# Understanding Your QC

**Presentation to:**

**KWWOA**

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**Department for Environmental Protection**
**Energy & Environment Cabinet**

***To Protect and Enhance Kentucky's Environment***

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# Outline

- Define QA/QC
- Define types of QC sample analysis
- Acceptable laboratory QC practices


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# Definitions

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# Definitions

- **<u>Quality Assurance</u>**: An integrated system of management activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.


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# Definitions

- **Quality Control**: The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of the users; operational techniques and activities that are used to fulfill requirements for quality.

# Definitions

- **Contamination**: The act of contaminating, or of making something impure or unsuitable by contact with something unclean, bad, etc.

# Definitions

- **<u>Accuracy</u>**: A measure of the closeness of an individual measurement or the average of a number of measurements to the true value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations. Refer to *Standard Methods, Data Quality Section for a more detailed explanation.*


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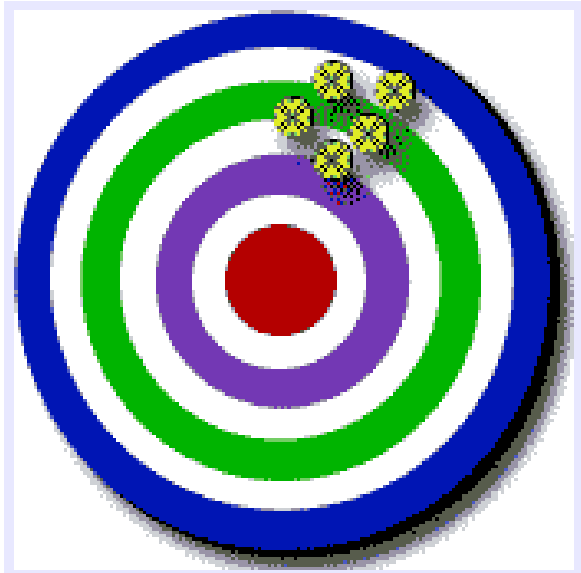
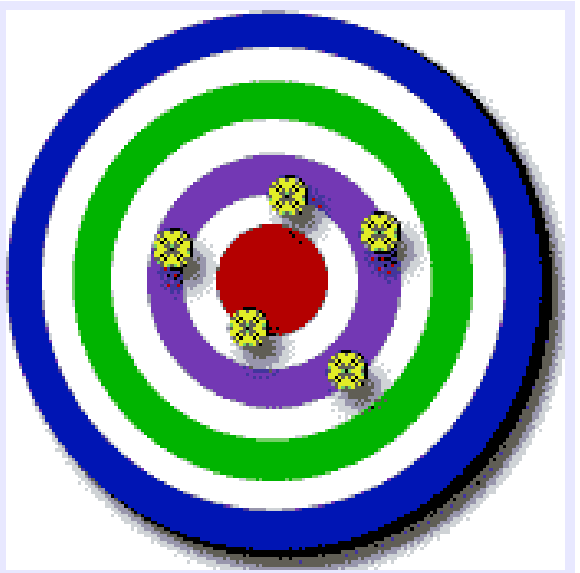
# Definitions

- **Precision**: The closeness of repeated measurements on the same parameter within a sample.

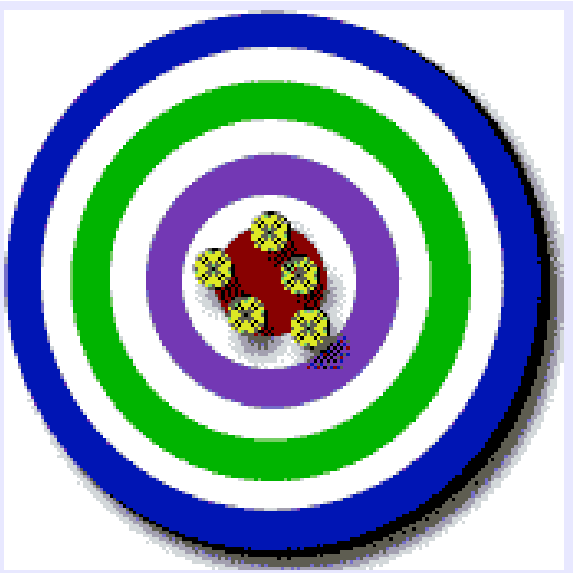
# Precision vs. Accuracy



Low Accuracy
**High Precision**



**High Accuracy**
Low Precision



**High Accuracy**
**High Precision**

# Types of QC Analysis

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# QC Samples

- QC before beginning analysis
  - Initial Demonstration of Capability (IDC)
  - Method Detection Limit (MDL)

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# QC Samples

- Blanks
  - Reagent Blanks/Method Blanks
  - Calibration Blank
  - Field Blanks
  - Equipment Blank
  - Trip Blanks
  - Instrument Blank
  - Temperature Blank

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# QC Samples

- Standards
  - Calibration Standards
  - Calibration Verification Standards
    - Initial (ICV)
    - Continuing (CCV)
  - Laboratory Control Spikes/Lab Fortified Blanks (LCS/LFB)
  - Quality Control Standard (QCS)
  - Proficiency Test (PT)

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# QC Samples

- Sample QC
  - Duplicates
  - Laboratory Fortified Matrix Spikes (LFMS)
  - Laboratory Fortified Matrix Spike Duplicates (LFMSD)
  - Field Spikes
  - Surrogate/Internal Standard

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# Acceptable Laboratory QC Practices

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# Initial QC

- IDC
  - 4 replicates
  - Mid range (can be LCS/QCS)
  - Analyzed by each analyst under the supervision of an experienced analyst
  - Demonstrates accuracy and precision

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# Initial QC

- MDL
  - 40 CFR 136, Appendix B
  - 7 replicates
  - Analyzed on 2-3 nonconsecutive days
  - Can be performed on multiple instruments and by multiple analysts
  - Calculated MDL < 10 x Spike Level  </=Regulatory Required Minimum Reporting Level
  - MDL calculation = (S)(t-value)
    - S = standard deviation
    - t-value for 7 replicates = 3.143

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# Initial QC

- Revised MDL Procedures
  - Proposed procedures in 2015 MUR
  - 7 spikes and 7 blanks
  - Analyzed in at least 3 different batches
  - It includes instructions for multiple instruments
  - Requires ongoing (quarterly) spikes
  - Recalculate every year (but not redo)
  - Includes matrix specific MDL instructions
  - Calculate standard deviation of spikes and blanks; choose whichever is higher as MDL

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# Reagent Blanks/Method Blanks

- Reagent Blanks/Method Blanks (LRB/MB)
  - It is an aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

# Calibration Blank

- Calibration Blank
  - A volume of reagent water acidified with the same acid matrix as in the calibration standards. The calibration blank is a zero standard and is used to auto-zero the AA instrument.

# Field Blanks

- Field Blanks (FRB)
  - An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.

# Equipment Blanks

- Equipment Blanks
  - A sample of analyte free water poured over or through decontaminated field sampling equipment prior to the collection of environmental samples. It is used to assess the adequacy of the decontamination process. It is also used to assess contamination from the total sampling, sample preparation and measurement process, when decontaminated sampling equipment is used to collect samples.

# Trip Blanks

- Trip Blanks (TB)
  - Analyte free water that is taken from the laboratory to the sampling site and transported back to the laboratory without having been exposed to sampling procedures. Typically, analyzed only for volatile compounds. It is used to assess contamination introduced during shipping and field handling procedures.
  - Typically analyzed only when an associated sample has a positive result.

# Instrument Blanks

- Instrument Blanks (IB)
  - A blank analyzed with field samples. It is used to assess the presence or absence of instrument contamination.

# Temperature Blanks

- Temperature Blanks
  - Not actually a blank but a VOA vial or other small sample bottle filled with distilled water that is placed in each cooler. Upon arrival at the laboratory, the temperature of this vial is measured. The temperature indicator or blank is not analyzed and does not measure introduced contamination, therefore, is not a blank. It is used  to evaluate if samples were adequately cooled during sample shipment

# Blank Acceptance Criteria

- Typical blank acceptance criteria is < ½ MRL
- Can be as high as < MRL
- Some methods specify < Calculated MDL

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# Failed Blank Corrective Actions

- Identify and eliminate the source of contamination
- Analysis of samples is halted until contamination is eliminated; if not, all results must be qualified because they did not meet the performance criteria of the test method
- Samples analyzed with an associated contaminated blank must be re-prepped and re-analyzed

# Calibration Standards

- Calibration Standards
  - Minimum of 3 (prefer 5) + blank (See method for exact requirements)
    - If using Quadratic, MUST be 5
  - Cover entire reporting range
  - 0.995 correlation coefficient
  - Do not force through 0
  - Should have positive y-intercept
  - Low standard must be at or below the reporting limit
  - Certain standards may be eliminated as long as the required minimum number is still met
    - If you drop from the low end, the MRL must be adjusted accordingly
    - If you drop from the high end, the retained highest standard defines the range for reporting results without having to dilute
    - Standards from the middle must not be dropped

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# Initial Calibration Verification (ICV)

- Initial (ICV)
  - Second source (preferably)
  - % difference = (actual ÷ theoretical) x 100
  - Acceptance criteria varies by method
  - Corrective action for failures include: instrument maintenance, re-preparation of standards or recalibration

# Continuing Calibration Verification (CCV)

- Continuing (CCV)
  - Same source as calibration standards
    - When internal standards are used to quantitate samples, a continuing calibration verification standard shall be performed:
      - At the beginning of each analytical run, unless a calibration curve has been generated prior to samples being analyzed on the same day
      - After each group of 10 or 20 samples or after a 12-hour period, whichever comes first

Continued…

# Continuing Calibration Verification (CCV) Continued

- Continuing (CCV)
  - Same source as calibration standards
    - When internal standards are not used to quantitate samples, a continuing calibration verification shall be performed:
      - At the beginning of each analytical run, unless a calibration curve has been generated prior to samples being analyzed on the same day
      - After each group of 10 or 20 samples or after a 12-hour period, whichever comes first
      - At the end of each analytical run

Continued…

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# Continuing Calibration Verification (CCV) Continued

- Acceptance criteria (unless otherwise specified by method)
  - Within 10% of the standard concentration for reportable inorganic analytes and metals
  - Within 15% of the standard concentration for reportable organic analytes
- If the CCV fails:
  - The laboratory may take corrective action and re-analyze
  - After 2 consecutive failed CCVs, the laboratory must recalibrate
  - Bracketed samples must be re-prepped and re-analyzed

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# Standards

- Control Spikes/Lab Fortified Blanks (LCS/LFB)
  - Typically second source from calibration standards
    - 1/20 samples
    - Acceptance criteria varies by method
  - Quality Control Standard (QCS)
    - Same as LCS/LFB but purchased or made at analyzed concentration
      - Cannot prove matrix interference with QCS analysis
- Reporting Limit Standard (RLS)
  - A procedural standard that is analyzed to evaluate instrument performance at or below the minimum reporting limit
    - Analyzed after each calibration or at least once per quarter

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# Proficiency Test

- Proficiency Test (PT)
  - Must pass annually
  - Must be analyzed exactly like samples
  - Must be submitted to state by the PT provider to be valid
  - PT should be in range of normal sample results
  - Separate studies for DW & KPDES
  - 2 consecutive analyte failures must be reported to the State

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# Duplicates

- Duplicates
  - May be sample or QC
    - A duplicate sample is a second portion (aliquot) of the same sample that is tested by using the same analytical procedures
    - 1/20 samples
    - ± 20% RPD
    - % RPD = $\frac{(|\text{difference}|)}{\text{average}} \times 100$

# Matrix Spikes

- Laboratory Fortified Matrix Spikes (LFMS)
  - Used to evaluate the performance of an analytical procedure when testing a specific sample (matrix) type
  - Made by adding a known amount (a spike) of analyte to a sample, testing the spiked sample, and determining if you have recovered the amount that you added

# Matrix Spikes

- Spiking Solution
  - A standard that is chosen for preparing a matrix spike
  - The concentration of the analyte in the spiking solution is usually 50 -100 times higher than the concentration found in the unspiked sample
    - This concentration is chosen to ensure that the spike does not significantly change the sample volume
    - This simplifies the calculations and avoids introducing unknown effects from dilution of the matrix
    - The spike should not increase the sample volume by more than 5%. Otherwise, it must be taken into account

# Matrix Spikes

- Matrix Spike concentration
  - For routine quality control purposes, it is recommended to prepare and analyze one matrix spike sample for each batch of 10 or 20 samples. In this case, the spike concentration should be chosen to fall in the middle of the analytical method range
  - It is important to remember that the concentration of the spiked sample should be within the method range(e.g. within the calibration curve). If necessary, dilute the spiked sample (after spiking) to bring the measurement within the calibration curve

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# Matrix Spikes

## Preparing a Matrix Spike

- Choose a spiking level
- Determine the concentration of the spiking solution to be used
- Determine the amount of spiking solution to be added to the sample as follows:

$$\text{Volume of Spiking Solution} = \frac{\text{spike concentration desired} * \text{volume of sample to which spike is added}}{\text{concentration of the spiking solution}}$$

- Add the calculated volume of spiking solution
- Analyze the spiked sample using the same analytical procedure as for the unspiked sample
- Calculate the percent recovery of the spike as follows:

$$\%R = \frac{(\text{spiked sample result} - \text{unspiked sample result}) * 100\%}{\text{known spike added concentration}}$$

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# Traceability

- The ability to follow the history, application, or location of an item or activity by means of recorded identifications; for calibration, this relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials, and, for data collection processes, this relates to calculations and data generated throughout the project back to the requirements for the quality of the project.

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# QED

The Latin phrase ***quod erat demonstrandum***

***'What I set out to prove'***

- ***Data of Known Quality***
  - ***Properly documented or recorded***
  - ***Free of contamination***
  - ***Free of any bias***
  - ***Reproducible***

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# Questions?? Comments??

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