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NPL 



Sampling for radioactive waste characterization

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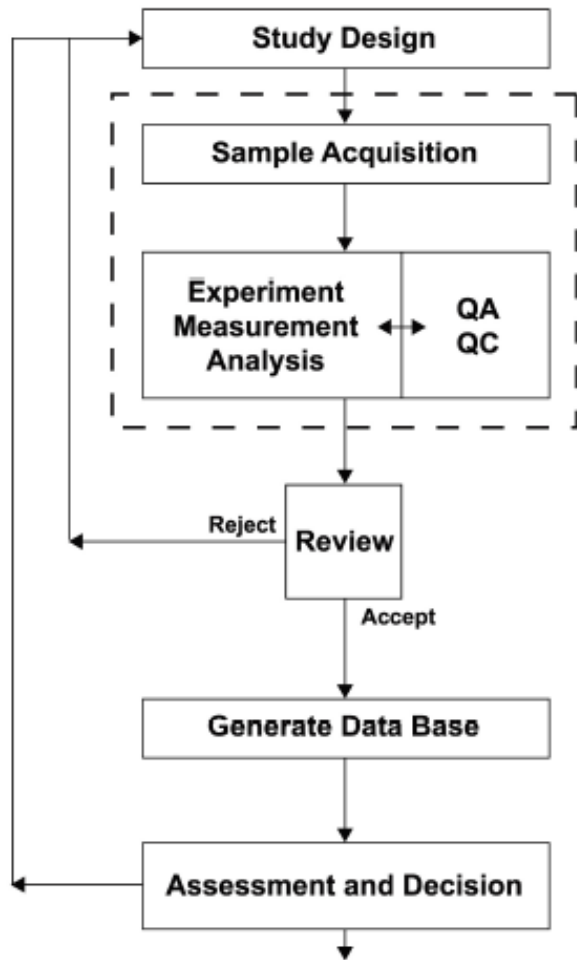
Overview

- **Introduction**
- **Homogeneity**
- **Precision and Bias**
- **Data Quality Objectives (DQO) method**
- **Sampling strategy**
- **Sampling equipment**
- **Sample dissolution**
- **References**

Introduction

- **Sampling** - first physical step in any measurement of a sample population.
- The characteristics of collected samples are used **to make estimates of the characteristics of the population.**
- The assumption is that the samples both accurately and precisely **represent the population.**

Sampling and measurement process

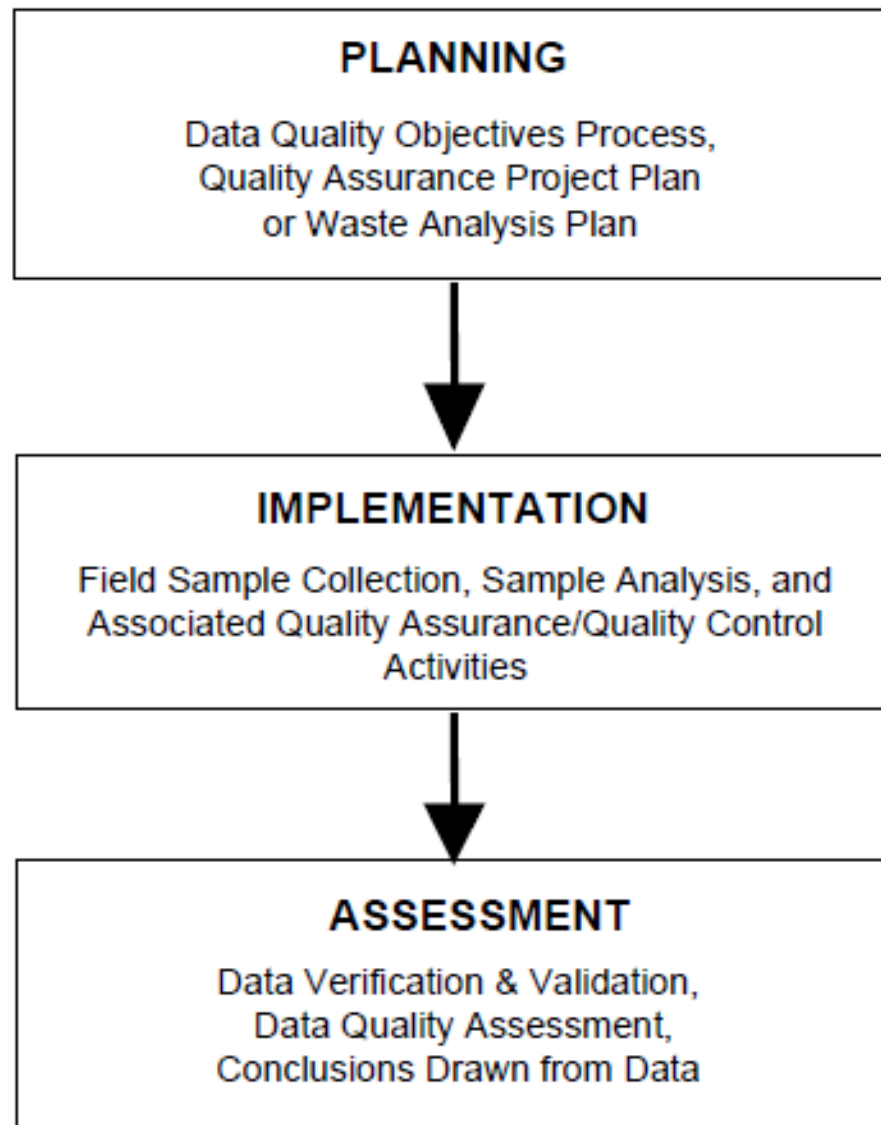


Data Quality Objectives (DQO)

Planning - asking the right questions

Implementation involves using the field **sampling procedures** and **analytical methods** specified in the plan and taking measures to **control errors** that might be introduced along the way.

Assessment - **evaluate the results** against the original objectives and **make decisions** regarding management or treatment of the waste.

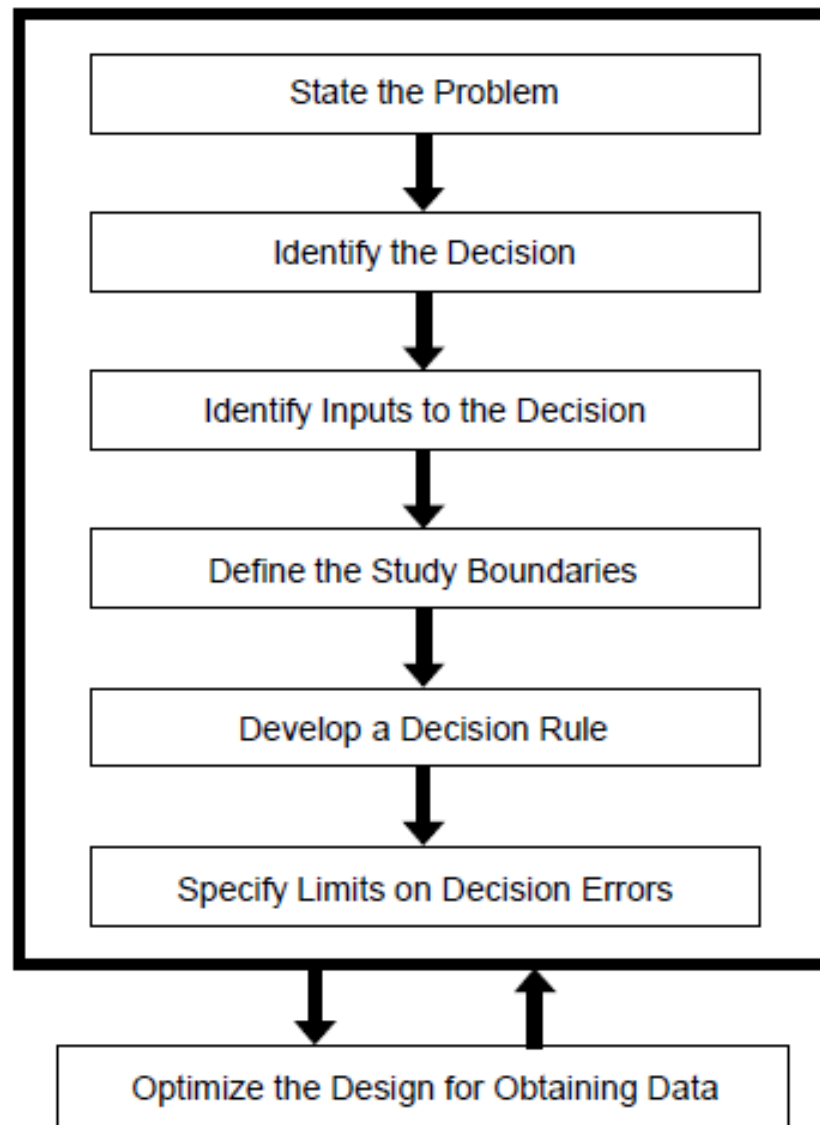


Data Quality Objectives (DQO) Process

The DQO Process comprises **seven planning steps**.

One of the most important features of the process- its **iterative** nature. You don't have to "get it right the first time." You can use existing information to establish DQOs.

This way, you can evaluate **sampling designs and related costs *in advance*** before significant time and resources are expended to collect and analyze samples.





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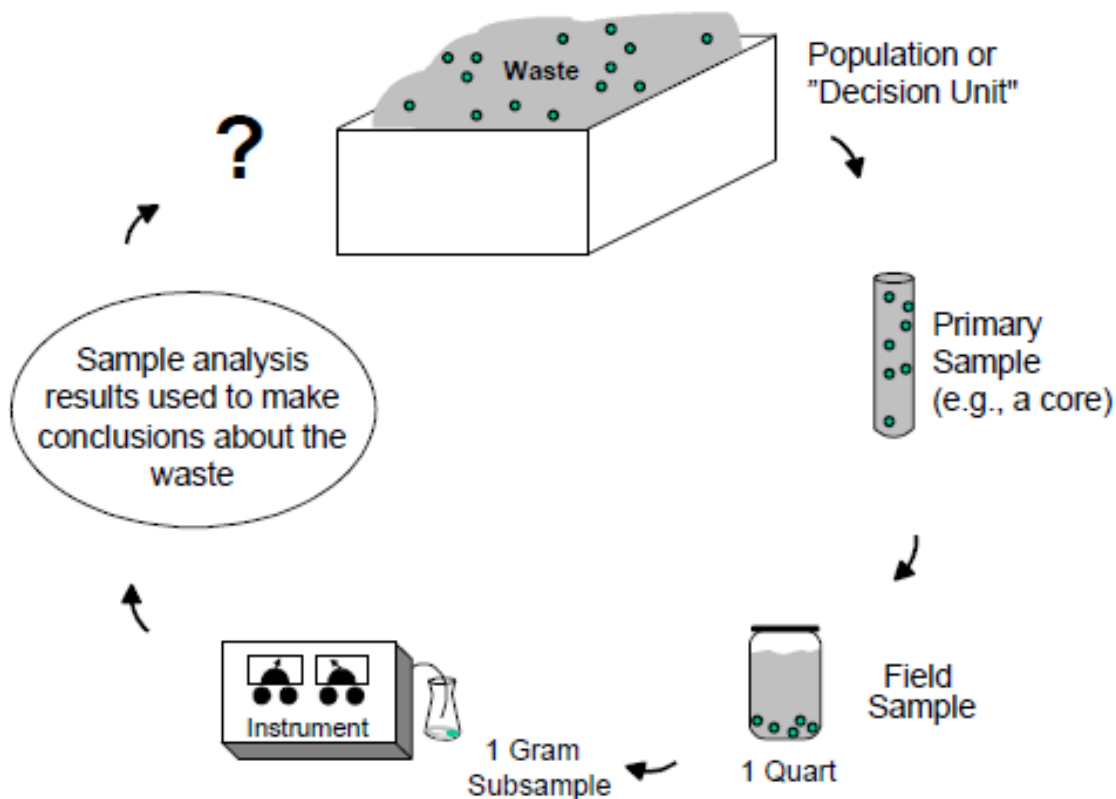
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Samples

Population: The entire selection of interest for study, i.e. a set of waste packages or a waste stream.

Sample: A portion of material that is taken from a larger quantity for the purpose of **estimating properties** or composition **of the larger quantity**.

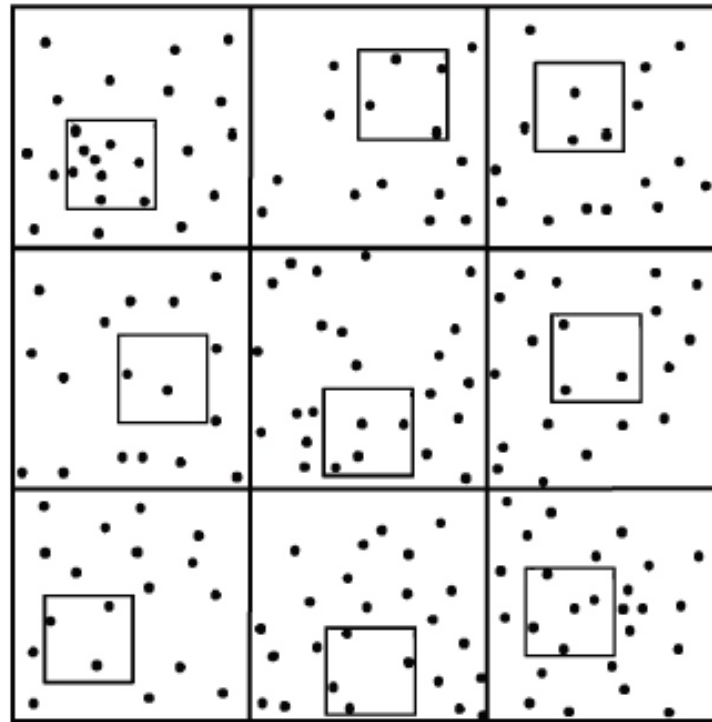
Statistical sample: A set of samples or measurements elected by probabilistic means (i.e., by using some form of randomness).



Heterogeneity

Heterogeneity is the condition of a population when all of **the individual items** are **not identical with respect to the characteristic of interest**.

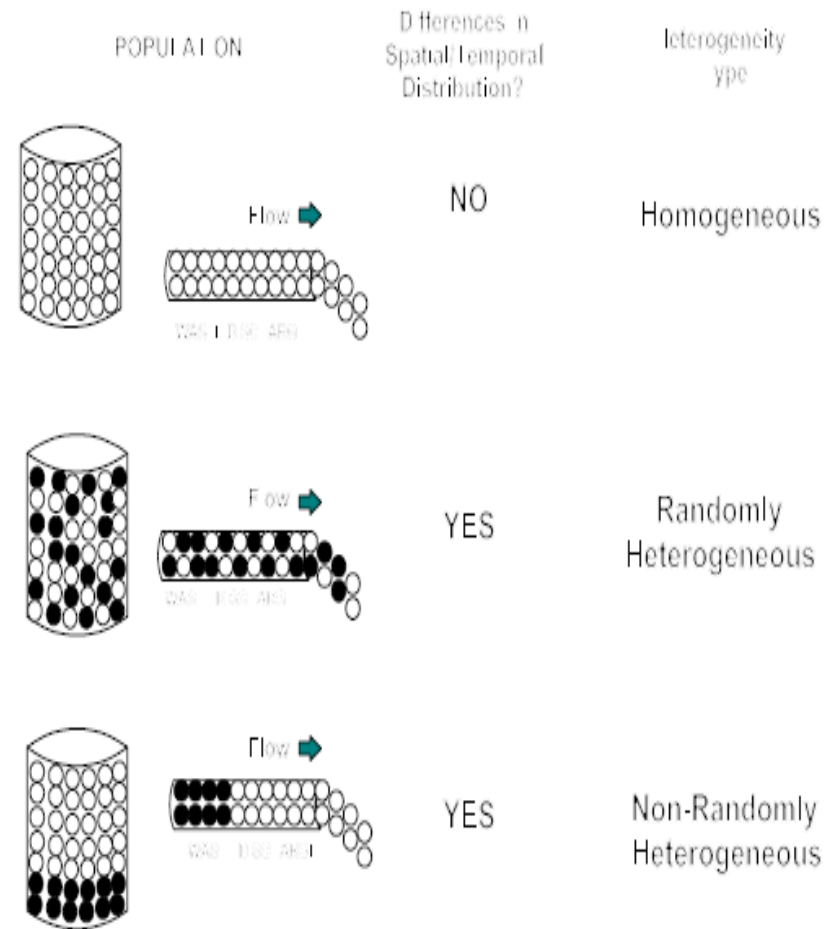
Since a sample cannot be completely identical to the lot, the next best goal is for it to be **as similar to the lot as possible**.



Heterogeneity

Items, particles, or phases are **distributed in various ways to create distinctly different types of heterogeneity.**

- Random heterogeneity— occurs when dissimilar items are **randomly distributed throughout the population.**
- Non-random heterogeneity— occurs when **dissimilar items are non-randomly distributed, resulting in the generation of strata.**



Strata- subgroups of a population separated in space or in time, which are **internally consistent.**

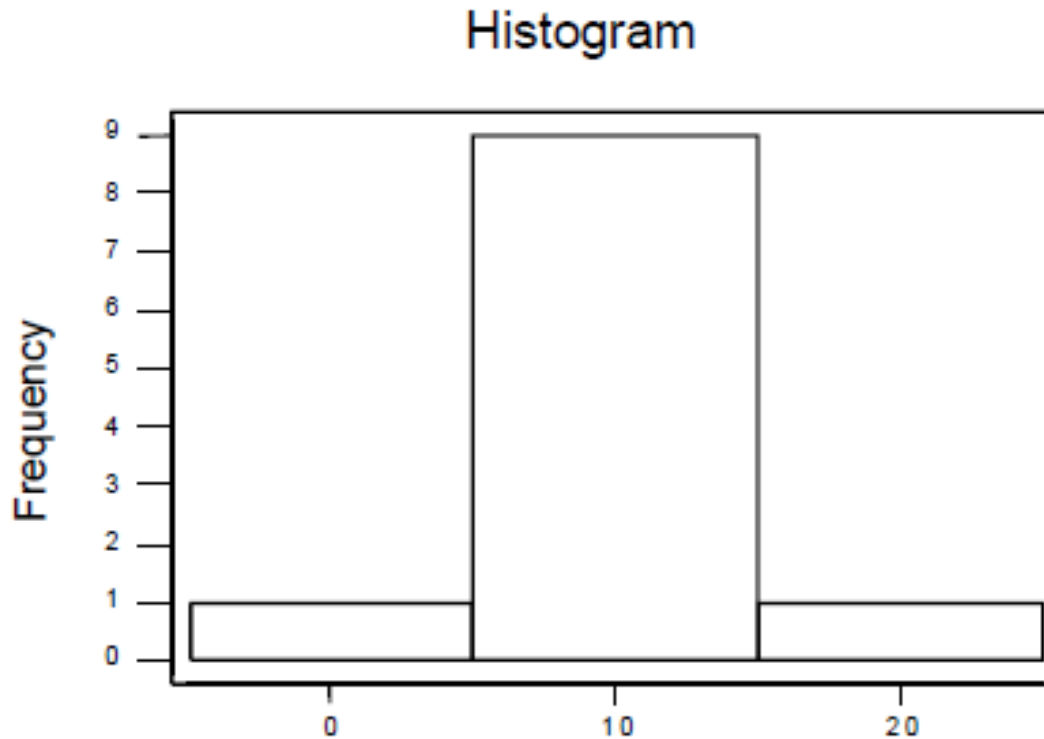
Heterogeneity

To reduce the heterogeneity:

- **Increasing the sample mass** - the distribution of the different particles will more likely closely match the original particle distribution and the relative influence of any given particle on the property of interest is smaller.
- **Breaking up the larger particles into several smaller particles** (reducing the scale of heterogeneity) by crushing or grinding the sample. The smaller the particle size, the smaller the effect of including or excluding any type of particle in the sample

Distributions

The **concentration of radionuclides of concern will not be the same** for every individual sample, there must be a ***distribution of concentrations among the population***.



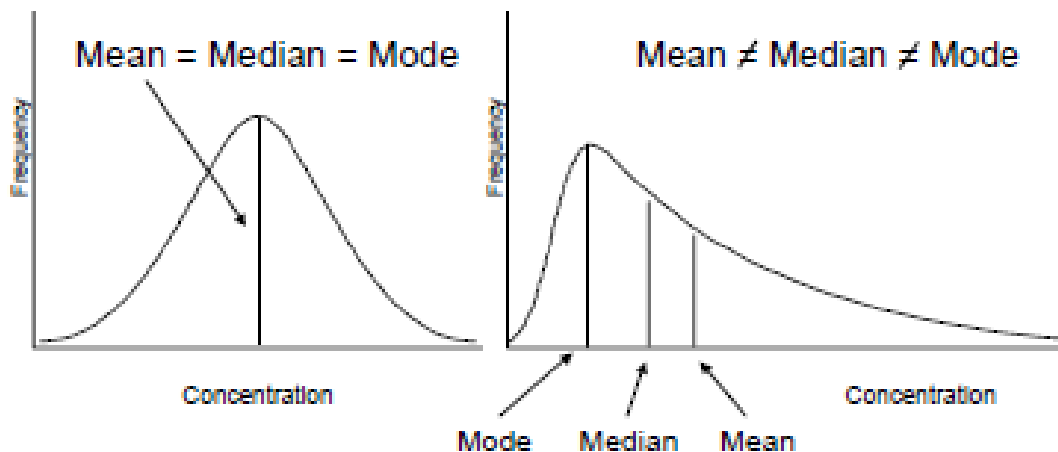
Histogram, [²³⁵U], Bq.g⁻¹

Distributions

Sufficiently large number of samples- **probability - density function.**

(a) Normal Distribution

(b) Lognormal Distribution



(a) **Normal distribution** - symmetrical mound-shape.

(b) **Log-normal distribution**, indicates that a relatively small proportion of the population includes some relatively large values

Statistical characteristics

The average of measurements taken on random samples is the **sample mean**.

$$\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i$$

Sample variance (s^2) includes random variation in the population as well as random variation that can be introduced by sample collection and handling, sample transport, preparation and analysis:

$$s^2 = \frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2$$

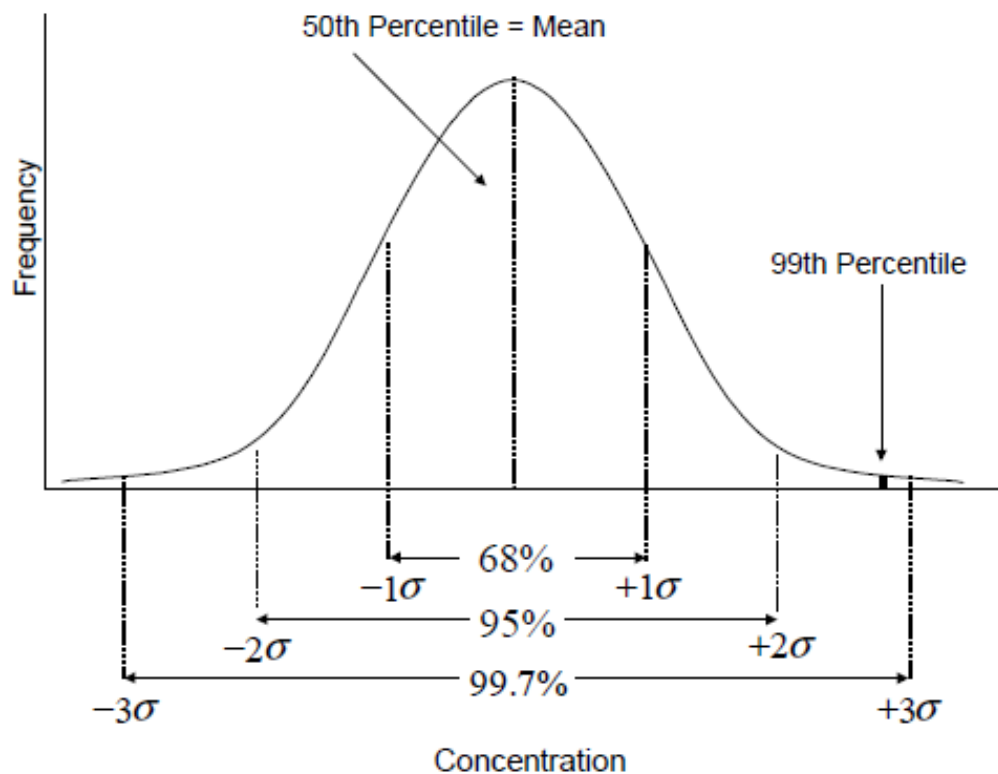
The **standard deviation** σ is used to measure the variability in a data set:

$$\sigma = \sqrt{s^2}$$

Standard deviation

For a normal distribution, we know the following:

- Approximately **68 percent** of measurements will fall within **± 1 standard deviation** of the mean;
- Approximately **95 percent** of the measurements will fall within **± 2 standard deviations** of the mean;
- Almost all (**99.74 percent**) of the measurements will fall within **± 3 standard deviations** of the mean.



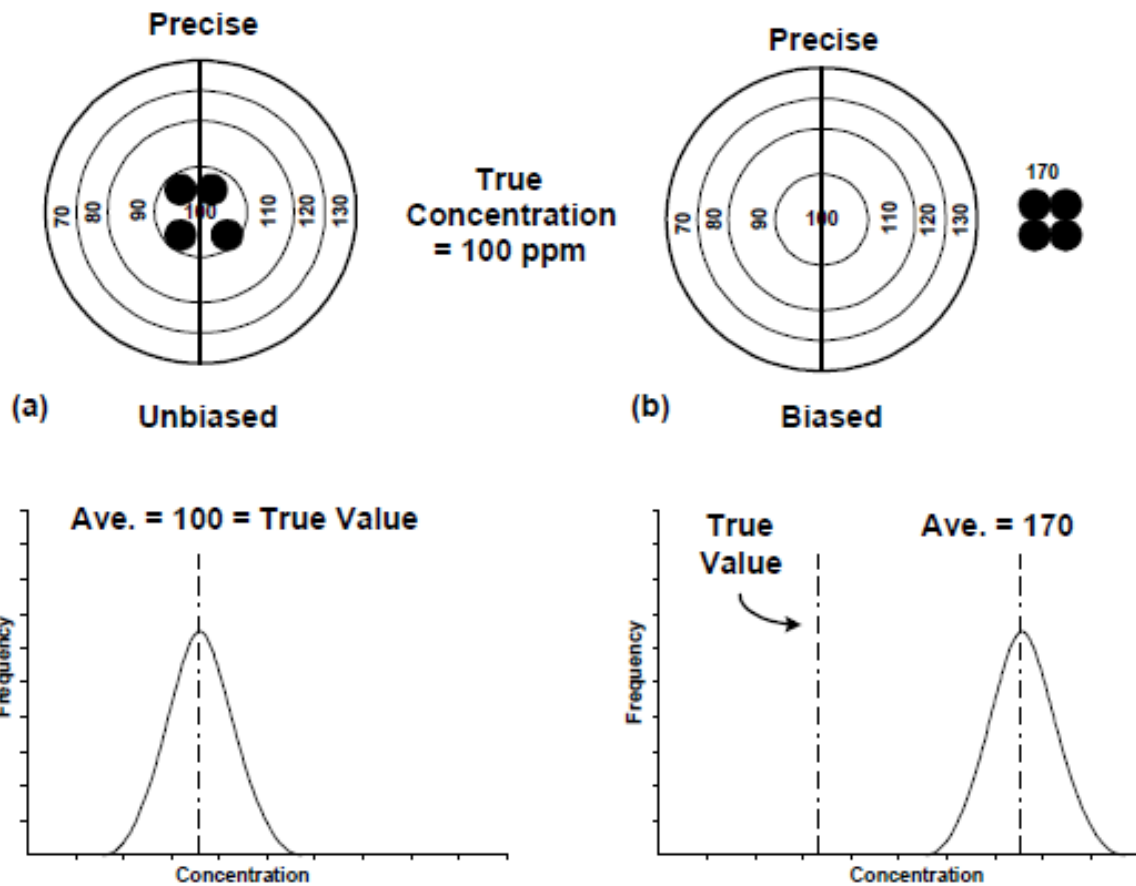
The larger the spread in the data, the less certainty we have in estimates or decisions made from the data.

Precision and Bias

Precision - *closeness of agreement between repeated measurements.*

Bias is over- or underestimation of the true value

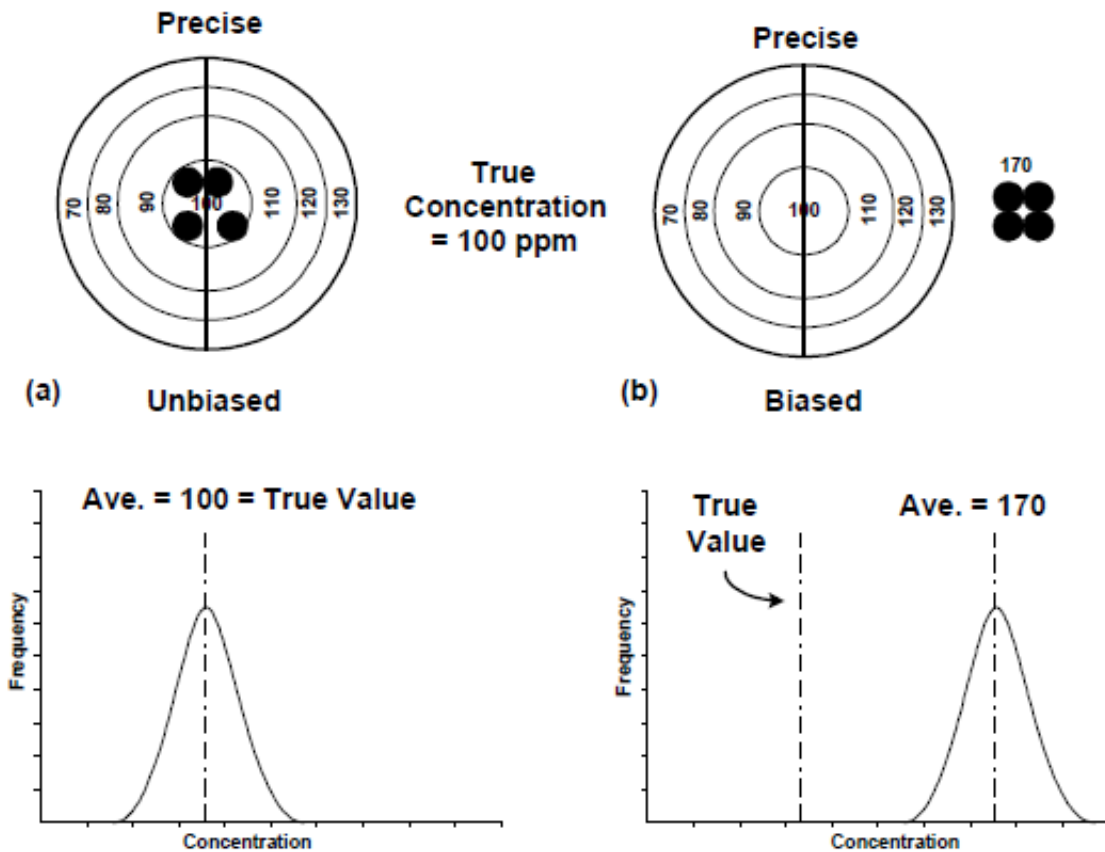
The “shots” in targets through **represent measurement results** from samples taken to estimate the true concentration.



Precision and Bias

(a) High precision and low bias; (b) High precision and high bias

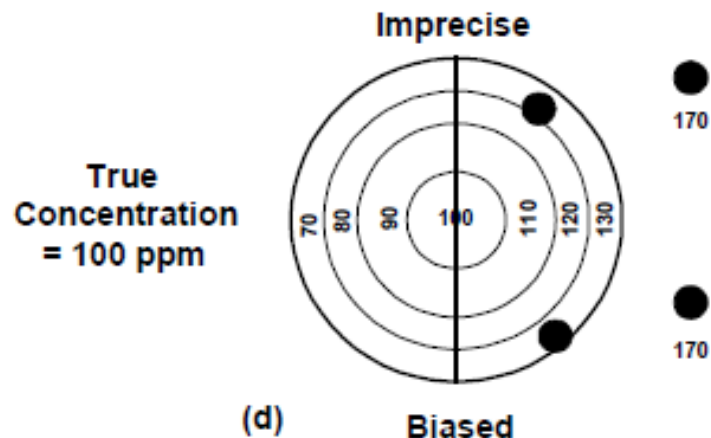
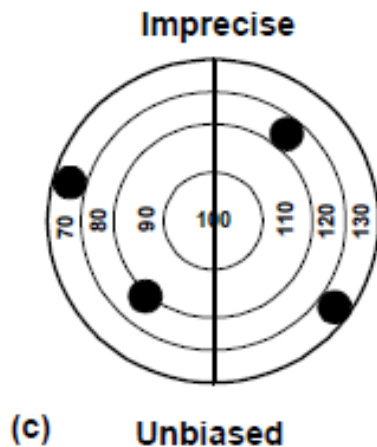
If the sampling and measurement process is **very precise but suffers from bias** (such as use of an incorrect sampling procedure or contamination of an analytical instrument), the situation could be as pictured in (b) in which the **repeated measurements are close to one another but not close to the true value.**



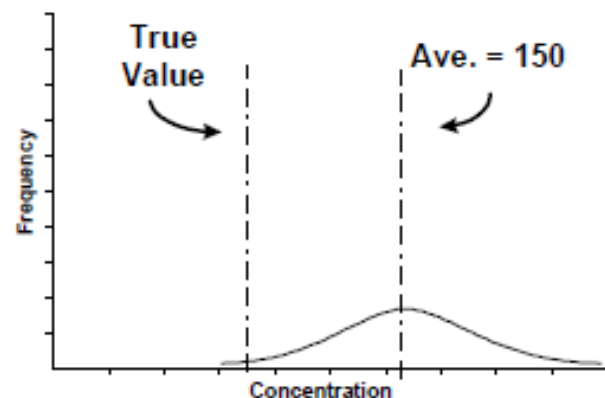
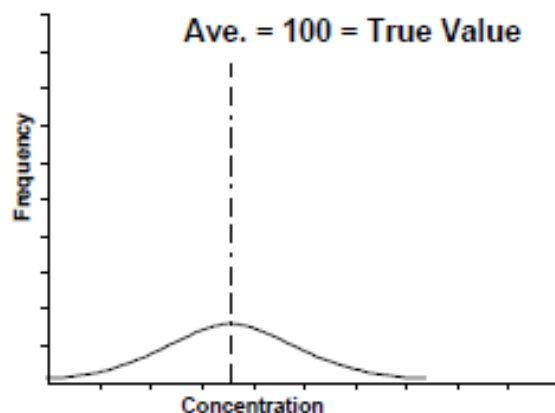
Precision and Bias

d) Low precision and high bias

(c) low precision and low bias
(high dispersion around the mean) indicates lack of systematic error and the average of the measurements reflects the true average concentration.



True
Concentration
= 100 ppm



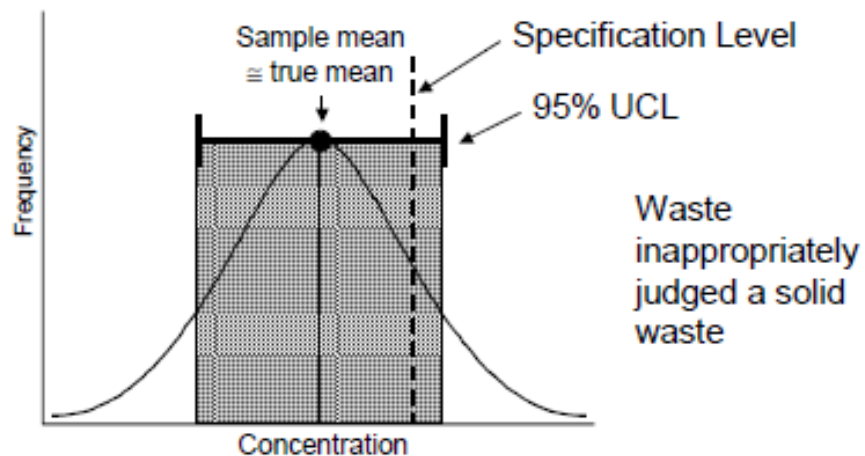
Compare with the Applicable Standard

Sampling precision can affect the ability to claim an exclusion from the definition of solid.

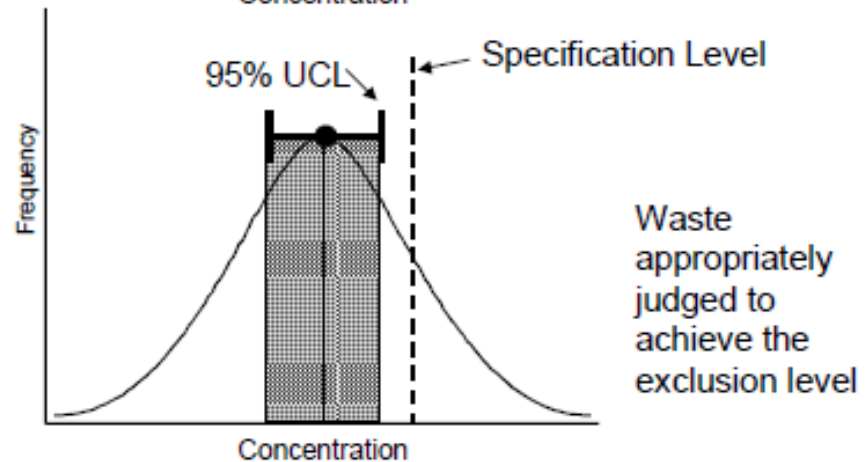
In A the sampling results are unbiased, but they are not sufficiently precise. Imprecision can be caused by the heterogeneity of the material sampled, by random errors in the field and laboratory, and by too few samples.

In B the results also are unbiased, but significant improvement in precision is observed (e.g., because more or larger samples were analyzed and errors were kept within acceptable limits), allowing us to conclude that the mean is indeed below the specification level.

A



B



Example of how sampling precision could impact a waste exclusion.

Sampling Design

Consider various strategies for selecting the locations, times, and components for sampling

Examples of sampling designs include:

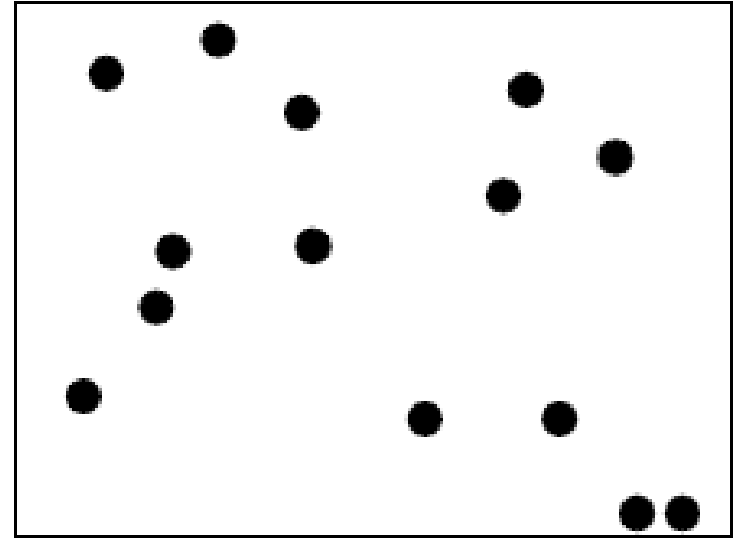
- **Simple random** sampling
- **Stratified random** sampling
- **Systematic** sampling
- **Ranked set** sampling
- **Sequential** sampling.

Standard operation procedures (SOPs) - describes the **steps to be followed when implementing a sampling** activity (e.g., equipment preparation, sample collection, decontamination).

Simple Random Sampling

The **simplest type** in which the **sampling unit** from the **target population** are selected **randomly**.

Appropriate **at an early stage of an investigation** in which little is known about the waste



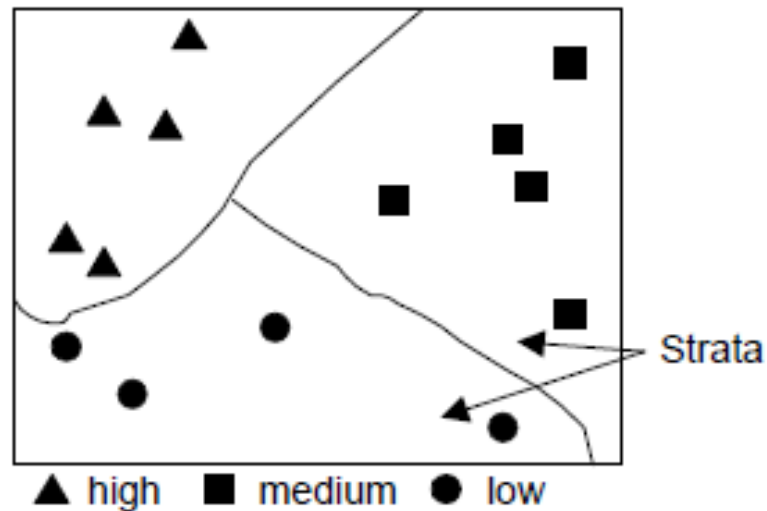
- Useful when the **population of interest is relatively homogeneous** (i.e., there are **no major patterns** or “**hot spots**” expected).
- Provides **statistically unbiased estimates** of the mean, proportions, and the variability.
- **Easy to understand** and implement.
- **Least preferred if patterns or trends** are known to exist.

Stratified Random Sampling

Sample population is divided into **strata**.

Each stratum should be **internally relatively homogeneous**.

Simple random sampling is used within each stratum.



- Requires some **prior knowledge of the waste**

- **May reduce costs** over simple random and systematic sampling designs because **fewer samples may be required**.

- **Statistical procedures** for calculating are **more complicated than for simple random sampling**.

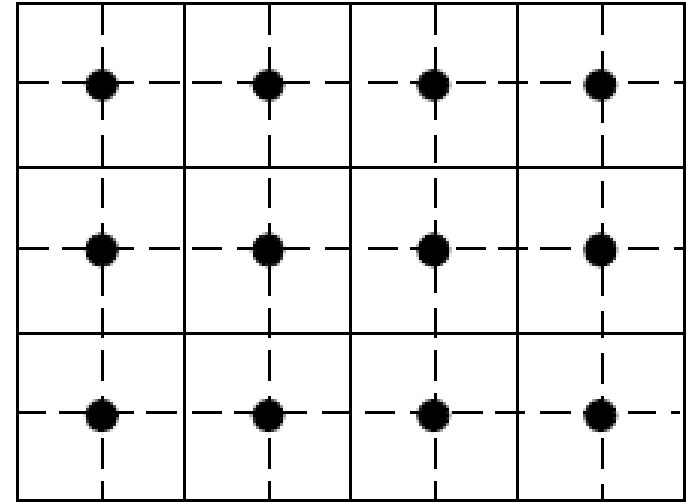
- Ensures **more uniform coverage** of the entire target population.

Systematic Sampling

Involves **establishing a two-dimensional grid** of the unit or waste under investigation.

Points may be arranged **in a pattern of squares** (rectangular grid sampling) **or a pattern of equilateral triangles** (triangular grid sampling).

The result of either approach is a **simple pattern of equally spaced points** at which sampling is to be performed.



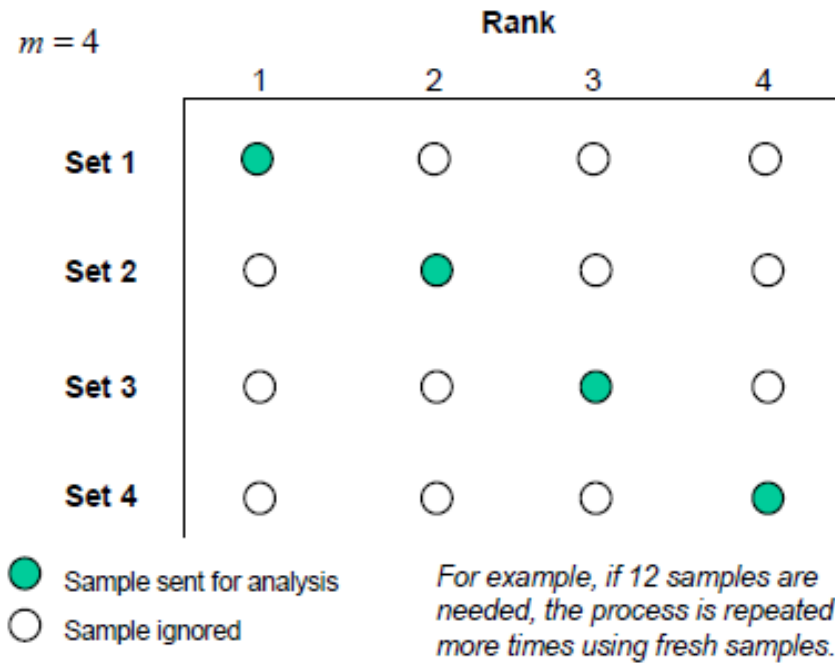
- **Practical and easy method** for designating sample locations.
- **Ensures uniform coverage** of site, unit, or process.
- May be **lower cost than simple random sampling** because it is easier to implement.

Ranked Set Sampling (RSS)

RSS uses the availability of **expert knowledge** or an **inexpensive surrogate measurement**

Auxiliary variable, such as visual inspection for colour or an inexpensive quantitative measure (i.e. **dose rate**).

Exploits this correlation **to obtain a sample that is more representative of the population than would be obtained by random sampling**

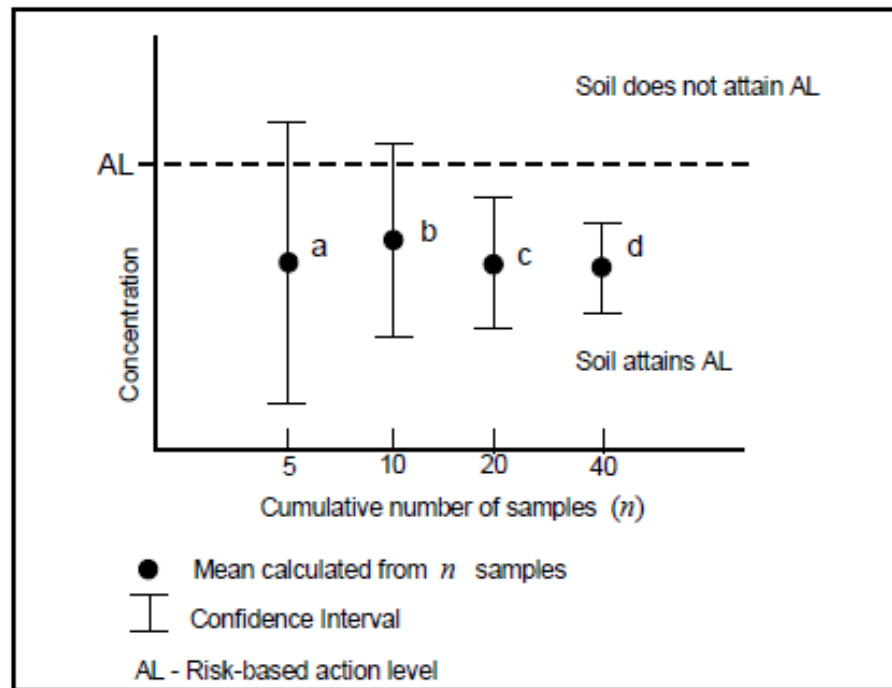


- **Reduces the number of samples**
- **Could be misleading if not properly implemented**

Sequential Sampling

Analysing **one sample at a time** until enough data have been collected to meet the statistical confidence level that the material does not exceed the critical level.

30 to 60 % lower samples needed than a fixed sample size test with the same power.

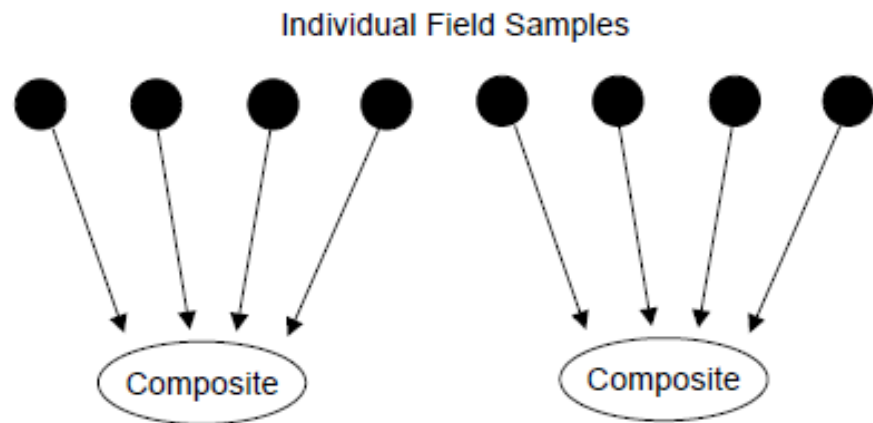


- It may not be especially useful if multiple waste characteristics are of interest or if rapid decision making is necessary.

Composite Sampling

Multiple individual or “grab” samples are physically combined and mixed into a single sample so that a physical, rather than a mathematical, averaging takes place.

A single measured value should be similar to the mean of measurements of the individual components of the composite.



- Useful when the cost of analysis is higher than the cost of sampling

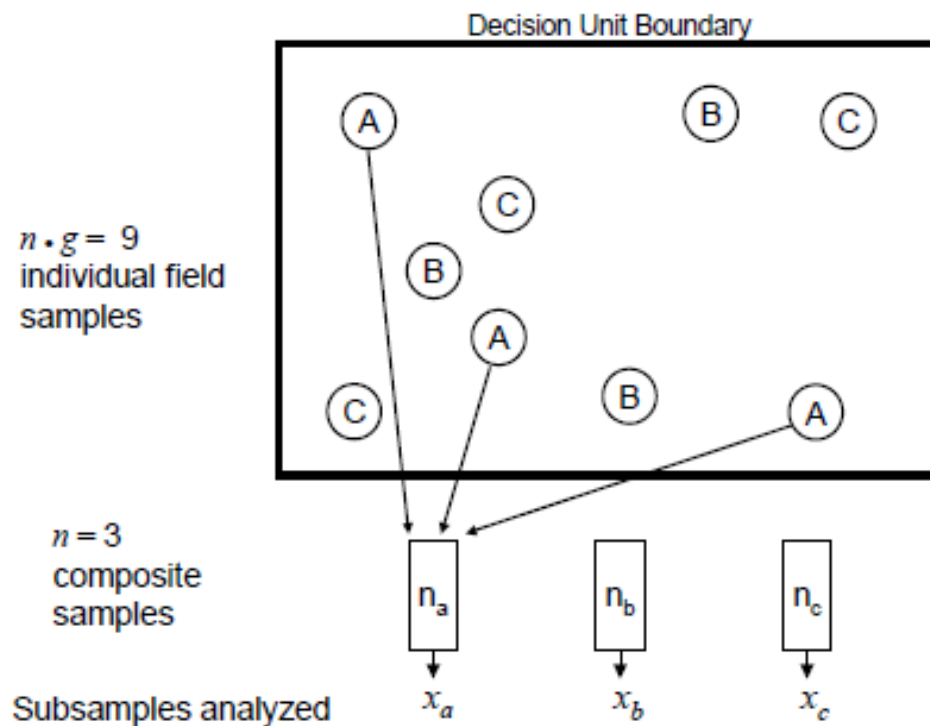
Simple Random Composite Sampling

Randomly positioned field samples are **randomly grouped together** into composite samples.

The set of composite samples can then be used to **estimate the mean and the variance**.

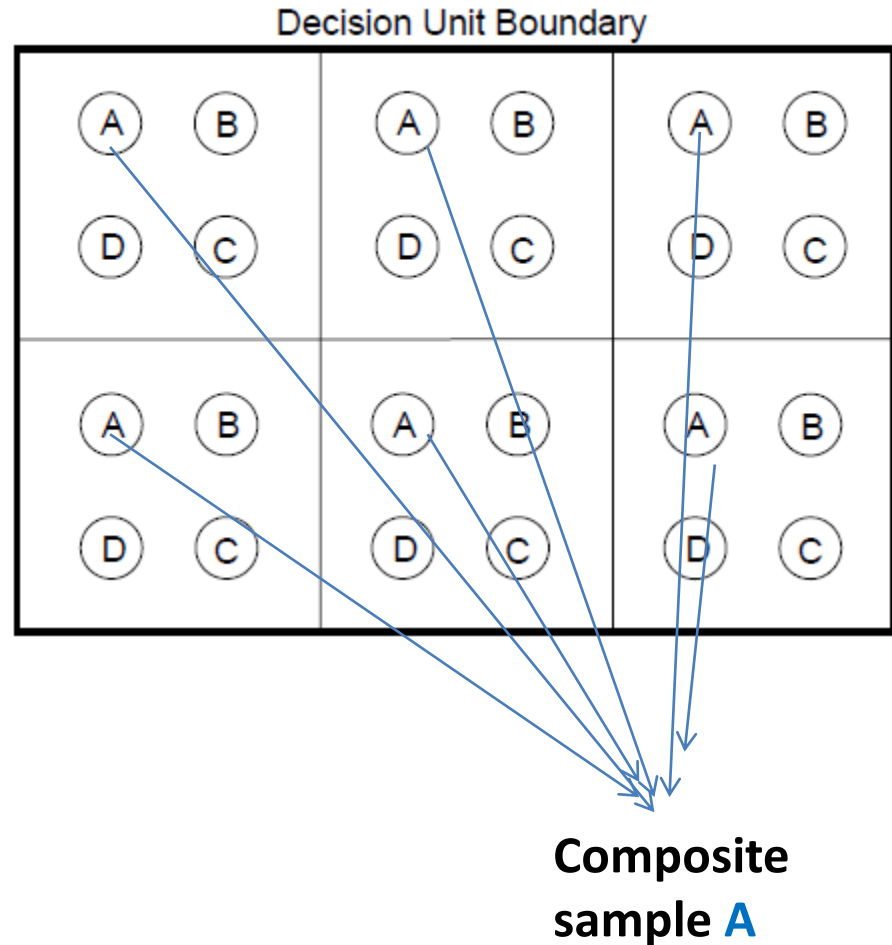
Because the compositing process is a **mechanical way of averaging** out

The **resulting concentration data should tend to be more normally distributed** than individual samples.



Systematic Composite Sampling

- Used to **estimate the mean concentration**
- The **relative location** of each individual field sample (such as “A”) **should be the same** within each block.
- **Easy to implement** and explain
- **Provides even coverage** of the unit



Using Composite Sampling To Obtain Estimate of the Mean

The population mean (μ) can be estimated from the analysis of n composite samples (each made from g individual samples). The population mean (μ) is estimated by the sample mean \bar{x} by:

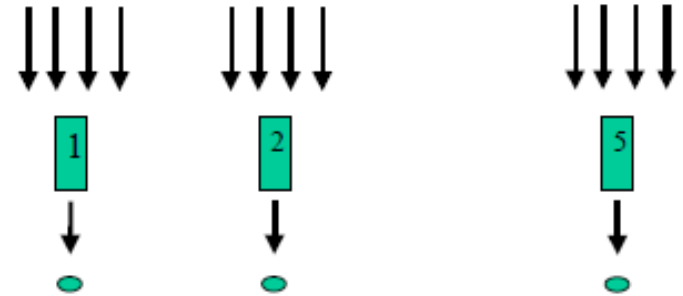
$$\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i$$

The sample variance (s^2) can then be calculated by:

$$s^2 = \frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2$$

- Use of these equations **assumes equal numbers of individual field samples (g) are used to form each composite.**
- **By increasing the number of individual field samples (g) per composite sample, there will be a corresponding decrease in the standard error (s).**

Example



Five composite samples. **Sample mean** is:

2.75 Bq
3.71 Bq
3.28 Bq
1.95 Bq
5.10 Bq

$$\bar{x} = \frac{1}{n} \sum_{i=1}^n x_i = \frac{16.79}{5} = 3.36 \text{ Bq}$$

The **sample variance** (s^2) is:

$$s^2 = \frac{1}{n-1} \sum_{i=1}^n (x_i - \bar{x})^2 = \frac{1}{4} (0.3721 + 0.1225 + 0.0064 + 1.9881 + 3.0276) = 1.38$$

The **standard error** is obtain as follows:

$$s_{\bar{x}} = \frac{s}{\sqrt{n}} = \frac{0.17}{\sqrt{5}} = 0.52$$

$$\bar{x} = 3.36 \pm 0.52 \text{ Bq}$$

Sampling Tools and Devices

Sampling device and sampling method **depends on:**

- **the sampling objectives**
- the **physical characteristics** of the waste or media
- **the chemical constituents**
- the sampling **location**
- **technology** limitations
- **safety** issues

Selecting Sampling Tools and Devices

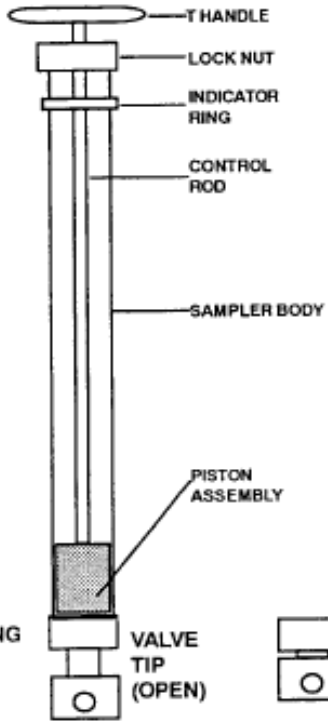
Media	Sampling point	Sampling device
Liquids	Drums, Tanks, Pipes, Discharge points	Dipper , Peristaltic pump , Syringe sampler, Plunger type sampler, Swing jar sampler
Sludges , slurries, and solid- liquid suspensions	Drums, Tanks, Pipes, Conveyors, Surface impoundment	Dipper , Bucket , Scoop, Shovel, Swing jar sampler, Syringe sampler, Plunger type sampler
Unconsolidated solids (soil, sand)	Waste pile, Drums, Bags, Conveyors, Storage bins,	Scoop , Shovel, Bucket auger, Coring type sampler
Consolidated solids (concrete)	Storage bins, Waste piles, Structures	Penetrating probe sampler Rotating coring device



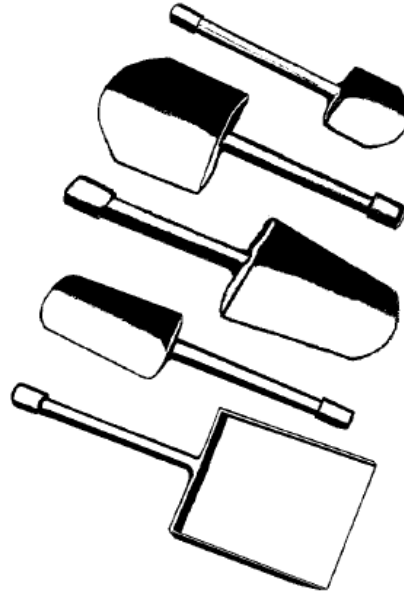
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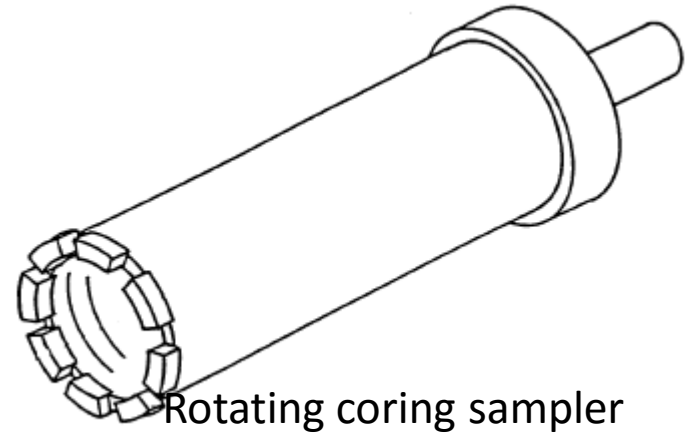
Sampling Devices



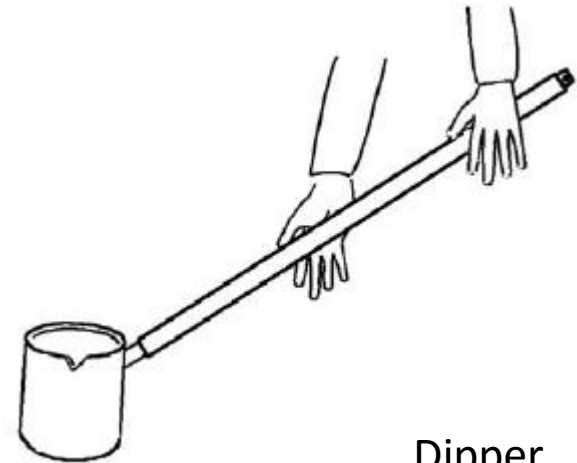
Syringe sampler



Scoops and shovels



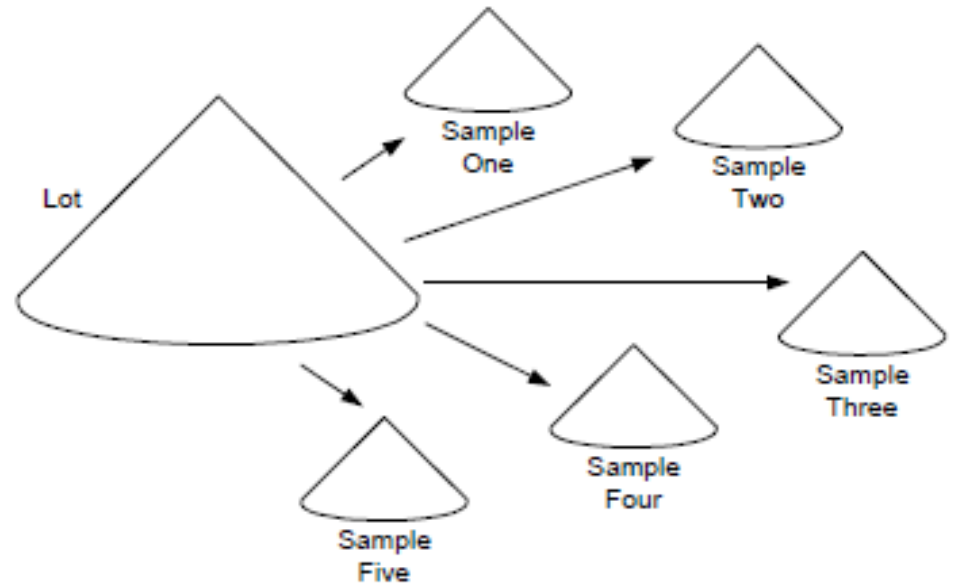
Rotating coring sampler



Dipper

Sample Splitting

- **A field sample is significantly larger than the required analytical sample.**
- Splitting is to reduce the mass of the retained sample and **obtain an aliquot of the field sample** that reflects the average properties of the entire field sample.



Fractional shoveling as a sample splitting method

To perform fractional shoveling, deal out small increments from the larger sample in sequence into separate piles, **randomly select one of the piles** and retain it as the subsample

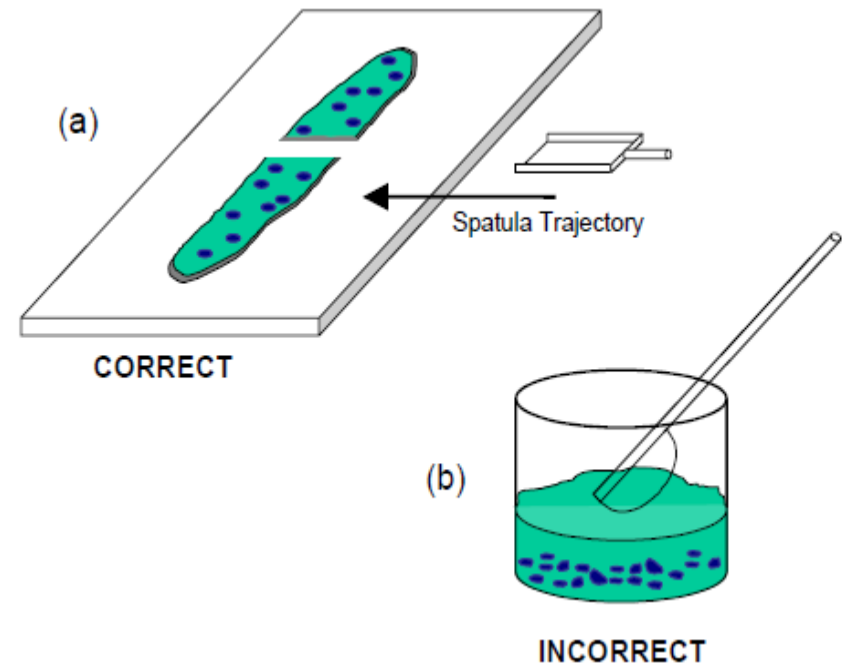
Subsampling

Subsampling of **liquids** (containing solids and/or in multiple phases)

- **Mixing the sample** such that all phases are homogenized, and then taking a subsample
- Allowing all of the **phases to separate** and **analysis of each phase separately**.

Subsampling

- Empty the sample from the sample container onto a smooth and clean surface or appropriate material.
- Do not try to homogenize the sample, as this may promote segregation of particles.
- Reduce the sample by using the fractional shoveling technique until a sample 5 to 10 times larger than the analytical sample is obtained.
- Shape the remaining material into an elongated pile with uniform width and thickness.
- Take increments all across the pile through the entire thickness.
- Reshape the pile perpendicular to its long axis, and continue to take increments across the pile until the appropriate sample weight is reached.



Correct (a) and incorrect (b) laboratory techniques for obtaining subsamples of granular solid media



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Sample dissolution

In which cases:

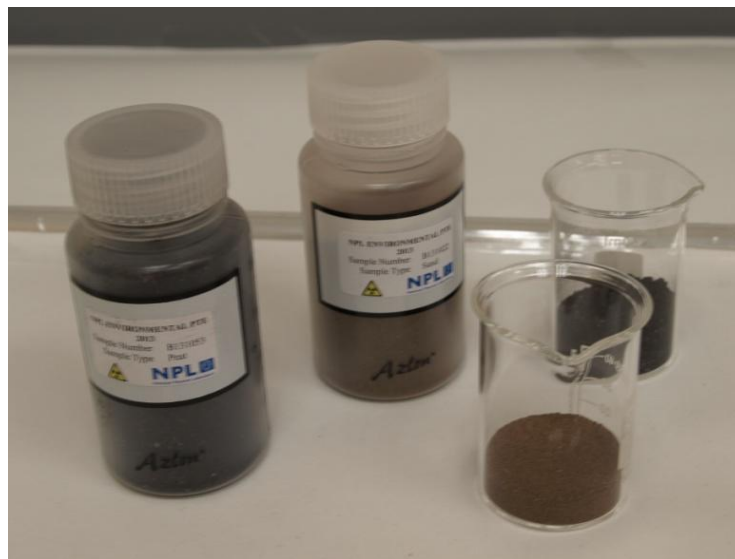
- destructive analysis;
- alpha, beta emitters, ICP-MS;
- adding tracers;

What kind of samples:

- Soil
- Sand
- Oxides
- Precipitates
- Concrete etc.

How:

- Acid leaching
- Ashing
- Acid dissolution
- Fusion: fluoride, sulfate, boron fusion
- MW digestion



Chemistry of Dissolution

In order to dissolve a sample completely, each **insoluble component must be converted into a soluble form.**

Chemical **processes behind sample dissolution:**

- Chemical **Exchange**
- **Decomposition**
- **Rearrangement** Reactions
- **Oxidation-Reduction** Processes
- **Complex formation**

Chemical Exchange, Decomposition, and Simple Rearrangement Reactions

Chemical exchange, decomposition, and simple rearrangement reactions **to convert insoluble components to a soluble** chemical species



Sodium pyrosulfate fusion, for example, converts zirconia (ZrO_2) into zirconium sulfate [$\text{Zr}(\text{SO}_4)_2$], which is soluble by a simple rearrangement of oxygen atoms:



Oxidation-Reduction Processes

Many oxidizing agents used in sample dissolution **convert metals to a stable oxidation state**

Oxidation to a higher state is common when dissolving uranium samples in acids or during treatment with fusion fluxes. The uranyl ion (UO_2^{2+}) forms **soluble salts such as chloride, nitrate, and perchlorate** with anions of the common acids.



Perchloric acid offers a particular advantage because very soluble metal perchlorate salts are formed.

Complex formation

Complexation **increases solubility in the dissolution** mixture and helps to minimize hydrolysis of the cations.

Numerous **stable complexes** of anions from solubilizing acids (HCl, HF, HNO₃, H₂SO₄, HClO₄) **contribute to the dissolution** of other elements, such as americium, cobalt, technetium, thorium, uranium, and zirconium.

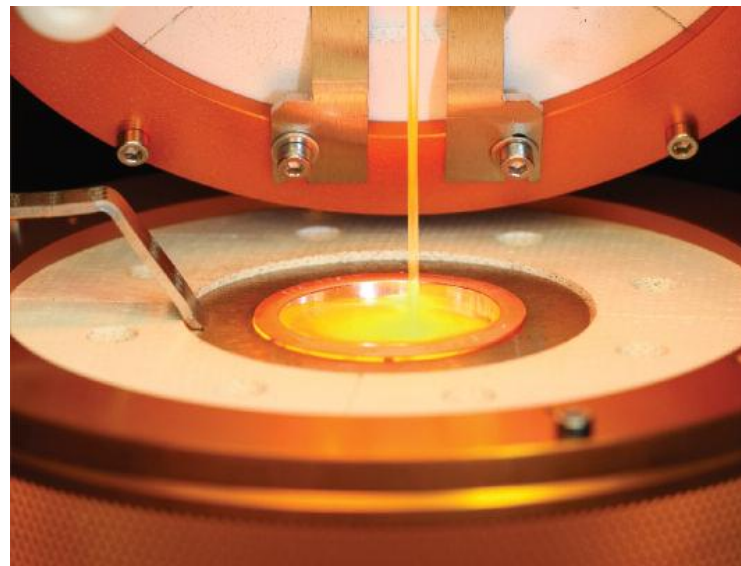


Fusion Techniques

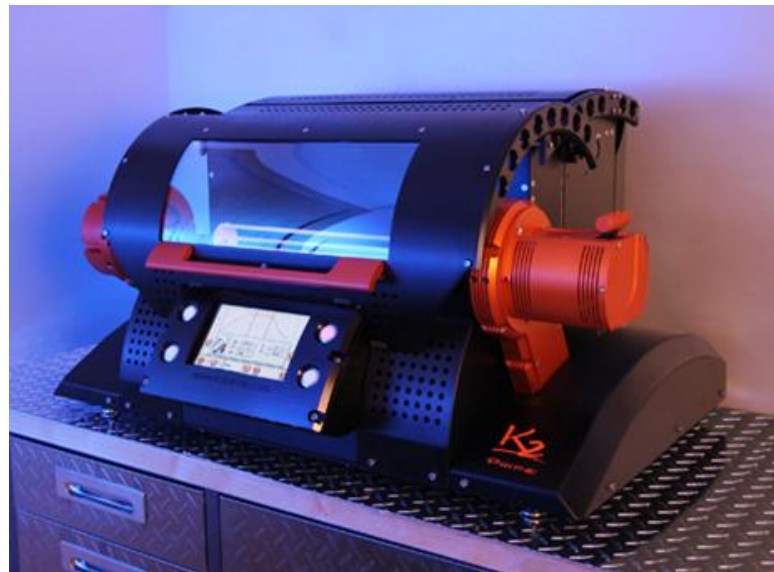
For **samples that are difficult to dissolve in acids** such as soils, sludges, silicates, and some metal oxides.

Fusion is accomplished by heating a salt (the flux) mixed with an appropriate amount of sample.

The mixture is heated to a temperature **above the melting point of the salt**, and the sample is allowed to react in the molten mixture.



Source: www.katanax.com





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Fusion Techniques

After the sample has returned to room temperature, the **fused material is dissolved**. The solvent is usually **warm water or a dilute acid solution**, depending on the salt.

The **fused sample is then dissolved**, and the analysis is continued. **Any residue** remaining may be treated by **repeating the fusion** with the same salt, performing a fusion with a different salt, **acid treatment**, or any combination of the three.



Source: www.katanax.com





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Fusion Techniques

Flux (mp, °C)	Fusion Temperature, °C	Type of Crucible	Types of Sample Decomposed
$\text{Na}_2\text{S}_2\text{O}_7$ (403°) or $\text{K}_2\text{S}_2\text{O}_7$ (419°)	Up to red heat	Pt, quartz, porcelain	For insoluble oxides and oxide-containing samples, particularly those of Al, Be, Ta, Ti, Zr, Pu, and the rare earths.
NaOH (321°) or KOH (404°)	450-600°	Ni, Ag, glassy carbon	For silicates, oxides, phosphates, and fluorides.
Na_2CO_3 (853) or K_2CO_3 (903)	900-1,000°	Ni Pt for short periods (use lid)	For silicates and silica-containing samples (clays, minerals, rocks, glasses), refractory oxides, quartz, and insoluble phosphates and sulfates.
Na_2O_2	600°	Ni; Ag, Au, Zr; Pt (<500 °C)	For sulfides; acid-insoluble alloys of Fe, Ni, Cr, Mo, W, and Li; Pt alloys; Cr, Sn, and Zn minerals.
H_3BO_3	250°	Pt	For analysis of sand, aluminum silicates, titanite, natural aluminum oxide (corundum), and enamels.
$\text{Na}_2\text{B}_4\text{O}_7$ (878°)	1,000-1,200°	Pt	For Al_2O_3 ; ZrO_2 and zirconium ores, minerals of the rare earths, Ti, Nb, and Ta, aluminum-containing materials; iron ores and slags.
$\text{Li}_2\text{B}_4\text{O}_7$ (920°) or LiBO_2 (845°)	1,000-1,100°	Pt, graphite	For almost anything except metals and sulfides. The tetraborate salt is especially good for basic oxides and some resistant silicates. The metaborate is better suited for dissolving acidic oxides such as silica and TiO_2 , and nearly all minerals.
NH_4HF_2 (125°) NaF (992°) KF (857°) or KHF_2 (239°)	900°	Pt	For the removal of silicon, the destruction of silicates and rare earth minerals, and the analysis of oxides of Nb, Ta, Ti, and Zr.

Fusion Techniques- crucibles

Several types of materials are used for fusion crucibles, **platinum** and **graphite** are most common.

Platinum crucibles:

- ✓ **High resistance** toward molten alkali metal, borates, fluorides, nitrates, and bisulfates.
- ✓ **Avoid *aqua regia***, sodium peroxide, sulphur dioxide
- ✓ Platinum crucibles can be **cleaned in boiling HNO_3** or by performing a blank fusion with sodium hydrogen sulfate.

Fusion Techniques- crucibles

Graphite crucibles

- ✓ **Cost-effective** alternative to metal crucibles
- ✓ **Disposable**, which eliminates the need for cleaning and the possibility of cross-sample contamination
- ✓ Chemically inert and heat-resistant, although they do **oxidize slowly at temperatures above 430°C**
- ✓ Unaffected by most of the usual acids, including hydrofluoric
- ✓ Dissolve in *aqua regia*

Boron Fusions

Recommended for **analysis of:**

- **Soil, sand, concrete**
- **Silicates**
- **Metal oxides**
- **Iron and rare earth ores**
- **Zr, Ti, Nb, Ta**

Relatively **large amounts of flux are required** for these types of fusions.

Platinum crucibles should be used

Fluxes:

- **Boric acid (H_3BO_3)**
- **Sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7$)**
- **Lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$)**
- **Lithium metaborate (LiBO_2)**

Boron Fusions

LITHIUM TETRABORATE ($\text{Li}_2\text{B}_4\text{O}_7$)

- Dissolves **oxides and silicates**.
- **Platinum dishes** are normally used.
- The fusion melt typically is **dissolved in dilute acid**, usually nitric.
- When **easily hydrolyzed metal** ions are present, dissolution should be carried out in the **presence of ethylenediamine tetracetic acid (EDTA)** or in acid.

LITHIUM METABORATE (LiBO_2)

A mixture of the meta- and tetraborates, is a **more basic flux**

dissolving **highly acidic oxides or very insoluble ones**, such as **silica (SiO_2)** or **TiO_2** .

Suitable for dissolving most metal oxides.

SODIUM CARBONATE FUSION

FUSION WITH SODIUM CARBONATE (Na_2CO_3)

- **Decomposing silicates** (clays, rocks, mineral, slags, glasses, etc.), refractory **oxides** (magnesia, alumina, beryllia, zirconia, quartz, etc.), and **insoluble phosphates and sulfates**.
- This is the **method of choice when silica in a silicate is to be determined**, because the fusion converts an insoluble silicate into a mixture that is easily decomposed by hydrochloric acid:



Acid Leaching

- Use of **HCl** or **HNO₃** to transfer RN **into solution**.
- The **acid concentration** may vary **up to** and include **concentrated acid**.
- **HF** and **aqua regia** are **not included** in this definition.
- **Sample size** is usually relatively **much larger** than that used for fusion.
- Although mineral acids **might not totally break down all matrices**, they have been shown to be **effective leaching solvents** for metals, oxides, and salts in some samples.
- **Excess reagents can be removed through evaporation**.
- **Complete dissolution of a sample is not necessary** if it can be demonstrated confidently that the **radionuclide of interest is completely leached** from the sample medium.

Wet Ashing and Acid Dissolution

Wet ashing and acid dissolution describe **sample decomposition using hot, concentrated acid solutions.**

Different acids are used **alone or in combination** to decompose specific compounds that may be present in the sample.

Acid	Typical Uses
Hydrofluoric Acid, HF	Removal of silicon and destruction of silicates; dissolves oxides of Nb, Ta, Ti, and Zr, and Nb, and Ta ores.
Hydrochloric Acid, HCl	Dissolves many carbonates, oxides, hydroxides, phosphates, borates, and sulfides; dissolves cement.
Hydrobromic Acid, HBr	Distillation of bromides (e.g., As, Sb, Sn, Se).
Hydroiodic Acid, HI	Effective reducing agent; dissolves Sn^{+4} oxide and Hg^{+2} sulfide.
Sulfuric Acid, H_2SO_4	Dissolves oxides, hydroxides, carbonates, and various sulfide ores; hot concentrated acid will oxidize most organic compounds.
Phosphoric Acid, H_3PO_4	Dissolves Al_2O_3 , chrome ores, iron oxide ores, and slag.
Nitric Acid, HNO_3	Oxidizes many metals and alloys to soluble nitrates; organic material oxidized slowly.
Perchloric Acid, HClO_4	Extremely strong oxidizer; reacts violently or explosively to oxidize organic compounds; attacks nearly all metals.

Dissolves silica and silicates



whereby the fluorosilicic acid formed dissociates into gaseous silicon tetrafluoride and hydrogen fluoride upon heating:



- HF **prevents hydrolysis** of radionuclides (Ti, Zr, Hf, Nb, Ta, Pa..)
- HF should **never be used or stored in glass**, or porcelain containers
- Digestion **in Teflon containers** as long as the temperature does not exceed 250 C

HCl

HCl forms strong complexes with Au^{3+} , Ti^{3+} , and Hg^{2+} . The concentrated acid will also complex Fe^{3+} , Ga^{3+} , In^{3+} , and Sn^{4+} . Most chloride compounds are readily soluble in water except for AgCl , PbCl_2 , HgCl_2 , TiCl_4

While HCl dissolves many carbonates, oxides, hydroxides, phosphates, borates, sulfides, and cement, it **does not dissolve**:

- **Silicates**
- Transition metal **oxides**
- **Sulfates** of Sr, Ba, Ra, or Pb;
- Group II **fluorides**;
- Ores of U, Th, Nb, Ta.



High boiling point (340 C).

Dissolves oxides, hydroxides, carbonates, and sulfide ores

H_2SO_4 is **not appropriate for Ca, Sr, Pb, Ba, Ra** because of the low solubility of the sulfates.

Glass, quartz, platinum, and porcelain are resistant to H_2SO_4

Teflon should not be used above 250 C, and, therefore, it is not recommended for applications involving concentrated H_2SO_4 that require elevated temperature.

HNO₃

Widely used **oxidizing acids for sample decomposition.**

Most **metals and alloys are oxidized to nitrates**, which are usually **very soluble**

Nitric acid **does not attack Hf, Ta, Zr, Au, and Pt-group**

Al, Cr, Ga, In, Nb, Th, Ti, Ca, Mg, and Fe form a layer of insoluble oxide when treated with c.HNO₃, thereby **passivating the metal surface.**

Complexing agents (e.g., Cl⁻, F⁻, citrate, oxalate) can assist HNO₃ in dissolving most metals.

Although nitric acid is a good oxidizing agent, it usually boils away before sample oxidation is complete. Oxidation of organic materials proceeds slowly and is usually accomplished by repeatedly heating the solution to HNO₃ fumes.

HClO₄

A **powerful oxidizer**. Hot concentrated HClO₄ will attack nearly all metals (except **Au and Pt-group** metals) and oxidize them to the highest oxidation state.

Excellent solvent for **stainless steel and Cr**

Because of the violence of the oxidation reactions, HClO₄ is rarely used alone for the destruction of organic materials.

The concentrated acid is a **dangerous oxidant that can explode violently**.

Perchloric acid vapor should **never be allowed to contact organic materials** such as rubber stoppers. The acid should be **stored only in glass bottles**.

Aqua regia

One part concentrated HNO_3 and **three parts** concentrated HCl (by volume) are combined to form aqua regia:



The solution is **more effective if allowed to stand for 10 to 20 minutes** after it is prepared.

Dissolves sulfides, phosphates, and many metals and alloys including gold, platinum, and palladium.

Aqua regia volatilizes osmium as the tetroxide; has little effect on rhodium, iridium, and ruthenium; and **has no effect on Ti.**

Oxidic **uranium compounds** and synthetic mixed oxides (U_3O_8) are **dissolved in aqua regia**, with oxidation of the U^{4+} to UO_2^{2+} ions.

Microwave Digestion

- **Higher reaction rates** when using microwave radiation.
- **Takes minutes**
- **Faster, cleaner, more reproducible**, and more accurate than traditional hot-plate digestion
- High temperatures and pressures.
- **Dissolve most materials.**
- The three main approaches to microwave dissolution are:

-focused open-vessel

-low-pressure closed-vessel

-high-pressure closed-vessel



References

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