QA/QC in the Wastewater Laboratory

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What is Quality Assurance?

- Per Standard Methods for the Examination of Water and Wastewater, 21st Ed.
  - "Quality Assurance – a definitive plan for laboratory operation that specifies the measures used to produce data of known precision and bias."

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What is Quality Control?

- Per Standard Methods for the Examination of Water and Wastewater, 21st Ed.
  - 'Quality Control – set of measures within a sample analysis methodology to assure that the process is in control."

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Code of Federal Regulation

- 136.7: Quality Assurance and Quality Control
  - Requires the use of suitable QA/QC procedures when analyzing compliance samples
  - Most will be listed in the approved methods
  - If they aren't, then refer to the "equivalent" EPA method
  - If that fails, 136.7 lists the requirements

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40 CFR 136.7

- Twelve QC elements where suitable.
  - Demonstration of capability
  - Method detection limit study
  - Lab reagent blank
  - Lab fortified blank
  - Matrix spike/matrix spike duplicate
  - Internal standards/surrogate standards/tracers

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40 CFR 136.7 Cont.

- Calibration/calibration verification/continuing calibration verification
- Control charts
- Corrective action
- QC acceptance criteria
- Definitions of preparatory and analytical batches
- Minimum frequency for conducting all QC elements

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Demonstration of Capability

- Can vary from method to method
  - Determination of linearity
  - Analysis of QC and blank samples
  - Method detection limit study

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Method Detection Limit Study

- Estimate the detection limit for the method
- Spike reagent water at 1-5 times the EDL
- Process at least seven aliquots through the entire method
- Times the standard deviation of the measurements by the student's T value
- If the level of analyte in the sample was below the determined MDL or exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.
- Repeat the study if it fails

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MDL Study with 2015 MUR

- Requires analysis of both 7 spiked and 7 blank samples
- Both preparation and analysis of these samples must include at least three batches on three separate calendar dates
- If multiple instruments are used for the analysis, the study must be spread across all of them

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2015 MUR MDL cont.

- Calculate both the MDL_s and the MDL_b
- If none of the blank aliquots provide a numerical result, the MDL_b does not apply
- If some, but not all of the blank aliquots provide numerical results, the MDL_b is set at the highest blank value
- Whichever MDL is greater (MDL_b or MDL_s) will now be the initial MDL

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2015 MUR MDL cont.

- During any quarter in which samples are being analyzed, prepare and analyze a minimum of two spiked blanks on each instrument, in separate batches if available
- Ensure that at least 7 spiked blanks and 7 method blanks are completed for the annual verification
- At least once per year, re-calculate MDL_s and MDL_b from the collected spiked blank and method blank results

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Per batch QC

- Lab reagent blank
  - Reagent water with the same preservatives and prep as a regular sample
- Lab fortified blank
  - A sample of reagent water that has been spiked with the analyte/s of concern treated exactly as a regular sample

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Per batch QC cont.

- Matrix spike/matrix spike duplicate/duplicate
  - $\%\ Recovery = \frac{(MS\ results - sample\ result)}{Known\ spike\ concentration}$ X 100%
- Internal standards/surrogates/tracers
  - Internal standards are primarily for organics and some metals analysis
  - Surrogates are used for organics analysis
  - Tracers are used for radiochemistry analysis

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Calibration

- Initial calibration
  - A minimum of three for linear curves
  - A minimum of five for non-linear curves
- Calibration verification
  - Frequency can vary

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Control Charts

- Two types of charts are primarily used
  - Accuracy charts
  - Precision charts

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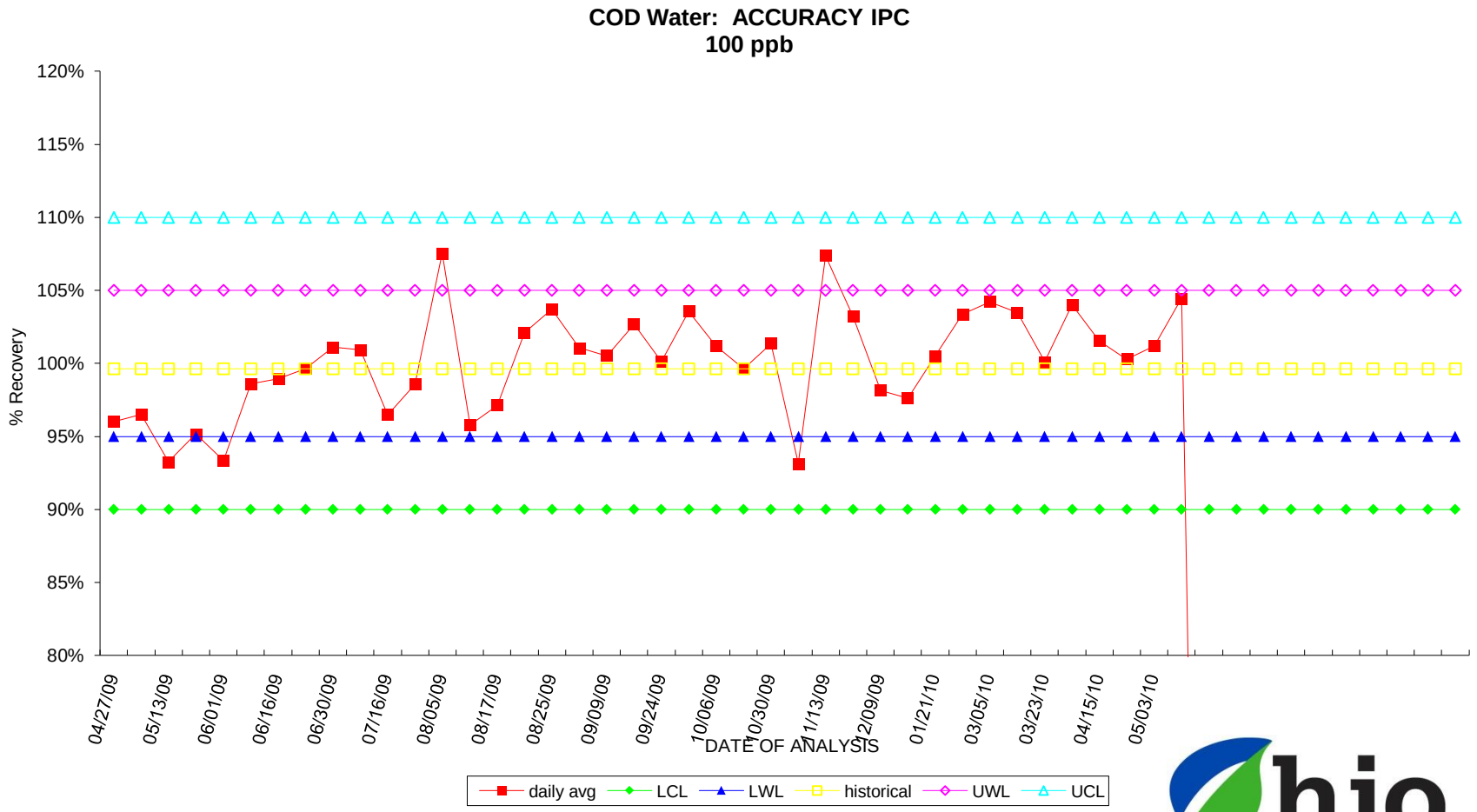
Example of an accuracy chart



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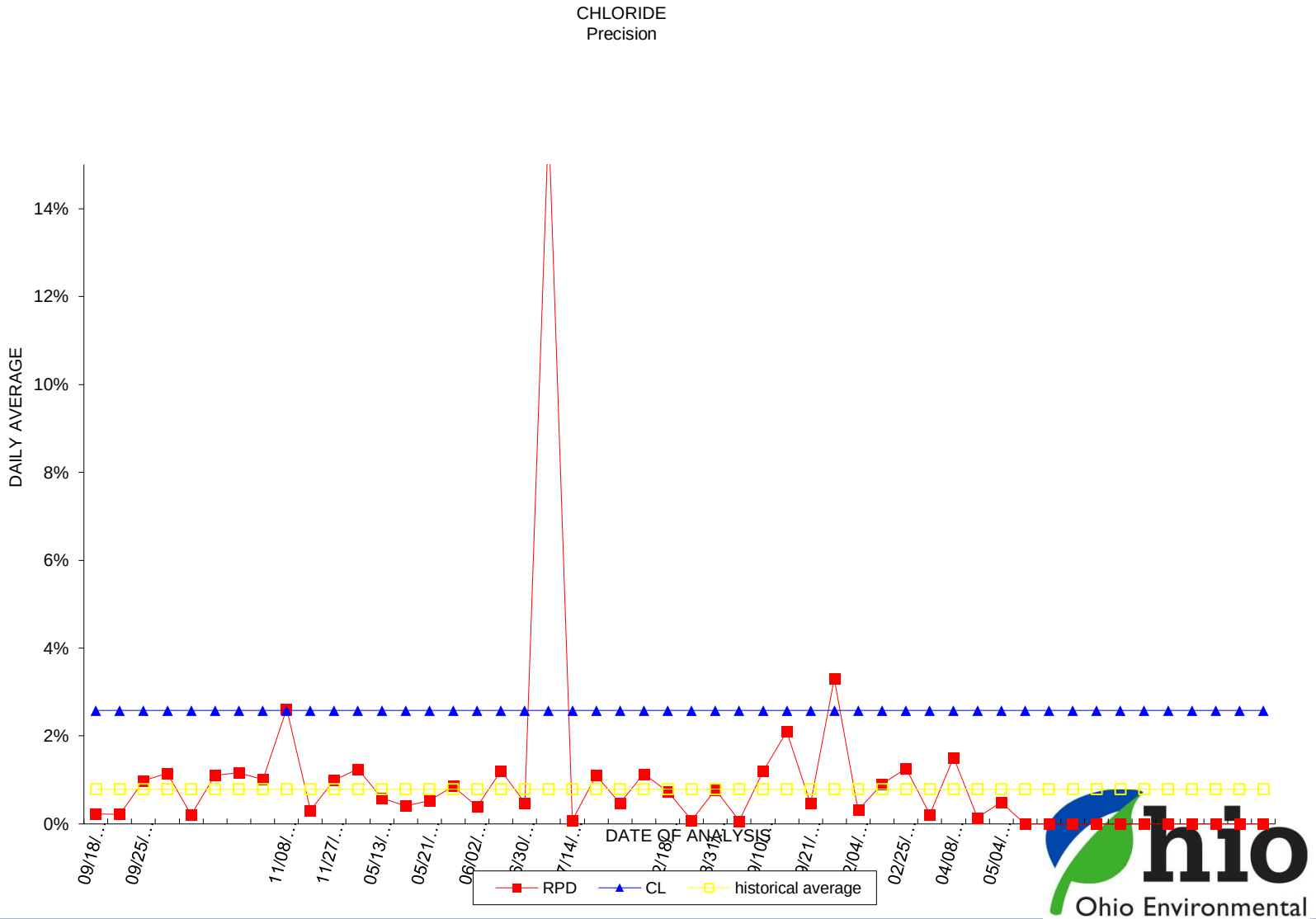
Example of a precision chart



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Corrective Action

- Corrective action must be taken when data is out of control
- Examples:
  - Check data for calculation/transcription error
  - Check calibration standards against a second source or QCS
  - If LFB fails, analyze a second
  - If the second LFB fails, check against a secondary source or QCS
  - If a LFM fails, check LFB. If the LFB is acceptable qualify the data for the LFM sample or use another method

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Other 40 CFR 136.7 requirements

- QC acceptance criteria
  - Check the individual methods
  - Check the QCS certificate of analysis
  - You may have to develop certain ones based on your data
- Definition of prep and analytical batches
  - Lab and sample volume dependent

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Quality Controls in Standard Methods

- Large QA/QC section in the front of the book
- Not all QC requirements are covered in the large QC section
- Check individual methods
- Check the beginning portion of each series of method.

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Documentation

- Bench sheets
- Logbooks
  - What should be written in logbooks?
- Temperature logs
- Laboratory Information Management Systems (LIMS)

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Questions?

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