Industrial Waste

Information Brochure

For further information, please visit our website at http://www.watercorporation.com.au/indwaste
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SAMPLING OF INDUSTRIAL WASTES – IWPUB16

A typical Industrial Waste sampling and monitoring facility

All sampling of industrial waste for subsequent analysis to determine the nature and proportions of its component constituents is to be carried out in accordance with the guidelines in this Brochure. These guidelines apply both to samples collected for surveillance purposes by Water Corporation staff, and to self-monitoring carried by dischargers (or their contractors).

Sampling Equipment and Facilities

Sample Containers

For the sampling and analysis of:

- Biochemical Oxygen Demand (BOD5),
- Chemical Oxygen Demand (COD),
- Suspended Solids (SS),
- pH, and
- Many other parameters,

a single sample in a 1 L polyethylene bottle is preferred.

For a determination of:

- Oil and Grease

a separate sample in a 1 L polyethylene bottle should be supplied to the analyst.

For the sampling and analysis of:

- Heavy metals,

a separate 125mL sample in a polyethylene bottle is preferred.

Sampling for:

- Cyanide and Mercury

requires special sample bottles and special procedures (see section below). Sample bottles for these variables should be obtained from the analysing laboratory.

Note: All samples should be filled to within about 5 mm of the top of the bottle, leaving a small air space.

Manual Sampling Devices

A typical manual sampling device consists simply of a stainless steel or plastic container of 500 mL to 1 L capacity, attached to a length of pipe or rod such as a broom handle.

Automatic Samplers

Automatic sampling machines can provide a significant saving in labour. An automatic sampler should possess the following features:

- It should be capable of collecting samples on a time-proportional basis, or on a flow-proportional basis when connected to an external flow meter.
- The sample chamber should be able to accommodate a single large sample bottle of at least 5 L capacity (for composite sampling) or at least 24 smaller bottles of at least 5 L total capacity (for discrete grab sampling). The sampler should have the facility to collect at least 4 samples per bottle when using the 24 small bottles.
- The sample chamber should have cold storage facilities. Portable samplers should be able to hold sufficient crushed ice for a 24 hour sampling period. Fixed samplers should have refrigerated sampling compartments. Dedicated fixed, refrigerated samplers are highly desirable for major industries. The cold storage facility for the sample chamber should be utilised throughout the sampling period.

Sampling Sites

Full details of monitoring point requirements are explained in a separate brochure ("Industrial Waste Monitoring Points" - IWPUB11). With respect to sampling, the key points are shown below:

- Mains power is to be available within 2 metres of the sampling point to eliminate the need to run autosamplers on battery power.
- It is highly desirable that a flow meter pulse output is to be available for direct connection to the autosampler, enabling direct flow proportional sampling. Ideally the flow meter output connection should be located with the mains power outlet box. If direct connection to the autosampler is not available, flow meter readings will need to be taken at no greater than hourly intervals during the period of autosampler operation.
- Sample points should be precisely defined and chosen to ensure that representative samples are taken. Typical sample points include sedimentation tanks, settling or pump pits and disconnecter gullies.

Sampling Procedures

General

Extreme care should be taken to avoid contamination:

- Remove caps from sample bottles ONLY at the time of sampling.
- NEVER put sample bottle caps on the ground.
- DO NOT touch the inside of the bottles or caps with the hand or sampling equipment.
- DO NOT RINSE sample bottles.
- Bottles should be capped IMMEDIATELY on collection of sample.
- Label bottles BEFORE taking samples. This avoids possible mix-ups and also avoids the difficulty of trying to write on wet bottles.

Manual Sampling - Grab Samples

When sampling from open water systems such as channels or tanks, collect sample from the middle of the stream or body of water and at mid-depth. Avoid skimming from the surface, scraping the sides or bottom, or sampling in stagnant corners.

When sampling from distribution lines, the lines should be flushed before taking the sample to ensure that the sample collected is representative of the flowing stream and not simply material which has collected in the sampling point. Regulate the flow so that no splashing occurs when filling the bottle.

Composite Sampling Using Automatic Samplers

Automatic samplers should be protected from heat, direct sunlight and sources of contamination. The sample probe should be maintained in such a position in the flow stream that representative samples are obtained (mid-depth is usually ideal). Wherever possible, avoid having the probe lying on the surface or edges of the stream.

When collecting samples for charging purposes, the flow meter reading must be recorded at the commencement of sampling.

The preferred form of samples, especially when collecting samples for charging, are flow proportional composite type, typically taken over a 24 hour period. There are two ways of collecting flow-proportional composite samples:

- The first and preferred method requires a flow meter to be directly linked to the autosampler. The sampler is programmed to collect samples at regular intervals of flow. The single large sample bottle is used in the sample chamber.

  The sampling interval should be chosen so that a minimum of 80 samples would be taken over a 24 hour period. The volume of each sample should not be less than 25 mL, and preferably 50-100 mL. A final volume of 5 L of composite sample is desirable, and in some cases essential, depending on the analysis required.
- The second method is used where an electronic link to an external flow meter is not available. It involves collection of a series of grab samples on a time-proportional basis in individual bottles. With a sampler holding 24 bottles, each bottle would represent a 1 hour sampling period. The sampler should be typically programmed so that samples are collected at least every 15 minutes (ie. 4 sub samples will be collected in each bottle). The volume of each subsample should not be less than 25 mL and preferably be about 100 mL. A final volume of 5 L of composite sample is desirable, and in some cases essential, depending on the analysis required.

  Flow readings are taken hourly using a flow meter which is not linked to the sampler. At the end of the sampling period a composite sample is prepared from the 24 one-hourly samples by bulking them in proportion to the amount of flow during the hour each sample is taken to represent.

Sample Bulking and Splitting

Sample Bulking

It is of paramount importance that subsamples are thoroughly mixed before taking portions for bulking. Most industrial wastes contain suspended solids, which are usually readily settleable. If samples are allowed to stand even briefly after mixing, these solids will settle out. If the subsample is then decanted into another container, some of the solids will be left behind and the subsample ceases to be representative of the sample from which it is being taken.

When bulking subsamples, always:

- Thoroughly mix subsamples by capping, shaking and inverting bottles several times before taking portions for bulking.
- Pour out the required portion immediately after mixing the subsample
- Preferably keep the subsample mixed by agitating the bottle while pouring from it.
- Pour subsamples rapidly into bulk container. Use a funnel to make this easier and to avoid loss of sample.

Transferring Samples from Bulking Containers to 1 L Sample Bottles

The comments above regarding the need to keep samples mixed when making up the bulk sample apply in every respect when transferring samples from a bulking container to the 1 L sample bottles which will be sent for laboratory analysis.

Splitting Samples

Sometimes there is a need to split samples into two, for example so that analysis can be performed by two different laboratories. Since the aim of this is usually to compare the performance of the laboratories, it is essential that both receive identical splits of the sample.

To ensure that this occurs the following steps should always be followed when splitting samples:

- Always split the bulked sample - never prepare splits by preparing two separate bulked composites from the subsamples.
- Thoroughly mix the bulk sample by capping and shaking the bottle before pouring out splits.
- Pour out splits immediately after mixing the bulk sample.
- Preferably keep the bulk sample mixed by agitating the bottle while pouring from it.
- Ideally use a dual-necked funnel to fill both split containers simultaneously.

Special Collection Procedures for Cyanide and Mercury

Sampling for cyanide requires the following special procedures:

- Samples should only be collected in bottles provided by the analysing laboratory.
- Samples must be preserved at the time of collection by addition of sodium hydroxide to achieve a pH of 12 or more.
- If samples contain oxidising agents or sulphide, other on-site pretreatment procedures will be necessary. Refer to the analysing laboratory for advice.

Note that because of the special preservation procedures needed for cyanide samples, composite samples collected as described in the sections above cannot be used for cyanide determinations.

Sampling for mercury requires the following special procedures:

- Samples should only be collected in bottles provided by the analysing laboratory.
- Samples must be preserved at the time of collection by addition of potassium dichromate to achieve a final concentration of 0.05%, and addition of concentrated nitric acid to achieve a pH of 2 or less.

As with cyanide sampling, composite samples collected as described in the sections above cannot be used for mercury determinations.

Wherever possible, the analysing laboratory should be advised at least several days in advance of cyanide and mercury samples being collected.

Sample Preservation, Storage and Delivery

All sample bottles should be labelled in a manner which clearly identifies the sample and the sampling point from which it was taken, and distinguishes it from all others in the batch. All bottles which may contain cyanide (not just bottles preserved for cyanide determination) should have 'SAMPLE MAY CONTAIN CYANIDE' warning labels or marks affixed to them as soon as they are prepared.

Samples should be maintained chilled from the time of collection until delivered to the laboratory conducting the analysis. The only exception to this requirement is samples for metals analysis only, which may be transported unchilled. Composite samples should be kept chilled throughout the compositing period.

Samples should be delivered to the laboratory as soon as possible after collection, and certainly within 24 hours. Longer delivery times may necessitate more complex preservation procedures. Parameters such as biochemical oxygen demand (BOD5) cannot be

reliably determined if storage time is significantly greater than 24 hours.

Sampling personnel should liaise with the analysing laboratory to determine sample receival requirements. In particular, the days and times that the laboratory will accept BOD samples should be ascertained, as restricted times may apply, because of the special nature of the test.

Laboratory Tests Required

These should be determined in consultation with the Industrial Waste Section. For samples for charging purposes, the minimum requirement will be pH, conductivity, suspended solids (SS), biochemical oxygen demand (BOD) and chemical oxygen demand (COD). Other tests may be required depending on the industry.

Laboratories

Generally, only laboratories holding NATA accreditation for the appropriate wastewater parameters should be used for analysing industrial waste samples. This is especially the case for samples on which industrial waste charges are being determined.

The Industrial Waste Section will from time to time submit quality assurance samples to laboratories to evaluate their performance. If results on quality assurance samples from a laboratory are not considered acceptable, routine industrial waste results will not be accepted from that laboratory until it can demonstrate acceptable performance.

Only in special circumstances will the requirement for NATA registration be waived. In such cases, additional requirements to demonstrate competence will be required. The laboratory will be responsible for bearing the cost of these requirements.

Data Management and Reporting

Reports containing the results of self-monitoring analyses should generally include the following information:

- Name of testing laboratory
- Sample type (composite or grab)
- Sample location
- Date of sampling (or date sampling commenced if a 24 hour composite)
- Any deviations from the sample collection, preservation or storage procedures outlined in these guidelines.

In cases where industries are carrying out self-monitoring for the determination of industrial waste charges, the results may be required to be submitted in summary form, along with flow or other data, in a format prescribed by the Industrial Waste Section.

In such cases the original laboratory reports (and flowmeter data where relevant) must be held by the industry for at least 12 months, and be available for inspection by the Industrial Waste Section as requested.

More Information?

You can find more information about the Industrial Waste service on our website at http:\\www.watercorporation.com.au/indwaste

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