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COMMUNICATION

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FROM MACRO-SOURCE TO MICRO ANALYSIS: TAKING SAMPLES FROM WASTESTREAMS FOR CHEMICAL ANALYSIS

1. INTRODUCTION

New analytical equipment and techniques have lowered detection levels of many critical elements and compounds to points undreamed of twenty years ago. Where parts per million seemed almost beyond comprehension, during the past decade, analytical results in parts per billion or even parts per trillion have become routine. The sensitivity of mass spectrophotometers and gas chromatographs has opened a whole new world to analytical chemists. Whether this is a blessing or a Pandora's box filled with terrifying prospects remains to be seen.

With the increases in analytical sensitivity, the importance of the standard laboratory practices of quality control, cross checking with chemical standards, and instrument calibration has spiraled.

Unfortunately it is not possible to control or standardize the environment from which samples are being collected for analysis. The fact remains that the sources from which samples are to be collected are rarely homogeneous or static. Wastestreams are frequently very large and continually changing. Materials of concern may be floating, dissolved, settling, volatilizing or in various other changing states. It is therefore essential to select site conditions, sample extraction methods and equipment so that samples may be as representative as possible of the larger universe from which they are drawn.

If someone arrived at your laboratory with an unidentified bottle of liquid to be analyzed, you would certainly not just perform every test possible. You would want to know what analyses were required and what materials, with some idea of their concentration range, you might be expected to find. This would help you to determine what analytical methods should be used ant the sensitivity levels to select. Similarly, if the results of wastewater analyses are to have any validity it is necessary to know and control, as much as possible, the conditions under which samples are collected. Where, how and what time period is represented, as well as, by what means the collected samples are preserved, are factors you must consider. To select sampling location and methods of collection you should be aware of the general character of the wastewater flows and have some idea of the variability of pollutant concentrations.

2. SAMPLING LOCATIONS

1. Be sure the site provides the information desired. It must include all streams that represent the area under study and exclude streams from other sources.

2. Locate the sampling point in an area of maximum turbulence to provide complete mixing and avoid areas were material can settle out or build up floating layers.

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3. Provide maximum accessibility and safety for personnel and equipment. Avoid, if possible areas known to surcharge or flood, manholes in busy streets, or locations that invite vandalism.

3. TYPES OF SAMPLE

Samples may be collected either manually or by a variety of automatic sampling devices. The method used is usually determined by the frequency of sampling required and the availability of funds for trained personnel and equipment.

The type of sample to be collected depends on a number of factors such as rate of change in flow and character of the wastewater, and the accuracy required. The most common types of sample are: 1) discrete, 2) simple composite, 3) flow proportioned composite, and 4) sequential composite.

1. **The discrete sample** (may also be called individual, "grab" or "snap") is retained as a separate entity in its own container. It represents conditions at a specific time and place. Discrete samples are required for tests such as pH, VOC (volatile organic compounds) and DO (dissolved oxygen).

2. **A simple composite sample** (sometimes called a timed composite) is made up of a series of aliquots (smaller samples) of constant volume (*Vc*) collected at regular time intervals (*Tc*) and combined in a single container. Such a sample is denoted by *TcVc* meaning time intervals between aliquots and volume of each aliquot are constant.

3. **A flow proportioned composite** is collected in relation to the flow volume during the period of compositing to indicate the "average" conditon during the period. Sampling in proportion to flow may be done in several ways:

1. Pacing a sampler by a flow recorder signal so that time between constant volume samples is varied in relation to flow (*TvVc*).

2. By varying the volme of each aliquot in proportion to flow volume at the time of collection (*TcVvq*) or to flow since last sample (*TcVvQ*).

4. **A sequential composite** is made up of a series of (usually 2 to 8) individual samples per container, each container representing a specific time period, generally one hour. This type of sampling is particularly useful where the character of the wastewater may vary significantly from hour to hour, where batch dumping is expected, or where self-cancelling conditions occur, such as alternating high and low pH, which would not be apparent in a single composite. May be collected *TvVc*, *TvVc*, *TcVv* by *q* or *Q**.

4. MANUAL AND AUTOMATIC SAMPLING

Although samples can be collected manually, if the sampling period is to be longer than a few hours, it is probably more reliable and more cost effective to collect composite samples using an automatic sampler.

SELECTING SAMPLING EQUIPMENT

There are a wide variety of automatic samplers available commercially. The best equipment to take the type of samples required is not always the most expensive or elaborate device on the market. Each type of equipment must be judged according to its capabilities and limitations, as well as for its operational characteristics, as applied to the particular stream to be sampled.

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* *T* — time, *v* — variable, *q* — instantaneous flow rate, *V* — volume, *c* — constant, *Q* — flow since last sample.

The relative importance of any of the following criteria will depend on the application and the type of data required. Ideally, an automatic sampler should be:

Simple to operate and calibrate.
Resistant to clogging.
Repeatable in both volume size and timed intervals.
Easy to clean.
Field repairable or with plug-in replaceable parts.
Rugged and corrosion resistant.
Adaptable to pacing by a suitable flowmeter.

If nature of the wastes or analyses requires it:

Provide for cooling or chemical preservation.
Made of materials that will not alter sample characteristics such as low range organics.

If portable:

Light weight, easy to handle and install.
Operate on available line power or a battery.

SAMPLE EXTRACTION METHODS

Samples may be taken from a stream by three basic methods: mechanical, forced flow, or suction lift. The method, or variation, selected should take into account the variables previously mentioned, accessibility to the site, general character of the wastes, flow patterns, and the type of analyses to be made.

Mechanical

A. Cup or bucket on a chain or oscillating arm.
B. Self closing pipe "core".
C. Scoop(s) revolving or oscillating.
1. Shape characterized to primary element (weir/flume) for automatic flow proportioning (*TcVv*).
2. Wheel driven by flow-fixed volume cup on periphery. Number of samples, thus composite size, will vary as function of flow (*TcVc*).

Forced flow

A. Pneumatic ejection – requires pressurized gas or motor driven air compressors and/or electrical power.
B. Submersible pumps – possible obstruction to flow – maintenance problems – must be used if DO monitoring is involved.
C. Valve extraction from a pressurized line.

Suction Lift

A. Evacuated bottles – inherently explosion proof – limited lift.
B. Prssure vacuum pump/metering chamber – good volumetric control at high flows but may "air lift" excessive solids.
C. Positive displacement pumps – flexible impeller, progressive cavity, or peristaltic pumps. Speeds may be varied – some may be reversed to purge system.

5. SAMPLE COLLECTION SYSTEM

Pumped sample collection systems should maintain solids in suspension.
Intake lines should be of sufficient size to allow particles to pass through, but not so large as to allow them to settle out.

Low velocities result in loss of suspended solids due to settling. Excessive inlet velocities produce unrepresentatively high suspended solids or loss of dissolved gasses.

Sample intake line should be placed so that it is not collecting from a bed load or floating on the surface. Where water level is likely to change, such as in a sump, precautions should be taken to assure inlet will remain in the water. A screen should be over the intake that will exclude particles which would not be representative or clog the mechanism. If a scoop, dipper or cup is used it should be raised above the flow level except when sample is being collected.

Sample containers

The type and size of sample containers must also be considered. Obviously the container should be able to hold enough sample for the analyses to be made. Where sample containers will be reused, they should be easy to clean. If samples are to be analyzed for low range organics, fiberglass or other plastics should not be used as plasticizers may interfere. Glass containers are required for oily materials determinations.

Sample preservation

Some samples may need to be chilled to preserve biological integrity or retard volatilization. Chemical preservatives or pH adjustments may be required to stabilize certain parameters. Samples should not be exposed to excessive heat or light.

All sample containers should have waterproof labels with information regarding their source, method of collection, date and time collected, preservatives used, and any other information that may be required by laboratory to assure proper identification and handling. If samples are to be used as legal evidence, chain of custody logs must be maintained.

6. SUMMARY

The analysis of any sample, no matter how accurate or sensitive, is of no value unless the sample is representative of the source from which is was taken.

Micro analysis is providing valuable information about the condition of our environment. An understanding of how to collect samples from a macro-source can add greater reliability to the data and value to its application.