1. PURPOSE

1.1 To detail the policy and procedures for sampling, sample handling and sample receipt.

2. SCOPE

2.1 This procedure applies to the laboratory personnel for sampling, sample handling and sample receipt.

3. NORMATIVE REFERENCES

3.1 ISO 8402, 1994. Quality Management and Quality Assurance - Vocabulary.
3.2 SANS ISO/IEC 17025:2017. General requirements for the competence of testing and calibration laboratories.
3.3 SANS 9000: 2005 Quality Management Systems - Fundamentals and vocabulary.
3.4 SANAS Documentation Manual.
3.5 UNITED STATES ENVIRONMNETAL PROTECTION AGENCY (US EPA), 2002. Method for measuring the acute toxicity of effluent and receiving waters to freshwater and marine organisms. EPA-821-R-02-012, 5th Edition. U.S. Environmental Protection Agency, Office of Water (4303T), 1200 Pennsylvania Avenue, NW Washington, DC 20460.

Note: This Standard Operating Procedure is an adaptation of the US EPA, 2002 protocol.

4. TERMS AND DEFINITIONS

4.1 See "TERMS AND DEFINITIONS" in Quality Manual.
4.2 ***Sampling.*** Sampling is a defined procedure whereby a part of a substance, material or product is taken to provide for testing or calibration of a representative sample of the whole. Sampling may also be required by the appropriate specification for which the substance, material or product is to be tested or calibrated. In certain cases, the sample may not be representative, but is determined by availability.

5. RESPONSIBLE PEOPLE

5.1 Responsibilities for the following people are described in this procedure:

a) Management
b) Quality Manager
c) Technical/Laboratory Manager
d) Laboratory personnel
e) Samplers
f) Client Coordinator (In this case the Managing/Director)

6. PROCEDURES

6.1 Effluent Sampling for licence conditions

6.1.1 The effluent sampling point must be the same as that specified in the licence.

6.1.2 Conditions for exception would be:

a) Better access to a sampling point between the final treatment and the discharge outfall
b) If the effluent is chlorinated prior to discharge to the receiving waters, it may also be desirable to take samples prior to contact with the chlorine to determine toxicity of the un-chlorinated effluent, if required, if not the total effect will be assessed for the effluent as is, depending on the purpose of the testing
c) In the event that there is a desire to evaluate the toxicity of the influent to publicly owned treatment works or separate process waters in industrial facilities prior to them being combined with other process waters or non-contact cooling water, additional sampling points may be chosen

Any such exception must be noted on the analysis request form (QM7.1/R-26) in the space provided.

6.1.3 The decision on whether to collect grab or composite samples is based on:

- The licence,
- The objectives of the test, and
- An understanding of the short and long-term operations and schedules of the discharger.

6.1.4 If the effluent quality varies considerably with time, which can occur where holding times within the treatment facility are short, grab samples may seem preferable because of the ease of collection and the potential of observing peaks (spikes) in toxicity. However, the sampling duration of a grab sample is so short that full characterisation of an effluent over a 24 hour period would require a prohibitive number of separate samples and tests.

6.1.5 Collection of a 24 hour composite sample, however, may dilute toxicity spikes, and average the quality of the effluent over the sampling period.

6.1.6 Sampling recommendations would include:

- Aeration during collection and transfer of effluents should be minimised to reduce the loss of volatile chemicals.
- Ensure that the bottle is filled to the top
- Date and name of the sample should be recorded on the sample bottle. The procedures used for effluent sample and dilution water collection should be recorded on the sample request sheet (see Request for Analysis, QM7.2/R-26), if applicable.

6.2 The Advantages and Disadvantages of Effluent Grab and Composite Samples

6.2.1 Grab samples

6.2.1.1 Advantages

- Easy to collect; requires a minimum of equipment and on-site time
- Provide a measure of instantaneous toxicity
- Toxicity spikes are not masked by dilution

6.2.1.2 Disadvantages

- Samples are collected over a very short period of time and on a relatively infrequent basis
- The chances of detecting a spike in toxicity would depend on the frequency of sampling, and the probability of missing spikes is high.

6.2.2 Composite samples

6.2.2.1 Advantages

- A single effluent sample is collected over a 24 hour period
- The sample is collected over a much longer period of time than grab samples and contains all toxicity spikes.

6.2.2.2 Disadvantages

- Sampling equipment is more sophisticated and expensive, and must be placed on-site for at least 24 hours
- Toxicity spikes may not be detected because they are masked by dilution with less toxic wastes.

6.3 Effluent Sampling Recommendations

6.3.1 Sufficient sample must be collected to perform the required toxicity and chemical tests.

6.3.2 A 1 ℓ container will provide sufficient sample volume for screening toxicity tests and 2 ℓ for definitive testing (basic battery of tests including a bacteria, algae, invertebrate and vertebrate), unless otherwise specified and/or requested.

6.3.3 Care should be taken that sediments are not contained in the bottles during water sampling.

6.3.4 For Ostracod and Phytotox analyses, as well as for leachate testing, at least 500g of dry sediment/soil/product should be submitted. Care should be taken to avoid large debris.

6.4 Sampling Methods

6.4.1 Continuous discharges (US EPA, 2002)

- If the facility discharge is continuous, but the calculated retention time of the continuously discharged effluent is less than 14 days and the variability of the effluent toxicity is unknown, at a minimum, 4 grab samples or 4 composite samples are collected over a 24 hour period. For example, a grab sample is taken every 6 hours (total of four samples) and each sample is used for a separate toxicity test, or four successive 6 hour composite samples are taken and each is used in a separate test.
- If the calculated retention time of a continuously discharged effluent is greater than 14 days, or if it can be demonstrated that the wastewater does not vary more than 10% in toxicity over a 24 hour period, regardless of retention time, a single grab sample is collected for a single toxicity test.
- The retention time of the effluent in the wastewater treatment facility may be estimated from calculations based on the volume of the retention basin and rate of wastewater inflow. However, the calculated retention time may be much greater than the actual time because of short-circuiting in the holding basin. Where short-circuiting is suspected, or sedimentation may have reduced holding basin capacity, a more accurate estimate of the retention time can be obtained by carrying out a dye study.

6.4.2 Intermittent discharges (US EPA, 2002)

- If the facility discharge is intermittent, a grab sample is collected midway during each discharge period.
- Examples of intermittent discharges are:
    - When the effluent is continuously discharged during a single 8 hour work shift (one sample is collected), or two successive 8 hour work shifts (2 samples are collected).
    - When the facility retains the wastewater during an 8 hour work shift, and then treats and releases the wastewater as a batch discharge (1 sample is collected).
    - When the facility discharges wastewater to an estuary only during an outgoing tide, usually during the 4 hour following flow high tide (one sample is collected).
- At the end of a shift, clean up activities may result in the discharge of a slug of toxic waste, which may require sampling and testing.

6.5 Receiving Water Sampling

6.5.1 Logistical problems and difficulty in securing sampling equipment generally preclude the collection of composite receiving water samples for toxicity tests. Therefore, it is common practice to collect a single grab sample and use it throughout the test.

6.5.2 The sampling point is determined by the objectives of the test.

6.5.3 In rivers, grab samples should be collected at mid-stream and mid-depth, if accessible.

6.5.4 At estuarine and marine sites, samples should be collected at mid-depth.

6.5.5 To determine the extent of the zone of toxicity in the receiving water downstream from the outfall, receiving water samples are collected at several distances downstream from the discharge, where possible and/or applicable.

6.5.6 The time required for the effluent receiving-water mixture to travel to sampling points downstream from the outfall, and the rate and degree of mixing, may be difficult to ascertain. Therefore, it may not be possible to correlate downstream toxicity with effluent toxicity at the discharge point unless a dye study is performed.

6.6 General Sampling Recommendation

6.6.1 Sample containers

6.6.1.1 Samples are collected in clean plastic or glass (where practical) bottles.

6.6.1.2 Sample bottles do not necessarily have to be sterile (see method for requirements), unless otherwise specified (e.g. for microbiological analyses, as outsourced).

6.6.1.3 To minimise the loss of toxicity due to volatilisation of toxic constituents, all sample containers should be "completely" filled, leaving no air space between the contents and the lid as far as is possible.

6.6.1.4 Sample bottles are labelled at a minimum with date and sample name, located on the side of the bottle as well as on the cap.

6.6.2 Sample handling

6.6.2.1 Unless the samples are analysed at an on-site toxicity testing laboratory, the day of collection (or hand delivered to the testing laboratory for use on the day of collection), it is recommended that they be held at 1 - 7°C until used to inhibit microbial degradation, chemical transformations, and loss of highly volatile toxic substances.

6.6.2.2 Composite samples should be chilled as they are collected.

6.6.2.3 Grab samples should be chilled immediately following collection.

6.6.2.4 If the effluent has been chlorinated, total residual chlorine must be measured immediately following sample collection if possible and if required. The presence/potential presence of chlorine must be noted with a “(c)” next to the sample name on the sample request sheet (QM7.1/R-26).

6.6.3 Sample holding

6.6.3.1 Sample holding time begins when the last grab sample in a series is taken (i.e., when a series of 4 grab samples are taken over a 24 hour period), or when a 24 hour composite sampling period is completed.

6.6.3.2 If the data from the samples are to be acceptable for use in the licence, the lapsed time (holding time) from sample collection to first use of each grab or composite sample must not exceed 72 hours as far as is practicably possible. Should this specified retention time not be met, it should be noted in the report and is applied in order to reduce the time that the samples are out of the control of the laboratory.

6.6.4 Shipment

6.6.4.1 Samples collected for off-site toxicity testing are to be chilled to 1-7°C during or immediately after collection.

6.6.4.2 Sample bottles should be transported in a cooler box with ice bricks or ice packs (not frozen).

6.6.4.3 Sufficient ice should be placed with the sample in the shipping container to ensure that ice will still be present when the sample arrives at the laboratory and is unpacked. Insulating material should not be placed between the ice and the sample in the shipping container unless required to prevent breakage of glass sample containers.

6.6.4.4 Place the ice bricks vertically next to the sample, or pack the ice pack around the sample.

6.6.4.5 Ensure that ice bricks/ice packs are solidly frozen.

6.6.4.6 There should be sufficient ice bricks to keep samples cold during transportation.

6.6.4.7 To prevent contamination do not mix various types of samples in one cooler box, such as effluents and control waters, as far as is practicably possible.

6.6.4.8 All sample containers should be rinsed with source water before being filled with sample. After use with receiving water or effluents, sample bottles are punctured to prevent reuse.

6.6.4.9 Samples should be in an upright position and the lid securely placed to prevent leaking.

6.6.4.10 Several sample shipping options are available, including road mail, air express, bus, and courier service.

6.7 Sample Receipt

6.7.1 Samples are received in a dedicated area by authorised staff.

6.7.2 Upon arrival at the laboratory, samples are logged in. It is noted on the Request for Analysis sheet if the samples have been transported in a cooler box. If the samples are not immediately prepared for testing, they are stored at 1-7°C until used. It is of utmost importance that the relevant documentation (sample request form, order number etc) is in order at the time of sample delivery in order to prevent further time laps before testing commences (sampling may not commence until all paperwork is in order, unless otherwise approved by the Managing Director).

6.7.3 Every effort must be made to initiate the test with an effluent sample on the day of arrival in the laboratory, and the sample holding time should not exceed 72 hours before first use as far as is practicably possible (refer to section 6.6.3.2).

6.7.4 The required toxicity test to be performed is specified on the Request for Analysis form, QM7.1/R-26).

6.7.5 Date and time of receipt of sample in the toxicity laboratory is noted.

6.8 Access

6.8.1 Access to samples and toxicity laboratory is restricted to authorized laboratory staff. Samples are stored in the laboratory which is access controlled.

6.9 Safety

6.9.1 All samples should be treated as potentially toxic and personal protective equipment should be worn at all times when handling samples.

6.10 Retention

6.10.1 Samples are retained in the relevant refrigerator at 1-7 °C until the tests have been completed and all data have been classified.

6.10.2 After the report has been submitted to the Client, the samples are retained for a maximum period of 2 weeks during which time queries can be raised. After this, the samples will be discarded. Retention of samples is purely for verification/confirmation purposes (e.g. extremely low/high pH/EC values) as a result of potential instability and re-testing will require resampling

7. RELEVANT QUALITY RECORDS

7.1 Request for Analysis, QM7.1/R-26
7.2 Test Methods
7.3 Test Reports.

8. BIBLIOGRAPHY

8.1 None

9. APPENDICES

9.1 None

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