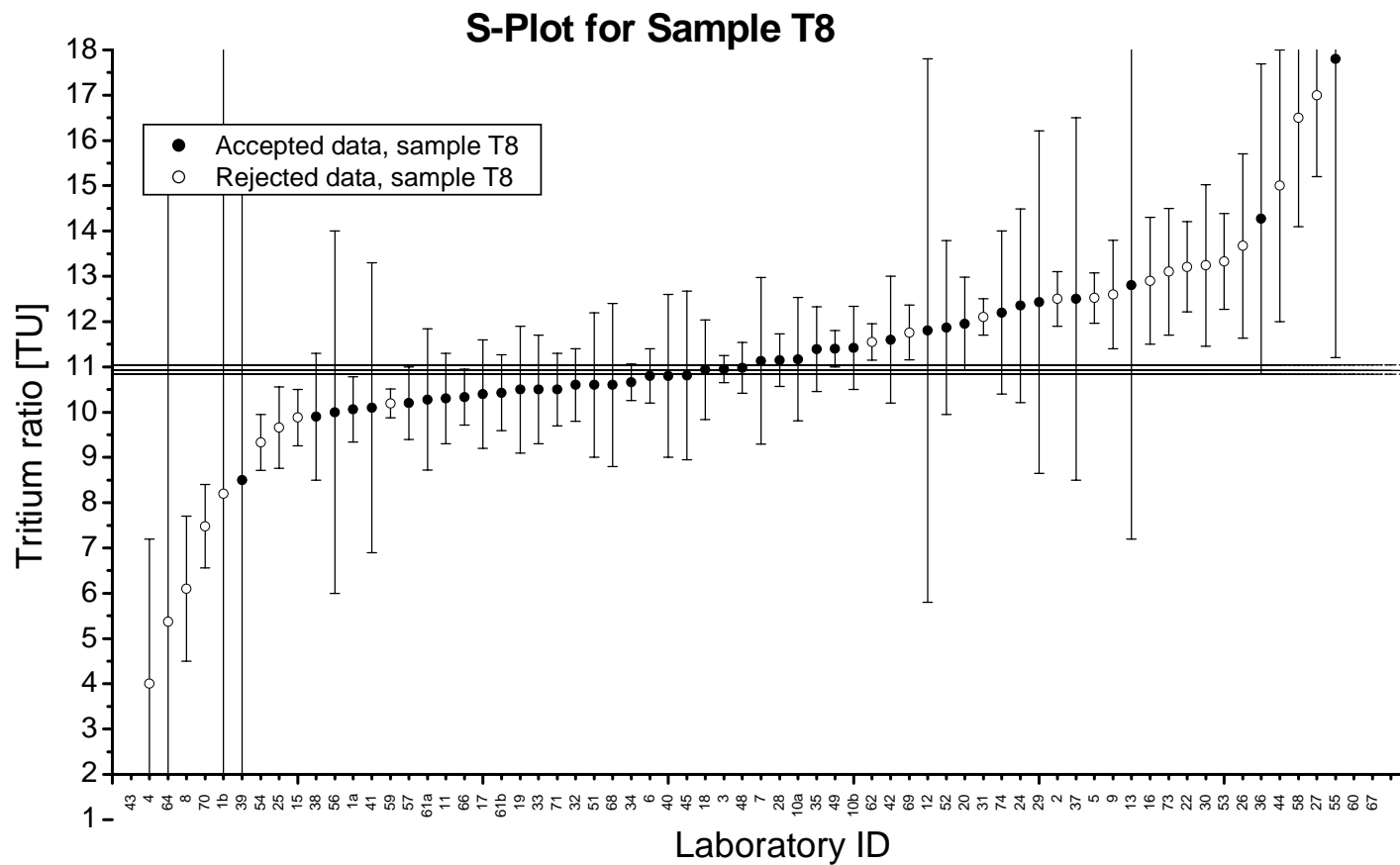


Fig.2: S-plot for sample T8. Laboratory results at the utmost left and right sides are outside the chosen scale. Rejected values are marked with open symbols.

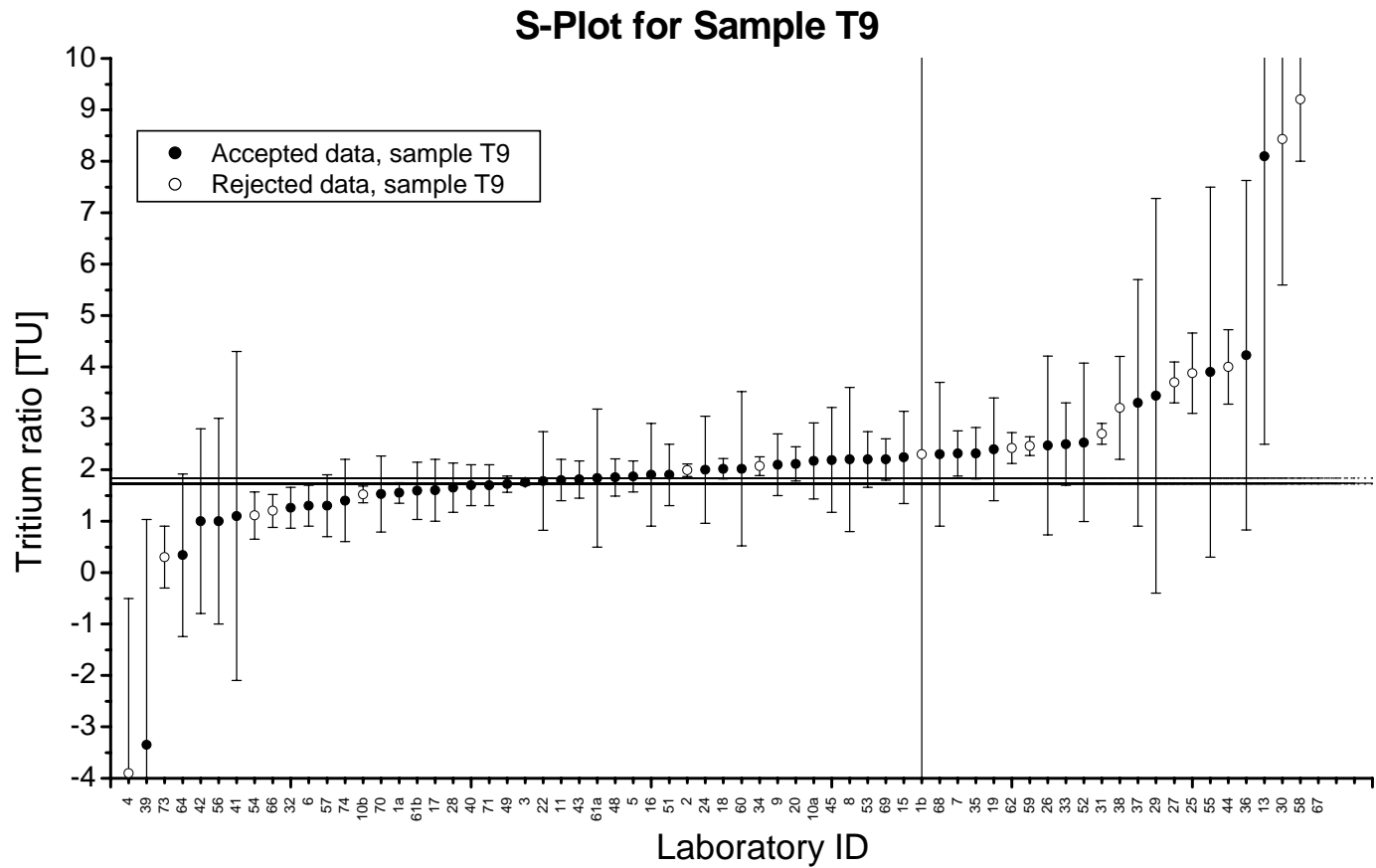


Fig.3: S-plot for sample T9. Laboratory results at the utmost left and right sides are outside the chosen scale. Rejected values are marked with open symbols.

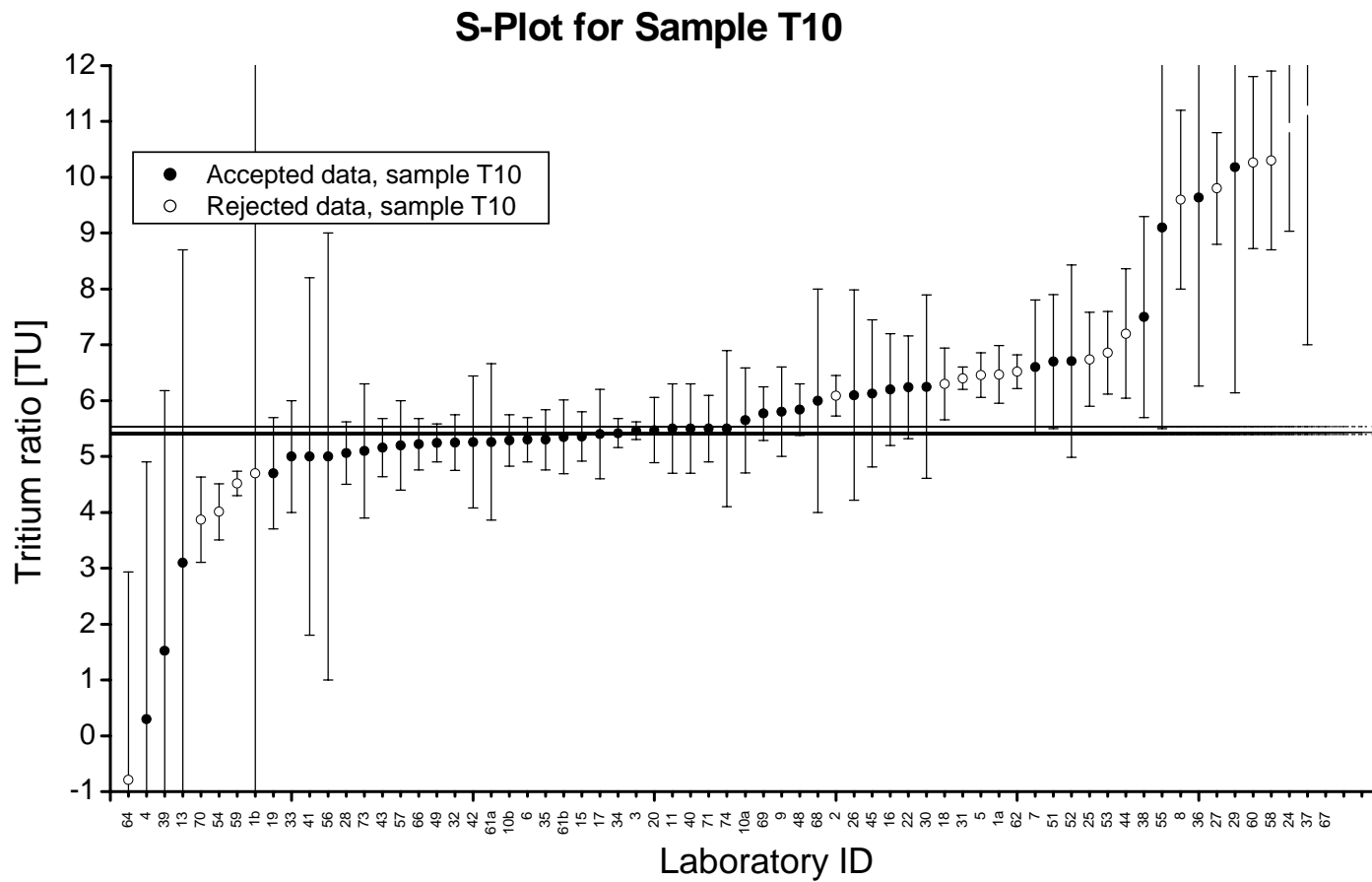


Fig.4: S-plot for sample T10. Laboratory results at the utmost left and right sides are outside the chosen scale. Rejected values are marked with open symbols.

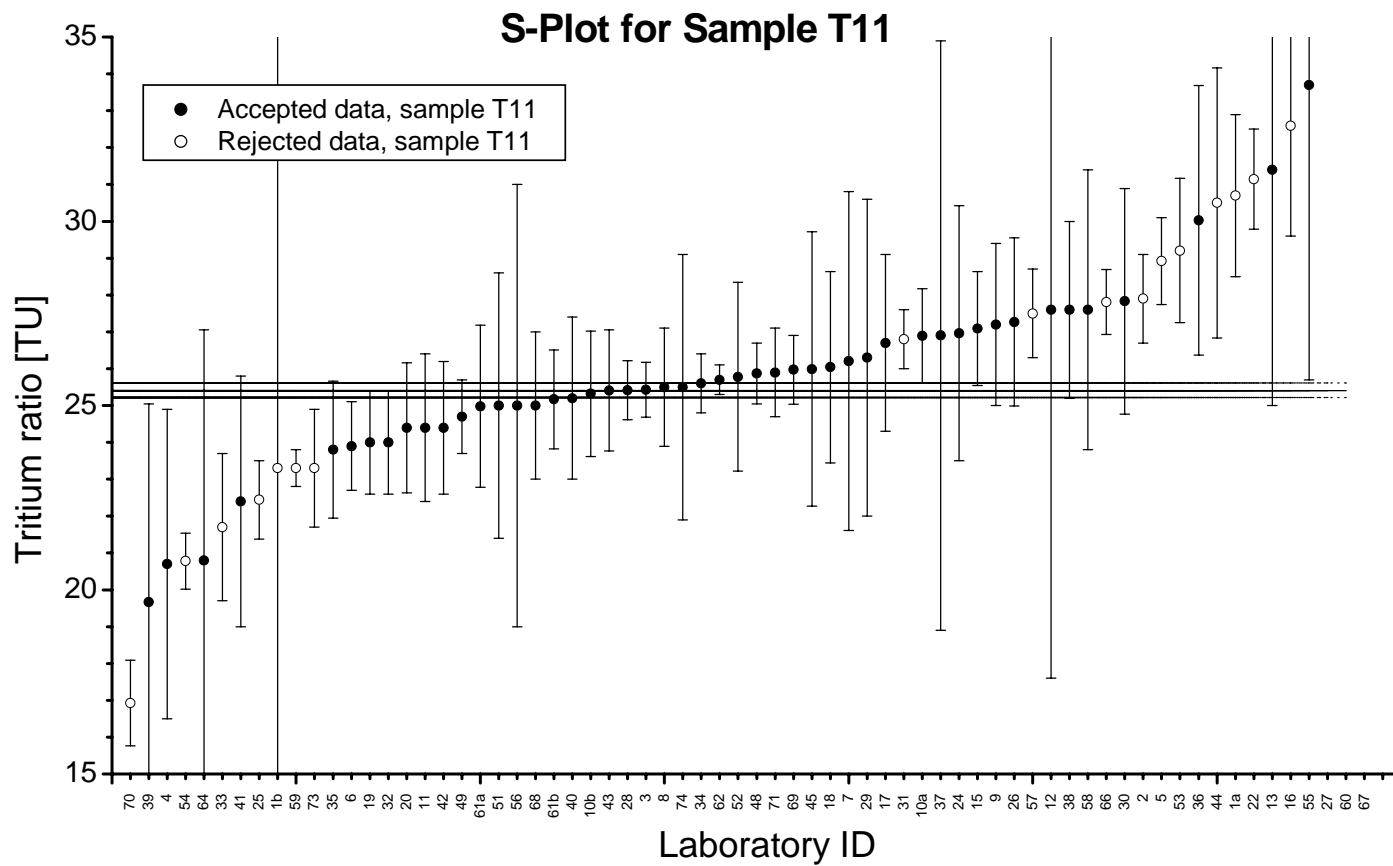


Fig.5: S-plot for sample T11. Laboratory results at the utmost left and right sides are outside the chosen scale. Rejected values are marked with open symbols.

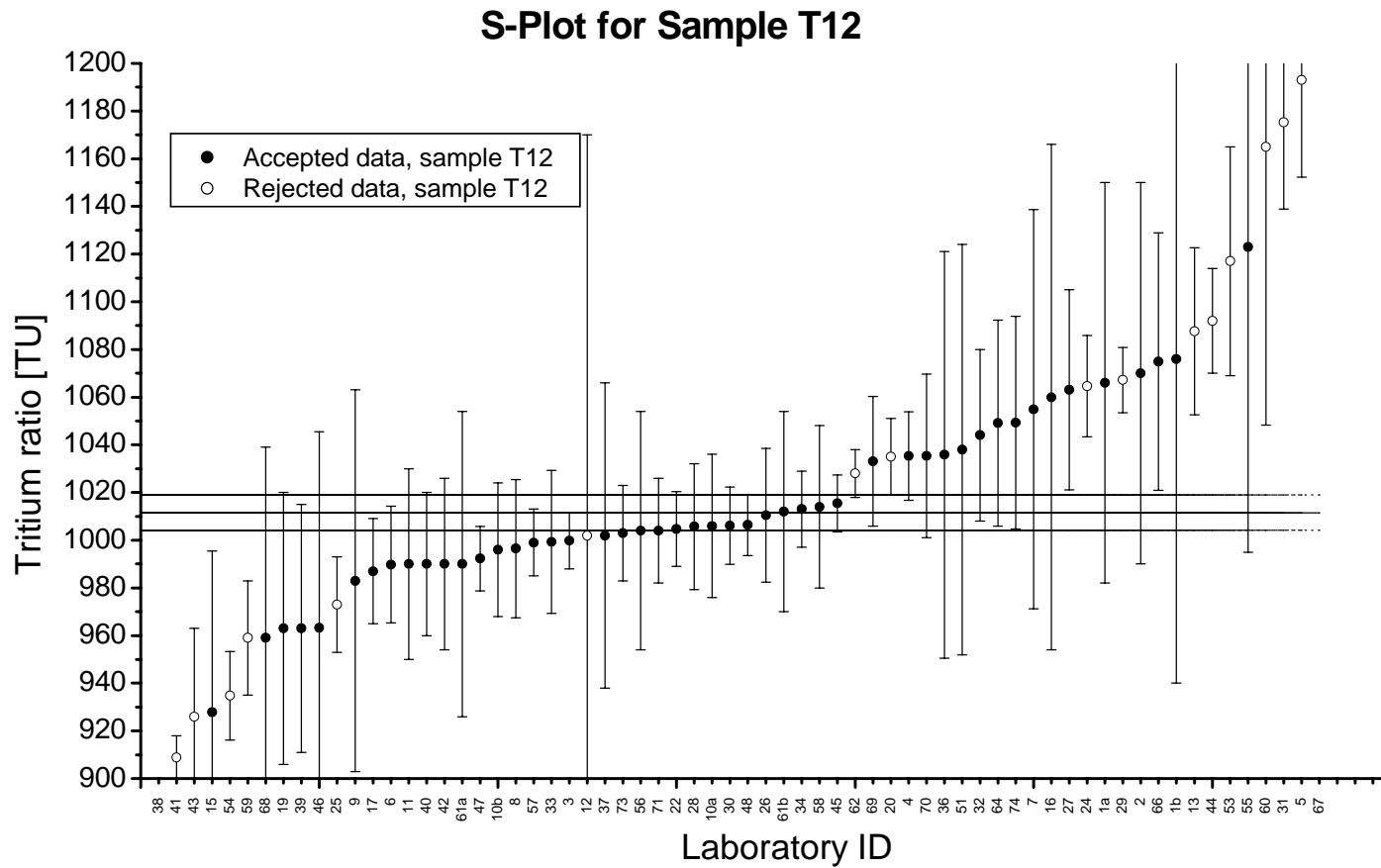


Fig.6: S-plot for sample T12. Laboratory results at the utmost left and right sides are outside the chosen scale. Rejected values are marked with open symbols.

### 3.2 Implications of Results for Sample T7

Compared to the last tritium intercomparison exercise (TRIC2000), much less laboratories showed effects of tritium contaminations, as visible by high tritium values for the tritium free sample T7.

64 out of the 65 tritium results for sample T7 could be evaluated. From this data set only 6 results (9%) were significantly on the positive side, while 3 more were significantly too negative. This is much less than the 15 (20%) positive outliers of the last exercise and is a quite positive development for the group of participating laboratories.

The average value evaluated from 61 laboratory results was  $0.00 \pm 0.16$  TU. This value, however, is considerably biased by a series of measurements without tritium enrichment and a much higher uncertainty. Beside the 5  $^3\text{He}$  laboratories, 14 other participating laboratories did not use any tritium enrichment.

### 3.3 Results for the High Level Sample T12

This sample can be measured accurately by direct gas or liquid scintillation counting, and was intended to be measured in this way by the participating laboratories. Although the spread of the submitted results can be slightly greater due to the various used calibrating standard waters and half-lives, it should be much easier to achieve conformity with the reference value than for samples requiring tritium enrichment procedures. However, as in previous intercomparisons, a disappointingly high number of laboratories submitted results which were gross outliers. From 66 provided results, 18 (27%) had to be identified as outliers. 14 of these applied liquid scintillation counting, and 2 used the  $^3\text{He}$  method. 5 results were on the low side (range 117 – 959 TU) and 9 were too high (1064 – 1397 TU). 4 further outliers are close to the reference value, but indicate problems with measurement precision by the laboratories concerned.

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. For at least 19 laboratories the used standards seem not to be appropriate according to the information given. Reference is made to the discussion of this topic in the TRIC2000 report [6].

Only 13 out of 67 reported tritium half-life values used the old outdated 4540 days, and the majority used the new recommended value of  $4500 \pm 8$  days or similar. This is a substantial improvement versus the situation during the last exercise.

### 3.4 Comparison of Reported Uncertainties

To enable laboratories to judge how their reported precision compares with that of other participating laboratories, two distributions of the reported uncertainties were compiled, one for samples T7 to T11 (Fig.7) and one for sample T12 (Fig.8).

Several parameters, which are highly variable among laboratories, have to be considered in assessing 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) [19];
- (b) longer counting times (accumulation times for  $^3\text{He}$ ), 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.

In Fig. 7, for each laboratory the cumulative reported uncertainties for five samples at environmental tritium levels are stated. In cases where less than five samples were measured, the number of samples is indicated in brackets at the laboratory ID number. The large increase of reported uncertainties towards the right side of Fig.7 indicates the direct counting laboratories without using tritium enrichment.

For sample T12, all laboratories should have measured the sample in similar manner directly without tritium enrichment (Fig.8). There is no obvious reason for the largely discrepant results except the use of liquid scintillation analysers in non low-level counting mode.



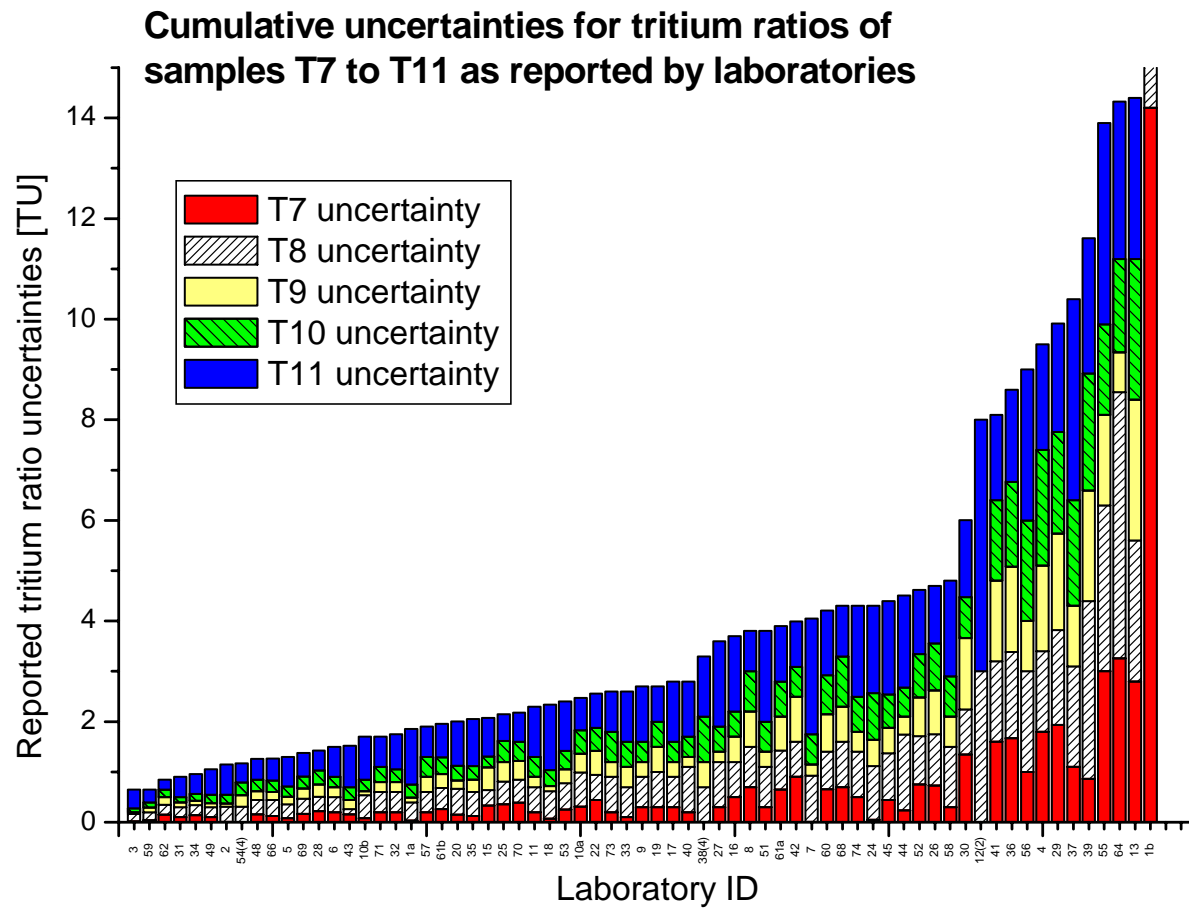


Fig.7: Cumulative uncertainties for samples T7 to T11 as reported by laboratories in increasing order. For laboratories with less than 5 analysed samples (biasing the ranking), the respective number is added in brackets to the Lab ID.

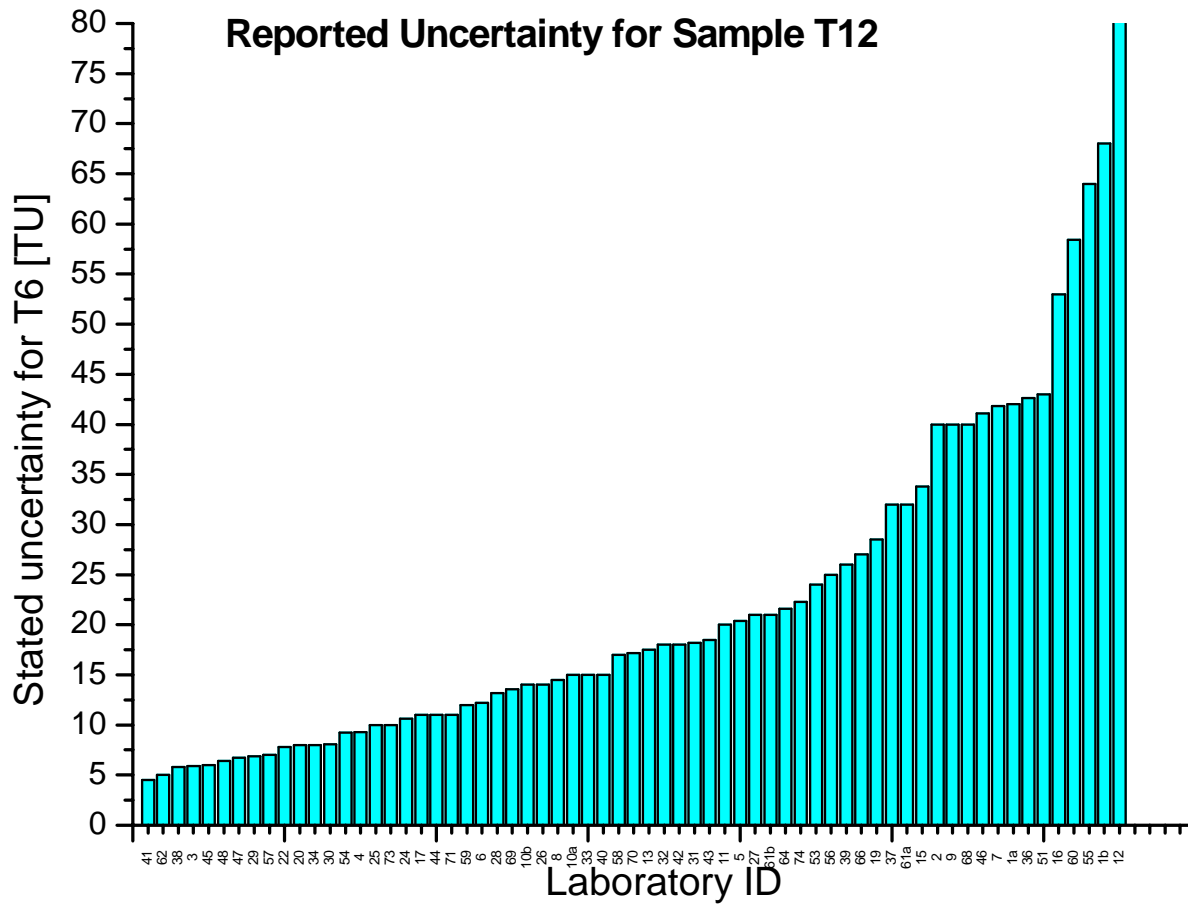


Fig.8: Reported uncertainty for sample T12 as stated by laboratories.

### **3.5 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.

#### **3.5.1 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 T7 to T11 for each laboratory is shown in Fig. 9 (sorted according to the 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 TRIC2004.

Several features can be derived from Fig. 9. The sum of deviations from the reference value ranges from 0.1 TU (lab 3) to 50 TU (lab 60), not considering lab 67, which was totally excluded from evaluation due to its huge offset. 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 less than 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 T7 to T11. Those 3 laboratories 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 T12 and the commonly used direct measurement approach without enrichment and with much higher uncertainties, the deviations from the reference tritium ratio for sample T12 are presented separately in Fig.10. In this figure, the laboratories are sorted from most negative deviation to most positive deviation of sample T12.

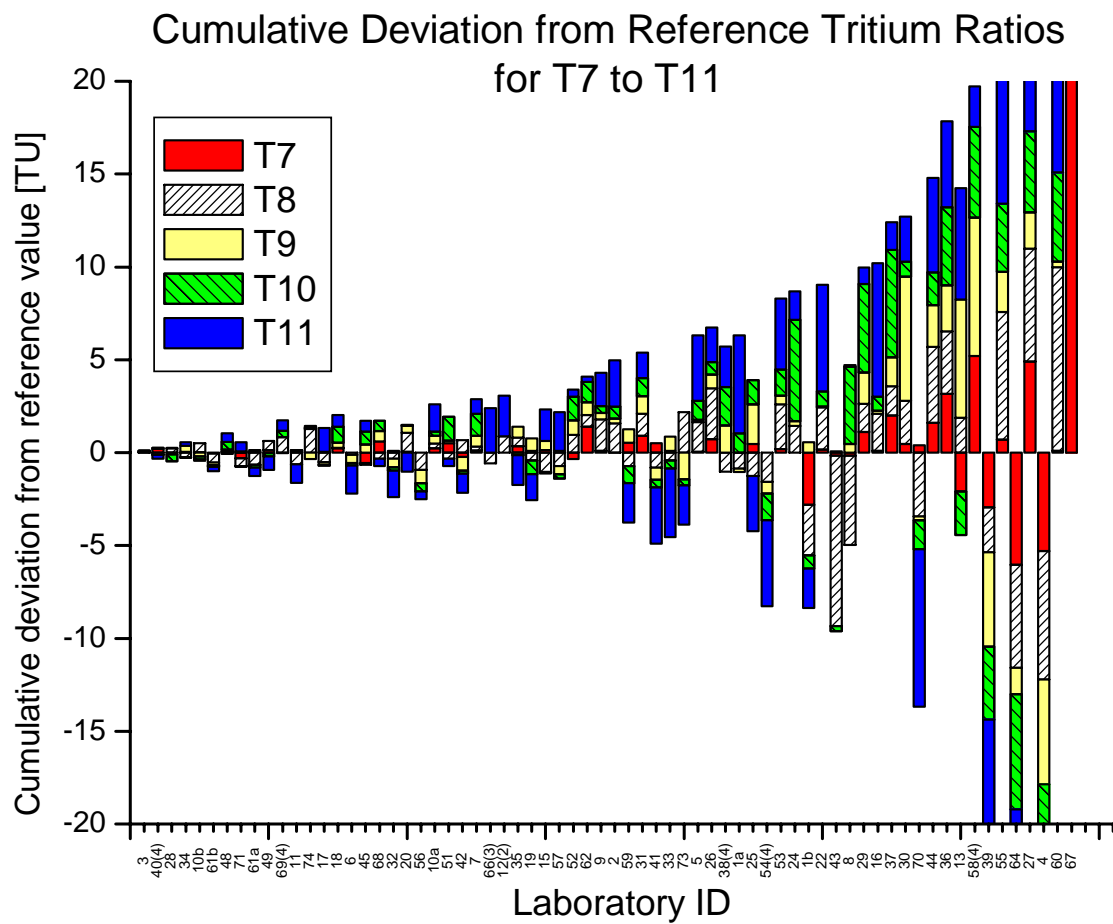


Fig.9: Sum of deviations from the reference tritium ratios for samples T7 to T11 in increasing absolute order in TU.

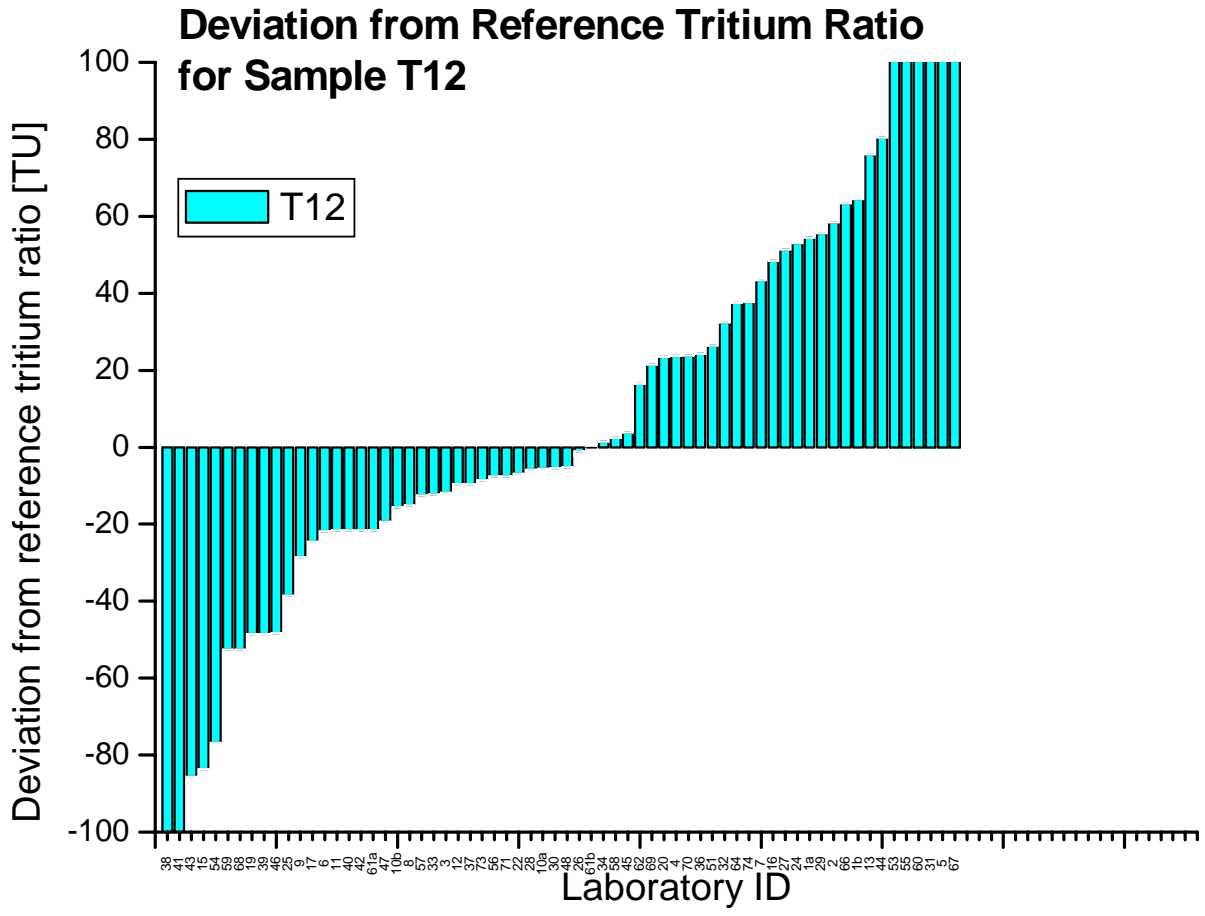


Fig.10: Deviation from the reference tritium ratio for sample T12 in increasing order.



### 3.5.2 Precision (Reliability of the Precision Statement):

The actual performance of a laboratory for sample analysis can be estimated by the deviation of results from known reference values. The performed measurements on six different samples are very helpful in this respect to provide a better estimate of the performance, as such deviations can be of random nature or due to a systematic bias. Additional information is provided by the comparison of these deviations to the laboratory's statement on its precision for the 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 calculated, and then divided by the reported uncertainty (Z-score). Therefore, the deviation for each sample is expressed in a sigma scale, directly comparing the reported  $1\sigma$ -uncertainty for each sample of a laboratory with the actual deviation from the reference value. Each laboratory's "sigma-deviations" for the six samples T7 to T12 are presented in Fig. 11, ordered by increasing sum of absolute "sigma-deviation" values. Here all six results can be combined due to the use of the sigma scale.

Assuming the absence of any systematic bias for each individual laboratory, all results should be normal distributed. Under this assumption the respective reference value should lay with 95% probability within  $2\sigma$  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  $1\sigma$ -level (67% probability) for each of the 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, statistically the sum of deviations should not exceed  $8\sigma$  and it should not fall below  $4\sigma$ . As it can be seen from Fig. 11, 54% of all laboratories exceed the value of  $8\sigma$ , which clearly is an indicator for an underestimation of uncertainty in those laboratories (including the possibility of systematic biases). On the other side, 16% of all laboratories provided data deviating in sum less than  $4\sigma$  from the reference values of samples T7 to T11, which signals their quite conservative approach in stating significantly too high uncertainties. About 30% of all laboratories are found in the acceptable medium range of  $4\text{--}8\sigma$ .

It has to be stressed, however, that the results of Fig. 11 do not tell all about the actual suitability of reported data for a certain purpose. This is illustrated in Fig. 12, where a similar plot as in Fig. 11 is presented for samples T7 to T11. In addition to the sum of absolute "sigma-deviations" for the five samples, the cumulated reported uncertainty for the five samples in TU 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".

This indicates the strong need to further improve the evaluation and uncertainty statements for tritium measurements. The uncertainty evaluation for tritium at environmental low-level activities using electrolytic enrichment and liquid scintillation counting is discussed in [12], providing numerical examples to facilitate the application of the described principles.

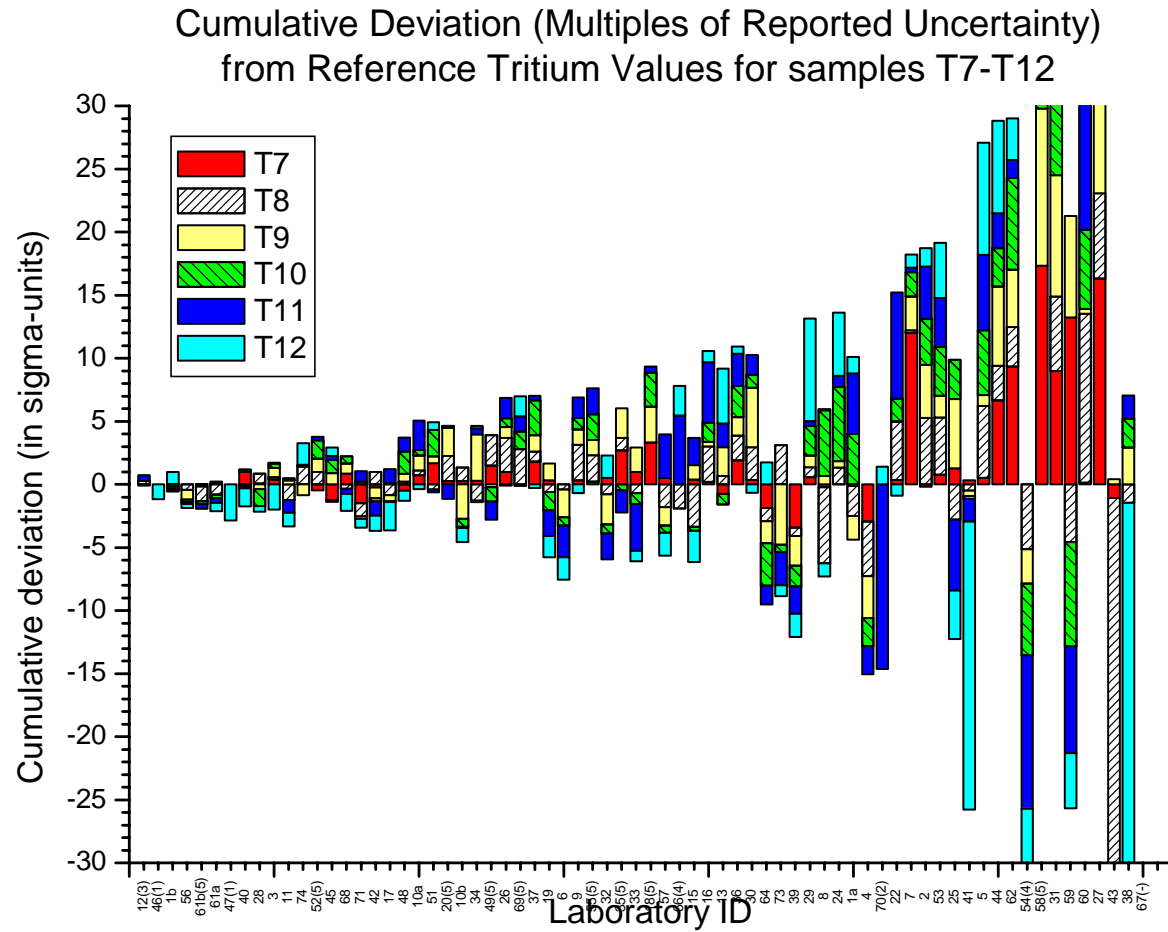


Fig.11: Sum of deviations from the true values for samples T7 to T12 in increasing absolute order, here reported as sigma-deviation by dividing the calculated deviation for each sample by the stated uncertainty. For details see text.



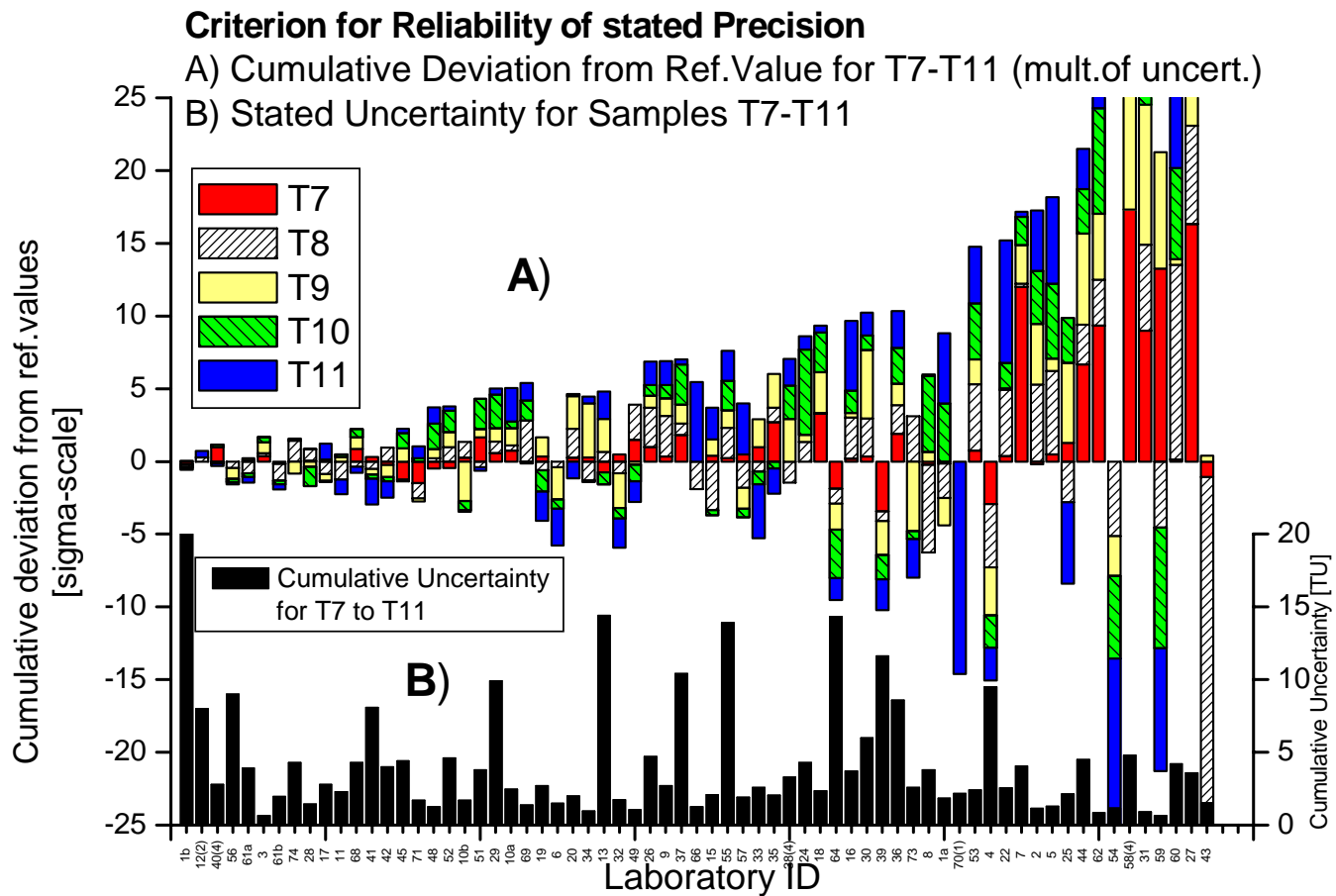


Fig.12: A) Sum of deviations from the reference value for samples T7 to T11 in increasing absolute order, reported as sigma-deviation by dividing the calculated deviation for each sample through the stated uncertainty. B) Reported cumulative uncertainty for samples T7 to T11.

### 3.6 Reporting of Results

Due to some problems in result reporting for TRIC2004 some basic guidelines are discussed here again.

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 TRIC2004, 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<sup>-1</sup>).

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. 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. It is encouraging that the majority of laboratories reported the used tritium half-life to be the recent value of 4500±8 days. 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.

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We would like to acknowledge the contribution of Ms. Andrea Howe on a Safeguards internship and of Mr. Matthias Rogner on an internship at our laboratory in a first compilation of tritium activities and uncertainties of the prepared six water samples. We would like to thank our colleagues Ms. Daniela Brummer and Mr. Chinmaya Sambandam for performed tritium measurements.

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## APPENDIX A: DETAILS OF THE SAMPLE PREPARATION

Since from the perspective of low-level tritium laboratories, SRM4927F is an extremely active tritiated water, the first two dilution stages were not performed at the Isotope Hydrology Laboratory, but at the distant IAEA Laboratories in Seibersdorf. A de-ionised “tritium-free background water” was used to dilute the standard in two stages. This water from the artesian well Grafendorf G4 (near Graz) dates from 1992 and was used for the previous tritium interlaboratory comparisons. Repeat analyses of the de-ionised water indicated that its tritium ratio is less than 0.1 TU. Its massic activity is less than  $5 \times 10^{-9}$  that of the resulting daughter water after 2 dilution stages, and its contribution to the prepared values can therefore be neglected.

Table 4: Dilution procedure for the tritiated water standard SRM4927F (reproduced from [6]).

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

In the recent exercise a sufficient supply of water was sampled from another artesian well G3 in Grafendorf, which is at the same location as the formerly used well G4, but tapping a slightly shallower aquifer. Therefore that water was tested again for its upper margin of any tritium content. This water was then used as diluting water T7 for preparation of the remaining five samples T8 to T12. The possible tritium concentration range of this water was judged to be between 0 - 0.050 TU at the 1  $\sigma$ -level, using the submitted results.

The diluting water T7 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 262 mg/kg water, i.e. – 0.0262%.

On the reference date 2004-07-01 the standard daughter water (“D2-2000”) had a tritium activity of 385959.2 TU. The weighing parameters for its dilution to create the five samples are summarised in the accompanying table 5. 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 five samples T8 to T12 used in this interlaboratory comparison exercise, using as tritiated standard the ‘Dilution Stage 2’ of Table 4 and as dilution water the sample T7 (Grafendorf well G3). Values in brackets are uncertainties at the 1- $\sigma$  level.

	T8	T9	T10	T11	T12
Weight of SRM4927F daughter “D2-2000” [g]	2.3256 (0.002)	0.3684 (0.002)	1.1455 (0.002)	5.2612 (0.002)	21.2329 (0.002)
Weight of T7 water [g]	82213.57 (0.17)	81811.77 (0.17)	81392.73 (0.17)	79935.85 (0.17)	8082.14 (0.06)
Correction of above T7 weight for TDS [g]	82192.04 (0.17)	81790.35 (0.17)	81371.42 (0.17)	79914.92 (0.17)	8080.02 (0.06)
Dilution factor	35343.3	222016	71036.7	15190.5	381.543

APPENDIX B: INDIVIDUAL LABORATORY RESULTS

Table 5: All sample results as reported by the individual participating laboratories. The uncertainties  $u(\text{sample})$  were requested to be stated at the 1- $\sigma$  level. Blank fields indicate not-submitted data. The three measurement methods are indicated: LSC – Liquid Scintillation Counting; Gas – Gas Proportional Counting;  $^3\text{He}$  – Mass Spectrometric  $^3\text{He}$  analysis. All outlier values in the tables are gray-shaded, for details on the outlier determination procedure see text. The TRIC2004 reference values are stated in the header lines.

Lab	T7	u(T7)	T8	u(T8)	T9	u(T9)	T10	u(T10)	T11	u(T11)	T12	u(T12)	Meas. Method
Ref./-u	<b>0</b>	0.000	<b>10.92</b>	-0.040	<b>1.738</b>	-0.006	<b>5.433</b>	-0.020	<b>25.410</b>	-0.092	<b>1011.58</b>	-3.68	
/+u		0.050		0.064		0.050		0.054		0.105		3.68	
All tritium values and uncertainties are stated as tritium ratio in [7]													
1a	-0.004	0.034	10.06	0.36	1.55	0.1	6.47	0.26	30.7	1.1	1066	42	$^3\text{He}$
1b	-2.8	14.2	8.2	15	2.3	15	4.7	13.8	23.3	15.3	1076	68	LSC
2	-0.002	0.01	12.5	0.3	1.99	0.06	6.09	0.18	27.9	0.6	1070	40	$^3\text{He}$
3	0.006	0.016	10.95	0.15	1.76	0.03	5.46	0.08	25.43	0.37	999.8	5.9	LSC
4	-5.3	1.8	4	1.6	-3.9	1.7	0.3	2.3	20.7	2.1	1035.3	9.3	LSC
5	0.04	0.08	12.52	0.28	1.87	0.15	6.46	0.2	28.92	0.59	1193	20.4	$^3\text{He}$
6	0	0.2	10.8	0.3	1.3	0.2	5.3	0.2	23.9	0.6	989.8	12.2	LSC
7	0.12	0.01	11.13	0.92	2.32	0.22	6.6	0.6	26.21	2.3	1054.95	41.84	LSC
8	-0.16	0.7	6.1	0.8	2.2	0.7	9.6	0.8	25.5	0.8	996.5	14.5	LSC
9	0.1	0.3	12.6	0.6	2.1	0.3	5.8	0.4	27.2	1.1	983	40	LSC
10a	0.23	0.31	11.17	0.68	2.17	0.37	5.65	0.47	26.89	0.64	1006	15	LSC
10b	0.02	0.08	11.42	0.46	1.52	0.08	5.29	0.23	25.32	0.85	996	14	Gas
11	0	0.2	10.3	0.5	1.8	0.2	5.5	0.4	24.4	1	990	20	LSC
12			11.8	3					27.6	5	1002	84	LSC
13	-2.1	2.8	12.8	2.8	8.1	2.8	3.1	2.8	31.4	3.2	1087.7	17.5	LSC
15	0.13	0.33	9.88	0.31	2.24	0.45	5.36	0.22	27.09	0.77	927.91	33.8	LSC
16	0.1	0.5	12.9	0.7	1.9	0.5	6.2	0.5	32.6	1.5	1060	53	LSC
17	0.04	0.3	10.4	0.6	1.6	0.3	5.4	0.4	26.7	1.2	987	11	LSC
18	0.23	0.07	10.94	0.55	2.02	0.1	6.3	0.32	26.04	1.3			$^3\text{He}$
19	0.1	0.3	10.5	0.7	2.4	0.5	4.7	0.5	24	0.7	963	28.5	LSC
20	0.038	0.153	11.95	0.514	2.115	0.168	5.473	0.291	24.397	0.881	1035.07	7.99	LSC
22	0.16	0.44	13.21	0.5	1.78	0.48	6.24	0.46	31.14	0.68	1004.71	7.8	LSC
24	0	0.05	12.35	1.07	2	0.52	10.89	0.93	26.96	1.73	1064.59	10.61	LSC
25	0.46	0.36	9.66	0.45	3.88	0.39	6.74	0.42	22.44	0.53	973	10	LSC
26	0.72	0.73	13.67	1.02	2.47	0.87	6.1	0.94	27.27	1.14	1010.42	14.02	LSC
27	4.9	0.3	17	0.9	3.7	0.2	9.8	0.5	35.8	1.7	1063	21	LSC
28	0.01	0.22	11.15	0.29	1.65	0.24	5.06	0.28	25.42	0.4	1005.7	13.2	LSC
29	1.12	1.93	12.43	1.89	3.44	1.92	10.18	2.02	26.3	2.15	1067.19	6.86	LSC
30	0.46	1.35	13.24	0.89	8.43	1.42	6.25	0.82	27.83	1.53	1006.1	8.1	Gas
31	0.9	0.1	12.1	0.2	2.7	0.1	6.4	0.1	26.8	0.4	1175.2	18.2	LSC
32	0.1	0.2	10.6	0.4	1.26	0.2	5.25	0.25	24	0.7	1044	18	LSC
33	0.1	0.1	10.5	0.6	2.5	0.4	5	0.5	21.7	1	999.3	15	LSC
34	0.04	0.14	10.66	0.2	2.07	0.09	5.42	0.13	25.6	0.4	1013	8	Gas
35	0.35	0.13	11.39	0.47	2.32	0.25	5.3	0.27	23.8	0.93			LSC

Lab	T7	u(T7)	T8	u(T8)	T9	u(T9)	T10	u(T10)	T11	u(T11)	T12	u(T12)	Meas. Method
Ref./-u /+u	<b>0</b>	0.000 0.050	<b>10.92</b>	-0.040 0.064	<b>1.738</b>	-0.006 0.050	<b>5.433</b>	-0.020 0.054	<b>25.410</b>	-0.092 0.105	<b>1011.58</b>	-3.68 3.68	
All tritium values and uncertainties are stated as tritium ratio in [7]													
36	3.17	1.67	14.27	1.71	4.23	1.7	9.64	1.69	30.03	1.83	1035.85	42.63	LSC
37	2	1.1	12.5	2	3.3	1.2	11.2	2.1	26.9	4	1002	32	LSC
38			9.9	0.7	3.2	0.5	7.5	0.9	27.6	1.2	117.5	5.8	LSC
39	-2.95	0.86	8.5	3.54	-3.35	2.19	1.52	2.33	19.67	2.69	963	26	LSC
40	0.2	0.2	10.8	0.9	1.7	0.2	5.5	0.4	25.2	1.1	990	15	LSC
41	0.5	1.6	10.1	1.6	1.1	1.6	5	1.6	22.4	1.7	908.9	4.5	LSC
42	-0.23	0.9	11.6	0.7	1	0.9	5.26	0.59	24.4	0.9	990	18	LSC
43	-0.173	0.16	1.75	0.1	1.81	0.18	5.16	0.26	25.41	0.82	926	18.5	LSC
44	1.60	0.24	15	1.5	4	0.36	7.2	0.58	30.5	1.83	1092	11	LSC
45	-0.55	0.44	10.81	0.93	2.19	0.51	6.13	0.66	25.99	1.86	1015.46	5.97	LSC
46											963.2	41.1	LSC
47											992.25	6.75	LSC
48	-0.08	0.16	10.98	0.28	1.85	0.18	5.84	0.23	25.87	0.41	1006.4	6.4	LSC
49	0.15	0.1	11.4	0.2	1.72	0.08	5.24	0.17	24.7	0.5			<sup>3</sup> He
51	0.5	0.3	10.6	0.8	1.9	0.3	6.7	0.6	25	1.8	1038	43	LSC
52	-0.35	0.75	11.87	0.96	2.53	0.77	6.71	0.86	25.78	1.28			LSC
53	0.19	0.25	13.33	0.53	2.2	0.27	6.86	0.37	29.21	0.98	1117	24	LSC
54			9.33	0.31	1.11	0.23	4.01	0.25	20.78	0.38	934.8	9.26	LSC
55	0.7	3	17.8	3.3	3.9	1.8	9.1	1.8	33.7	4	1123	64	LSC
56	0	1	10	2	1	1	5	2	25	3	1004	25	LSC
57	0.1	0.2	10.2	0.4	1.3	0.3	5.2	0.4	27.5	0.6	999	7	LSC
58	5.2	0.3	16.5	1.2	9.2	0.6	10.3	0.8	27.6	1.9	1014	17	LSC
59	0.53	0.04	10.19	0.16	2.46	0.09	4.52	0.11	23.3	0.25	959	12	LSC
60	0.09	0.66	20.83	0.74	2.02	0.75	10.26	0.77	60.61	1.29	1165	58.4	LSC
61a	0.04	0.65	10.28	0.78	1.84	0.67	5.26	0.7	24.98	1.1	990	32	LSC
61b	-0.04	0.26	10.43	0.42	1.59	0.28	5.35	0.33	25.17	0.67	1012	21	Gas
62	1.4	0.15	11.55	0.2	2.42	0.15	6.52	0.15	25.7	0.2	1028	5	Gas
64	-6.05	3.26	5.37	5.29	0.34	0.79	-0.79	1.86	20.79	3.13	1049.1	21.6	LSC
66	0	0.13	10.33	0.31	1.2	0.16	5.22	0.23	27.81	0.44	1074.94	27	LSC
67	622		129		340		622		622		1397		LSC
68	0.6	0.7	10.6	0.9	2.3	0.7	6	1	25	1	959	40	LSC
69	-0.02	0.17	11.76	0.3	2.2	0.2	5.77	0.24	25.97	0.47	1033.04	13.58	LSC
70	0.4	0.39	7.48	0.46	1.53	0.37	3.87	0.38	16.93	0.58	1035.39	17.16	LSC
71	-0.3	0.2	10.5	0.4	1.7	0.2	5.5	0.3	25.9	0.6	1004	11	LSC
73	0	0.2	13.1	0.7	0.3	0.3	5.1	0.6	23.3	0.8	1003	10	LSC
74	0	0.5	12.2	0.9	1.4	0.4	5.5	0.7	25.5	1.8	1049.3	22.3	Gas



## APPENDIX C: LIST OF PARTICIPANTS

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