FIFTH IAEA INTERCOMPARISON OF LOW-LEVEL
TRITIUM MEASUREMENTS IN WATER

REPORT

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FOREWORD

The environmental isotope intercalibration programs of the International Atomic Energy Agency (IAEA) were initiated more than three decades ago. In fact, two parallel programs have been implemented: (i) for stable isotopes of light elements, and (ii) for radioactive isotope of hydrogen, tritium. This involvement of the IAEA resulted in the preparation of a number of reference and intercomparison materials of various types, now stored in the Agency and available upon request. The Agency's current environmental isotope intercalibration program is much wider in scope than initially conceived, and is a part of the Analytical Quality Control Service (AQCS) instituted by the IAEA in order to assists laboratories engaged in various fields of scientific research to check and maintain the quality of their work.

The IAEA has organized five tritium measurement intercomparisons during the past three decades. The results of the first four exercises were published in the scientific literature (Cameron, 19667; Florkowski, Payne and Sauzay, 1970; Taylor, 1978; Hut, 1986a, 1986b). This report summarizes the results of the most recent exercise completed in December 1994. The basic features of these intercomparisons are summarized in below:

Basic features of tritium intercomparison programs carried out by IAEA

<table>
<thead>
<tr>
<th>Year</th>
<th>Number of participating laboratories*</th>
<th>Water samples distributed for intercomparison</th>
</tr>
</thead>
<tbody>
<tr>
<td>1965</td>
<td>12 (29)</td>
<td>Sample A: &lt; 100 TR**</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample B: 200 - 800 TR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample C: 1000 - 6000 TR</td>
</tr>
<tr>
<td>1970</td>
<td>35 (38)</td>
<td>Sample A: ~ 10 TR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample B: ~ 50 TR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample C: ~ 250 TR</td>
</tr>
<tr>
<td>1975</td>
<td>41 (79)</td>
<td>Sample A: &lt; 0.2 TR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample B: 8.37 ± 0.04 TR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample C: 33.1 ± 0.1 TR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample D: 678.5 ± 2.2 TR</td>
</tr>
<tr>
<td>1985</td>
<td>57 (85)</td>
<td>Sample A: &lt; 0.2 TR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample B: 5.94 ± 0.03 TR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample C: 25.66 ± 0.13 TR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample D: 100.2 ± 0.5 TR</td>
</tr>
<tr>
<td>1994</td>
<td>57 (91)</td>
<td>Sample A: ~ 0 TR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample B: ~ 1 TR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample C: ~ 5 TR</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Sample D: ~ 19 TR</td>
</tr>
</tbody>
</table>

* Number in parentheses in the second column indicate the number of identified active laboratories to which the announcement was sent.

** TR = 1, corresponds to a $^3$H/$^1$H ratio of $10^{-18}$
In each intercomparison exercise, a set of water samples covering the actual range of environmental tritium concentrations was distributed. For the first two intercomparisons, the tritium concentration in the distributed samples was known only approximately. The results were published with full identification of the participating laboratories. For the next three exercises the tritium activity of the distributed samples was established by gravimetric dilution of the tritium standard NBS 4926. The participating laboratories were informed about the approximate concentration of tritium in the distributed samples and were requested to provide detailed information on the measurement techniques applied. The results were reported anonymously. The anonymity was maintained also during the most recent exercise.

Participation in the IAEA intercomparison exercises is voluntary. However, leading laboratories of recognized experience and quality are usually requested to participate. Often they are also involved in preparation of the intercomparison samples. The results are discussed in meetings of invited experts, and the decisions and recommendations are usually accepted by the scientific community.

Most laboratories applying environmental isotopes in geochemistry, hydrology, agriculture, plant physiology, life sciences, environmental studies, etc., have participated in the Agency's intercomparison exercises. These are considered unique opportunities to check the agreement of the measurement performed with those of other laboratories, to discover the concurrence of errors and systematic deviations, and eventually to try to correct them. They constitute a continual incentive to improve the quality of the measurements. Thus, the participation in such exercises is considered not only useful but necessary in order to prove and demonstrate the quality and reliability of isotopic analyses.
INTRODUCTION

Many laboratories around the world now measure tritium (as HTO) at currently very low concentration levels in environmental waters. The results are applied with particular benefit in hydrological, oceanographic and geothermal research. Technical difficulties associated with low-level tritium measurements are well known; these are now more difficult to overcome because concentrations have declined by an order of magnitude and more below those which prevailed during the peak years of bomb tritium in the 1960s. IAEA have encouraged intercomparability between laboratories by recommending a uniform measurement scaled based on a single water standard and uniform half-life (Taylor and Roether, 1982) and have conducted four previous laboratory intercomparison exercises. A fifth such intercomparison has been conducted during 1994 and is reported here.

Invitations were sent to 91 laboratories, with 81 responding positively. Four water samples (A,B,C,D) were sent to each, with tritium concentrations typical of present environmental waters.

CONCENTRATION UNITS

Results are quoted here as Tritium Ratios (TR), where TR = 1 corresponds to a T/H ratio of $10^{18}$. This follows the IAEA recommendation (Taylor and Roether, 1982). Results submitted as TR or TU (Tritium Unit) are given here as quoted by the measuring laboratories, many of whom did not report their measurement scale or standardisation procedures. Quite a number of laboratories reported results in various specific activity units, not as TR or TU. For the purposes of this report, such results have been converted to TR using the conversion: $TR = 1 \equiv 0.11815 \text{ Bq/kg}$.

PREPARATION OF SAMPLES

None of the samples were distilled before sending.

**Sample A:** this water is from a deep artesian aquifer near Linz in Upper Austria. From the known geological conditions and carbon-14 measurement, water from this aquifer should have tritium concentration very close to zero.

**Sample B:** this water was prepared to have concentration very close to TR = 1, by diluting a daughter standard of US National Bureau of Standards (now NIST) tritium water standard #4926 with sample A.

**Sample C:** this water was prepared in the same way as Sample B, with TR close to 5.

**Sample D:** this sample is Vienna tap water. Several measurements by the IAEA laboratory indicated that TR is close to 19.
DISTRIBUTION OF SAMPLES

The samples were sent in heavy-walled polyethylene bottles early in July 1994. Laboratories received 1100ml of each sample. Parcels within Europe were sent by surface transport, elsewhere by air. By 12 December 1994, 90 sets of results had been returned by 57 laboratories. No results are yet available from 5 laboratories using the $^3\text{He}$ ingrowth measurement method; these laboratories also received an extra sample of near-zero tritium concentration from a deep aquifer under Christchurch, New Zealand, sent by air in glass bottles.

RADIOMETRIC MEASUREMENT METHODS

Concentrations of tritium in these samples and in present-day environmental waters are such that direct beta-counting (by liquid scintillation or gas proportional counters) can yield at best only marginally useful results; the concentration of the most active sample D corresponds to only 0.135 dpm/g water. Adequate accuracy at these levels can only be achieved by enriching the samples before counting. A high proportion of the participating laboratories included this step, and all of these used the electrolytic enrichment technique; several laboratories counted samples both before and after enrichment.

PRESENTATION OF RESULTS

Each laboratory was assigned a confidential number to identify its results from the following sections of the report. Table 1 lists the results as TR values ± 1 standard error. The host column identifies the measurement technique used.

Method Codes:

D = direct count (without pre-enrichment); E = counted after electrolytic enrichment; G = gas counting; L = liquid scintillation counting; * = result supplied is mean value of more than one measurement, individual results were not received in some of these cases; # = information supplied suggests the error does not contain all contributing components; $ = measurements made in Bq/l and converted to TR by the laboratory using a factor different to that used at IAEA; & = enrichment factors calibrated by deuterium measurement before and after enrichment; 2 = results from second set of samples; ? = no technique information supplied by laboratory.

STATISTICAL ANALYSIS OF RESULTS

59 laboratories returned data in time for this report. 91 sets of results were received (some incomplete), because several laboratories submitted more than one set. 11 laboratories used gas proportional counting, and 14 counted without prior enrichment. The distribution of results by counting method and enrichment is given below. (The information supplied by two laboratories was insufficient to classify the methods used).
<table>
<thead>
<tr>
<th>Counting method</th>
<th>Enrichment</th>
<th>Direct</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas</td>
<td>9</td>
<td>13</td>
</tr>
<tr>
<td>Liquid Scintillation</td>
<td>48</td>
<td>18</td>
</tr>
</tbody>
</table>

For the statistical analyses, all results reported as < values were ignored; this included all results for laboratory 14.

**Graphical summaries of the data**

Figs. 1a-d summarize the results for samples A, B, C and D in the form of histograms and box and whisker plots. The central box covers the middle 50% of the data, a central line within the box shows the median, and extreme observations appear outside the box as stars. For A, B and C, the frequency distribution histogram is skewed to the right with a long tail (particularly for A and B). Sample D shows a more symmetric distribution. Some outlying results are apparent for all four samples.

Figs. 2a-d are further box and whisker diagrams of the results received, but now categorised according to the measurement methods: liquid scintillation (LSC), gas proportional counting (GPC); direct counting (D) enrichment (E).

For samples A and B (Figs. 2a-b), the results obtained by direct counting are inevitably highly scattered; the net count rates are not statistically significant relative to background, so the scatter merely indicates the range of statistical counting errors. A higher scatter is observed for samples counted by liquid scintillation, but this is likely to be due to greater statistical counting error resulting from higher background and lower counting efficiency.

For sample C (Figs. 2c), the net count rates are still very small, but sufficient to narrow the direct count box. In the case of sample D (Fig. 2d), the direct and enriched boxes have similar width; for this sample, the spread caused by enrichment error becomes roughly equivalent to the statistical error of a direct count.

**Numerical summaries**

The first table of numerical summaries includes the weighted mean and median (which is more resistant to any outlying or extreme values), the range (minimum and maximum), and the standard deviation (which gives a measure of the spread of the results).
<table>
<thead>
<tr>
<th>sample</th>
<th>mean</th>
<th>median</th>
<th>range</th>
<th>number of observations</th>
<th>std dev</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1.030</td>
<td>0.17</td>
<td>-5.04, 15.40</td>
<td>53</td>
<td>2.584</td>
</tr>
<tr>
<td>B</td>
<td>2.340</td>
<td>1.20</td>
<td>-0.44, 22.0</td>
<td>67</td>
<td>3.228</td>
</tr>
<tr>
<td>C</td>
<td>5.965</td>
<td>5.00</td>
<td>2.4, 25.21</td>
<td>69</td>
<td>3.305</td>
</tr>
<tr>
<td>D</td>
<td>19.750</td>
<td>19.53</td>
<td>7.2, 33.61</td>
<td>88</td>
<td>3.00</td>
</tr>
</tbody>
</table>

For samples A and B, there are large differences between mean and median, and the standard deviations are large relative to the mean; this again emphasises the wide scatter, with presence of extreme observations. These features are not so apparent for samples C and D.

**Removal of gross outliers**

A number of extreme observations were identified from the box and whisker plots, and these were removed from the subsequent calculations. The criterion for exclusion is that any observation greater (or less) than the upper (lower) quartile + (-) 1.5 times the interquartile range is removed. In this way 4, 6, 7, and 2 values were omitted for samples A, B, C and D respectively. The summary statistics were then recalculated as follows.

<table>
<thead>
<tr>
<th>sample</th>
<th>mean</th>
<th>median</th>
<th>number of observations</th>
<th>std dev</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>0.676</td>
<td>0.170</td>
<td>49</td>
<td>1.088</td>
</tr>
<tr>
<td>B</td>
<td>1.507</td>
<td>1.16</td>
<td>61</td>
<td>1.007</td>
</tr>
<tr>
<td>C</td>
<td>5.06</td>
<td>4.95</td>
<td>62</td>
<td>0.052</td>
</tr>
<tr>
<td>D</td>
<td>19.735</td>
<td>19.53</td>
<td>86</td>
<td>2.253</td>
</tr>
</tbody>
</table>

Omission of these outliers makes only small changes to the median, but does significantly alter the means for samples A, B and C; all the standard deviations are considerably reduced. The median values are at this point taken as preliminary estimates of the TR values of the samples.

The errors quoted by the laboratories have not so far been taken into account. In the following stage of the analysis, a consistent set of results (i.e. those in agreement with the preliminary estimate within the quoted errors) will be defined, and used to calculate average weighted TR values.
Inclusion of quoted experimental errors

All results for which the above preliminary estimates do not lie within the interval reported TR ± twice the quoted error will be omitted from the final calculation. Using this criterion, a further 14, 17, 8 and 21 results were excluded from samples A, B, C and D respectively. Weighted averages were then evaluated, together with estimates of their errors.

<table>
<thead>
<tr>
<th>sample</th>
<th>number of observations</th>
<th>Average TR</th>
<th>estimated error</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>35</td>
<td>0.139</td>
<td>0.018</td>
</tr>
<tr>
<td>B</td>
<td>45</td>
<td>1.074</td>
<td>0.026</td>
</tr>
<tr>
<td>C</td>
<td>54</td>
<td>4.926</td>
<td>0.029</td>
</tr>
<tr>
<td>D</td>
<td>65</td>
<td>19.551</td>
<td>0.085</td>
</tr>
</tbody>
</table>

A relatively large number of results were excluded in calculating these average values. This suggests problems with calibration and standardisation, contamination, and unrealistic error estimates in some cases. The weighted averages are in agreement with the a-priori expectations, but the result for sample A is clearly positively biased. But it is noticeable that most laboratories, although quoting a finite result with error, are unable to distinguish sample A from 0 at the 2 sigma level.

Envelope diagrams

In order to illustrate the spectrum of performance and relative capabilities of the participating laboratories at these low concentration levels, the data for each sample has been assembled into "envelope" diagrams. In these, the laboratories are graded from left to right in order of increasing reported standard measurement error. A horizontal line represents the weighted average result calculated by the above statistical assessment. Envelope lines above and below show the measurement precision as deviations +/- 2 sigma relative to the weighted average. The position of the experimental values within or outside the envelope provides in most cases a good indication of the quality of each measurement. Table 2 links the laboratory code numbers to their positions in these diagrams.

To avoid making the diagrams of unmanageable length, values reported by some laboratories as more than one result using a common method were first transformed to one result which is a weighted average. However, the standard errors accompanying these mean values are average values from the set which provide an indication of the routine precision of a single measurement. The presence of two or more points for a laboratory indicates that separate measurements were performed using different methods.
All the diagrams (Sample A - Fig. 3; Sample B - Fig. 4; Sample C - Fig. 5; Sample D - Fig. 6) reveal a wide range of precision amongst the participants. Those laboratories with poor precision are, in many cases, those which generally operate in higher concentration ranges than usually found in the environment; their precision may be suited to their own needs, and the samples provided for this exercise have not provided any indication of their capabilities in their accustomed range.

About 1/3 of the higher precision results for sample D fall outside the envelope, suggesting either that the errors are quite appreciably under-estimated, or alternatively that the laboratory working standards may not be accurately calibrated. For sample C, calibration errors contribute less significantly to the overall error. There are here fewer outliers at the high precision end of the diagram, suggesting that the high proportion of outliers for sample D is due in many cases to unrigorous calibration.

The diagrams for samples A and B show a noticeably skew distribution. In both cases, results at the high precision end of the diagram lie predominantly below the weighted average. In the case of sample A, 4 of the first 5 results were excluded as outliers by the statistical evaluation leading to the indicated mean result, although they lie much closer to the expected near-zero tritium concentration. The first 7 results for B all lie below the average value. It is thus apparent that the weighted averages of A and B have been affected by slight positive bias (of order TR = 0.1 on average) at some of the laboratories further right in the diagrams. These laboratories may not be particularly concerned about attaining very high precision as TR approaches zero; in such cases, this degree of positive bias would not show up in the course of routine operation. The outliers in both A and B diagrams reveal that several laboratories have inherent contamination effects which are serious cause for concern if they are normally measuring within the range of concentrations covered by this exercise. A few laboratories submitted results which were uniformly too high.

Reanalysis using the weighted average as preliminary estimate

Further statistical analysis was suggested by the results for sample A and B, which showed that the labs quoting smaller errors were generally systematically below the weighted average (Figs. 3-4). The procedure is as before, but with the median now replaced by the weighted average as the preliminary estimate. Two stages are involved: in the first, some of the lower results are still outliers, but no longer become so when related to the newly calculated weighted average; in the second stage, this new average is used as initial value, allowing reincorporation of most of the low outliers into the set for calculation of a final estimate.

<table>
<thead>
<tr>
<th>sample</th>
<th>Weighted average TR values</th>
<th>final estimate of new average TR</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>previous weighted TR</td>
<td>first estimate of new average TR</td>
</tr>
<tr>
<td>A</td>
<td>0.139</td>
<td>0.067</td>
</tr>
<tr>
<td>B</td>
<td>1.074</td>
<td>1.058</td>
</tr>
<tr>
<td>C</td>
<td>4.926</td>
<td>4.967</td>
</tr>
<tr>
<td>D</td>
<td>19.551</td>
<td>19.058</td>
</tr>
</tbody>
</table>
This analysis has made a significant difference to the calculated weighted averages for samples A B and D, but much less in the case of sample C.

Both the approaches used can be justified on statistical grounds, but they differ in their treatment of the errors quoted by the laboratories. For the latter approach to be valid, then the errors need to be strictly comparable, and it was clear that this was not the case. The latter approach was also not robust, because a few laboratories with very small errors dominate the calculated TR averages.

Comparison of results by method

After exclusion of outliers, 2-sample t-tests (not assuming equal variances) were made to establish any differences between methods.

<table>
<thead>
<tr>
<th>sample</th>
<th>counting method</th>
<th>direct/enriched</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>p = 0.22</td>
<td>p = 0.67</td>
</tr>
<tr>
<td>B</td>
<td>p = 0.006*</td>
<td>p = 0.79</td>
</tr>
<tr>
<td>C</td>
<td>p = 0.055**</td>
<td>p = 0.23</td>
</tr>
<tr>
<td>D</td>
<td>p = 0.93</td>
<td>p = 0.68</td>
</tr>
</tbody>
</table>

The * result for sample B indicates a significant difference at the p<0.05 level for sample B, i.e. between the mean concentrations obtained by liquid scintillation and gas counting. Reasons for this are not obvious. The ** result for sample C is very close to being significant at the same level. In general, however there is not convincing evidence of real differences between the different approaches to the radiometric measurement. The apparent differences for samples A, B and C in the earlier diagrams are undoubtedly due in large part to the large measurement errors (as a fraction of the actual concentration) reported at these very low concentrations; the differences are seen to be generally insignificant when the results are analyzed as here. Further consideration would only be possible if comprehensive information on each laboratory's procedures became available.

GENERAL CONCLUSIONS, COMMENTS AND RECOMMENDATIONS

The more precise results for samples A and B appear to indicate lower real concentrations than the calculated average of results. This indicates that there was no significant contamination during preparation and handling at the Agency. There appears therefore to be a small degree of contamination, in the order of 0.1 TR on average, inherent in the procedures at many of the participating laboratories. This does not affect the average result for sample C, which is close to the value expected from the sample preparation details. No expected value is available for sample D. The main feature of this sample is thus to establish the extent of agreement between laboratories for a sample in which the accuracy of the enrichment procedures and the reliability of calibration/standardisation are more important to the precision. This sample is also at the level where direct counting begins to yield useful information.
Not all the laboratories achieve satisfactory sensitivity at the concentration levels now pertaining in environmental waters. Those involved only in radiation safety and similar projects do not routinely require this degree of performance.

Comparison is possible between the number of outliers (envelope diagrams) for samples C and D with those of samples B and C (roughly equivalent concentrations) in the 1976 intercomparison (Taylor, 1978):

- 1976, sample B, 31% outliers; 1994, sample C, 10% outliers
- 1976, sample C, 24% outliers; 1994, sample D, 22% outliers

Although this suggests some improvement overall, it is disturbing that the outliers for 1994 sample D all lie among the laboratories claiming better precision.

The outliers for samples A and B (high average result in the case of sample A; for sample B, all, with just one marginal exception, lie above the envelope) indicate the presence of contamination preventing attainment of reliable results at the very lowest concentrations.

These problems can be addressed by: (i) rigorous attention to standardisation and reproducibility, e.g. by all laboratories adopting the measurement scale (standard water, half-life) recommended by IAEA (Taylor and Roether, 1982) (ii) by acquiring a supply of tritium-free water to assess the presence, cause and consistency of laboratory blanks (iii) by stringent repeatability and quality control checks on counting and enrichment procedures (iv) by inclusion of all components in the quoted errors, and (v) by operating the laboratories to certified standards of quality control.

In respect of the reporting of results, it is recommended that the scale used should be indicated always, with the reported errors being one standard error accurately reflecting all error contributions.

Comparison with the earlier intercomparison exercises indicates that many laboratories have not yet moved to compensate for the continuing drop in environmental concentrations by improving sensitivity and lowering detection limits. Only 3 of the laboratories (23, 48 and 72) routinely achieve detection limits below TR = 0.1, but this demonstrates that the traditional radiometric methods can approach the sensitivity achieved by the $^3$He mass spectrometric technique. Such sensitivity at concentration levels below TR = 1 significantly enhances the present and extents the future applicability environmental tritium as a tracer of the hydrological cycle. We add the strong recommendation that radiometric laboratories involved in such applications should attempt to improve their detection limits by applying a higher degree of enrichment and optimising all aspects of their counting procedures; this should be accompanied by steps to ensure that procedures avoid any significant contamination.
ACKNOWLEDGEMENTS

The authors gratefully acknowledge the willingness of the participating laboratories to expose themselves to the scrutiny of this project. The technical and administrative staff of the IAEA Section of Isotope Hydrology are thanked for their diligent efforts in setting up and carrying it through.

REFERENCES


Table 1. The results for sample A, B, C, D, as received from the participating laboratories, reported in TR.

<table>
<thead>
<tr>
<th>Lab Code</th>
<th>Sample A</th>
<th>Sample B</th>
<th>Sample C</th>
<th>Sample D</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>TR</td>
<td>sigma</td>
<td>TR</td>
<td>sigma</td>
<td>TR</td>
</tr>
<tr>
<td>0</td>
<td>&lt;1</td>
<td></td>
<td>&lt;1</td>
<td></td>
<td>4.4</td>
</tr>
<tr>
<td>1</td>
<td>≤0.4</td>
<td>0.8</td>
<td>0.31</td>
<td>5.23</td>
<td>0.34</td>
</tr>
<tr>
<td>1</td>
<td>1.16</td>
<td>0.24</td>
<td>5.11</td>
<td>0.37</td>
<td>19.2</td>
</tr>
<tr>
<td>1</td>
<td>0.87</td>
<td>0.25</td>
<td>4.79</td>
<td>0.36</td>
<td>18.5</td>
</tr>
<tr>
<td>1</td>
<td>0.76</td>
<td>0.31</td>
<td></td>
<td></td>
<td>20.8</td>
</tr>
<tr>
<td>1</td>
<td>0.85</td>
<td>0.32</td>
<td></td>
<td></td>
<td>19.2</td>
</tr>
<tr>
<td>1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>20.3</td>
</tr>
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<td></td>
<td></td>
<td></td>
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<td>23.5</td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
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<td>23.0</td>
</tr>
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<td></td>
<td></td>
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<td></td>
<td></td>
<td>20.2</td>
</tr>
<tr>
<td>2</td>
<td>0.04</td>
<td>0.23</td>
<td>1.1</td>
<td>0.24</td>
<td>4.9</td>
</tr>
<tr>
<td>2</td>
<td>-0.02</td>
<td>0.15</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.09</td>
<td>0.08</td>
<td>0.96</td>
<td>0.12</td>
<td>4.69</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
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All results for sample A

Figure 1a
All results for sample B

Figure 1b
All results for sample C

Figure 1c
All results for sample D

Figure 1d
Results for sample A by method (1:LSC, 0: GPC)

Results for sample A by method (1: direct, 0: enriched)

Figure 2a
Results for sample B by method (1:LSC, 0: GPC)

Results for sample B by method (1:direct, 0: enriched)

Figure 2b
Results for sample C by method (1:LSC, 0: GPC)

![Box plot for LSC/GPC](image)

Results for sample C by method (1:direct, 0: enriched)

![Box plot for D/E](image)

**Figure 2c**
Results for sample D by method (1:LSC, 0: GPC)

Results for sample D by method (1:direct, 0: enriched)

Figure 2d
Result distribution for sample A
arranged in order of increasing sigma

Upper line is average TR + 2 sigma reported by labs.
Dotted line is x-axis

Result distribution for sample A
arranged in order of increasing sigma

Envelope is weighted average TR +/- 2 sigma reported by labs.
Dotted line is x-axis

Figure 3
Result distribution for sample B
arranged in order of increasing sigma

Envelope is weighted average TR +/- 2 sigma reported by labs.

Figure 4
Result distribution for sample C
arranged in order of increasing sigma

Envelope is weighted average TR +/- 2 sigma reported by labs.

Figure 5
Result distribution for sample D
arranged in order of increasing sigma

Envelope is weighted average TR +/-
2 sigma reported by labs.

Figure 6
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