Sampling of industrial waste

Industrial waste - information sheet 16

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 out by dischargers (or their contractors).

Sampling equipment and facilities

Sample containers
The following bottle information is general only and may vary depending on the particular laboratory’s preference. The laboratory should be consulted for analyses of variables not covered in this list.

A single 1 L polyethylene bottle may be used for analysis of:
- Biochemical Oxygen Demand (BOD5),
- Chemical Oxygen Demand (COD),
- Suspended Solids (SS),
- pH, and various other parameters including nutrients

An extra 1 L polyethylene bottle should be supplied for the determination of:
- Oil and Grease

A separate 125mL sample in a polyethylene bottle should be provided for analysis of:
- Heavy metals

Sampling for:
- Cyanide and Mercury requires special sample bottles and 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 unless otherwise directed by the laboratory.

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 string, pipe or rod (such as a broom handle).

Automatic samplers - Autosamplers
Autosamplers provide a significant saving in labour and should possess the following features:
- Be capable of collecting samples on a time or 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 24 small bottles with 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.

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**Sampling sites**
Full details of monitoring point requirements are explained in a separate brochure (Industrial Waste Monitoring Points - PUB11), for sampling, the key points are:

- Mains power should be available within 2 metres of the sampling point to eliminate the need to run autosamplers on battery power.
- A flow meter pulse output should 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 must be precisely defined and chosen to ensure that representative samples are taken. Typical sample points include sedimentation tanks, settling or pump pits and disconnector gullies.

**Sampling procedures**

**General**
Extreme care should be taken to avoid contamination:
- Label bottles BEFORE taking samples. This avoids possible mix-ups and also avoids the difficulty of trying to write on wet bottles.
- 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.

**Manual sampling - grab samples**
When sampling from open water systems such as channels or tanks, collect the 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 sampling to ensure 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 autosamplers**
Autosamplers 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 and the date of the reading recorded as the sample date.

The preferred form of sampling, especially when collecting samples for charging, is using flow proportional composites, typically taken over a 24 hour period.

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There are two ways of collecting flow-proportional composite samples.

**Flow meter linked directly to autosampler**
The referred 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.

**Electronic link to an external flow meter is not available**
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 immediately pouring out the required portion.
- Where possible keep the subsample mixed by agitating the bottle while pouring from it.
- Pour subsamples rapidly into the bulk container. Use a funnel to make this easier and to avoid loss of sample.

**Transferring samples from bulking containers to 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 sample bottles which will be sent for laboratory analysis.
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**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 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 should 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 pre-treatment procedures will be necessary. Refer to the analysing laboratory for advice.

Please note, 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 several days in advance of cyanide and mercury samples being collected.
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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 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 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, as restricted times may apply.

Laboratory tests required
These should be determined in consultation with Commercial & Industrial Services (C&IS). The minimum requirement of samples used for charging purposes will be pH, conductivity, suspended solids, biochemical oxygen demand and chemical oxygen demand. Other tests may be required depending on the waste producing industry.

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

Commercial and Industrial Services will from time to time submit quality assurance samples to laboratories to evaluate their performance. Results will only be accepted from laboratories who have demonstrated acceptable performance.

Only in special circumstances will the requirement for NATA accreditation be waived. In such cases, additional requirements to demonstrate competence may be required. The laboratory, or a discharger wishing to use them, will be responsible for bearing the cost of these requirements.
Data Management and Reporting
Reports containing the results of self-monitoring analyses will be as agreed in a self monitoring protocol and may include:

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

In cases where industries are carrying out self-monitoring for the determination of industrial waste charges, the results will be required to be submitted in a format and time interval prescribed by Commercial and Industrial Services (usually a spreadsheet provided by C&IS) and agreed in the self monitoring protocol.

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 Commercial & Industrial Services on request.

More information
For more information about Sampling of Industrial Waste please call 13 13 95 or visit your nearest Water Corporation office.

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