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SCIENTIFIC TECHNICIAN MANUAL

SAMPLING

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SAMPLING

Sampling is one of the most important means of determining information about a chemical process. Therefore, it is imperative that the most representative samples possible are taken, and that they are properly bottled, labeled, and analyzed.

Included in this report is the basic information necessary for a technician to carry out a good sampling program.

1. Methods

a. Solids

Solids are usually sampled directly from their containers by selecting varying solids at random. An average of the analyses of several such random samples will be a good indication of all the solids in the containers.

b. Liquids

The ideal liquid sample is the direct sampling of a constantly flowing stream. If a flowing stream cannot be sampled directly, enough liquid must be withdrawn, by running a stream from the sample point, to completely remove the stagnant liquid before any sample is collected. When sampling a drum or tank, the technician should first thoroughly mix the solution (this can be done by recycling, using an electric agitator, or stirring with a rod.) Then a sample can be removed by dipping the sample containers into the solution, by drawing solution into a glass tube with a rubber bulb, or by drawing solution through a valve on the tank if one is available.

c. Gases

The method often used is that of connecting a gas sample bottle to a vacuum source with flexible tubing and evacuating the gas sample bottle. Then connect evacuated sample bottle to the gas container and withdraw a gas sample. Many times a gas sample bottle is evacuated filled with the gas being sampled, then re-evacuated to insure that no residual gas remains except the gas being sampled.

d. Radioactive Solutions

Radioactive solutions many times have a high level of radiation which makes it undesirable to sample them in the usual manner. Special devices are used for this purpose, an example of which is here discussed (see Figure 1). This sampler uses an air lift pump principle plus a vacuum to remove solution from the tank being sampled, carry it through a sample bottle, and back

TYPICAL RADIOACTIVE SOLUTION SAMPLING

into the original tank. A controlled volume of air is fed into a line leading from the tank being sampled to a hypodermic needle (which penetrates a self sealing neoprene rubber diaphragm in the top of the sample bottle) which reaches nearly to the bottom of the sample bottle. The solution then flows out through another hypodermic needle near the top of the sample bottle and through a float control (which prevents radioactive solution from being drawn into the vacuum system) back into the original solution tank. This arrangement allows the solution to be recycled through the sample bottle until a representative sample is collected. The entire sampling device can be located behind shielding, the neoprene rubber top on the sample bottles prevents splatters and spilling, and the entire sampling operation can be controlled from a remote point. Should the float (which shuts off the vacuum supply) fail to seat, there is a thermometer which actuates a solenoid which shuts off the vacuum supply and prevents contamination of the vacuum system.

2. Quantity

Several factors influence the quantity of sample necessary. These include: (a) number of types of analyses desired; (b) concentration of substances to be analyzed; and (c) type of analysis. One factor that plays a very important part is quantity of material involved in the chemical operation. Many times it is desirable to take duplicate samples so that one may be kept for a future recheck.

3. Containers

a. Solids

Paper or plastic bags or glass sample bottles are usually used to contain solid samples.

b. Liquids

Glass sample bottles are usually used unless the liquid is hydrofluoric acid. Polyethylene bottles are used many times especially when the liquid sample would be dangerous if the sample bottle were broken.

c. Gases

Special glass bottles with stopcocks on each end are available for gas samples.

4. Labels and Label Codes

Special pens (that write on glass and plastics in ink), tapes, and small paper rectangles with adhesive on one side (though they should be secured with scotch tape for protection) are in widespread use for labels on samples. Most label codes include: name of the process or equipment, run number, date (month, day, and year), a means of determining what time of day it was taken, and the sample point. Here is a typical sample label:

T - 10
AW - 5
Oct. 10, 1955

Thorex process and run number 10.

Fifth sample taken from AW sample point.

Date

The run log or data sheets should give the time of day that sample 5 was taken in this case.

5. Submitting for Analysis

Various special forms are available. The one presently in use at ORNL is included (see Figure 2.) here with an explanation of the terms in the form. If the technician works where the budget system is in use, it will be necessary to obtain a charge number which is placed on the analysis sheet. The charge number can be obtained from the problem leader. A typical analysis sheet is filled out as an example (see Figure 3).

REQUEST FOR CONTROL ANALYSIS

This form is to be used only for samples containing less than 50 milligrams concentration of fissionable material.

NAME Name of Problem LeaderDATE SUBMITTED DateSERIES NUMBER Run NumberCHARGE NUMBER PLACED HERE

SAMPLE CODE		DESIRED ANALYSIS	ESTIMATION OF CONCENTRATION	PREVIOUS HISTORY OF SAMPLE	NATURE AND ESTIMATION OF ACTIVITY	CONCENTRATION OF ALL CONSTITUENTS IN SAMPLE
Code used on labels of the individual samples		Analyses desired on samples listed in first column	Estimation of concentration of substances for which analysis is desired	Give a brief description of what the process involves and where the sample was obtained in relation to the process	List types and amount of radiation (if any) given off by sample	List all known materials in the sample and estimate their concentrations

REQUEST FOR CONTROL ANALYSIS

This form is to be used only for samples containing less than 50 milligrams concentration of fissionable material.

NAME R. J. McNameeDATE SUBMITTED 10-10-55SERIES NUMBER T -10CHARGE NUMBER 3370-6-1354

SAMPLE CODE		DESIRED ANALYSIS	ESTIMATION OF CONCENTRATION	PREVIOUS HISTORY OF SAMPLE	NATURE AND ESTIMATION OF ACTIVITY	CONCENTRATION OF ALL CONSTITUENTS IN SAMPLE
AW - 1 2 3 4 5		$\begin{Bmatrix} \text{Th} \\ \text{Al}^+ \\ \text{H}^+ \end{Bmatrix}$ ↓	$\begin{Bmatrix} 0.05 \text{ g/l} \\ 27 \text{ g/l} \\ 1.0 \text{ M} \end{Bmatrix}$ ↓	Waste Sol'n Removed From Bottom of Extraction Column.	γ 10 mr/hr	Al Th HNO_3
AP - 1 2 3 4 5		Th ↓	70 g/l ↓	Thorium Extracted From Feed	α	42% TBP 58% AMSCO
BP - 1 2 3 4 5		$\begin{Bmatrix} \text{Th} \\ \text{H}^+ \end{Bmatrix}$ ↓	$\begin{Bmatrix} 100 \text{ g/l} \\ 0.008 \text{ M} \end{Bmatrix}$ ↓	Thorium Stripped From AP	α	Th HNO_3
BW - 1 2 3 4 5		$\begin{Bmatrix} \text{Th} \\ \% \text{ TBP} \end{Bmatrix}$ ↓	$\begin{Bmatrix} 0.0001 \text{ g/l} \\ 42\% \end{Bmatrix}$ ↓	Stripped Solvent	Slight Trace α	42% TBP 58% AMSCO Th
Feed		$\begin{Bmatrix} \text{Th} \\ \text{Al}^+ \\ \text{H}^+ \end{Bmatrix}$	350 g/l 1M 1M		γ 10 mr/hr α	Th Al HNO_3
Solvent		% TBP	42%		None	42% TBP 58% AMSCO
Scrub		$\begin{Bmatrix} \text{Al}^+ \\ \text{H}^+ \end{Bmatrix}$	1M 1M		None	Al HNO_3
Strip		H^+	0.008M		None	HNO_3

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Requestor

((Figure 3))

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