

## PRETREATMENT CORNER

# Things “Not To Do” When Sampling

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Valid test results are dependent on a sample being representative of the source from which it is taken. Inaccurate conclusions about wastewater treatment process efficiency or industrial wastewater discharges are more often related to inadequate sampling protocol than to analytical error. The quality of the data is also dependent on maintaining the integrity of the samples from sample collection through analysis, following proper chain-of-custody protocol and ensuring quality assurance and quality control procedures have been adhered to in all phases of sample collection and analysis.

Everyone is probably aware of the contents of Table II in 40 CFR §136.3, *Required Containers, Preservation Techniques and Holding Times*, which stipulates whether a sample should be placed in a glass or plastic sample bottle, what chemicals should be added prior to or after the sample is added to the sample bottle to prevent further degradation or to prevent interferences during testing, and how long a sample can be maintained before analysis is conducted. For instance, there is a logical reason why samples collected for semi-volatile analysis should be collected using glass containers. Many phthalate compounds, such as Bis (2-Ethylhexyl) Phthalate, are used as plasticizers and may be detected in the wastewater sample if plastic bottles are used, giving a false positive test result. This means that plastic tubing or plastic sampling devices should also not be used for samples collected for semi-volatile analysis.

Grab samples are required for the collection of wastewater samples for volatile organic analysis as the VOCs may volatilize if collected with a composite sampler. Samples for oil and grease analysis must not only be collected in glass containers since the oil and grease may adhere to the sidewalls of plastic containers, but must also be grab samples, as oil and grease loss may occur on the walls of the composite sampler tubing.

Speaking of tubing, the piping or tubing through which a sample is collected is crucial for valid test results. Ever wonder why the effluent copper concentrations were so high? Could it have been due to the new copper piping that replaced the old plastic tubing in the permanent automatic composite sampler? That rusty old bucket is also not a good idea to collect samples in for metals analysis. A brand new galvanized bucket would not work well either for metals sampling. It would be best to stick to glass or plastic. Certain glassware and plastic materials contain metals too, so make sure the sample equipment and containers are shown to be free of the metals of interest.

How many wastewater treatment plants have *No Smoking* signs posted in the administration/control building where the laboratory is located? Tobacco smoke contributes to higher ammonia concentrations in wastewater samples, especially when an ammonia electrode is used as opposed to the colorimetric method for ammonia nitrogen. Tobacco smoke may also interfere with Mercury analysis. This warning also includes those persons who collect samples. Therefore, smoking is not only hazardous to one's health but also to the wastewater test results as well.

EPA has developed “clean” sampling techniques. One example of these clean sampling techniques is found in EPA Method 1669, *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels*. Clean sampling techniques are critical to the defensibility of data, which is especially important in industrial pretreatment programs. In the case of sampling for metals, contamination may come from such simple sources as dust, hand lotions, make-up, clothing and breathing over samples, to name a few. Recall that mercury amalgam the dentist used to fill your tooth! Clean sampling techniques are required in several of the newer EPA test methods (1600 series) that have been developed during the past few years, such as EPA Method 1631 for Mercury.

The clean sampling techniques are also known in some circles as Clean Hands/Dirty Hands sampling techniques. In this scenario, two or more people conduct the sampling. One pair of hands is designated as Clean

Hands and has specific responsibilities, while the second pair of hands is designated as Dirty Hands and has separate responsibilities. For instance, Clean Hands takes care of all sampling operations that involve equipment that comes into contact with the wastewater sample. Dirty hands, on the other hand (no pun intended), takes care of all sampling operations that involve contact with potential sources of contamination.

Clean sampling carries over from the field sampling to the laboratory during sample preparation and analysis. There are several areas of a laboratory in which contamination of wastewater samples may occur, including, but not limited to, glassware, labware (careful of those talc gloves that contain high levels of zinc), reagents, lab pure water, indoor air quality, and instrumentation carryover from one sample to the next.

Remember, above all, the monitoring is conducted to ensure compliance either by the POTW or the industry and it is necessary to follow and document all necessary Quality Assurance and Quality Control procedures. Please share your sampling successes and failures with other pretreatment coordinators.