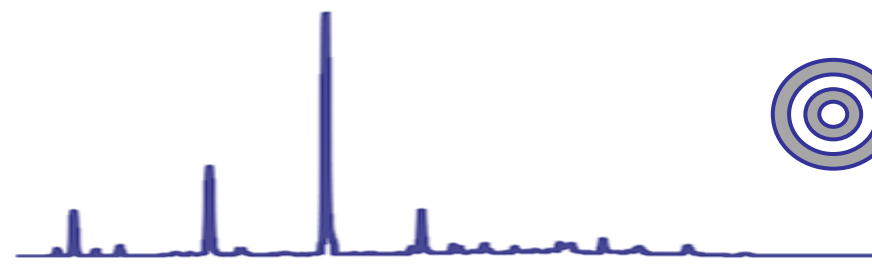
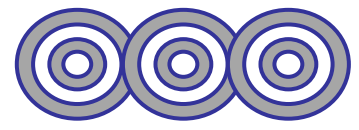


Environmental Analytical Workflow



Ahmad Aqel Ifseisi

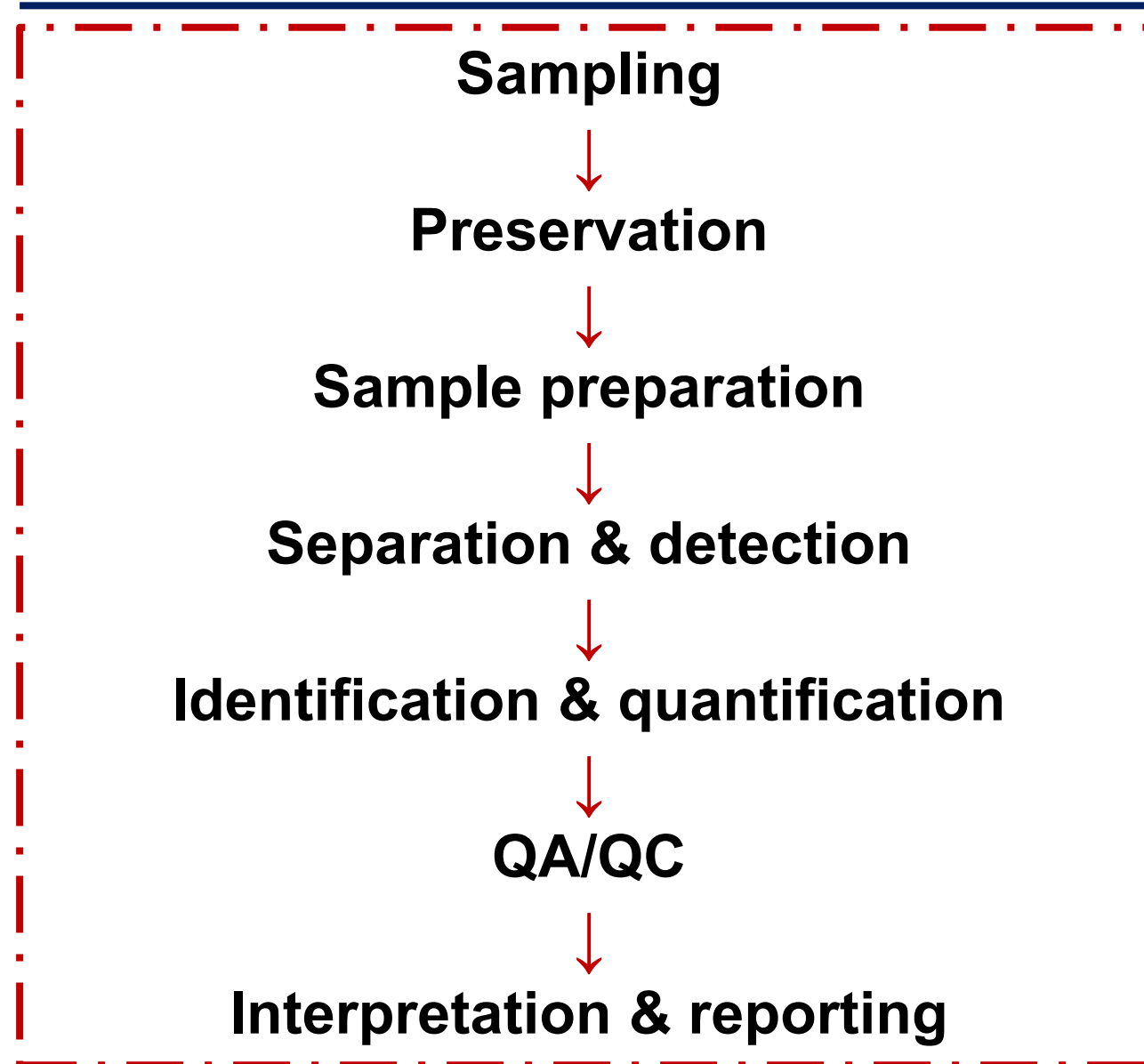
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The Environmental Analytical Workflow



Environmental Sampling

Sampling obtains a portion of the environment that represents the system under investigation.

A representative sample is essential because analytical accuracy cannot compensate for poor sampling.

Sampling design considers location, depth, frequency, season, sample number, variability, and study objectives.

Common approaches include grab, composite, passive, and continuous or automated sampling.



Sample Preservation and Handling

Environmental samples can change through reactions, volatilization, adsorption, precipitation, or biological activity.

Preservation may involve cooling, filtration, pH adjustment, chemical preservation, protection from light, or specialized containers.

Holding times and storage conditions should be controlled.

Documentation and chain-of-custody procedures help maintain sample identity and data integrity.



Environmental Sample Preparation

Sample preparation converts a complex environmental sample into a form suitable for measurement.

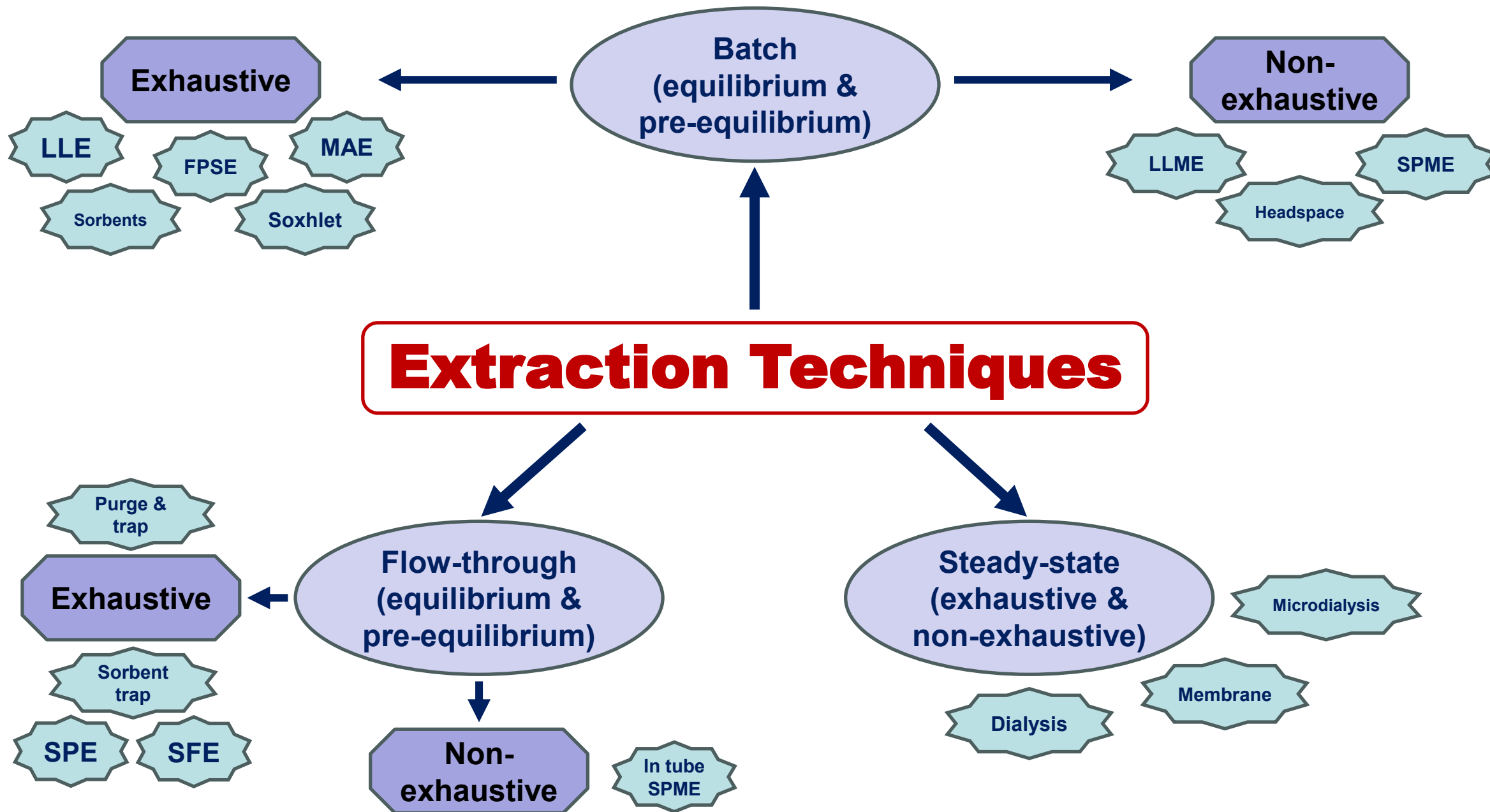
Common operations includes filtration, dilution, digestion, extraction, concentration, cleanup, and derivatization.

Examples: liquid-liquid extraction (LLE), solid-phase extraction (SPE), and solid-phase microextraction (SPME).

Sample preparation can improve selectivity and sensitivity but may introduce contamination, analyte loss, or matrix effects.

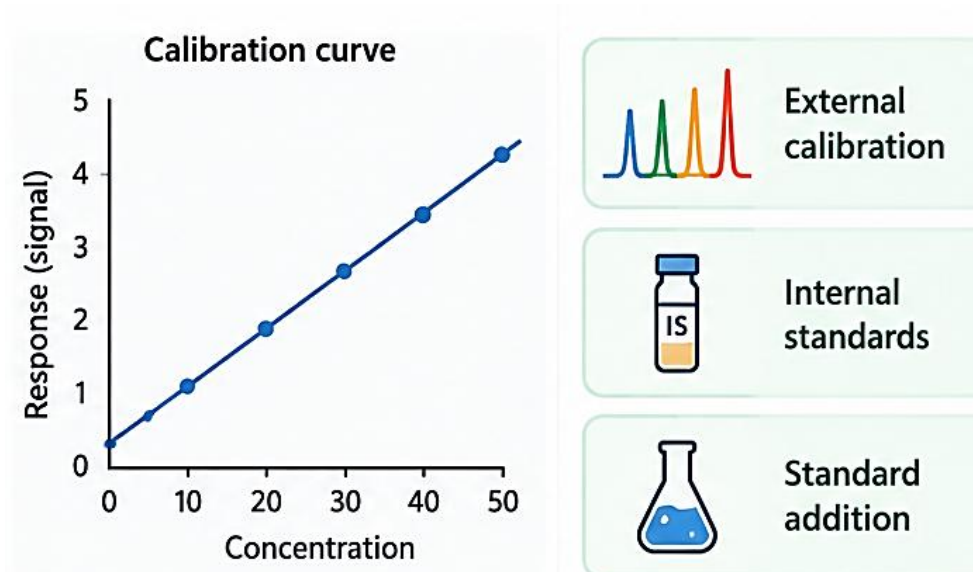
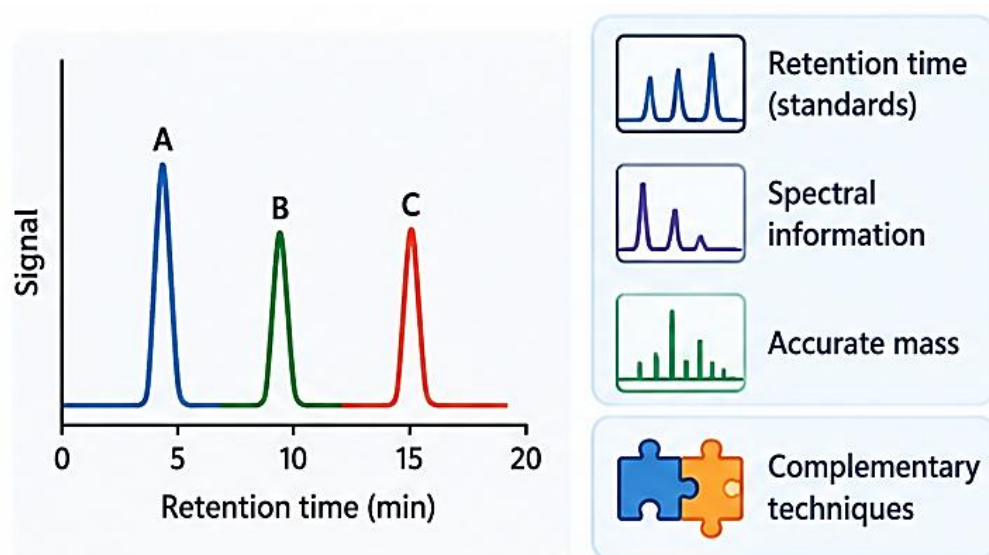


From a complex environmental sample → to a clean, concentrated, and prepared sample → ready for accurate and reliable analysis



Qualitative and Quantitative Analysis

Qualitative analysis	Quantitative analysis
Determines what substances are present	Determines how much analyte is present
Identification may use standards, retention time, spectral information, accurate mass, or complementary techniques.	Quantification commonly uses external calibration, internal standards, or standard addition.



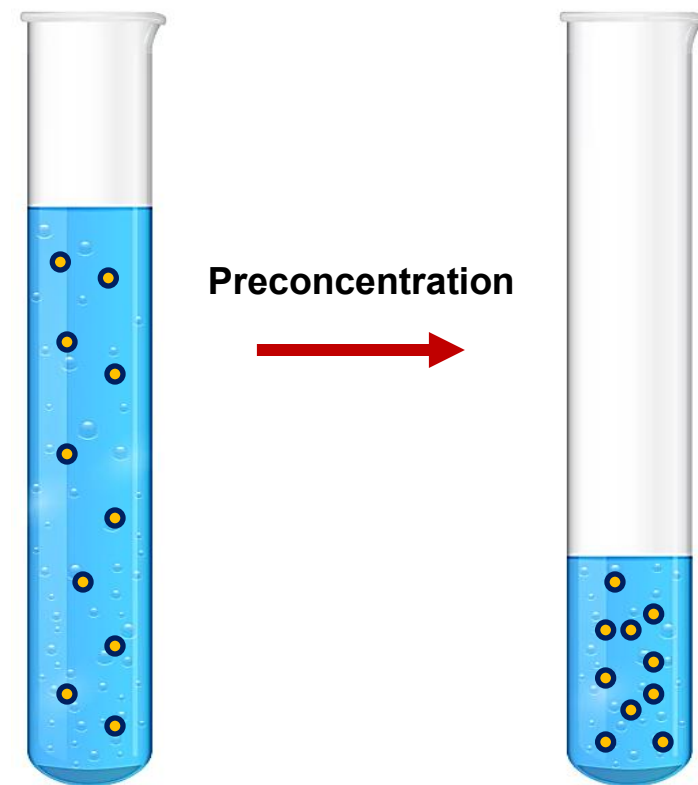
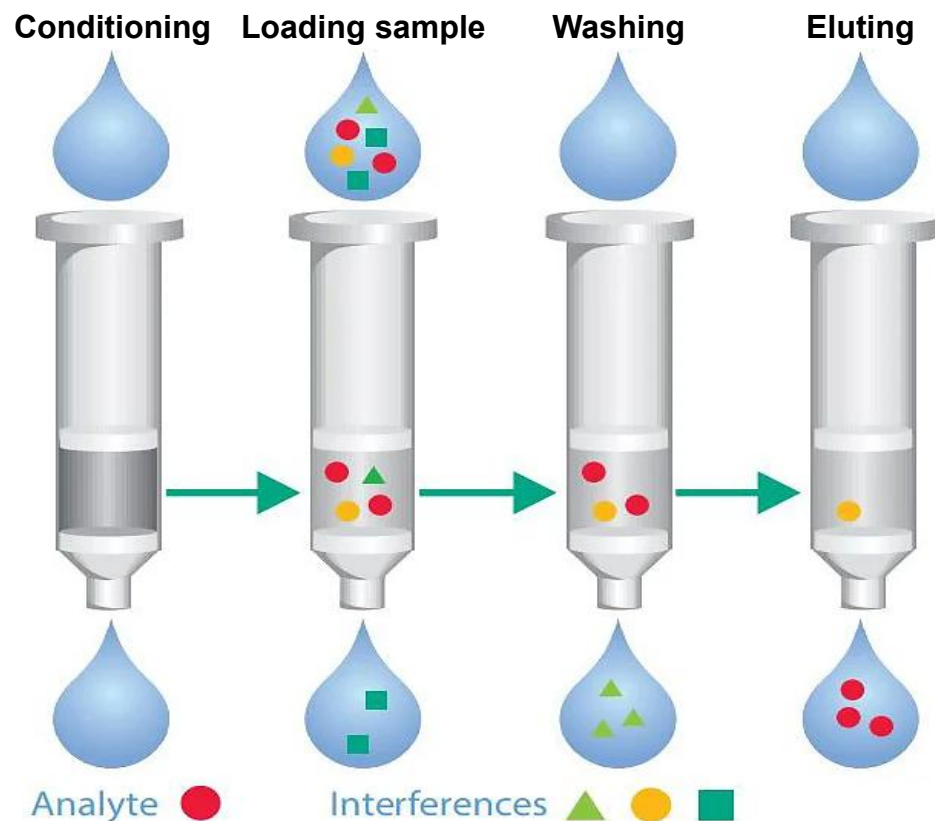
Trace and Ultra-Trace Environmental Analysis

Environmental contaminants often occur at very low concentrations.

Trace analysis may involve $\mu\text{g/L}$, $\mu\text{g/kg}$, or similar levels.

Ultra-trace analysis may require ng/L , ng/kg , or lower detection capability.

High sensitivity, selectivity, preconcentration, contamination control, efficient preparation, and rigorous QA/QC are essential.



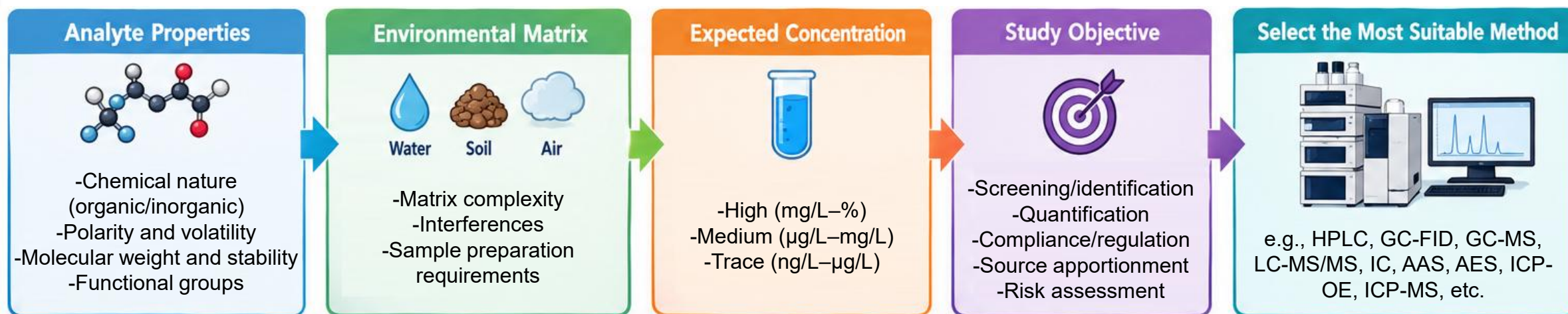
Selecting an Analytical Method

The selected method should match the analyte chemical and physical properties, environmental matrix, expected concentration, and study objective.

Important factors include selectivity, sensitivity, accuracy, precision, detection capability, throughput, cost, and available instrumentation.

Matrix complexity may determine the need for separation, extraction, digestion, or cleanup.

Standardized or regulatory methods may be required for compliance monitoring.



The best method is the one that provides reliable, accurate, and cost-effective results while meeting the study objectives and considering the environmental matrix and analyte characteristics.

Major Analytical Methods

Analytical methods can be separated into **classical** and **instrumental** methods of analysis



Major Analytical Techniques

Spectroscopy: UV-Vis, FTIR, Raman.

Atomic spectroscopy: AAS, AES, ICP-OES, ICP-MS.

X-ray methods: XRF and related elemental techniques.

Chromatography: TLC, GC, HPLC, IC, SFC.

Mass spectrometry: GC-MS, LC-MS, high-resolution MS.

Electrochemical methods: ion-selective electrodes, voltammetry, potentiometry, sensors ... etc.

Thermal methods: TGA, DSC, DEA, DTA ... etc.

Optimization of the Analysis Conditions

Optimizing analytical conditions involves systematically adjusting experimental parameters to obtain the best possible analytical results in terms of accuracy, precision, sensitivity, selectivity, resolution, and analysis time.

Each analytical instrument and analytical process requires its own optimization strategy, because the parameters affecting performance differ depending on the technique, analyte, sample matrix, and analytical objective.



General optimization workflow

Define the analytical objective



Select critical parameters



Choose an optimization strategy



Conduct experiments



Evaluate responses



Identify optimal conditions



Validate performance

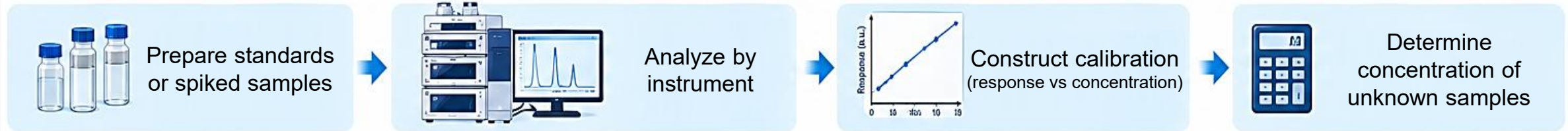
The goal is to achieve reliable, accurate, reproducible, and efficient analytical results.

Calibration and Quantification

Calibration establishes the relationship between analytical response and analyte concentration.

Calibration should cover the intended concentration range and use an appropriate mathematical model.

Key considerations include linearity, sensitivity, range, matrix effects, and calibration verification.

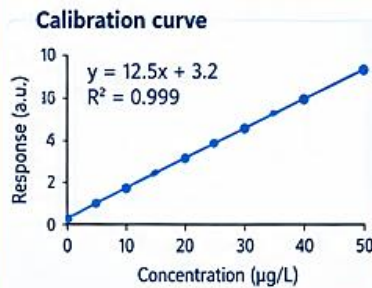
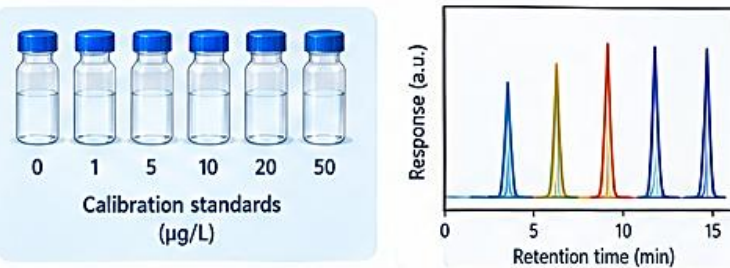


Calibration establishes the relationship between the analytical response produced by an instrument and the known concentration of an analyte. **Quantification** uses this relationship to determine the analyte concentration in an environmental sample.

Common approaches include **external calibration**, **internal standard calibration**, and **standard addition**.

1 External Calibration

Uses a series of standards prepared in the same matrix as the samples (without internal standard).

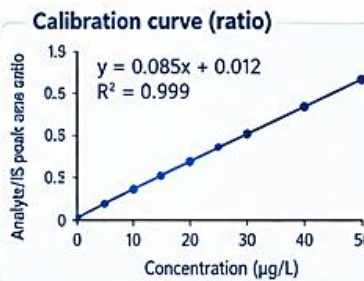
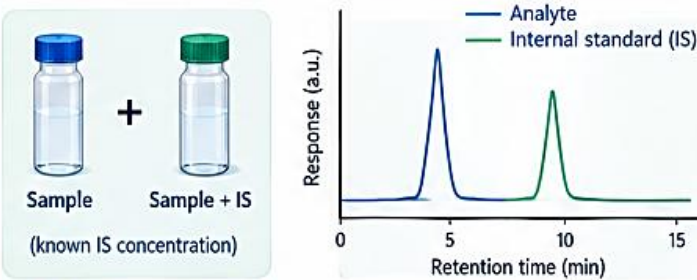


Use when:

- Matrix is clean or similar to standards.
- No internal standard is available.
- Simple and cost-effective.

2 Internal Standard Calibration

Uses a fixed amount of an internal standard (IS) added to all standards and samples.

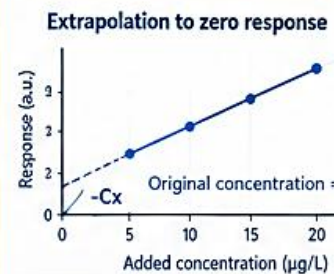
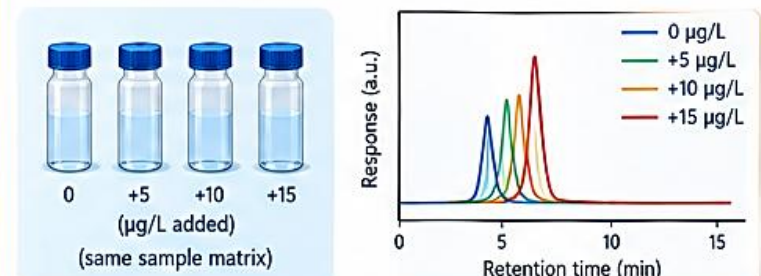


Use when:

- Matrix effects are a concern.
- Higher accuracy and precision are required.
- For trace-level analysis.

3 Standard Addition

Adds known amounts of analyte to the sample and extrapolates to find the original concentration.

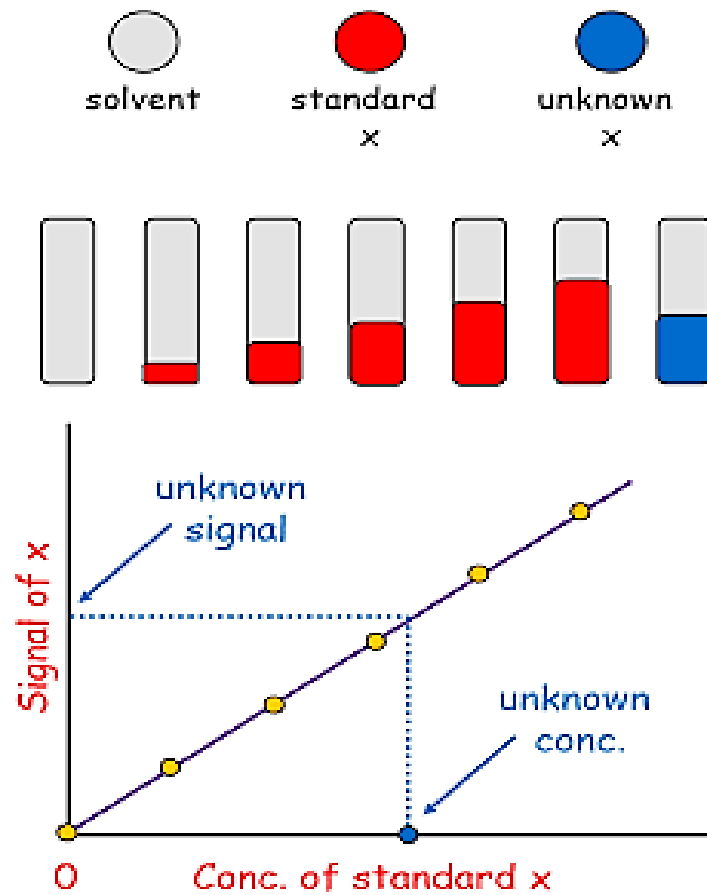


Use when:

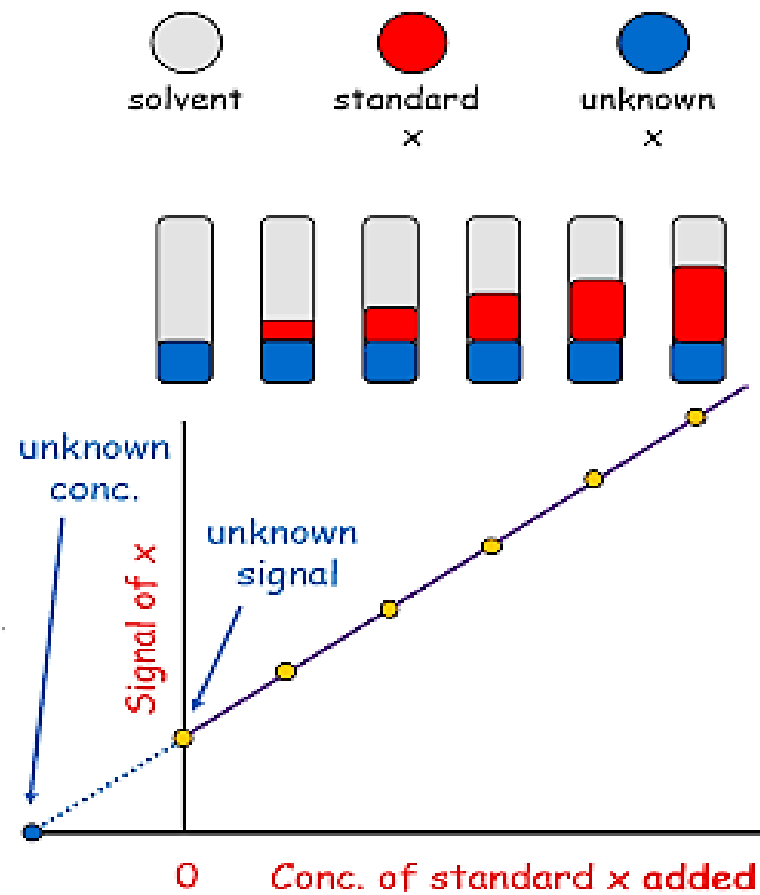
- Matrix is complex or unknown.
- Significant matrix effects are expected.
- High accuracy is required for difficult samples.

Common calibration approaches

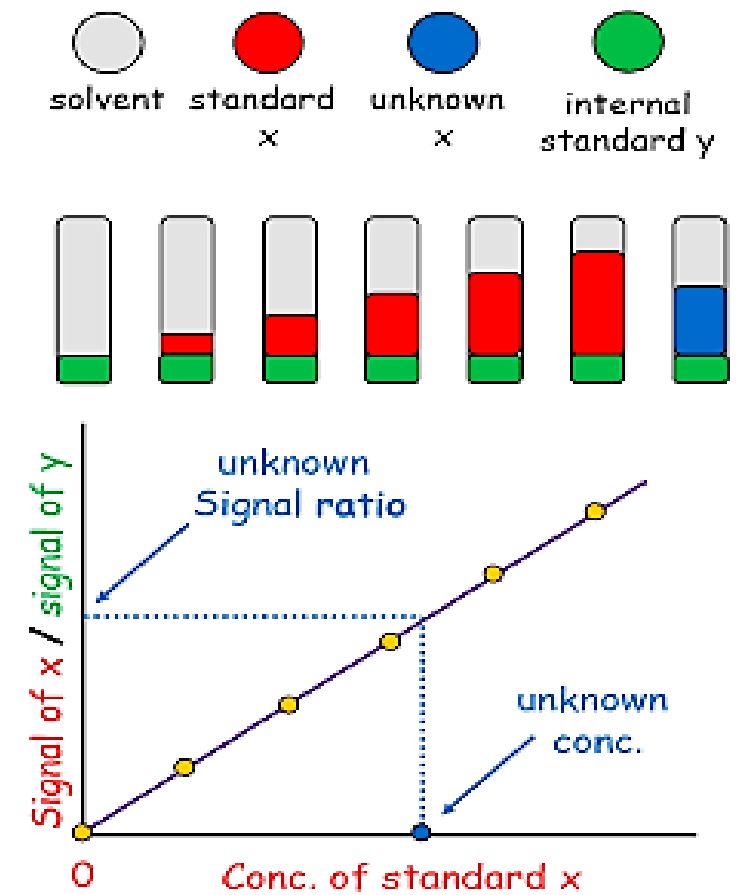
External Calibration



Standard Addition



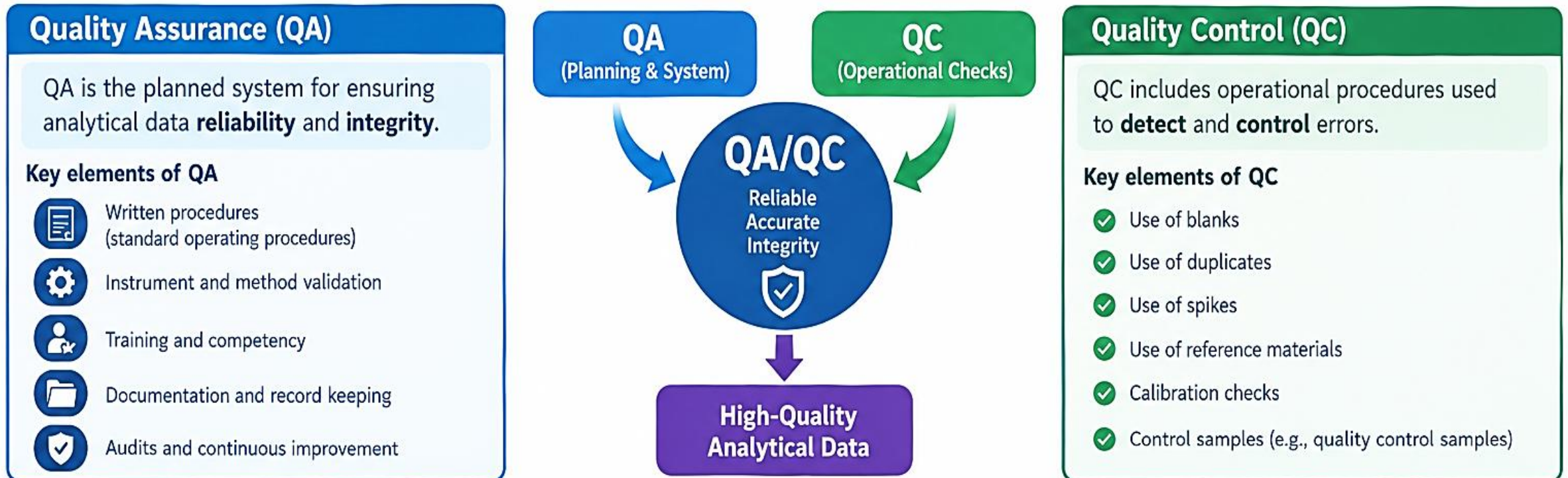
Internal Standard



Quality Assurance & Quality Control (QA/QC)

Quality Assurance (**QA**) is the planned system for ensuring analytical data reliability and integrity.

Quality Control (**QC**) includes operational procedures used to detect and control errors.



QA/QC helps identify contamination, drift, poor recovery, matrix effects, and other sources of error.

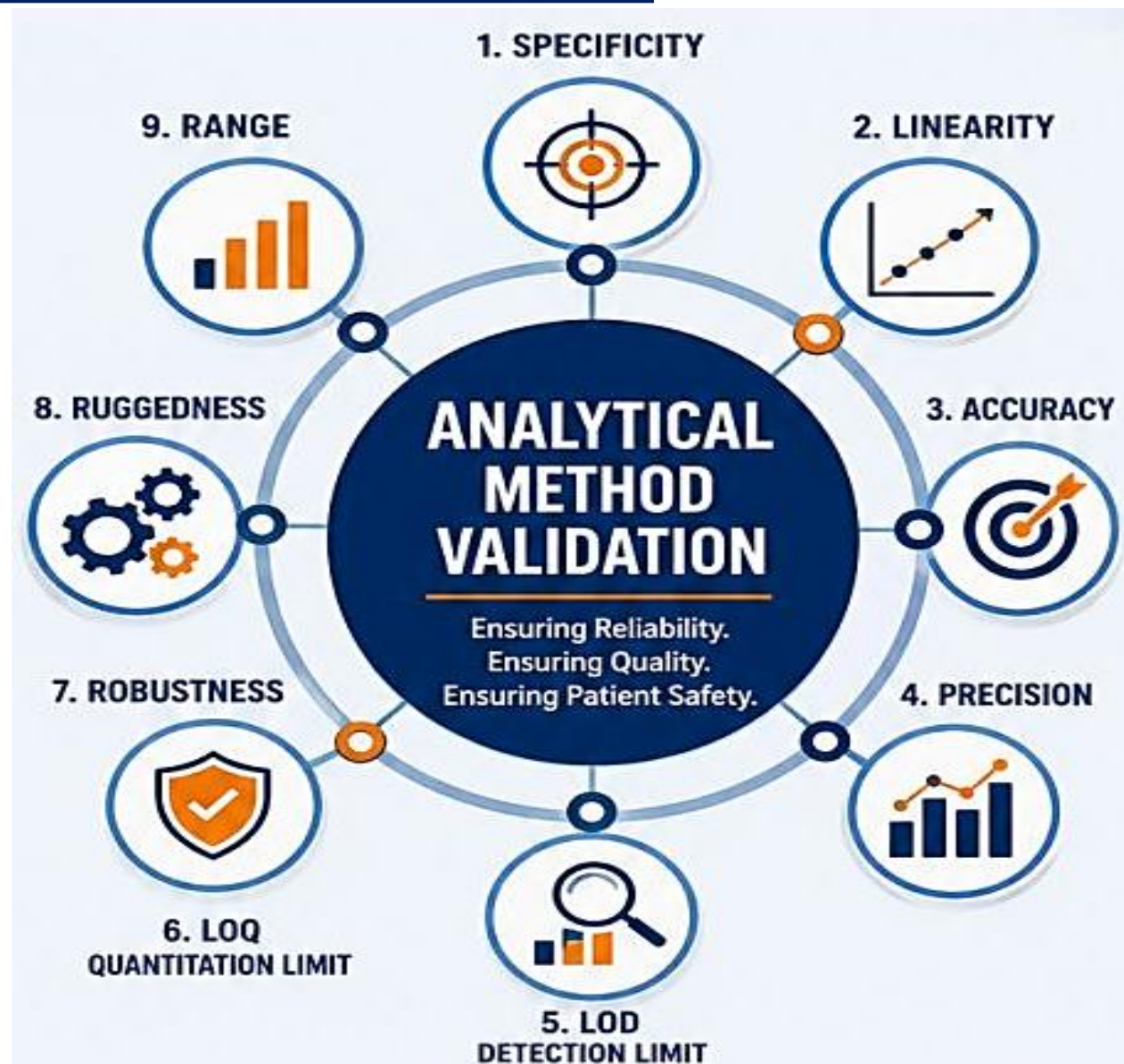
Analytical Method Validation

Validation demonstrates that an analytical procedure is fit for its intended purpose.

Common characteristics include accuracy, precision, selectivity, sensitivity, linearity, range, recovery, robustness, LOD, and LOQ.

Validation should consider the actual environmental matrix and expected concentration range.

Validated methods provide scientifically defensible and reproducible results.

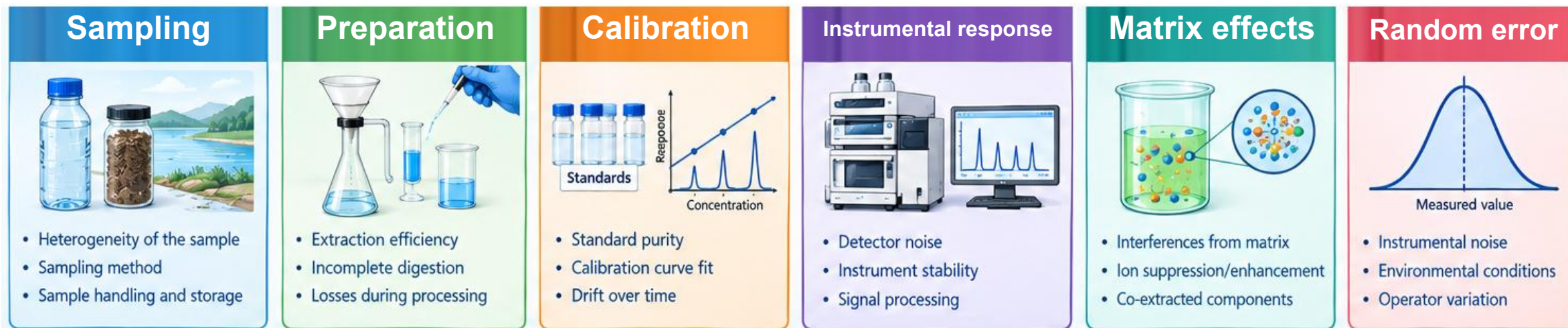


Measurement Uncertainty and Data Quality

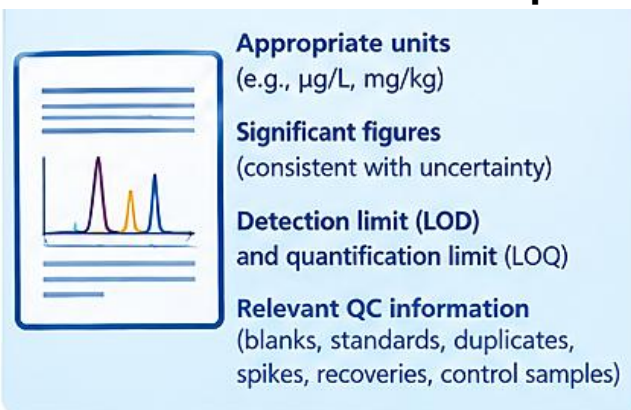
Every analytical measurement contains some degree of uncertainty.

Uncertainty reflects the doubt about the exact value of the result and is expressed as a range or confidence interval.

Sources of uncertainty



What to include in the report



Example of reporting

Analyte	Concentration ($\mu\text{g/L}$) $\pm$ Uncertainty
Benzene	2.34 ± 0.23
Toluene	5.67 ± 0.45
Ethylbenzene	1.28 ± 0.12

LOD = $0.05 \mu\text{g/L}$ LOQ = $0.15 \mu\text{g/L}$

QC: Method blank (ND), Spike recovery (92%),
RSD (3.2%), Calibration $R^2 = 0.999$

From Analytical Results to Environmental Information

Results should be interpreted with sampling design, QA/QC information, uncertainty, and environmental context.

Concentrations may be compared with applicable environmental standards or guideline values.

Spatial and temporal patterns can reveal pollution sources and environmental trends.



**Supports
Informed
Decisions**



**Risk
Assessment**



**Pollution
Control**



**Treatment
and Remediation**



**Regulatory
Decisions**

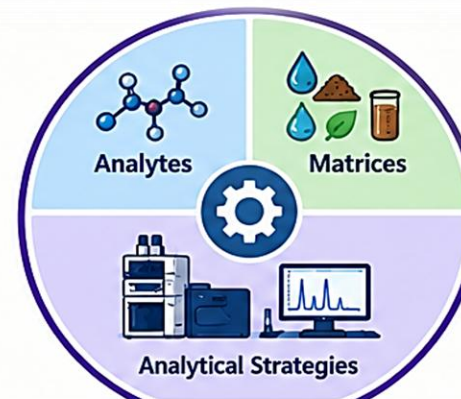
Key Concepts and Conclusions



Environmental analysis provides chemical evidence about environmental quality.



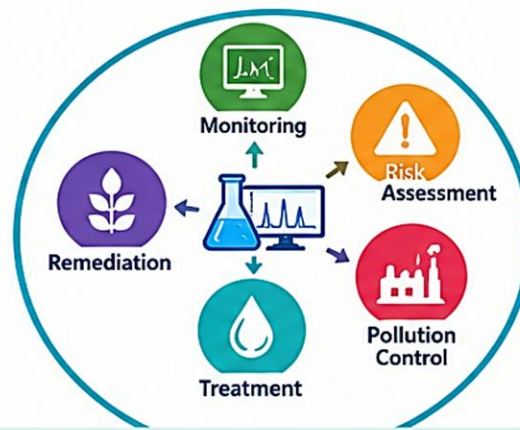
Sampling and sample preparation are as important as instrumental measurement.



Different analytes and matrices require different analytical strategies.



Reliable results require calibration, QA/QC, validation, and appropriate interpretation.



Environmental analysis connects analytical chemistry with monitoring, risk assessment, pollution control, treatment, and remediation.

Thank You!

