Laboratory Certification Update
Part 2
Common Findings

KWWOA

Louisville

April 15, 2015

Presented by Frank Hall,
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Overview

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Overview

- Applications
- Field Procedures
- Chain of Custody (C-O-C)
- Sample Storage / Integrity
- DOC/MDL
- Microbiology
- WET
- Organics
- Metals
- Inorganics
- QA/QC
- Proficiency Test PT

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Applications

Applications

- **QAP**
  - Not addressing all areas
    - Scope
    - Organization Charts
  - If using the templates
    - Make certain you are deleting out the portions that don't apply to you
    - Make certain you are completing all the required areas
- **SOP**
  - Must follow the method as written
  - Analyst must be performing the analysis as written in the SOP
- **Completeness**
  - Make sure all attachments are available
    - Method-Analyte list request
    - QAP
    - SOP
    - DOC/MDL
    - PT

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Field Parameters

Field Parameters

- **Calibration**
  - All calibrations for field parameters is “daily, in use”
  - pH
    - 95% - 105% slope
    - 0.1 S.U. accuracy
    - Must bracket readings
  - Conductivity
    - In range of use

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Field Parameters

- **QC**
  - Calibration Verification
  - Blank
  - Duplicate
  - QCS
- **PT**
  - pH
  - Chlorine
  - Conductivity
  - Turbidity
- Records

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Field Parameters

- **QC**
  - Calibration Verification
    - Daily for pH, conductivity, chlorine, & turbidity
  - Blank
    - Daily for conductivity, chlorine, & turbidity
  - Duplicate
    - 1/ every 20 sample for pH, conductivity, chlorine, turbidity, DO, & temperature (may be over multiple days)
  - QCS
    - Quarterly for conductivity, chlorine, & turbidity
- **PT**
  - Annually for pH, conductivity, chlorine, & turbidity

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Chain of Custody (C-O-C)

C-O-C

- A C-O-C is a legal document!
- Gaps in C-O-C
  - Relinquished/Received by
- Missing information
  - Sampler
  - Date
  - Time
  - Preservative
  - Number and type of bottles
  - Sample ID
  - Parameters requested
- Verification upon receipt by the laboratory

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Sample Storage / Integrity

Sample Storage/Integrity

- Reagent logs
  - Every reagent needs to be tracked
  - Every time a reagent is used it needs to be tracked
  - Need a certificated of analysis for all purchased reagents
- Standard logs
  - Every standard needs to be tracked
  - Every time a standard is used it needs to be tracked
  - Need a certificated of analysis for all purchased standards

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Sample Storage/Integrity

- Temperature logs
  - Ovens
    - Thermometer in sand
    - Recorded each day in use
    - Adjusted for correction factor
    - Documentation of corrective action
  - Refrigerator
    - Thermometer in liquid
    - Recorded each day in use
    - Adjusted for correction factor
    - Documentation of corrective action

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Sample Storage/Integrity

- Internal sample tracking
  - Procedures must be in place to assure that sample remains secure
  - Procedures must be in place to assure that sample preservation conditions are met and maintained

- **If you didn't write it down, you didn't do it!**

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DOC/MDL

DOC/MDL

- Must be performed annually
- DOC must be performed by every analyst
  - 4 replicates
    - Must be at the same concentration
- MDL must be performed or verified for every instrument
  - Perform on 2-3 non consecutive days
  - New criteria proposed in the new MUR

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Microbiology

Microbiology

- Signatures on the BARF
  - Need to be signed after the
- Expired Colilert/Colilert-18 comparators
- Must utilize a SW PT for drinking water *E.coli* enumeration (not WW PT)
- Insufficient pre-warming for Colilert-18 presence/absence testing
  - 7 minutes for sample load of 10 or more is typically not sufficient to bring those samples up to 33-38^{o}C. Most labs have had to increase to 8 or 9 minutes.

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WET

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WET

- Routine chemical and physical analyses associated with WET testing
  - Must be treated the same as a normal chemical analysis
    - Must calibrate meters
    - Must analyze associated QC
    - Must be documented

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Organics

Organics

- Manual integrations
  - Allowable
  - Must be documented
  - Print original chromatograph and the new chromatograph
  - Give brief reason for manual integration

Metals

Metals

- Digestion
  - Must be performed if turbidity is not < 1 NTU
    - Turbidity analysis must be preformed utilizing an approved method and must be documented.
  - The digestion must be documented
- Linear Dynamic Range (LDR)
  - If not performed then analysis results can only be performed up to 90% of calibration range
- Spectral Interference Check
  - Must be performed to assure that various metals are not creating interferences with the analytes of concern.

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Inorganics

Inorganics

- Gravimetric Methods
  - Balance must be verified, in range of use each day
  - Reporting limit is a function of minimum weight requirements and volume
- Titration Methods
  - Titrant must be verified
  - Reporting limit is a function of titrant concentration and volume
- Colormetric Methods
  - Spectrometer must be verified annually
  - Calibration must be verified

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Inorganics

- Potentialmetric Methods (probes)
  - Probe maintenance
  - Probe storage
  - Calibration must be verified
  - Documentation
- Physical Properties
  - Method defined parameters
    - Must be analyzed exactly as written

QA/QC

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QA/QC

- QC must be documented
- QC frequency must be defined
- QC acceptance criteria must be established and reviewed
- Both passing and failing QC must be charted
  - Even if analysis results for the failed QC are not used
- Analyst should know what the purpose of each type of QC.
  - Kevin Stewart is giving a presentation on QC later today.
- RLS analysis is required for all WW parameters at the time of calibration or at least quarterly
- **QC is a requirement not a recommendation**
  - **WW must address all of the 12 mandatory elements for every method**

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PT

PT

- Send PT results to:
  - **dowptresults@ky.gov**
- Recommend performing these in 1st or 2nd quarter to allow to adequate time for repeats if necessary
- DOW only allows clerical revisions for the PTs submitted with the initial application
- PT samples must be analyzed the same as all samples.

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Questions?? Comments??

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