# Proficiency test SPIL-1 (2026)

## Organic matter, phosphorus, chloride, sulphate and suspended solids in wastewater (effluent)

**Quality Documentation**
**May 2026**

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## *CONTENTS*

# *1 INTRODUCTION*

A proficiency test on the analysis of organic matter, phosphorus, chloride, sulphate and suspended solids in wastewater was conducted on 12 March 2026. The proficiency test was organised by Eurofins Miljø A/S.

The present report contains Eurofins' documentation for the quality of the proficiency test. Results of the proficiency test including data from participating laboratories and statistical analysis of these data were issued in a report to all participants /1/ on 21 April 2026.

# 2 FEATURES OF THE PROFICIENCY TEST

Participants in the proficiency test were a total of 58 laboratories from Brazil, Croatia, Denmark, Finland, France, Germany, Greece, Hungary, Ireland, Japan, Netherlands, Spain and Sweden.

The closing date for submission of results was 27 March 2026. All participants had submitted their results before the deadline.

## 2.1 Sample preparation

The parameters covered in the proficiency test are listed in Table 2 as are the abbreviations used in this report.

Six samples were dispatched for the proficiency test. The samples were sample pairs covering the parameters as described in Table 1. The matrix of the samples represented wastewater, in this case effluent. Sample preparation is described in Appendix A.

*Table 1 Samples in the proficiency test*

| Sample name | Parameters |
|---|---|
| A1/B1 | $COD_{Cr}$, BOD (w. ATU) and NVOC/TOC |
| A2/B2 | TP, Cl and $SO_4$ |
| A3/B3 | TSS |

## 2.2 Statistical analysis of participants' data

A split-level design was used. The data analysis was performed in accordance with ISO 5725-2:2025 /2/, ISO 13528:2022 /3/ and as described in detail in Spliid (1992) /4/. A short introduction to the statistics and a list of symbols and abbreviations used is given in Eurofins document "Schedule for a proficiency test", which is available at Eurofins' home page /5/.

The statistical model used assumes that the variances for the two samples in a sample pair are identical. The assumption was tested (F-test, 95% confidence level) and the result was that the two variances may be assumed to be identical for all parameters.

## 2.3 Assigned and spike value

An overview of the concentrations in the samples (the assigned values) and the difference in concentration between the two samples of a sample pair (spike value) are shown in Table 2 compared to the range of concentrations normally encountered in effluent. The table also gives the expanded uncertainty of the assigned values. Assigned values, spike values and uncertainty of the assigned values were calculated in accordance with ISO 13528:2022 /3/. The Uncertainty of the assigned values are the expanded uncertainty with coverage factor, $k = 2$.

*Table 2 Assigned and spike value*

| Parameter | Abbreviation | Unit | Typical Range | Assigned value | Uncertainty of assigned value | Spike value |
|---|---|---|---|---|---|---|
| Chemical oxygen demand – dichromate method | $COD_{Cr}$ | mg/l $O_2$ | 5-75 | 58 | 2.9 | 8 |
| Five day biochemical oxygen demand (w. ATU) | $BOD_5$ (w. ATU) | mg/l $O_2$ | 2-6 | 4.8 | 0.35 | 0.8 |
| Seven day biochemical oxygen demand (w. ATU) | $BOD_7$ (w. ATU) | mg/l $O_2$ | 2-6 | 5.5 | 0.23 | 0.8 |
| Non-volatile/Total organic carbon | NVOC/TOC | mg/l C | 2-30 | 22.7 | 0.86 | 3.0 |
| Total phosphorus | TP | mg/l | 0.2-2 | 0.96 | 0.015 | 0.19 |
| Chloride | Cl | mg/l | 50-700 | 171 | 2.4 | 42 |
| Sulphate | $SO_4$ | mg/l | 20-200 | 55 | 1.8 | 16 |
| Total suspended solids | TSS | mg/l | 20-100 | 37.2 | 2.1 | 4.5 |

### *2.3.1 Assigned and spike values*

The content of each parameter in each sample is given an assigned value for the sample with the lower content and a spike value, the spike value being the difference in concentration between the two samples of the sample pair.

To ensure optimal use of the data, the assigned value is calculated as the average of the median for both samples in the sample pair after subtraction of the spike value. The spike values are calculated from sample preparation.

The assigned values for all parameters except NVOC, Cl and $SO_4$ are operationally defined and are consensus values based upon the median for method no. 3, 5, 63, 76, 77, 91, 92 and ISO 15705 ($COD_{Cr}$), method no. 1, 2, 5 and 7 (TP), or method no. 1 and 2 (TSS), which are the methods required by the Danish EPA /6/. Assigned values for BOD are based upon standardised methods (method no. 1 - 4). A list of method identification numbers is found in the report to participants /1/. Assigned values for Cl and $SO_4$ are consensus values for all laboratories based on the median.

### *2.3.2 Test of spike values*

A comparison was made (t-test, 95% confidence level) between the spike value and the difference in concentration between the two samples in the sample pair found from the laboratories' results, see Appendix B. The test revealed a significant difference between the two for $COD_{Cr}$. The difference could be attributed to influence from a few laboratories. The test was repeated after exclusion of the results from laboratory no. 40 and now showed no significant difference. Furthermore, the results of control measurements at Eurofins confirmed the assigned value (Appendix D). No significant differences were found for the remanding parameters. The test also revealed a significant difference between the two for $BOD_5$. However, the difference is numerically small and has insignificant influence on the general quality of analyses estimated from the data as well as on the evaluation of accuracy of participating laboratories. The remaining parameters did not indicate a significant difference for difference between sample pairs and spike value.

### *2.3.3 Test of assigned values*

The assigned value and the average of the results obtained from all laboratories were also compared (t-test, 95% confidence level), see Appendix C. The test showed no significant difference between the two and the control of assigned value at Eurofins confirmed the value (Appendix D).

# 3 HOMOGENEITY AND STABILITY OF SAMPLES

The homogeneity and stability of samples were tested using the following parameters as indicators:

| | |
|---|---|
| $COD_{Cr}$ | Combined homogeneity and stability test |
| TP | Combined homogeneity and stability test |
| TSS | Combined homogeneity and stability test |

The results of control measurements are shown in Appendix E. The appendix also gives the results of the statistical evaluation of the control data. The data are analysed by analysis of variance (ANOVA) giving:

1. the standard deviation/variance for replicates (the contribution from analytical variability),

2. the between bottle standard deviation/variance (the contribution from heterogeneity) and

3. the between days concentration difference (the contribution from instability).

Homogeneity is evaluated by comparing the between bottle variance to 0.3 * the standard deviation for evaluation of participants' performance ($0.3 \cdot \hat{\sigma}$) specified by the Danish EPA /6/, whereas the stability is evaluated by comparing the concentration change of the samples to $0.3 \cdot \hat{\sigma}$ or $0.3 * \hat{\sigma} + 2\sqrt{u_x^2 + u_y^2}$ where the precision of the measurement method contribute to the inability to meet the criterion. This test ensures that heterogeneity and instability will not have negative influence on the evaluation of participant performance /3/.

The appendix also shows the standard deviation within and between laboratories from the proficiency test to allow comparison between tests performed and average quality from participating laboratories.

The test for stability and homogeneity show that samples A1/B1 and A3/B3 are stable and homogeneous. Samples A2/B2 can be considered stable.

The homogeneity test performed on sample B2 could not verify that the samples are homogeneous. The found standard deviation between bottles exceeds the limit with a nominally small value and the standard deviation between laboratories are below $\hat{\sigma}$, hence the samples are still deemed suitable for evaluation the participant's results.

# 4 CONCLUSION

The quality control performed, including test of sample stability and homogeneity as well as test of recovery of spike and assigned values, shows that the samples and their assigned values are suitable for testing the proficiency of the participating laboratories for all parameters. The results are also suitable for estimation of the general quality of analyses among all participating laboratories.

For $COD_{Cr}$ and $BOD_5$ the participants could not recover the spike value (shown with t-test, 95% confidence level). The difference between the calculated spike value and that found by the participants is small and the influence on evaluation of participant performance or estimation of general quality of analyses is insignificant.

The homogeneity test performed on sample B2 could not verify that the samples are homogeneous. The found standard deviation between bottles exceeds the limit with a nominally small value and the standard deviation between laboratories are below $\hat{\sigma}$, hence the samples are still deemed suitable for evaluation the participant's results.

The test for stability and homogeneity show that samples A1/B1 and A3/B3 are stable and homogeneous. Samples A2/B2 can be considered stable.

# 5 REFERENCES


/1/ Eurofins A/S, *Proficiency test SPIL-1 (2026)*, Report to participants, April 2026.

/2/ ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*, 2025.

/3/ ISO 13528, *Statistical methods for use in proficiency testing by interlaboratory comparison*, 2022.

/4/ Spliid, H., *Procedure and analysis of data for proficiency tests and environmental analyses*, Report to Danish Environmental Protection Agency, 1994 (in Danish).

/5/ Eurofins A/S, *Schedule for a proficiency test*, document may be downloaded from www.eurofins.dk/proficiencytest.

/6/ Ministry of Environment and Equality regulation no. 1275 on *quality criteria for environmental measurements,* 31 October 2025 (in Danish).

# *A N N E X E S*

# *APPENDIX A SAMPLE PREPARATION*

| **Stock solution** | **Prepared from** | | **Concentration** |
|---|---|---|---|
| Concentrate A1 | 0.925 g<br>0.925 g | D-glucose<br>L-glutamic acid<br>milli-Q water up to 1000 g | $COD_{Cr}$: 1.892 g/kg<br>NVOC: 0.747 g/kg<br>$BOD_5$: 1.295 g/kg<br>$BOD_7$: 1.344 g/kg |
| Concentrate B1 | 0.7385 g<br>0.7385 g | D-glucose<br>L-glutamic acid<br>milli-Q water up to 1000 g | $COD_{Cr}$: 1.510 g/kg<br>NVOC: 0.597 g/kg<br>$BOD_5$: 1.034 g/kg<br>$BOD_7$: 1.073 g/kg |
| Stock TP | 1.507 g | Na-B.glycerophosphate<br>milli-Q water up to 1000.0 g | TP: 0.153 g/kg |
| Stock Cl | 10.003 g | Sodium chloride (NaCl)<br>milli-Q water up to 1000.0 g | Cl: 6.068 g/kg |
| Stock $SO_4$ | 5.006 g | Sodium sulphate ($Na_2SO_4$)<br>milli-Q water up to 1000.2 g | $SO_4$: 3.385 g/kg |
| Stock TSS | 15.000 g | Microcrystalline cellulose<br>milli-Q water up to 1000.0 l | TSS: 15.000 g/l |

| Sample | Sample prepared from | $COD_{Cr}$ mg/l $O_2$ | NVOC mg/l C | $BOD_5$ (w. ATU) mg/l $O_2$ | TP mg/l P | Cl mg/l | $SO_4$ mg/l | TSS mg/l |
|---|---|---|---|---|---|---|---|---|
| A1-COD/NVOC | At the laboratory 2.00 mL of concentrate A1 is diluted up to 100.0 mL with filtered water from Vejen sewage treatment plant | a + 37.8 | b + 14.9 | | | | | |
| B1-COD/NVOC | At the laboratory 2.00 mL of concentrate B1 is diluted up to 100.0 mL with filtered water from Vejen sewage treatment plant | a + 30.2 | b + 11.9 | | | | | |
| A1-BOD | At the laboratory 6.00 mL of concentrate A1 is diluted up to 2000.0 mL with filtered water from Vejen sewage treatment plant | | | c + 3.89 | | | | |
| B1-BOD | At the laboratory 6.00 mL of concentrate B1 is diluted up to 2000.0 mL with filtered water from Vejen sewage treatment plant | | | c + 3.10 | | | | |
| A2 | 150.0 g stock TP<br>280.0 g stock Cl<br>200.0 g stock $SO_4$<br>filtered water from Vejen sewage treatment plant up to 60.0 kg | | | | d + 0.380 | e + 28.22 | f + 11.3 | |
| B2 | 40.0 g stock TP<br>220.0 g stock Cl<br>150.0 g stock $SO_4$<br>Sample A2 up to 30.0 kg | | | | 0.986 · (d + 0.380) + 0.203 | 0.986 · (e + 28.22) + 44.34 | 0.986 · (f + 11.3) + 16.9 | |
| A3 | At the laboratory 1000.0 mL of filtered water from Vejen sewage treatment plant is added to 2.8 mL stock TSS | | | | | | | g + 41.9 |
| B3 | At the laboratory 1000.0 mL of filtered water from Vejen sewage treatment plant is added to 2.5 mL stock TSS | | | | | | | g + 37.4 |

## *APPENDIX B CONTROL OF SPIKE VALUES*

## *Chemical oxygen demand with dichromate ($COD_{Cr}$), mg/l $O_2$*

Control of differences within sample pairs

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 1 | 0.0 | |
| 2 | 12.9 | C |
| 4 | -23.1 | C |
| 5 | -1.5 | |
| 7 | -0.2 | |
| 8 | -4.1 | |
| 9 | -1.1 | |
| 10 | -2.4 | |
| 11 | -0.4 | |
| 12 | 4.0 | |
| 13 | 1.1 | |
| 15 | -1.4 | |
| 16 | 0.0 | |
| 17 | -1.3 | |
| 18 | -2.3 | |
| 19 | -6.5 | |
| 20 | 1.0 | |
| 21 | 0.0 | |
| 22 | -1.4 | |
| 23 | -2.0 | |
| 24 | 2.0 | |
| 25 | 1.6 | |
| 26 | 3.2 | |
| 27 | -4.6 | |

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 28 | 0.7 | |
| 29 | -2.0 | |
| 30 | -1.0 | |
| 31 | -0.4 | |
| 32 | 1.5 | |
| 33 | 1.3 | |
| 34 | -3.2 | |
| 35 | -2.8 | |
| 36 | -0.2 | |
| 37 | -4.8 | |
| 39 | -5.0 | |
| 40 | -10.0 | |
| 41 | -1.8 | |
| 43 | 0.8 | |
| 45 | 1.8 | |
| 46 | -3.9 | |
| 49 | -3.7 | |
| 50 | -0.7 | |
| 51 | -1.2 | |
| 53 | - | U |
| 55 | -1.0 | |

| Statistical data – Control of spike value | | |
|---|---|---|
| No of labs | 42 | **Notes** |
| No of repl | 2 | ** denotes that there is a significant difference (t-test, 1 %-level)<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| d | -1.2369 | |
| $s^2$ | 7.0698 | |
| s | 2.6589 | |
| t | -3.0148 ** | |
| Sign. level 99.9% | 3.5442 | |
| Sign. level 99% | 2.7012 | |
| Sign. level 95% | 2.0195 | |

## *Five day biochemical oxygen demand ($BOD_5$ w. ATU), mg/l $O_2$*

Control of differences within sample pairs

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 4 | -1.03 | G |
| 7 | -0.30 | |
| 11 | -0.40 | |
| 19 | -0.37 | |
| 20 | -0.20 | |
| 21 | 0.00 | |
| 23 | - | |
| 24 | -0.50 | |
| 25 | -0.14 | |
| 26 | 0.11 | |
| 28 | - | |
| 29 | -0.08 | |
| 30 | 0.10 | |
| 33 | -0.20 | |
| 37 | 3.00 | C |
| 40 | -1.20 | G |
| 41 | -0.19 | |
| 46 | -0.45 | |
| 51 | 0.09 | |
| 53 | 0.10 | |
| 55 | -0.11 | |

| Statistical data – Control of spike value | | | |
|---|---|---|---|
| No of labs | 16 | | **Notes** |
| No of repl | 2 | | ** denotes that there is a significant difference (t-test, 1 %-level)<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| d | -0.1591 | | |
| $s^2$ | 0.0419 | | |
| s | 0.2046 | | |
| t | -3.1097 | ** | |
| Sign. level 99.9% | 4.0728 | | |
| Sign. level 99% | 2.9467 | | |
| Sign. level 95% | 2.1314 | | |

## *Seven day biochemical oxygen demand ($BOD_7$ w. ATU), mg/l $O_2$*

Control of differences within sample pairs

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 2 | 0.00 | |
| 7 | -0.50 | |
| 12 | 0.03 | |
| 19 | 0.01 | |
| 20 | -0.11 | |
| 29 | -0.08 | |
| 32 | 0.55 | |
| 38 | 2.12 | C |
| 41 | -0.05 | |
| 42 | -0.01 | |
| 44 | -0.20 | |
| 52 | 0.17 | |
| 56 | 0.05 | |
| 57 | -0.07 | |

| Statistical data – Control of spike value | | |
|---|---|---|
| No of labs | 13 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| d | -0.0162 | |
| $s^2$ | 0.0540 | |
| s | 0.2323 | |
| t | -0.2507 | |
| Sign. level 99.9% | 4.3178 | |
| Sign. level 99% | 3.0545 | |
| Sign. level 95% | 2.1788 | |

## *Non-volatile/total organic carbon (NVOC/TOC), mg/l C*

Control of differences within sample pairs

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 2 | -0.80 | |
| 3 | -0.38 | |
| 6 | -1.03 | |
| 7 | 0.65 | |
| 11 | -0.37 | |
| 13 | 0.65 | |
| 14 | -5.44 | C |
| 15 | -0.53 | |
| 16 | 0.00 | |
| 17 | -1.00 | |
| 19 | 0.50 | |
| 20 | 0.10 | G |
| 21 | -0.60 | |
| 23 | 0.28 | |
| 24 | -0.60 | |
| 25 | 0.50 | |
| 26 | -0.31 | |
| 27 | 1.00 | |
| 28 | -0.50 | G |
| 29 | 0.00 | |
| 30 | 1.80 | |

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 33 | -1.00 | |
| 34 | -0.30 | |
| 35 | -0.02 | |
| 36 | 0.08 | G |
| 38 | 0.20 | |
| 40 | -0.16 | |
| 41 | -0.20 | |
| 42 | -0.48 | |
| 43 | 0.11 | |
| 44 | 0.50 | |
| 46 | 0.60 | G |
| 47 | -1.48 | |
| 49 | 0.60 | |
| 51 | -0.30 | |
| 52 | -1.30 | |
| 54 | 0.10 | |
| 55 | -1.52 | |
| 56 | -3.29 | C |
| 57 | 0.60 | |
| 58 | -0.60 | |

| Statistical data – Control of spike value | | |
|---|---|---|
| No of labs | 35 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| d | -0.1569 | |
| $s^2$ | 0.5277 | |
| s | 0.7264 | |
| t | -1.2775 | |
| Sign. level 99.9% | 3.6007 | |
| Sign. level 99% | 2.7284 | |
| Sign. level 95% | 2.0322 | |

## *Total phosphorus (TP), mg/l*

Control of differences within sample pairs

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 1 | 0.006 | |
| 2 | -0.035 | |
| 3 | 0.009 | |
| 4 | 0.027 | |
| 5 | 0.450 | C |
| 6 | 0.004 | |
| 7 | -0.002 | |
| 8 | -0.010 | |
| 9 | 0.005 | |
| 10 | 0.020 | |
| 11 | 0.000 | |
| 13 | -0.011 | |
| 14 | -0.004 | |
| 15 | 0.007 | |
| 16 | 0.032 | |
| 17 | -0.010 | |
| 18 | -0.010 | |
| 19 | -0.072 | C |
| 20 | 0.007 | |
| 21 | -0.000 | |
| 22 | 0.011 | |
| 23 | 0.000 | |
| 24 | 0.080 | C |
| 25 | 0.038 | |
| 26 | 0.010 | |
| 27 | 0.011 | |
| 28 | -0.007 | |
| 29 | -0.007 | |
| 30 | -0.010 | |

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 31 | 0.013 | |
| 32 | 0.010 | |
| 33 | -0.110 | C |
| 34 | 0.030 | |
| 35 | 0.000 | |
| 36 | -0.005 | |
| 37 | -0.010 | |
| 38 | 0.050 | |
| 39 | -0.020 | |
| 40 | -0.010 | |
| 41 | 0.006 | |
| 42 | -0.005 | |
| 43 | -0.030 | |
| 44 | -0.003 | |
| 45 | 0.007 | |
| 46 | -0.026 | |
| 47 | -0.006 | |
| 48 | -0.010 | |
| 49 | -0.006 | |
| 50 | 0.010 | |
| 51 | 0.010 | |
| 52 | -0.020 | |
| 53 | -0.005 | |
| 54 | -0.010 | |
| 55 | 0.012 | |
| 56 | -0.015 | |
| 57 | -0.010 | |
| 58 | -0.030 | |

## *Total phosphorus (TP), mg/l*

| Statistical data – Control of spike value | | |
|---|---|---|
| No of labs | 53 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| d | 0.0002 | |
| $s^2$ | 0.0003 | |
| s | 0.0166 | |
| t | 0.0711 | |
| Sign. level 99.9% | 3.4877 | |
| Sign. level 99% | 2.6737 | |
| Sign. level 95% | 2.0066 | |

## *Chloride (Cl), mg/l*

Control of differences within sample pairs

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 4 | 2.0 | |
| 7 | 1.3 | |
| 8 | 84.8 | C |
| 9 | -10.5 | G |
| 10 | -2.0 | |
| 11 | 1.7 | |
| 13 | 0.9 | |
| 14 | 0.6 | |
| 16 | 0.0 | |
| 18 | -10.0 | |
| 19 | 0.0 | |
| 20 | -2.0 | |
| 21 | 4.0 | |
| 22 | -3.0 | |
| 23 | -1.0 | |
| 24 | 1.0 | |
| 25 | -2.9 | |
| 26 | 0.0 | |

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 28 | -1.5 | |
| 29 | -1.0 | |
| 30 | 6.0 | |
| 31 | -1.0 | |
| 33 | 0.1 | |
| 35 | -0.5 | |
| 36 | -1.1 | |
| 39 | 10.1 | |
| 40 | 6.2 | |
| 41 | -0.3 | |
| 42 | -4.2 | |
| 43 | -2.0 | |
| 46 | 17.1 | C |
| 47 | 0.3 | |
| 51 | 3.0 | |
| 53 | -9.0 | |
| 55 | -1.3 | |

| Statistical data – Control of spike value | | |
|---|---|---|
| No of labs | 32 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| d | -0.1743 | |
| $s^2$ | 14.4508 | |
| s | 3.8014 | |
| t | -0.2593 | |
| Sign. level 99.9% | 3.6335 | |
| Sign. level 99% | 2.7440 | |
| Sign. level 95% | 2.0395 | |

## *Sulphate ($SO_4$), mg/l*

Control of differences within sample pairs

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 7 | 0.9 | |
| 11 | 0.7 | |
| 13 | 1.8 | |
| 14 | 11.8 | C |
| 16 | 0.0 | |
| 19 | 0.8 | |
| 20 | 0.6 | |
| 21 | 0.3 | |
| 22 | 0.8 | |
| 23 | 2.7 | |
| 25 | 1.0 | |
| 28 | -0.5 | |
| 29 | -0.5 | |
| 30 | -1.9 | |
| 33 | 1.6 | |
| 35 | 1.6 | |
| 39 | 6.1 | C |
| 40 | -2.0 | |
| 41 | 0.1 | |
| 43 | -0.8 | |
| 46 | -0.7 | |
| 49 | -2.9 | |
| 51 | -0.3 | |
| 55 | 1.8 | |

| Statistical data – Control of spike value | | |
|---|---|---|
| No of labs | 22 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| d | 0.2307 | |
| $s^2$ | 1.8903 | |
| s | 1.3749 | |
| t | 0.7870 | |
| Sign. level 99.9% | 3.8193 | |
| Sign. level 99% | 2.8314 | |
| Sign. level 95% | 2.0796 | |

## *Total suspended solids (TSS), mg/l*

Control of differences within sample pairs

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 1 | -0.90 | |
| 2 | 0.00 | |
| 3 | 7.90 | |
| 4 | -17.10 | C |
| 6 | -0.44 | |
| 7 | 0.83 | |
| 8 | -0.10 | |
| 11 | -0.03 | |
| 12 | -1.20 | |
| 13 | 0.50 | |
| 14 | -3.35 | |
| 15 | 0.20 | |
| 16 | 3.50 | |
| 17 | 0.50 | |
| 19 | 0.30 | |
| 20 | -1.10 | |
| 21 | -0.50 | |
| 22 | -0.50 | |
| 23 | 0.00 | |
| 24 | 1.90 | |
| 25 | -0.90 | |
| 26 | 0.50 | |
| 27 | 7.70 | |
| 28 | 0.60 | |
| 29 | 0.30 | |
| 30 | 3.50 | |

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 31 | -0.40 | |
| 32 | 5.50 | |
| 33 | 3.20 | |
| 34 | 2.80 | |
| 35 | -1.00 | |
| 36 | 2.30 | |
| 37 | -1.10 | |
| 38 | -9.50 | |
| 40 | -4.10 | |
| 41 | 0.20 | |
| 42 | 0.20 | |
| 43 | -4.50 | |
| 44 | -2.20 | |
| 45 | 0.00 | |
| 46 | -0.10 | |
| 47 | 0.10 | |
| 49 | -1.00 | |
| 50 | -2.50 | |
| 51 | -2.50 | |
| 52 | 0.80 | |
| 53 | 1.50 | |
| 55 | 1.10 | |
| 56 | -3.70 | |
| 57 | 1.50 | |
| 58 | 2.10 | |

| Statistical data – Control of spike value | | |
|---|---|---|
| No of labs | 50 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| d | 0.1582 | |
| $s^2$ | 8.0197 | |
| s | 2.8319 | |
| t | 0.3950 | |
| Sign. level 99.9% | 3.5004 | |
| Sign. level 99% | 2.6800 | |
| Sign. level 95% | 2.0096 | |

## *APPENDIX C CONTROL OF RECOVERY*

## *Chemical oxygen demand with dichromate ($COD_{Cr}$), mg/l $O_2$*

Control of recovery, average of results

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 1 | 54.0 | |
| 2 | 67.4 | C |
| 4 | 33.8 | C |
| 5 | 36.5 | |
| 7 | 62.0 | |
| 8 | 54.0 | |
| 9 | 56.6 | |
| 10 | 54.7 | |
| 11 | 60.2 | |
| 12 | 52.0 | |
| 13 | 64.9 | |
| 15 | 65.1 | |
| 16 | 56.0 | |
| 17 | 58.6 | |
| 18 | 52.3 | |
| 19 | 59.3 | |
| 20 | 30.2 | |
| 21 | 52.0 | |
| 22 | 55.2 | |
| 23 | 58.0 | |
| 24 | 60.0 | |
| 25 | 56.8 | |
| 26 | 57.8 | |

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 27 | 64.5 | |
| 28 | 66.7 | |
| 29 | 62.0 | |
| 30 | 61.5 | |
| 31 | 57.6 | |
| 32 | 60.5 | |
| 33 | 59.6 | |
| 34 | 63.1 | |
| 35 | 53.8 | |
| 36 | 31.0 | |
| 37 | 50.6 | |
| 39 | 35.5 | |
| 40 | 57.0 | |
| 41 | 59.7 | |
| 43 | 57.8 | |
| 45 | 30.5 | |
| 46 | 55.3 | |
| 49 | 64.2 | |
| 50 | 61.8 | |
| 51 | 64.7 | |
| 53 | - | U |
| 55 | 57.5 | |

## *Chemical oxygen demand with dichromate ($COD_{Cr}$), mg/l $O_2$*

| Statistical data – Control of recovery | | |
|---|---|---|
| No of labs | 42 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| m | 55.4908 | |
| $s^2$ | 88.1328 | |
| s | 9.3879 | |
| Assigned value | 58 | |
| Recovery | 95.7 | |
| t | -1.7321 | |
| Sign. level 99.9% | 3.5442 | |
| Sign. level 99% | 2.7012 | |
| Sign. level 95% | 2.0195 | |

## *Five day biochemical oxygen demand ($BOD_5$ w. ATU), mg/l $O_2$*

Control of recovery, average of results

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 4 | 0.18 | G |
| 7 | 4.75 | |
| 11 | 4.56 | |
| 19 | 4.15 | |
| 20 | 4.39 | |
| 21 | 6.40 | |
| 23 | -999 | |
| 24 | 5.35 | |
| 25 | 4.88 | |
| 26 | 5.01 | |
| 28 | -999 | |
| 29 | 5.05 | |
| 30 | 5.15 | |
| 33 | 5.40 | |
| 37 | 2.50 | C |
| 40 | 8.32 | G |
| 41 | 4.35 | |
| 46 | 4.51 | |
| 51 | 4.53 | |
| 53 | 6.25 | |
| 55 | 5.63 | |

| Statistical data – Control of recovery | | |
|---|---|---|
| No of labs | 16 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| m | 5.0214 | |
| $s^2$ | 0.4313 | |
| s | 0.6567 | |
| Assigned value | 4.8 | |
| Recovery | 104.6 | |
| t | 1.3485 | |
| Sign. level 99.9% | 4.0728 | |
| Sign. level 99% | 2.9467 | |
| Sign. level 95% | 2.1314 | |

## *Seven day biochemical oxygen demand ($BOD_7$ w. ATU), mg/l $O_2$*

Control of recovery, average of results

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 2 | 5.40 | |
| 7 | 5.75 | |
| 12 | 5.68 | |
| 19 | 4.69 | |
| 20 | 5.46 | |
| 29 | 5.30 | |
| 32 | 6.72 | |
| 38 | 6.74 | C |
| 41 | 4.87 | |
| 42 | 5.56 | |
| 44 | 5.40 | |
| 52 | 6.37 | |
| 56 | 6.48 | |
| 57 | 5.66 | |

| Statistical data – Control of recovery | | |
|---|---|---|
| No of labs | 13 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| m | 5.6388 | |
| $s^2$ | 0.3495 | |
| s | 0.5912 | |
| Assigned value | 5.5 | |
| Recovery | 102.5 | |
| t | 0.8468 | |
| Sign. level 99.9% | 4.3178 | |
| Sign. level 99% | 3.0545 | |
| Sign. level 95% | 2.1788 | |

## *Non-volatile/total organic carbon (NVOC/TOC), mg/l C*

Control of recovery, average of results

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 2 | 21.10 | |
| 3 | 22.47 | |
| 6 | 22.63 | |
| 7 | 23.44 | |
| 11 | 22.26 | |
| 13 | 21.71 | |
| 14 | 21.73 | C |
| 15 | 22.35 | |
| 16 | 22.00 | |
| 17 | 23.70 | |
| 19 | 22.85 | |
| 20 | 12.15 | G |
| 21 | 21.00 | |
| 23 | 21.09 | |
| 24 | 22.60 | |
| 25 | 18.85 | |
| 26 | 23.38 | |
| 27 | 23.30 | |
| 28 | 65.95 | G |
| 29 | 23.50 | |
| 30 | 22.20 | |

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 33 | 22.20 | |
| 34 | 21.45 | |
| 35 | 24.69 | |
| 36 | 12.65 | G |
| 38 | 21.60 | |
| 40 | 24.31 | |
| 41 | 23.21 | |
| 42 | 24.27 | |
| 43 | 23.07 | |
| 44 | 22.75 | |
| 46 | 29.58 | G |
| 47 | 26.02 | |
| 49 | 22.80 | |
| 51 | 21.65 | |
| 52 | 23.25 | |
| 54 | 22.35 | |
| 55 | 22.94 | |
| 56 | 25.85 | C |
| 57 | 24.00 | |
| 58 | 22.90 | |

| Statistical data – Control of recovery | | |
|---|---|---|
| No of labs | 35 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| m | 22.6816 | |
| $s^2$ | 1.6111 | |
| s | 1.2693 | |
| Assigned value | 22.7 | |
| Recovery | 99.9 | |
| t | -0.0859 | |
| Sign. level 99.9% | 3.6007 | |
| Sign. level 99% | 2.7284 | |
| Sign. level 95% | 2.0322 | |

## *Total phosphorus (TP), mg/l*

Control of recovery, average of results

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 1 | 0.933 | |
| 2 | 0.999 | |
| 3 | 0.925 | |
| 4 | 0.914 | |
| 5 | 1.113 | C |
| 6 | 0.962 | |
| 7 | 0.949 | |
| 8 | 1.025 | |
| 9 | 0.972 | |
| 10 | 0.890 | |
| 11 | 0.950 | |
| 13 | 0.954 | |
| 14 | 0.953 | |
| 15 | 0.954 | |
| 16 | 0.976 | |
| 17 | 1.005 | |
| 18 | 1.095 | |
| 19 | 0.974 | C |
| 20 | 0.962 | |
| 21 | 0.930 | |
| 22 | 0.951 | |
| 23 | 0.960 | |
| 24 | 1.020 | C |
| 25 | 0.924 | |
| 26 | 0.965 | |
| 27 | 0.936 | |
| 28 | 0.998 | |
| 29 | 0.907 | |
| 30 | 1.005 | |

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 31 | 0.956 | |
| 32 | 0.905 | |
| 33 | 1.055 | C |
| 34 | 0.980 | |
| 35 | 1.010 | |
| 36 | 0.968 | |
| 37 | 0.965 | |
| 38 | 1.025 | |
| 39 | 1.070 | |
| 40 | 0.905 | |
| 41 | 0.903 | |
| 42 | 0.971 | |
| 43 | 0.955 | |
| 44 | 0.939 | |
| 45 | 0.944 | |
| 46 | 0.931 | |
| 47 | 0.947 | |
| 48 | 1.020 | |
| 49 | 0.957 | |
| 50 | 0.965 | |
| 51 | 1.065 | |
| 52 | 0.930 | |
| 53 | 0.958 | |
| 54 | 0.961 | |
| 55 | 0.952 | |
| 56 | 0.943 | |
| 57 | 0.965 | |
| 58 | 1.035 | |

## *Total phosphorus (TP), mg/l*

| Statistical data – Control of recovery | | |
|---|---|---|
| No of labs | 53 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| m | 0.9644 | |
| $s^2$ | 0.0018 | |
| s | 0.0430 | |
| Assigned value | 0.96 | |
| Recovery | 100.5 | |
| t | 0.7474 | |
| Sign. level 99.9% | 3.4877 | |
| Sign. level 99% | 2.6737 | |
| Sign. level 95% | 2.0066 | |

## *Chloride (Cl), mg/l*

Control of recovery, average of results

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 4 | 151.0 | |
| 7 | 159.5 | |
| 8 | 173.4 | C |
| 9 | 140.8 | G |
| 10 | 181.0 | |
| 11 | 167.4 | |
| 13 | 169.6 | |
| 14 | 173.6 | |
| 16 | 165.0 | |
| 18 | 185.0 | |
| 19 | 173.0 | |
| 20 | 169.0 | |
| 21 | 175.0 | |
| 22 | 165.5 | |
| 23 | 170.9 | |
| 24 | 169.5 | |
| 25 | 186.0 | |
| 26 | 171.0 | |

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 28 | 174.5 | |
| 29 | 177.5 | |
| 30 | 171.0 | |
| 31 | 169.5 | |
| 33 | 172.9 | |
| 35 | 177.6 | |
| 36 | 173.1 | |
| 39 | 162.7 | |
| 40 | 166.3 | |
| 41 | 169.6 | |
| 42 | 173.0 | |
| 43 | 174.0 | |
| 46 | 169.8 | C |
| 47 | 173.6 | |
| 51 | 172.5 | |
| 53 | 162.5 | |
| 55 | 171.7 | |

| Statistical data – Control of recovery | | |
|---|---|---|
| No of labs | 32 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| m | 171.0512 | |
| $s^2$ | 47.0903 | |
| s | 6.8622 | |
| Assigned value | 171 | |
| Recovery | 100.0 | |
| t | 0.0422 | |
| Sign. level 99.9% | 3.6335 | |
| Sign. level 99% | 2.7440 | |
| Sign. level 95% | 2.0395 | |

## *Sulphate ($SO_4$), mg/l*

Control of recovery, average of results

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 7 | 52.9 | |
| 11 | 52.4 | |
| 13 | 50.6 | |
| 14 | 59.2 | C |
| 16 | 53.0 | |
| 19 | 56.4 | |
| 20 | 55.5 | |
| 21 | 58.3 | |
| 22 | 66.1 | |
| 23 | 61.7 | |
| 25 | 58.9 | |
| 28 | 53.8 | |
| 29 | 56.1 | |
| 30 | 55.2 | |
| 33 | 52.8 | |
| 35 | 51.1 | |
| 39 | 40.7 | C |
| 40 | 60.3 | |
| 41 | 55.1 | |
| 43 | 65.0 | |
| 46 | 57.8 | |
| 49 | 53.8 | |
| 51 | 54.4 | |
| 55 | 55.5 | |

| Statistical data – Control of recovery | | |
|---|---|---|
| No of labs | 22 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| m | 56.1944 | |
| $s^2$ | 17.1142 | |
| s | 4.1369 | |
| Assigned value | 55 | |
| Recovery | 102.2 | |
| t | 1.3542 | |
| Sign. level 99.9% | 3.8193 | |
| Sign. level 99% | 2.8314 | |
| Sign. level 95% | 2.0796 | |

## *Total suspended solids (TSS), mg/l*

Control of recovery, average of results

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 1 | 38.35 | |
| 2 | 38.25 | |
| 3 | 39.95 | |
| 4 | 45.42 | C |
| 6 | 37.75 | |
| 7 | 36.92 | |
| 8 | 37.45 | |
| 11 | 35.39 | |
| 12 | 37.10 | |
| 13 | 37.25 | |
| 14 | 38.31 | |
| 15 | 34.40 | |
| 16 | 38.75 | |
| 17 | 37.25 | |
| 19 | 37.85 | |
| 20 | 36.05 | |
| 21 | 36.75 | |
| 22 | 35.75 | |
| 23 | 36.50 | |
| 24 | 36.95 | |
| 25 | 37.45 | |
| 26 | 37.25 | |
| 27 | 40.95 | |
| 28 | 35.10 | |
| 29 | 37.45 | |
| 30 | 36.75 | |

| Laboratory | Sample pair AB | Outlier |
|---|---|---|
| 31 | 36.80 | |
| 32 | 35.45 | |
| 33 | 38.70 | |
| 34 | 35.90 | |
| 35 | 34.50 | |
| 36 | 38.55 | |
| 37 | 38.15 | |
| 38 | 40.45 | |
| 40 | 38.65 | |
| 41 | 37.40 | |
| 42 | 36.90 | |
| 43 | 37.75 | |
| 44 | 38.70 | |
| 45 | 36.40 | |
| 46 | 38.65 | |
| 47 | 36.95 | |
| 49 | 36.60 | |
| 50 | 35.75 | |
| 51 | 35.55 | |
| 52 | 37.70 | |
| 53 | 35.75 | |
| 55 | 35.75 | |
| 56 | 36.35 | |
| 57 | 36.85 | |
| 58 | 35.25 | |

| Statistical data – Control of recovery | | |
|---|---|---|
| No of labs | 50 | **Notes** |
| No of repl | 2 | No test statistics were found to be significant<br><br>U denote a manual selected outlier<br>C denotes a Cochran outlier<br>G denotes a Grubbs outlier |
| m | 37.1471 | |
| $s^2$ | 2.0018 | |
| s | 1.4149 | |
| Assigned value | 37.2 | |
| Recovery | 99.9 | |
| t | -0.2644 | |
| Sign. level 99.9% | 3.5004 | |
| Sign. level 99% | 2.6800 | |
| Sign. level 95% | 2.0096 | |

## *APPENDIX D CONCENTRATION LEVEL*

| Parameter | Unit | Sample | Bottle no. | Ampoule no. | I | II | Bottle Average | Sample Average | Assigned value | Spike Measured | Spike Assigned |
|---|---|---|---|---|---|---|---|---|---|---|---|
| $COD_{Cr}$ | mg/L $O_2$ | A1 | 15 | 7 | 62.4 | 61.4 | 61.9 | 60.7 | 66 | 5.0 | 8 |
| | | | 23 | 49 | 58.8 | 59.4 | 59.1 | | | | |
| | | | 68 | 82 | 58.4 | 63.6 | 61.0 | | | | |
| | | B1 | 27 | 4 | 55.0 | 55.8 | 55.4 | 55.6 | 58 | | |
| | | | 55 | 24 | 56.6 | 56.6 | 56.6 | | | | |
| | | | 70 | 67 | 55.0 | 54.8 | 54.9 | | | | |
| Total phosphorus | mg/L P | A2 | 3 | | 1.01 | 1.00 | 1.01 | 0.99 | 0.96 | 0.17 | 0.19 |
| | | | 31 | | 0.97 | 0.97 | 0.97 | | | | |
| | | | 58 | | 0.98 | 0.98 | 0.98 | | | | |
| | | B2 | 1 | | 1.16 | 1.16 | 1.16 | 1.16 | 1.15 | | |
| | | | 49 | | 1.15 | 1.15 | 1.15 | | | | |
| | | | 81 | | 1.17 | 1.17 | 1.17 | | | | |
| Chloride | mg/L | A2 | 3 | | 161 | 162 | 162 | 161 | 171 | 44 | 42 |
| | | | 31 | | 163 | 164 | 164 | | | | |
| | | | 58 | | 158 | 159 | 159 | | | | |
| | | B2 | 1 | | 204 | 205 | 205 | 205 | 213 | | |
| | | | 49 | | 202 | 203 | 203 | | | | |
| | | | 81 | | 207 | 207 | 207 | | | | |
| Sulphate | mg/L | A2 | 3 | | 61.7 | 61.3 | 61.5 | 62.0 | 55 | 15.3 | 16 |
| | | | 31 | | 62.4 | 62.2 | 62.3 | | | | |
| | | | 58 | | 62.4 | 62.1 | 62.3 | | | | |
| | | B2 | 1 | | 78.6 | 78.2 | 78.4 | 77.4 | 71 | | |
| | | | 49 | | 76.6 | 76.5 | 76.6 | | | | |
| | | | 81 | | 77.1 | 77.1 | 77.1 | | | | |

| Parameter | Unit | Sample | Matrix no. | Concentrate no. | I | II | Bottle | Sample Average | Assigned value | Spike Measured | Sample Average |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Total suspended solids | mg/L | A3 | 15 | 3 | 42.082 | | 42.08 | 42.45 | 41.7 | 4.94 | 4.5 |
| | | | 38 | 53 | 42.381 | | 42.38 | | | | |
| | | | 63 | 70 | 42.880 | | 42.88 | | | | |
| | | B3 | 2 | 5 | 37.905 | | 37.91 | 37.51 | 37.2 | | |
| | | | 38 | 44 | 37.107 | | 37.11 | | | | |
| | | | 75 | 73 | 37.506 | | 37.51 | | | | |

# *APPENDIX E HOMOGENEITY AND STABILITY*

**PT:** SPIL-1 2026
**Parameter:** COD - A1
**Unit:** mg/L $O_2$

**Sigma:** 10.8

**Responsible** for tests: O6HJ/DWY9/ DHBP/S7MS
**Approval of control test:** FYE3

**Homogeneity test** Date: 2026-02-25

| Sample | x(a) | x(b) | aver-age | sd | sd^2 |
|---|---|---|---|---|---|
| 1-5 | 66.0 | 66.8 | 66.4 | 0.566 | 0.320 |
| 12-8 | 66.6 | 64.2 | 65.4 | 1.697 | 2.88 |
| 16-18 | 66.4 | 67.2 | 66.8 | 0.566 | 0.320 |
| 25-23 | 64.8 | 64.2 | 64.5 | 0.424 | 0.180 |
| 27-27 | 65.6 | 64.4 | 65.0 | 0.849 | 0.720 |
| 36-37 | 68.8 | 65.6 | 67.2 | 2.263 | 5.120 |
| 46-47 | 68.2 | 65.8 | 67.0 | 1.697 | 2.880 |
| 51-54 | 68.8 | 66.4 | 67.6 | 1.697 | 2.880 |
| 58-55 | 63.4 | 64.2 | 63.8 | 0.566 | 0.320 |
| 67-67 | 66.0 | 65.2 | 65.6 | 0.566 | 0.320 |
| 70-72 | 62.4 | 61.4 | 61.9 | 0.707 | 0.500 |
| 77-77 | 63.0 | 63.4 | 63.2 | 0.283 | 0.080 |

**For homogeneity**

| | |
|---|---|
| General average (x) | 65.37 |
| Sample average sd ($s_x$) | 1.764 |
| Within-sample sd ($s_w$): | 1.173 |
| Between-samples sd (ss): | 1.5572 |
| $S_L$ in the Proficiency Test: | 9.275 |
| $S_R$ in the Proficiency Test: | 9.45 |

**Stability test** Date: 2026-03-12

| Sample | x(a) | x(b) |
|---|---|---|
| 15-7 | 62.4 | 61.4 |
| 23-49 | 58.8 | 59.4 |
| 68-82 | 58.4 | 63.6 |
| | | |

**For stability**

| | |
|---|---|
| General average (y): | 60.66667 |
| /x-y/ = | 4.7 |

**Conclusions**

| | |
|---|---|
| ss = | 1.5572 |
| /x-y/ = | 4.700 |
| 0.3*sigma= | 3.240 |
| $0.3 * \sigma_{pt} + 2\sqrt{u^2(x) + u^2(y)}$= | 8.412 |

**Analytical quality** Is $s_w$ < 0.15*sigma
**YES**

**Homoge-neity:** Is ss < 0.3*sigma?
**YES**

**Stability:** /x-y/ < 0.3*sigma?
**NO**

**Stability*:** $|x-y| < 0{,}3\sigma_{pt} + 2\sqrt{(u^2(x) + u^2(y))}$ ?
**YES**

**PT:** SPIL-1 2026
**Parameter:** TP - B2
**Unit:** mg/L P

**Sigma:** 0.064

**Responsible** for tests: O6HJ/DWY9/ DHBP/S7MS

**Approval of control test:** FYE3

**Homogeneity test** Date: 2026-02-25

| Sample | x(a) | x(b) | aver-age | sd | sd^2 |
|---|---|---|---|---|---|
| 6 | 1.19 | 1.19 | 1.19 | 0.000 | 0.000 |
| 7 | 1.18 | 1.18 | 1.18 | 0.000 | 0.00 |
| 18 | 1.22 | 1.22 | 1.22 | 0.000 | 0.000 |
| 21 | 1.17 | 1.17 | 1.17 | 0.000 | 0.000 |
| 32 | 1.19 | 1.19 | 1.19 | 0.000 | 0.000 |
| 36 | 1.18 | 1.18 | 1.18 | 0.000 | 0.000 |
| 44 | 1.18 | 1.18 | 1.18 | 0.000 | 0.000 |
| 52 | 1.17 | 1.17 | 1.17 | 0.000 | 0.000 |
| 57 | 1.17 | 1.17 | 1.17 | 0.000 | 0.000 |
| 65 | 1.17 | 1.17 | 1.17 | 0.000 | 0.000 |
| 71 | 1.23 | 1.23 | 1.23 | 0.000 | 0.000 |
| 75 | 1.18 | 1.18 | 1.18 | 0.000 | 0.000 |

**For homogeneity**

| | |
|---|---|
| General average (x) | 1.19 |
| Sample average sd ($s_x$) | 0.020 |
| Within-sample sd ($s_w$): | 0.000 |
| Between-samples sd (ss): | 0.0198 |
| $S_L$ in the Proficiency Test: | 0.042 |
| $S_R$ in the Proficiency Test: | 0.044 |

**Stability test** Date: 2026-03-12

| Sample | x(a) | x(b) |
|---|---|---|
| 1 | 1.16 | 1.16 |
| 49 | 1.15 | 1.15 |
| 81 | 1.17 | 1.17 |
| | | |

**For stability**

| | |
|---|---|
| General average (y): | 1.16 |
| /x-y/ = | 0.025833 |

**Conclusions**

| | |
|---|---|
| ss = | 0.0198 |
| /x-y/ = | 0.026 |
| 0.3*sigma= | 0.0192 |
| $0.3 * \sigma_{pt} + 2\sqrt{u^2(x) + u^2(y)}$= | 0.222 |

**Analytical quality** Is $s_w$ < 0.15*sigma
**YES**

**Homogeneity:** Is ss < 0.3*sigma?
**NO**

**Stability:** /x-y/ < 0.3*sigma?
**NO**

**Stability:** $|x-y| < 0{,}3\sigma_{pt} + 2\sqrt{(u^2(x) + u^2(y))}$ ?
**YES**

**PT:** SPIL-1 2026
**Parameter:** TSS - B3
**Unit:** mg/L

**Sigma:** 3.6

**Responsible** for tests: O6HJ/DWY9/ DHBP/S7MS

**Approval of control test:** FYE3

**Homogeneity test** Date: 2026-02-25

| Sample | x(a) | x(b) | aver-age | sd | sd^2 |
|---|---|---|---|---|---|
| 3-1 | 37.3 | | 37.3 | | |
| 8-7 | 36.9 | | 36.9 | | |
| 16-14 | 37.3 | | 37.3 | | |
| 23-20 | 37.0 | | 37.0 | | |
| 31-31 | 37.4 | | 37.4 | | |
| 39-37 | 37.2 | | 37.2 | | |
| 43-43 | 37.2 | | 37.2 | | |
| 49-51 | 37.4 | | 37.4 | | |
| 57-58 | 37.4 | | 37.4 | | |
| 62-61 | 37.4 | | 37.4 | | |
| 69-69 | 37.3 | | 37.3 | | |
| 78-76 | 37.4 | | 37.4 | | |

**For homogeneity**

| | |
|---|---|
| General average (x) | 37.3 |
| Sample average sd ($s_x$) | 0.167 |
| Within-sample sd ($s_w$): | |
| Between-samples sd (ss): | 0.167 |
| $S_L$ in the Proficiency Test: | 0.1753 |
| $S_R$ in the Proficiency Test: | 1.9932 |

**Stability test** Date: 2026-03-12

| Sample | x(a) | x(b) |
|---|---|---|
| 2-5 | 37.905 | |
| 38-44 | 37.107 | |
| 75-73 | 37.506 | |
| | | |

**For stability**

| | |
|---|---|
| General average (y): | 37.506 |
| /x-y/ = | 0.232517 |

**Conclusions**

ss = 0.17 0.3*sigma= 1.08
/x-y/ = 0.232517

**Analytical quality** Is $s_w$ < 0.15*sigma
**No data**

**Homoge-neity:** Is ss < 0.3*sigma?
**YES**

**Stability:** /x-y/ < 0.3*sigma?
**YES**