

1. Typical Quantitative Analysis

ex: if the sample already liquid → we can avoid dissolution step.

In measurement step → measure one of physical properties

In calculation step → find the relative amount of analyte present in the samples.

In final step → evaluate the quality of results & estimate reliability

Choosing Method

↳ Selection of method → essential 1st step

↳ One of the first question in selection process:

Level of accuracy required

↳ Selected method represent compromise between accuracy required & the time & money available for analysis.

↳ Second consideration related to economic factors

↳ number of samples that will be analyzed

! If there are many samples → spend significant amount of time in preliminary operations

→ ex: assembling & calibrating instruments & equipment

- preparing standard solutions

! If only have 1 sample / few sample → select a procedure that avoids/minimizes → ex: preliminary steps

→ The complexity of sample & number of components in the sample may influence the choice of method to some extent

Acquiring the Sample

↳ To produce meaning full information: analyst must be performed on sample that has the same composition as the bulk of material from its taken

↳ Heterogeneous 異質的

- required to have great effort to get representative sample
- A material is constituent parts can be distinguished visually / with the aid of microscope
- Ex: coal, animal tissue & soil.

↳ Assay 試驗

- perform on sample that weights abt 1 gram
- process of determining how much of given sample is the material by its indicated name
- Ex: zinc alloy is assayed for its zinc content & its assay is a particular numerical value.

↳ Sampling → process of collecting small mass of material whose composition accurately represent the bulk of material being sampled

試樣

- analyze sample & determine substance

ex: blood sample is analysed to determine concentration of various substance such as blood gases & glucose

↳ Analyst must be sure that laboratory sample is representative of the whole before proceeding

↳ Sampling is the most difficult step in analysis & source of greatest error

↳ The final analytical result will never be any more reliable than the reliability of sampling step.

- Processing the Sample

↳ Certain circumstance, no sample processing is required prior to measurement

↳ ex: When water sample is withdrawn from a stream, lake / ocean, the pH of sample can be measured directly.

↳ First step of processing the sample → preparation of lab. sample

↳ Step:

a) Preparing Laboratory Sample

- Solid lab. sample is ground to decrease particle size, mixed to ensure homogeneity & stored for various lengths of time before analysis begins.
- Absorption / Desorption of water may occur depend on humidity of environment
- Moisture content of sample can determine at the time of analysis in separate analytical procedure

• Liquid samples present slightly different but related set of problem during this step → If the sample allow to stay in open containers, solvent may evaporate & change the concentration of analyte.

* Volatile gases dissolve in liquid as our ex (blood gas)
the sample container must be kept inside second sealed container

to prevent contamination by atm gases

To preserve integrity of sample → required extraordinary measures include sample manipulation & measurement in inert atm.

- Defining Replicate Samples

↳ Replicate sample / replicates → portions of material of approximately the same size that're carried through analytical procedure at the same time & way.

複製試樣

→ masses / volumes have been determined by careful measurements with analytical balance / with precise volumetric device

↳ Replication improve quality of results & provides measure of reliability

- Preparing Solutions: Physical & Chemical Changes

↳ solvent should dissolve the entire sample, include analyte, rapidly & completely.

↳ Many Materials that must be analyze are insoluble in common solvent

↳ ex: Silicate materials, high molecular mass polymers & specimen of animal tissue

↳ Converting analyte into soluble form is most difficult & time consuming task in analytical process

↳ Sample may require heating with aqueous solution of:

- strong acid
- oxidizing agents
- combination of reagents
- strong bases
- reducing agents

↳ necessary to ignite sample in air / oxygen / to perform high temp fusion of sample in presence of various fluxes

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