TRAINING WORKSHOP "Training workshop & study tour for developing the capacity of prosecutors & investigators for the enforcement of water & environment legislations"
3-5 June 2013, Delft, The Netherland.

Presented by: Dr. Hosny Khordagui, Team Leader, SWIM-SM

Universally recognized & accredited methods of sampling, monitoring, measuring & reporting including the necessary precision, accuracy, reproducibility, sensitivity & detection limit of the analytical monitoring & measuring methods.

Monitoring Compliance with Water & Environment Regulations

- Monitoring compliance is the most important element of any enforcement program. Monitoring compliance by collecting & analyzing information on the compliance status of the regulated community is fundamental for the following reasons:

1. It detects & corrects noncompliance
2. It assesses the enforcement program progress
3. It provides evidence to support enforcement actions.

Evidences of Noncompliance

- Enforcement of these requirements will evidently necessitate the submission of **unchallenged & unquestionable** indictment evidences of violations & noncompliance to the court of law if deemed necessary.
- All aspects related to sampling & analyses procedures should be recorded, dated & signed by the person who might **testify** regarding personal participation in the action & personal knowledge of the presented facts.

What Are the Evidences of Noncompliance With Water & Environment Legislation?

1. Official inspection reports.
2. Recorded personal observations during official inspections appropriately dated & signed or initiated.
3. Video recording of the offences with time & date.
4. Dated photographs including remote sensing with clear landmarks.
5. Examination of self-monitoring reports.
6. Specific conversation with identified witnesses.
7. The collection of samples at a particular time in a particular day & similar information.

General Conditions For Accepting Evidence of Noncompliance

- Documentation of evidence must be accurate, authenticated by signature or initials, dated & complete.
- A universal rule is that hear-say is inadmissible (hear-say evidence that is based not on a witness' personal firsthand knowledge or direct involvement, but on matters told by others).

What Prosecutors Are Looking For?

- Traditionally, prosecutors & judges are fond of analysis & measurements. These are considered as “hard facts or evidences”, while oral descriptions of a damage to aquifers or public health condition are not accorded the same weight.
- In regular situation, an accredited monitoring system or laboratory will carry out the analyses or measurements.
- Quantitative values are then interpreted by regulating agency to show compliance or noncompliance with permits to define the need for additional sampling & analysis to confirm violations and/or impose sanctions.
- The court habitually attaches great importance to analyses being carried out as prescribed in the authorization.

What Is Needed?

- Regulating agencies in SWIM-SM countries need to develop monitoring and inspection systems that can furnish credible evidence for the purpose of legal enforcement of water & environmental regulations.
- SWIM-SM countries also need to develop methods & procedures to furnish credible evidence admissible in the court of law.

Methods For Monitoring Water & Environment

- ***Method*** – A body of procedures and techniques for performing an activity, systematically presented, in the order in which they are executed.
- ***Regulatory methods*** – Approved by the regulating authorities
- ***Ad.hoc methods (kits)*** – Not admissible in court
- ***Standard methods*** – Published by professional organizations (e.g. ASTM, SMEWW, USGS, AOAC, WHO, etc.)

ASTM = American Society for Testing Materials
SMEWW = Standard Methods for the Examination of Water & Wastewater
USGS = US Geological Survey
WHO = World Health Organization
EU = European Union
AOAC = Association of Official Analytical Chemists

Ad-hoc Methods (Kits)

- Field test kits are useful for educational monitoring & as a quick way to screen gross water quality problems but are not appropriate for studies designed to measure changes in water quality or to check if a water body is meeting water quality guidelines because they are not precise and/or accurate.

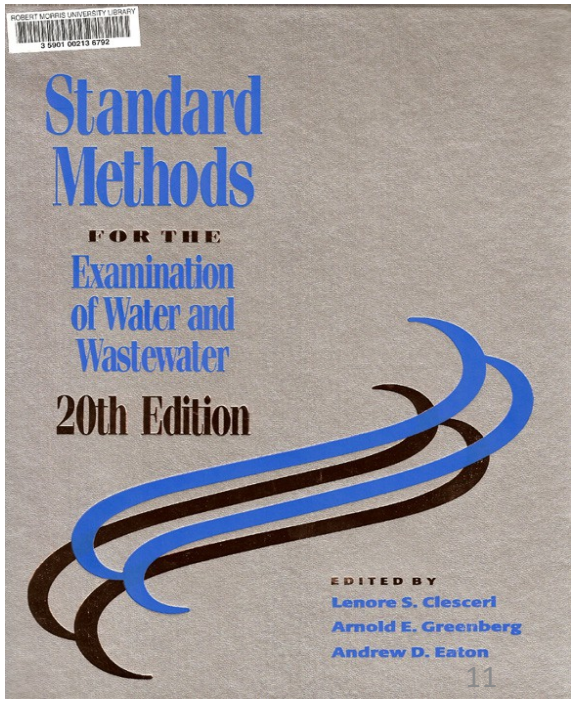
Standard Methods For The Examination of Water & Wastewater

APHA, WPCF & AWWA Methods

- First published 1905 (way before any regulating agency existed around the world)

'Mother' of all methods.

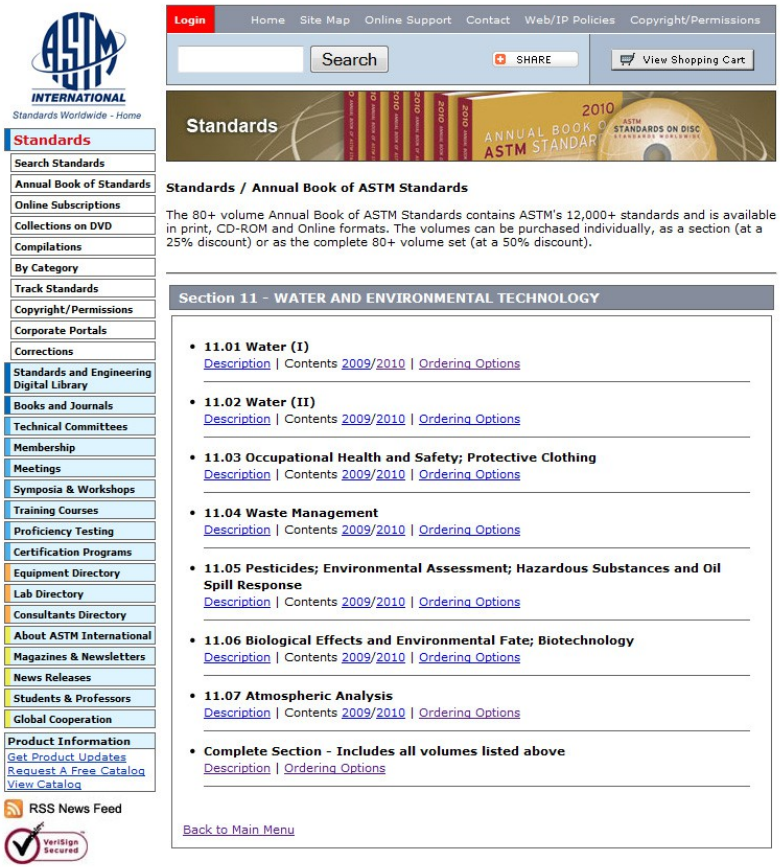
'Bible' of water & environment analysts



American Society For Testing Material

ASTM Methods

- 'Annual Book of ASTM Methoc
- Updated yearly
- 77 volumes,
- 12,000 standard methods



http://www.astm.org/BOOKSTORE/BOS/section11.htm

Specification of Standard Methods

- Recognized standard methods do specify the following:
  - Scope and application
  - Summary of method
  - Interferences
  - Safety
  - Apparatus and materials
  - Reagents
  - Calibration
  - Quality control
  - Sampling
  - Extraction
  - Instrumentation
  - Qualitative identification
  - Calculations
  - Method performance (MDLs)

Issues That Might Affect the Court Decision in Accepting the Evidences

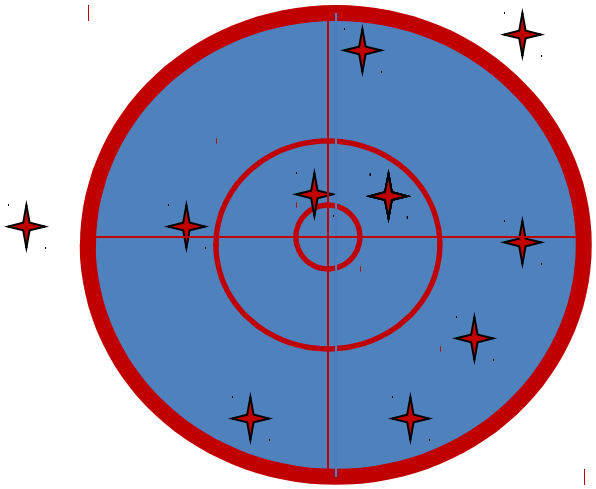
1. Precision & accuracy,
2. Reproducibility,
3. Sensitivity of the analytical methods,
4. Detection limit of the analytical methods.
5. Reliability including routine maintenance & operation of sampling gears and measuring instruments.
6. Adopted (QA) & (QC) programs.
7. Flawless chain of custody.
8. Qualifications, training and competence of inspectors, field & laboratory operators.

Precision

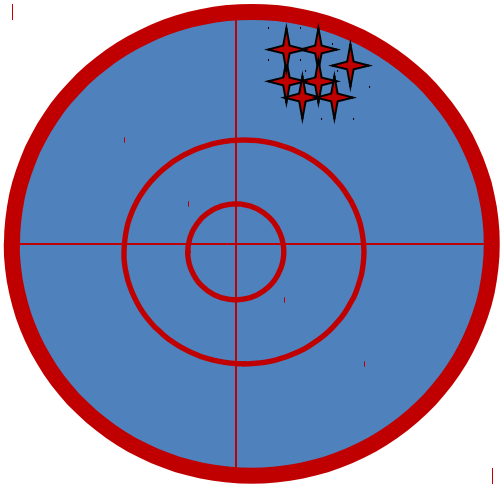
- Precision of an analytical procedure as the closeness of agreement (degree of scatter) between a series of measurements obtained from multiple sampling of the same homogeneous sample under the prescribed conditions.

Accuracy

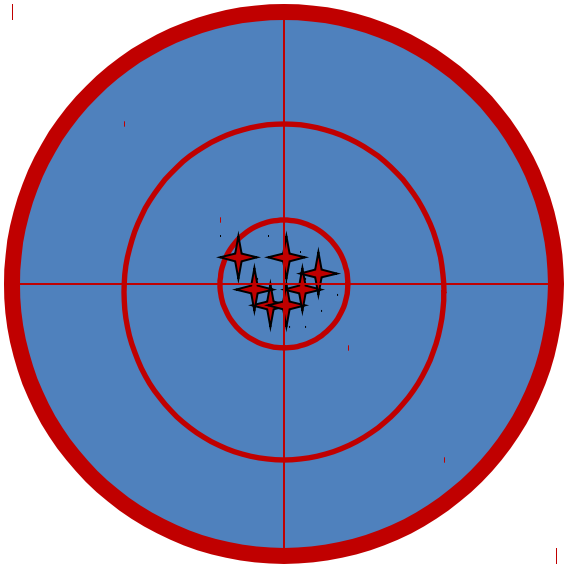
- Accuracy of an analytical procedure as the closeness of agreement between the true value and the value found.
- Accuracy can also be described as the extent to which test results generated by the method & the true value agree.



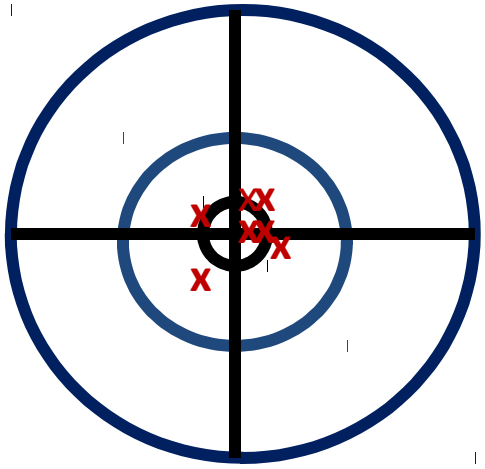
Neither accurate nor precise

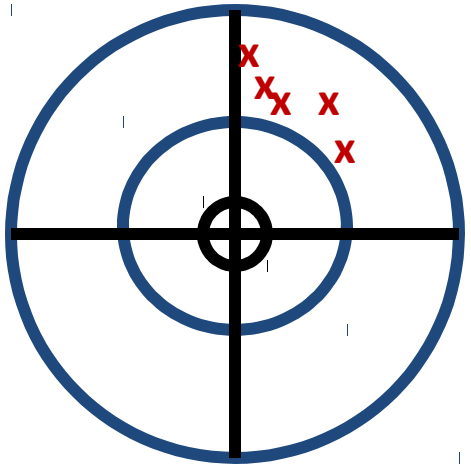


Precise but not accurate



Precise & accurate





High P

High A

No-low systematic error

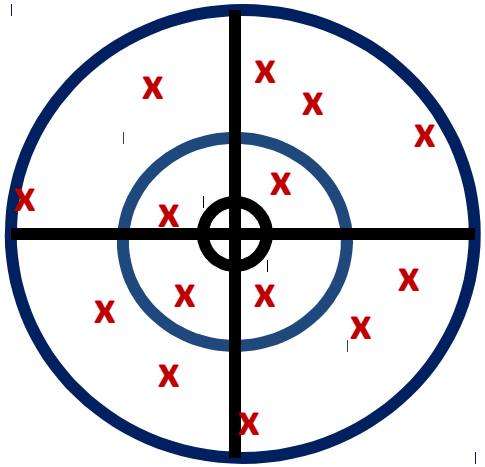
No-low random error

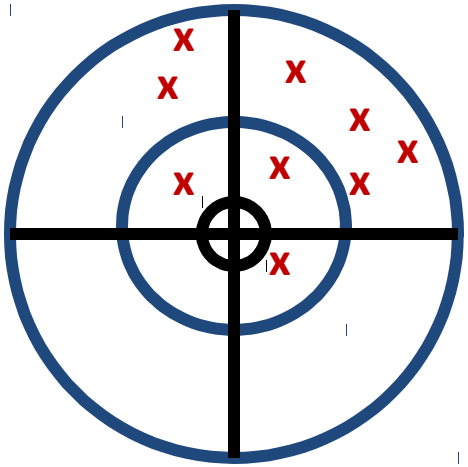
High P

Low A

High systematic error

low random error





Low P

Low A

Low systematic error

High random error

Low P

Low A

High systematic error

High random error

Reasons for Systematic & Random Error

- **Systematic Errors**

1. Bad calibration
2. Interfering substance
3. Overlooked blank
4. Malfunction of detector.
5. Defect in standard preparation
6. Error in calculation

•. **Random Errors**

1. Noise in instruments
2. Lack of experience
3. Different methods of analysis

Lower Limit of Detection (LOD)

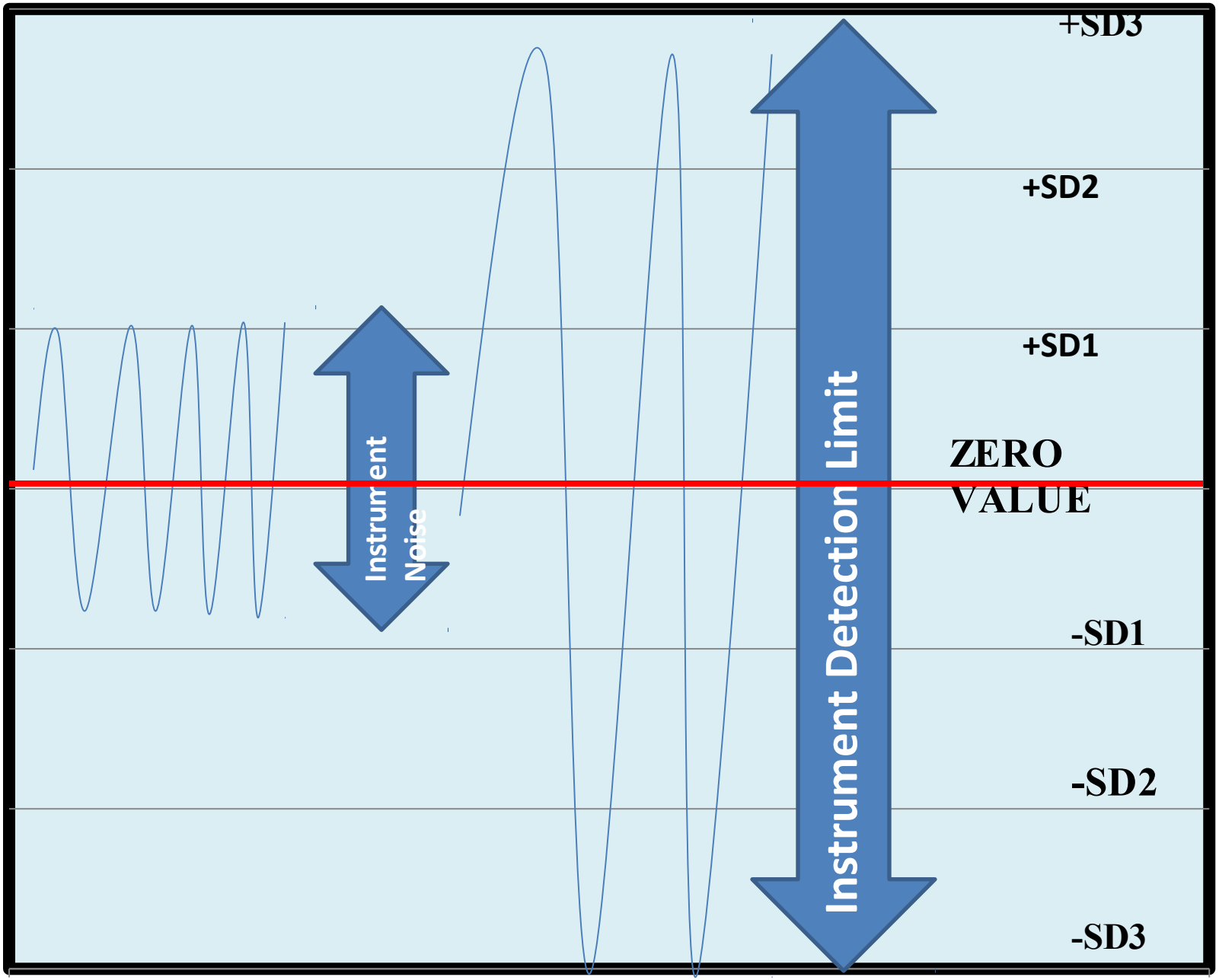
- the detection limit is the lowest quantity of a substance that can be distinguished from the absence of that substance (a blank value) within a stated confidence limit (generally 1%).

Commonly Known Detection Limits

1. **Instrument Detection Limit (IDL),**
2. **Method Detection Limit (MDL),** and the
3. **Limit of Quantification (LOQ).**

Instrument Detection Limit (IDL)

- Most analytical instruments produce a signal even when a blank (matrix without analyte) is analyzed. This signal is referred to as the noise level. The IDL is the analyte concentration that is required to produce a signal greater than three times the standard deviation of the noise level.


+SD3
+SD2
+SD1
ZERO VALUE
-SD1
-SD2
-SD3
Instrument Noise
Instrument Detection Limit

Method Detection Limit (MDL)

- Many times there is more to the analytical method than just submitting it to direct analysis. The sample may be diluted or concentrated prior to analysis on an instrument. Additional steps in an analysis add additional opportunities for error. Since detection limits are defined in terms of error, this will naturally increase the measured detection limit.
- Standard methods customarily provide you with the MDL.

Limit of Quantification (LOQ)

- Just because we can tell something from noise does not mean that we can necessarily know how much of the material there actually is. The LOQ is the limit at which we can reasonably tell the difference between two different values.

Example: Suppose you are at a noisy airport with your wife.

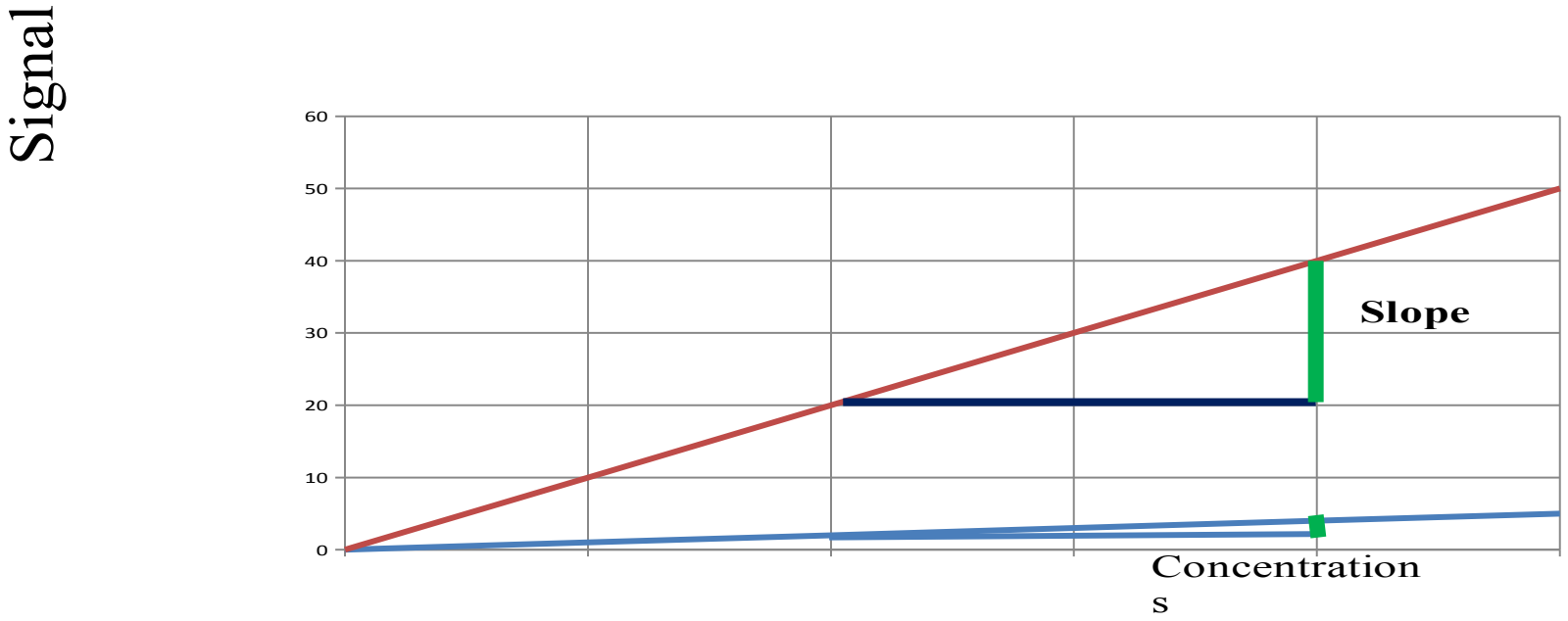
1. If she speaks softly, you will probably not hear her. Her voice is less <LOD.
2. If she speaks a bit louder, you may hear her but it is not possible to be certain of what she is saying. Her voice is >LOD but <LOQ.
3. If she speaks even louder, then you can understand her & take action. Her voice is then >LOD and >LOQ.
4. Likewise, her voice may stays at the same loudness, but the noise from jets may be reduced allowing her voice to become >LOD.

Why LOD is Important?

Assumed Pollutant	Reported Value in ppb	In Court
Mercury		X
Mercury	-	X
Mercury	zero	X
Mercury	Not Detected	X
Mercury	<MAL	X
Mercury	< than DL of 0.001 + Std. Method of analysis	Yes

Sensitivity

- Sensitivity is the slope of the calibration curve.



Sampling

- Sample collection is an important part of any compliance monitoring program. Without **proper** sample collection procedures, the results of such monitoring programs are neither useful nor valid, even with the most precise and accurate analytical measurements.

Evaluation of Sampling in Legal Investigations

- Any legal investigation will evaluate sampling procedures according to the following:

1. Sample collection techniques.
2. Field measurements.
3. Sample labeling (including location, date, time of the day, documentation, etc.)
4. Sample preservation & holding time
5. Transfer of custody & shipment of samples
6. QA/QC
7. Data handling & reporting.

Selection of Sampling Locations

- Normally, samples should be collected at the location specified in the permit issued by the regulating agency. In case not accessible, the inspector should determine the most representative sampling point available.
- that inspector must be familiar with the procedures & techniques necessary for accurate sampling of water & wastewaters.
- Two types of sample techniques are used: **grab** & **composite.**

Grab Sampling

- Grab samples are individual samples collected over a very short period of time and are representative of conditions at the time the sample is collected. The collection of a grab sample is appropriate when a sample is needed to:
  - Sample an effluent that does not discharge on a continuous basis
  - Provide information about instantaneous concentrations of pollutants at a specific time
  - Allow collection of a variable sample volume
  - Monitor parameters not amenable to compositing.

Composite Sampling

- Collect these samples over time, either by continuous sampling or by mixing discrete samples, and represent the average characteristics of the waste stream during the compositing period. Composite samples can be **proportional** & used when:

1. Average pollutant concentration during the compositing period is determined
2. Mass per unit time loadings is calculated
3. Wastewater characteristics are highly variable.

Preparation of Sampling containers might be a factor in dismissing your case in court of law

1. Wash with hot water and detergent.
2. Rinse with acid (e.g., nitric for metals).
3. Rinse with tap water, then rinse three or more times with organic-free water.
4. Rinse glass containers with an interference-free, redistilled solvent.
5. Dry in contaminant-free area.

Sample Identification Methods

- Identify each sample accurately & completely to eliminate any doubts around the case. bar codes, labels or tags should be used to identify the samples that are moisture-resistant & able to withstand field conditions. The information for each sample should include the following:

1. Facility name/location
2. Sample site location
3. Sample number
4. Name of sample collector
5. Date and time of collection
6. Indication of grab or composite sample with appropriate time and volume information
7. Identification of parameter to be analyzed
8. Preservative used.

Sample Preservation & Holding Time

- Water & wastewater samples contain one or more unstable pollutants that require immediate (e.g., within 15 minutes) preservation and/or analysis. Provide appropriate chemical preservation before transferring samples to the laboratory.
- Analysis of samples within one day ensures against error from sample deterioration. Where possible, provide sample preservation during compositing, usually by refrigeration to 4°C (or icing).

Transfer of Custody & Shipment of Samples

- To ensure the validity of the permit compliance sampling data **in court**, written records must accurately trace the custody of each sample through all phases of the monitoring program.
- The primary objective of this chain-of-custody is to create an accurate written record that can be used to trace the possession & handling of the sample from the moment of its collection through its analysis and introduction **as evidence**.

Credibility of Handling & Shipping

1. Use sample seals to protect the sample's integrity from the time of collection to the time it is opened in the laboratory.
2. Seal the shipping container to detect any evidence of tampering.
3. Place samples on synthetic ice to maintain sample temperature at 4°C throughout shipment.
4. Accompany all sample shipments with the chain-of-custody record and other pertinent forms.
5. When transferring possession of samples, the transferee must sign & record the date & time on the chain-of-custody record.

REPORTING

- The format and content of a data report depends on the government regulating agency's reporting format.
- Data are checked & approved by the unit supervisor. The final report is signed by the laboratory manager & includes:
  - Sample ID used by the laboratory.
  - Sample matrix type, description & method number.
  - Chemical/physical/biological parameters analyzed.
  - Reported values & units of measurement.
  - Method detection limits of the pollutant.
  - Data for all reported parameters.
  - Results of QC samples.
  - Footnotes to explain specific data.

مع خالص شكري وامتناني

Thank you for your attention

Merci pour votre attention

For additional information please contact:
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