Split Sample Audit Program
Guidance Document for Permit Holders

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Ministry of Environment and Climate Change Strategy's
Knowledge Management Branch

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1. ACRONYMS AND DEFINITIONS

Acronyms

EPD – Environmental Protection Division

EPO – Environmental Protection Officer

ENV – Ministry of Environment and Climate Change Strategy

FLNRO – Ministry of Forests, Lands, and Natural Resource Operations

LSQA – Laboratory Standards and Quality Assurance unit of the Ministry's Knowledge Management Branch of the Ministry.

QA – Quality Assurance

QC – Quality Control

QM – Quality Management

Definitions

Analyte: A substance whose chemical constituents are being identified and measured.

Audit event: The complete sampling and evaluation process of a single split sample audit.

Certified Reference Material (CRM): A stabilized material with parametric values established through inter-study testing. CRMs are used mainly to conduct Performance Tests by laboratories.

Drop Sign: The drop sign function is a manual function used in technical scoring (see Section 7.2). In the event that an analytical test result is reported at below Reportable Detection Limit (< RDL), the "<" sign is deleted to allow the tables functions to complete and evaluation of the result. For reporting purposes the "<" sign is replaced but the calculated results stand and are included in the Audit.

In-house laboratory: A laboratory that is operated by a permittee.

Parameter: A numerical characteristic of a population.

Ministry or ENV: The British Columbia Ministry of Environment and Climate Change Strategy.

Performance Test: A test of a laboratory's performance of a given analytical test method of certified reference materials.

Permittee: A company authorized to discharge effluent from their industrial operations under a permit provided by a director.

Reference data: Analytical test results of sample material obtained and submitted to a reference laboratory by a provincial ministry staff.

Reference laboratory: A laboratory, contracted by the Province to provide analytical testing of environmental media, whose performance is unbiased as demonstrated in inter-laboratory performance studies or blind audits.

Reference value: The concentration of an analyte reported by the Ministry.

Sample Set: Complete set of samples obtained during one audit. Complete set of samples required for a single monitoring event.

Sample splitter: A vessel made of an inert material which is fit with a manual churning device and spigot.

Split sample audit: The complete sampling, analysis and evaluation process of a single split sample audit event.

Split samples: Two aliquots of effluent obtained from a single contained source, typically a sample splitter, under constant agitation.

Test Suite: A suite of parameters belonging to a family of analytes such as metals or acids. Typically refers to a 'test' requested by either the Ministry or the permittee as opposed to individual analyte tests such as nitrate or colour.

2. INTRODUCTION

The Ministry of Environment and Climate Change Strategy (ENV) is charged with the responsibility of safeguarding the environmental integrity of the Provinces natural resources while accommodating the needs of industry. This responsibility is accomplished by managing and mitigating the effects of past, present and future activities on B.C.'s environment through leadership in science, governance, assessment, authorizations and compliance.

Industries that operate within BC are required to monitor and test the quality of the effluents they are permitted to discharge. Analytical data provided to ENV by permit holders is used to monitor and ensure compliance with the permits that authorize their discharge and ultimately account for the environmental protection they are designed to accomplish. For this reason it is incumbent upon ENV to ensure that the analytical data provided by permit holders meets an acceptably high level of quality.

The Split Sampling Program is designed to ensure that a high level of quality and confidence remains an integral component of the analytical data used by the Ministry of Environment and Climate Change Strategy. A high level of environmental data quality helps ensure that environmental protection is delivered through compliance permit criteria.

3. PURPOSE

The Split Sampling Program was established and implemented in 1990. In this year the program's objective was "to monitor and evaluate permit monitoring performance." The original guidance document went on to state "This encompasses both field sampling and laboratory analyses issues. The overall emphasis of the program will be to work co-operatively with permittees to improve and maintain data quality." This program objective has been upheld and remains the primary objective of this quality assurance program.

The Split Sampling Program provides compliance staff with a reliable mechanism to assess and monitor the quality of the data produced by permit holders. The results of the Split Sampling Audits ensure that the analytical data that is provided by permit holders to demonstrate compliance with their permit criteria

meets an acceptable level of precision and accuracy. The Split Sample Program includes samples obtained using a sample splitter and samples obtained during side-by-side sampling.

This program is designed for those permits that provide authorization to discharge effluent. The evaluation of analytical results reported in Split Sample Audit summary letters and tables are used to demonstrate a high level of quality control in a permittee's effluent monitoring. Specifically an audit pass demonstrates that the sampling techniques and sample handling methods deployed by a permittee, along with the sample handling, analytical techniques and equipment of their chosen lab/s meet the quality standards that ENV are responsible to uphold on behalf of the people of British Columbia.

4. SCOPE

All of the parameters listed within a permit may be included in whole or in part, in a Split Sample Audit. The analytical data included in a Split Sample Audit will be subjected to a quality assessment process regardless of the analytical service provider. The complete process of sample collection and handling by the permittee and all of the laboratories contracted by a permittee to test the quality of their effluent, including where applicable, the permittee's in-house laboratory, are within the scope of this program.

5. RESPONSIBILITIES

The Ministry's **Compliance** team is responsible for scheduling and conducting Split Sample Audits and ensuring that permittees scheduled for an audit are notified in advance of the audit. Permittees will receive program information and guidance from compliance staff where required. Provincial compliance staff will establish a list of analytes to be included each audit. The list of analytes will be based on the monitoring requirements of the permit. Regional compliance members who conduct split sample audits are ultimately responsible for interpreting and monitoring the performance of permittee results and assessing those results against the requirements of their permit. The completion of follow-up actions recommended in split sample summary reports will be determined by and monitored by the regional member who conducted the audit. Laboratory Standards will assist regional staff as required.

The **Laboratory Standards & Quality Assurance (LSQA)** unit is responsible for maintaining the Split Sample Program which requires an ongoing performance evaluation of the Province's reference laboratories, the audit assessment protocol and maintenance of individual permittee files and spreadsheets. LSQA is responsible for ensuring that the Province's reference laboratory maintains the highest achievable level of quality in the analytical services it provides to the Province. This is achieved through the administration of a quality assurance program called the Laboratory Blind Audit Program. The Provinces reference laboratories are subjected to monthly *Blind Audits*. LSQA is responsible for tabulating, evaluating, assessing, monitoring and reporting on the analytical data submitted by each permittee through the Split Sample Audit Program.

As stated in all effluent permits, permittees must sample in accordance with the British Columbia Field Sampling Manual which can be found online at the Laboratory Standards and Quality Assurance webpage available at https://www2.gov.bc.ca/gov/content/environment/research-monitoring-reporting/monitoring/laboratory-standards-quality-assurance/bc-field-sampling-manual.

Sampling must be carried out by competently trained staff. Samples collected for split sample audits will preferably be collected as split samples however side by side samples are permitted in situations where split sampling is not possible. It is the responsibility of the permittee to understand and include commensurate quality control measures in their sampling procedures. For more information on Quality Assurance and Quality Control (QA/QC) see Part A and Part E of the B.C. Field Sampling Manual.

In accordance with the Environmental Data Quality Assurance Regulation (EDQAR), **permittees** required to collect split samples must submit those samples to a qualified laboratory for analysis and provide to the LSQA unit of ENV, the results of the analysis no later than 45 days after the sample collection date. Permittee's must submit analytical results for each analyte included in their split sample audit. Permittee's are responsible for ensuring that a complete set of Laboratory reports is submitted to splitsampleaudits@gov.bc.ca within 45 days of sample collection. Where laboratory reports include analytical results for multiple samples it is the responsibility of the permittee to identify the sample/s to be assessed in their audit.

6. EFFLUENT SAMPLING

The quality of the analytical data produced in a laboratory is determined in part by the quality control procedures deployed during sampling. For this reason it is imperative that careful planning and preparations take place in advance of the sampling event. Planning should begin by identifying the objective/s of the sampling event which in turn will identify the parameters to be analysed and the sampling requirements necessary to achieve those objectives. Careful planning and competent quality control protocols must be deployed for all monitoring programs.

Sampling shall be carried out in accordance with the "British Columbia Field Sampling Manual". Sampling for Split Sample audits should follow the same procedures and or protocols deployed to satisfy the monitoring and reporting requirements of your permit. Similarly the samples collected for Split Sample audits should be analyzed by the same laboratories used for permit monitoring and reporting purposes.

Detailed information regarding sample planning and preparation and quality assurance and quality control is provided in Part A and Part E of the British Columbia Field Sampling Manual.

6.1 Side by Side Sampling

In some circumstances it will be necessary to conduct side-by-side sampling during a Split Sample Audit. Although this method of sampling shifts more of the causal outcomes of the audit findings onto sampling and handling it does not affect the validity of the assessed data or the audit results. It is important however that the method of sampling be recorded and provided to the Laboratory Standards and Quality Assurance unit along with the analytical reports.

6.2 Split Sampling

Prior to sampling the sample splitter must be cleaned. Cleaning will typically be conducted by Ministry staff however for information purposes the following steps outline an acceptable cleaning protocol:

a. Soak the vessel in deionized water.
b. If the vessel was previously used to split samples of water containing hydrocarbons, oils, or grease, or the vessel's interior walls exhibit an oily film

soak the vessel in a 0.1 to 2 percent non-phosphate, laboratory grade detergent solution;

c. Scrub all of the vessel's sample supporting parts using a non-metallic brush;

d. Rinse well with de-ionized water flushing some of the rinse water through the spigot; and;

e. Rinse twice more with deionized water flushing some of the rinse water through the spigot.

6.2.1 Split Sampling Procedure

1. Estimate the volume of effluent required for all of the 'split samples' included in the audit. The volume estimated must provide enough material to satisfy both the permittee's submission and the Ministry's submission and should include an additional volume equal to 25% of the combined submission volume to account for losses and incidentals.
2. Wearing nitrile gloves, fill the clean split sample vessel with effluent using a single container; preferably a 1L amber jar. Ensure the container used to fill the sample splitter is clean prior to filling.
3. Mix the contents of the sample splitter by raising and lowering the agitator rod through the height of the effluent contained in the splitter. Keep the churning plate submerged while mixing. Constant or near-continuous mixing will provide the most homogenous sample material. The rate at which the rod is raised and lowered does not have to be quick but should be uniform throughout the sampling process. The USGS suggests a rate of approximately 22 cm (9 in) per second. Mixing should be carried out for a minimum of 10 strokes before subsampling.
4. Pour off a small volume of effluent to purge the spigot and discard. Fill each sample bottle from the spigot while continuing to mix the contents of the sample splitter.
5. If multiple sample containers are required for the audit they should be filled in an alternating pattern. For example one bottle is filled by the permittee followed by one bottle filled by the ENV representative.
6. Sample preservation and filtering where required should be carried out in accordance with Part E of the British Columbia Field Sampling Manual.

7. PERFORMANCE CRITERIA

Samples collected by permittees can be tested by in-house laboratories, commercial laboratories, or a combination thereof provided they are 'qualified' to conduct the tests they are assigned. Please note that as of January of 2019 laboratories are unable to qualify to conduct Resin and Fatty Acid (RFA) tests due to a lack of certified reference materials. Until this issue changes permittee's are not required to seek a qualified laboratory for RFA analysis. Ministry submitted samples are tested by one of the Province's *reference laboratories* in accordance with the Environmental Data Quality Assurance regulation (EDQAR). Reference laboratories are subjected to monthly blind audits. Analytical test results produced by the Ministry's laboratory provide the *reference values* against which a permittee's test results are evaluated.

Performance criteria are used to evaluate each test result and an overall Performance Evaluation of the audit. Split sample audits conducted in 2018 will be recorded and presented in a new spreadsheet. Subsequent audits will be added to a permittees spreadsheet in a manner that easily exhibits potential trends.

With the exception of microbiological tests and toxicity tests, analytical results are evaluated using statistical equations which assign a performance score and a corresponding 'Pass' or 'Fail'. The performance score equation compares the absolute deviation between the test result and the reference value to an Acceptable Deviation (AD). The AD is calculated as a 95% confidence interval which is calculated for each test result.

Toxicity tests are evaluated on absolute deviation and microbiological test results are assessed using an industry standard grading table for drinking water. As with all other test results the evaluation of microbiology and toxicity test results produce a performance score for each individual analyte. The scored outcomes indicate the degree of variation between the permittee's test result and the reference value provided by the Ministry's test result.

The average of all test scores provides a *Performance Evaluation* score for the overall audit.

7.1 Test Result Outcomes

The *Table* provided with your *Audit Results letter* provides five evaluation outcomes as follows:

i. Absolute Deviation
ii. Acceptable Deviation
iii. Deviation Factor
iv. Points Assigned, and,
v. Pass/Fail/NC

i. The **absolute deviation** is the absolute difference between the concentration of the analyte reported by the permittee's laboratory and the reference value.

ii. With the exception of microbiological and toxicity test results, an '**acceptable deviation**' is calculated for each set of analytical test results included in the audit. The acceptable deviation is based on a 'Z' score calculation which takes into consideration the analyte being tested, the lowest or reportable detection limit (LDL/RDL) for that analyte reported by the reference laboratory and the concentration of the analyte reported by the reference laboratory.

iii. The **deviation factor** is a comparison of the absolute deviation to the acceptable deviation. The deviation factor provides a measure of the precision of a test result relative to the reference value.

iv. Calculable test results are assigned one of four **point-scores** (performance score). Performance scores are taken from a scoring table that is based on an Acceptable Deviation (AD) which is calculated for each test result.

Available performance scores are as follows:

< 0.5 AD **= 5** – [Pass] high precision, acceptable,

0.5 AD – 1.0 AD **= 4** – [Pass] good precision, acceptable,

> 1.0 AD – 1.5 AD **= 2** – [Fail] poor precision, unacceptable, and;

> 1.5 AD **= 0** – [Fail] major error/fail requires further scrutiny.

v. The average of all point-scores produced in an audit provide an overall **Performance Evaluation** of the audit.

7.2 Technical Scoring

Technical scoring will be applied to specific test results or test results that are incompatible with the programs automated functions. The majority of analytical test results are evaluated by a series of automated statistics-based equations however analytical results for toxicity and microbiology tests are evaluated in accordance with dedicated criteria which is explained in Section 7.2.1 and 7.2.2 of this guidance document.

Evaluations of test results that produce a 'non-calculable' (NC) designation will in some cases warrant further scrutiny. NC designations occur when one or both of the test results are 'not reported' (NR) or are reported at 'below detection limits' (<LDL). Under the following circumstances analytical test results will be omitted from an audit or assigned a technical performance score:

- Test result evaluations designated as NC in column P of the 'audit table' where an Audit Number is not included in column A are not assigned a technical score and are **not** included in the audit.
- Test result evaluations designated as *NR or SL (Not Required or Samples Lost) are **not** included in the audit.
- Test results for a parameter within a *test suite* not reported (NR) by the permittee or the Ministry will be omitted from the audit. For example if the Ministry requires dissolved metals to be included in the audit and the reference laboratory's 'metals suite' includes zinc but the laboratory contracted by the permittee does not include zinc in their metals suite, the test results for that parameter (zinc) will not be included in the audit unless the Ministry explicitly requested the analysis of that parameter.
- Where both the Permittee and the Ministry report a required analyte below the Reportable Detection Limit (RDL) and there is a significant difference in RDL values resulting from a laboratory requirement for dilution and not resulting from an inadequate volume of sample material the test will not be evaluated but will be included in the Audit.
- Additional technical scores will be assigned under the conditions presented in Table 1.

Table1. Schedule of Technical Scores

Condition	Performance Score
Permittee does not report the results of a required test	**0**
ENV does not report the results of a required test	**5**
Reference test results reported as **<** RDL / Permittee test results reported at a value of **>** RDL **Drop Sign**	**Function Result**
Permittee test result reported **<** RDL/ ENV test result reported at a value of **>** RDL **Drop Sign**	**Function Result**
Permittee and ENV test result reported as BDL where the Permittee RDL = ENV RDL	**5**

Note: For an explanation of "Drop Sign" refer to the definitions section of this document.

7.2.1 Toxicity Test Result Evaluations

Toxicity test results are reported as percent mortality (%mort). The absolute deviation (difference) of the test results is used to assign a performance score.

Scoring for toxicity test results is provided in the following table.

Table1. Toxicity Test Scoring

Absolute Deviation	Performance Score
0% to 10%	**5**
11% to 30%	**4**
31% to 50%	**2**
> 50%	**0**

7.2.2 Microbiological Test Result Evaluations

Microbiology test results are reported in Colony Forming Units (CFU). The growth of CFU's vary widely in standardized testing and as such the application of the regression method cannot be used to evaluate the deviation seen in these test results. A grading scale developed for the Performance Testing of laboratories conducting coliform testing for drinking water is used to evaluate microbiology test results.

The following table describes the scoring method for microbiology test results.

Table2. Microbiological Test Scoring

Reference cfu/100mL or MPN/100mL	0	1 - 24	25 - 29	30 - 39	40 - 59	60 - 69	70 - 79	80 +	**Score**
Permittee cfu/100mL or MPN/100mL	NG	1–29	10-42	16-59	21-62	46-119	49-139	52-159	**5**
		30-59	1-9 or 43-59	1-15	1-20	16-45 or >119	23-49 or >140	30-51	**4**
		>59	>59	>59	>62	1-15	1-22	1-29	**2**
	G	NG	NG	NG	NG	NG	NG	NG	**0**

Note: NG = No Growth, G = Growth

Example 1: ENV reports 31 cfu/100mL, Permittee reports18 cfu/100mL = 4.

Example 2: ENV reports 0 cfu/100mL, Permittee reports 2 cfu/100mL = 0.

Example 3: ENV reports 60 cfu/100mL, Permittee reports 112 cfu/100mL = 5.

7.3 Table Footnotes

The following footnotes are excerpted from the Split Sample Audit table:

- **NR:** Not Reported.
- ***NR/SL:** Not Required for submission or inclusion of the Audit/Samples Lost and are therefore not included in the audit.
- **NC:** Not Completed or Not Calculable. NC denotes that a test result was either 'Not Reported' or was 'Reported Below the Reportable Detection Limit (RDL)'.
- The precision of the permittee's analytical result is represented as the 'Deviation Factor' in column P of the table. A deviation factor that is encased in a dark **2.1** green cell indicates a significant deviation and as such warrants further scrutiny. In most cases these test results will be scrutinized by the Ministry which may result in follow up actions however it is strongly recommended that any such test result be reviewed by the permittee.

8. AUDIT PASS/FAIL CRITERIA

The performance score for each test evaluated indicates the precision of the permittee's analytical result with that of the reference value. When the precision of an evaluated test is unacceptable it is considered a failed test. When all of the tests have been evaluated the number of failed tests is added and averaged and the sum of the individual performance scores are averaged to provide an audit's overall Performance Evaluation (PE) score. An audit pass is based on the percentage of failed tests and the overall performance score. An audit that produces a PE score of 70% or greater and 'failed tests' of 25% or less constitutes an audit pass.

All of the test result evaluations are processed by functions in the audit table and presented in the format shown in figure 1 below.

Figure 1. Evaluation Summary

Client Name:	ABC Pulp Mill
Audit No:	18.1
Requisition No.:	S9999999999
Sample Date:	6-Mar-18
Total Tests Included:	40
Total Tests Evalutated:	22
Total Points Assigned:	81
Failed Test Results:	6
Percent of Failed Tests:	15%
Performance Evaluation:	74%
Audit Result:	PASS