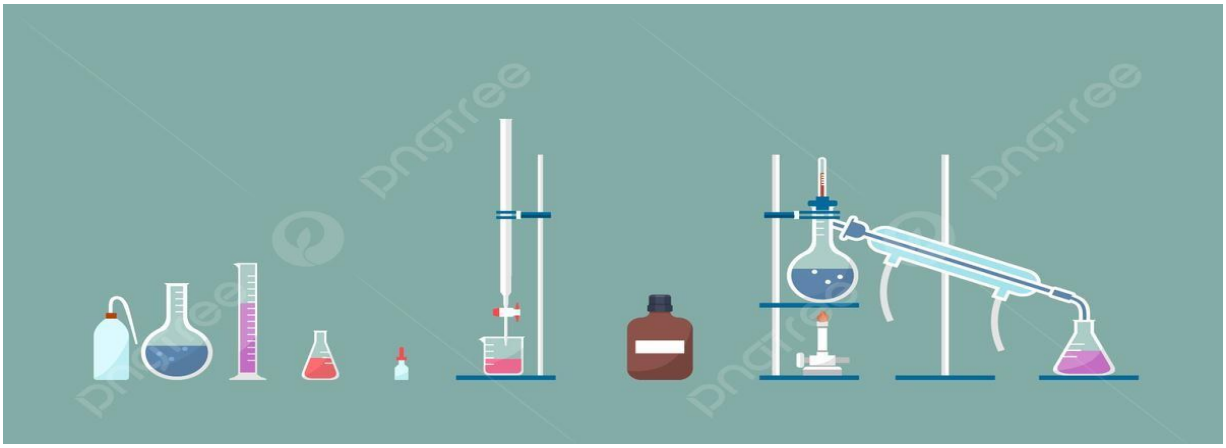
Al Mamoun University College
Laser and Optoelectronics Engineering Department
Chemistry

Analytical Chemistry



Chapter six & seven

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Obtaining and Preparing Samples for Analysis & Gravimetric Methods of Analysis

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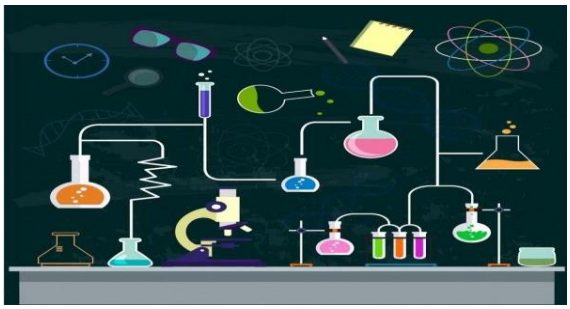

sampling plan
A plan that ensures that a representative sample is collected

1 *Designing A Sampling Plan*

A **sampling plan** must support the goals of an analysis. In characterization studies a sample's purity is often the most important parameter. For example, a material sci- entist interested in the surface chemistry of a metal is more likely to select a freshly exposed surface, created by fracturing the sample under vacuum, than a surface that has been exposed to the atmosphere for an extended time. In a qualitative analysis the sample's composition does not need to be identical to that of the substance being analyzed, provided that enough sample is taken to ensure that all components can be detected. In fact, when the goal of an analysis is to identify components present at trace levels, it may be desirable to discriminate against major components when sampling. In a quantitative analysis, however, the sample's composition must accurately represent the target population. The focus of this section, therefore, is on designing a sampling plan for a quantitative analysis.

Five questions should be considered when designing a sampling plan:

1. From where within the target population should samples be collected?
2. What type of samples should be collected?
3. What is the minimum amount of sample needed for each analysis?
4. How many samples should be analyzed?
5. How can the overall variance be minimized?

Each of these questions is considered below in more detail.

Sample Collection

Homogeneous solutions are easily sampled by siphoning, de- canting, or by using a pipet or syringe. Unfortunately, few solutions are truly homo- geneous. When the material to be sampled is of manageable size, manual shaking is often sufficient to ensure homogeneity. Samples may then be collected with a pipet, a syringe, or a bottle. The majority of solutions, however, cannot be sampled in this manner. To minimize the effect of heterogeneity, the method for collecting the gross sample must be adapted to the material being sampled

environmental sampling of waters and wastewaters provides a good illus- tration of many of the methods used to sample solutions. The chemical composi- tion of surface waters, such as streams, rivers, lakes, estuaries, and oceans, is influ- enced by flow rate and depth. Rapidly flowing shallow streams and rivers, and shallow (<5 m) lakes are usually well mixed and show little stratification with depth. Grab samples are conveniently collected by submerging a capped bottle below the surface and removing the cap. The air–water interface, which may be en- riched with heavy metals[9] or contaminated with oil, is avoided when collecting the sample. After the sample bottle is filled, the cap is replaced and the bottle removed. Slowly moving streams and rivers, lakes deeper than 5 m, estuaries, and oceans may show substantial stratification. Grab samples from near the surface can be collected as described earlier, whereas samples at greater depths are collected with a weighted sample bottle that is lowered to the desired depth. Once it has reached the desired depth, the sample bottle is opened, allowed to fill, and closed before retrieving. Grab samples can be analyzed individually, giving information about changes in the analyte's concentration with depth. Alternatively, the grab samples may be pooled to form a composite sample.

Wells used for collecting groundwater samples must be purged before the sam- ple is collected, since the chemical composition of water in the well-casing and in the adjacent matrix may be significantly different from that of the surrounding groundwater. These differences may result from contaminants introduced when drilling the well, or differences in the groundwater's redox potential when exposed to atmospheric oxygen. In general, wells are purged by pumping out a volume of water equivalent to several well-casing volumes, or until the water's temperature, pH, or specific conductance are constant. Samples collected from municipal water supplies must also be purged since the chemical composition of water left standing in

pipes may differ significantly from the treated water supply. Samples are collected at faucets after flushing the pipes for 2–3 min. Samples from municipal wastewater treatment plants and samples of industrial discharges often are collected as 24-h composites. Samples are obtained using an automatic sampler that periodically removes individual grab samples. The volume of each sample increment and the frequency of sampling may be constant or may vary in response to changes in flow rate.

Sample containers for collecting solutions are made from glass or plastic. Con-tainers made from Kimax or Pyrex brand borosilicate glass have the advantage of being sterilizable, easy to clean, and inert to all solutions except those that are strongly alkaline. The disadvantages of glass containers are cost, weight, and the likelihood of breakage. Plastic containers are made from a variety of polymers, in- cluding polyethylene, polypropylene, polycarbonate, polyvinyl chloride, and Teflon (polytetrafluoroethylene). Plastic containers are lightweight, durable, and, except for those manufactured from Teflon, inexpensive. In most cases glass or plastic bot-tles may be used, although polyethylene bottles are generally preferred because of their lower cost. Glass containers are always used when collecting samples for the analysis of pesticides, oil and grease, and organics because these species often inter- act with plastic surfaces. Since glass surfaces easily adsorb metal ions, plastic bottles are preferred when collecting samples for the analysis of trace metals.In most cases the sample bottle has a wide mouth, making it easy to fill and re- move the sample. A narrow-mouth sample bottle is used when exposing the sample to the container cap or to the outside environment is undesirable. Unless exposure to plastic is a problem, caps for sample bottles are manufactured from polyethylene. When polyethylene must be avoided, the container cap includes an inert interior liner of neoprene or Teflon.

1 Overview of Gravimetry

Before we look more closely at specific gravimetric methods and their applications, let's take a moment to develop a broad survey of **gravimetry.** Later, as you read through the sections of this chapter discussing different gravimetric methods, this survey will help you focus on their similarities. It is usually easier to understand a new method of analysis when you can see its relationship to other similar methods

sGravimetry

Any method in which the signal is a mass or change in mass

Types of Gravimetric Methods

1. precipitation gravimetry

2. electrogravimetry

3. volatilization gravimetry

4. particulate gravimetry

H.W
Why Gravimetric Is Important?


The End